In the name of Allah

Dear Colleagues and Friends:

On behalf of the organization committee of the "20th Iranian Conference of The Analytical Chemistry (20th IACC)" and Isfahan University of Technology authorities, it is a great honor and pleasure to warmly welcome you to Isfahan University of Technology. The conference will be held in the chemistry department, Isfahan University of Technology with the collaboration of Center of Excellence in Sensor and Green Chemistry and Iranian Chemical Society from Tuesday 25th Feb. through Thursday 27th Feb. 2014 (6–8 Esfand 1392). The conference will provides a national forum to share knowledge and exchange ideas with colleagues, artisans and scientist from many different universities, industries and research centers.

The scientific program covers a large variety of topics in analytical chemistry, including spectrometry, electrochemistry, separation, chemometrics, nanochemistry and all subjects related to the analytical chemistry and includes oral and poster presentations.

The abstracts submitted for presentation in 20th IACC were reviewed at least by two referees, and then the final decision was made by the scientific committee. As we received a huge number of abstracts, it was not possible to the organizing committee to perform the English editing for them. Therefore, the abstracts were published in the book of abstracts as the authors had sent those.

I would like to take the opportunity to express my deep gratitude to all authors who made contributed in this conference.

I hope that you will enjoy the 20th Iranian Conference of Analytical Chemistry with the scientific and relaxing social programs and have a wonderful experience in the cultural and natural heritage of Isfahan that will stay in your memories forever.

I would like to sincerely appreciate the president of Isfahan University of Technology, Center of Excellence in Sensor and Green Chemistry, Iranian Chemical Society, Mobarakeh Steel Company, scientific committee, faculty of analytical chemistry group, and the graduated students of analytical chemistry group for their valuable support, guidance and assistance in organizing this conference.

With the best wishes
Ali A. Ensafi, Professor
The Chairman of the 20th Iranians conference of Analytical Chemistry.


بیستمین کنفرانس شیمی تجزیه ایران

دانشگاه صنعتی اصفهان، ۶ الی ۸ اسفند ماه ۱۳۹۲

اعضای ستاد اجرایی بیستمین سمینار شیمی تجزیه ایران

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میثم نوروزی فر

ناهید پوررضا

هادی بیگی نژاد

هوشنگ پرهام

یداله یمینی

1,3-diaminopropane- N, N, N', N'- tetraacetic acid as selective and sensitive material for construction of a Nano-Composite carbon paste electrode

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Abstract

Potentiometric methods based on ion selective electrodes compared to other methods of analysis such as atomic absorption, chromatography and… have several advantages including: ease of application, speed and low cost [1]. In this work a membrane was prepared by use of 1,3-diaminopropane- N, N, N', N'- tetraacetic acid (DAPTA) as Ionophore, carbon nano tube and nano silica as modifier, paraffin oil as binder for construction the ion selective electrode for measurement of Tb^{+3} cation in solutions. The best performance was shown by the membrane of composition (w/w) of 3%DAPTA, 63.7%Graphit powder, 3%carbon nano tube, 0.3%nano silic, 30%paraffin oil. The proposed sensor exhibits linear potential response with a Nernstian slope of 20.1±0.3 mV/decade of activity in the Tb^{+3} concentration range of 1.0×10^{-9}M to 1.0×10^{-2}M and a detection limit of 9.0×10^{-10} M. It has a very short response time (~6s). This interference effect of various cations on the Nernstian response of the electrode to Tb^{+3} cation was investigated. The sensor exhibits a very good selectivity toward Tb^{+3} ions over a wide variety of cations [2]. The electrode is independent of pH at the pH=3.0-9.2. It was used succesfully as an indicator electrode in the potentiometric titration of Tb^{+3} ions with EDTA [3].

Keywords: ion selective electrode; potentiometric electrode; carbon paste

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دانشگاه صنعتی اصفهان، ۶ الی ۸ اسفند ماه ۱۳۹۲

3D-QSAR, pharmacophore modeling and virtual Screening of GPR119 agonist inhibitors from the ZINC diversity database

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G protein coupled receptor 119 (GPR119) is a class A type receptor that is expressed in pancreatic β-cells and incretion releasing cells in the GI tract. The attractive targeting of GPR119 with the small molecules inhibitors is one of the most promising new approaches for the treatment of type 2 diabetes and other elements of the metabolic syndrome. A 3D-QSAR namely CoMFA and COMSIA has been carried out on a series (43 compounds) of GPR119 agonists [1]. The all-orientation search (AOS) was used to achieve the best orientation and minimize the effect of initial orientation of the structures**.** Two alignment rules for the compounds were defined using maximum common substructure and field fit [2]. In addition to analysis of the independent test set; Leave-one-out cross-validation (LOO), bootstrapping and application domain analysis (AD) of the obtained model were performed in order to confirm the robustness of the model. The qualitative pharmacophore model with DISCOtech algorithm and refinement was carried out using GASP within SYBYL program 7.3 [3] to identify pharmacophore features necessary for potent GPR119 agonist inhibitors. We performed the virtual screening to filtering by Lipinski's rule of five, pharmacophore model and docking studies to refine the retrieved hits from ZINC database. In summary, we found eight candidate compounds with high scoring functions that all are more potent GPR119 agonist inhibitors.

Keywords: GPR119, CoMFA, COMSIA, pharmacophore, virtual screening, DISCOtech, GASP, ZINC database

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Carbon paste electrode modified with Ag nanoparticles- Polyaniline- Heteropoly acid for voltammetric determination of paracetamol

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Abstract

In this research,carbon paste electrode (CPE) was modified with nano Ag poly aniline heteropoly acid(Ag pani keggin) and the modified electrode was used for voltammetric determination of paracetamol.In the present work differential pulse voltammetry was studied in KCl as electrolyte at the surface of modified electrode,Also cyclic voltammetric result showed a one well defined oxidation peak .The proposed differential pulse voltammetric method was successfully applied to paracetamol in the potential range from 0.5 to 1 V ,under different pH condition but the best result were obtained at PH 2.0 .In addition the result revealed that the modified electrode shows an electrocatalytic activity toward the oxidation of paracetamol under the optimum condition.the anodic peak current showed a linear relation versus paracetamol concentration in the range of 6.62×10^{-7} to 9.92×10^{-5} M,with a regression equation of Y= $2 \times 10^{-5}x - 1 \times 10^{-6}$, ($R^2 = 0.9999$),and detection limit of 0.016 µM (signal to noise=3).Moreover the modified electrode demonstrated good reproducibility(RSD=2.6%, n=10). This method has been applied to the demonstration of paracetamol in waste water and also paracetamol tablet samples.

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Adsorption isotherms for removal of unsymetrical dimethylhydrazine (UDMH) from military industries wastewater using activated carbon

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1,1-Dimethylhydrazine (unsymmetrical dimethylhydrazine, UDMH) is one of the hydrazine derivatives with the formula $(CH_3)_2NNH_2$ that is mainly used as a high energy fuel in military applications as a rocket propellant and fuel for thrusters and small electrical power generating units. 1,1-Dimethylhydrazine is a hypergolic rocket fuel ingredient, often used as a bipropellant in combination with the oxidizer nitrogen tetroxide and Red Fuming Nitric Acid (RFNA). UDMH has higher stability than hydrazine, especially at elevated temperatures, and can be used as its replacement or together in a mixture [1]. This compound is volatile and toxic, and is readily absorbed by oral, dermal or inhalation routes of exposure. The International Agency for Research on Cancer (IARC) has classified 1,1-dimethylhydrazine in group B2 (human carcinogen) [2]. Its Threshold Limit Value (TLV) in working air is 0.1 mg/m^3; in community air, 0.001 mg/m^3; and in domestic water, 0.02 mg/l [3].This project aim to survey a new method to remove UDMH from wastewater using activated carbon (AC). Furthermore, some effective parameters in removal process including solution pH, temperature, contact time and adsorbent amount has been optimized. In an aqueous solution of UDMH at 25 ^{0}C and pH 4.0, the adsorption capacity was evaluated using both the Langmuir and Freundlich adsorption isotherm models. Equilibrium data fitted very well with Freundlich model with R^2 0.984 and the maximum predicted relative adsorption capacity of adsorbent (K_f) and the constant (1/n) indicated adsorption intensity was found to be 0.8912 (mg/g)(lit/mg)$^{1/n}$ and 1.893 ,respectively. Finally, the removal efficiency was obtained to be 89.9% . The results indicated that the proposed method was successfully applied for removal of UDMH in military industries wastewater.

Key words: Dimetylhydrazine, Removal, Adsorption isotherm, Activated carbon

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A Comparative Survey upon the Corrosion Behavior of the Platinum/Indium Oxide Nanocomposite Coatings Obtained from the Pulse Electro-Deposition Method

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Research in the scope of nanocomposite coatings has been paid attention by many researchers particularly within the recent 3 decades. the coating process and the surveying on the enterance of particles into coating and optimization of the mechanical & technical specifications are some samples of this case. In this research , the coating method of platinum along with indium oxide nano particles using the pulse electro deposition method is investigated. In addition ,we studied the morphology of two coating sample: 1-nano composite, 2-the usual composite, by using a “SEM”. The morphological test was performed by using the vicrose method and their abrasive behavior was investigated by “pin on plate” method . Then , the corrosion behavior of nanocomposite coatings was studied by “Impedance” method. The mechanical desirable characteristics can be obtained by establishing the desirable conditions exactly and studying the method of sedimentation of particles in coating . Also , we can modify and optimize the corrosion behavior in comparison with the net platinum coating by using the nanocomposite corrosion and abrasion mechanisms. In electro deposition bathing-tub, some substances such as carborendum(SiC),chromium sulfide(Cr_2S_3), molybdenum sulfide were used. In this research, as precating procedure, a copper-foil set with dimensions: 40*11*0.2 cm was prepared and the preparation related works such as polishing with emery, removing the grease by using the 30% industrial grease remover specific solution in 75^0C,washing with dilute sulfuric acid and distilled water and making dry was done. Then, the nano indium oxide powder with mean dimension which was 100 nm was added to bathing-tub 24 hours after the preparation of electro deposition specific solution and stirred in 90 minutes with an ultrasonic stirrer. The bath temperature was 65^0C.As a final result, we observed that coatings have the higher resistance versus corrosion in comparison with the net platinum coatings[1,2].The comparative aspect related upon the circuits is shown in Figure1.

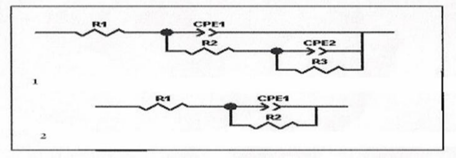

Figure1. The related circuit for 1-the cracked platinumized coatings, 2-The platinum-indium nanocomposite coatings.

Key words: coatings, corrosion, electro deposition, nanocomposite

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A combined liquid three-phase micro extraction and differential pulse voltammetric method for preconcentration and detection of ultra-trace amounts of buprenorphine using a modified pencil electrode

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Abstract

Buprenorphine is a strong semi-synthetic opiate painkiller with a sovereignty of 20-40 times higher than that of morphine, which is commonly used at higher doses for treating opioid addiction. In addition, buprenorphine at lower doses used for controlling moderate acute pain in non-opioid-tolerant individuals as well as moderate chronic pains [1]. A combination of polytetrafluorethylene (PTFE) film-based liquid three-phase micro-extraction and voltammetry was used for the micro-separation and determination of buprenorphine. Several essential factors affecting the liquid three-phase micro-extraction of buprenorphine including the type of organic solvent used, pH levels of the donor and acceptor phases, salt concentration, extraction time, stirring rate, and electrochemical factors were investigated. Differential pulse voltammetry exhibited two linear dynamic ranges of 1.0 – 109.0 pmol L^{-1} and 0.109 nmol L^{-1} – 0.11 µmol L^{-1} of buprenorphine. The detection limit was found to be as low as 0.6 pmol L^{-1} of buprenorphine. The effects of a number of common substances potentially interfering with the selectivity were studied. The results show that the proposed method is highly selective and sensitive for buprenorphine detection in real samples such as human urine and plasma of both drug-addict and non-addict human subjects.

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A Comparative Study for Adsorption of Eosin Yellow from Aqueous Solution by Magnetic Nanoparticles of Fe_3O_4 and Modified-Fe_3O_4 with 1-octyl-3-methylimidazolium Bromide

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The presence of dyes in wastewaters of industries is a major environmental problem as they are generally resistant to degradation by biological treatment methods [1]. Magnetic nanoparticles of Fe_3O_4 and modified-Fe_3O_4 with 1-octyl-3-methylimidazolium bromide were used for adsorption of eosin yellow from aqueous solution. The mean size and the surface morphology of the nanoparticles were characterized by TEM, FTIR and XRD techniques. Adsorption studies of eosin yellow were performed under different experimental conditions, such as nanoparticles amount, dye concentration, pH of the solution, ionic strength, and contact time. Experimental results indicated that Fe_3O_4 and modified-Fe_3O_4 with 1-octyl-3-methyl-imidazoliumbromide, respectively, had adsorbed more than 40%, 98% of dye under optimum condition. The isotherm evaluations revealed that the Langmuir model attained better fits to the equilibrium data than Freundlich model. Maximum adsorption capacity was 2.2 and 22.3 mg g^{-1} for Fe_3O_4 and modified-Fe_3O_4 with 1-octyl-3-methyl-imidazoliumbromide, respectively. The applicability of two kinetic models including pseudo-first order and pseudo-second order models was estimated. Adsorption processes follow to pseudo-second-order kinetic model. Both adsorption processes were exothermic [2]. The calculated values of ΔG at studied temperatures indicate that the adsorption processes are spontaneous. The dye was desorbed from nanoparticles by alkaline solution of 1 mol L^{-1} of NaCl and adsorbents were reused.

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A facile and one-pot electrochemical method for the synthesis of new benzofuran derivatives

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Electroorganic synthesis is a powerful tool for clean and safe organic synthesis. Its process is up to the standard of green chemistry [1]. Electrons are inherently environment-friendly reagents and the can substantially eliminate the waste treatment of used redox reagents [2]. Fused benzofuran derivatives are an important class of compounds because of their utility in biologically active compounds including pharmaceutical ingredients [3]. Therefore, their convenient as well as flexible syntheses have attracted much attention. Extending our interest to the research on electrochemical synthesis of new series of benzofuran, we studied anodic oxidation hydroquinone, 5,8-dihydroxy-1,4-naphthoquinone and quinizarin in the presence of dimedone as possible nucleophile in mixture of acetate buffer solution (c = 0.2 M, pH = 7.0) (50 ml)/acetonitrile (50 ml), by cyclic voltammetry and controlled-potential coulometry.

The mechanism of electrochemical reaction is confirmed as an ECEC pathway by voltammetric tracing in various times of controlled-potential coulometry. After recrystallization, products were characterized by: IR, ^{1}H NMR, ^{13}C NMR and MS.The present work describes a facile one-pot electrochemical method for the synthesis of some new benzofuran derivatives.

The results indicate that the dimedone participate in Michael type addition reaction with the oxidized form of quinizarin,5,8-dihydroxy-1,4-naphthoquinone and hydroquinone convert them to the corresponding new benzofuran derivatives .

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

A Generalization of MCR methods to Multivariate Calibration of First Order Data

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Second-order data sets are natural type of the data sets to be analyzed with soft-modeling methods. Recently these methods especially MCR-ALS are applied for analyzing first-order data [1, 2].

In analyte predictions in first-order data sets with standard methods such as PLS, PCR , etc. which are known as multivariate calibration methods, all the present components in unknown samples should also be included in calibration set.

In this work it was demonstrated that applying soft-modeling methods in analyzing first-order data results in unique concentration values for the analyte of interest under two main conditions: first, all the present components in the unknown set should be concluded in calibration set. Second, the known concentration of the analyte in calibration set should be fixed as a constraint during the iterations.

The novelty of this work is a generalization of soft-modeling methods specially MCR-ALS to multivariate calibration of first-order data sets.

A simulated chemical system was analyzed with MCR-ALS under the mentioned conditions and the rotational ambiguity in concentration space of the data was visualized with the help of systematic grid search method [3]. Also an experimental system was analyzed with the object of analyte determination in order to evaluate the results in real circumstances. The results confirmed the theoretical achievements.

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A High Sensitive Oxalic Acid Colorimetric Probe Based on Unmodified Silver Nanoparticle in Aqueous Media

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Abstract:

Nanoparticles exhibit several unique properties that can be applied to develop chemical and biosensors device. In fact, precious metal nanomaterials (e.g. Au and Ag) have gained a great deal of attention in the past decade because of their unique electronic and optical properties [1]. In particular, the surface plasmon resonance (SPR) absorption of metal nanoparticles is extremely sensitive to their size, shape, distances, and the surrounding media, based on which highly sensitive colorimetric sensors can be realized [2].In comparison with other nanoparticles especially gold nanoparticles, there are limited reports on the optical detection of molecules based on silver nanoparticles surface plasmon band [1].

Numerous methods have been suggested for the determination of oxalic acid in blood and other materials, but the procedures generally adopted are so inherently defective as to make investigators welcome the proposal of other process [3].

In this work, a highly sensitive and selective optical probe for oxalic acid was developed using unmodified silver nanoparticles. The introduction of oxalic acid (OA) reduced the overall surface charges of the AgNPs, resulting in aggregation of AgNPs and resulting a color change from bright yellow to pal green that can be measured these color changes by UV-vis spectrophotometry, and can be directly read out with the naked eye. For the detection, UV-visible absorption spectra were measured by the absorbance ratio A_{650nm}/A_{410nm}. These colorimetric response correlating with the concentration of OA and under the optimum conditions (time of reaction, temperature and pH) the analytical response was linear over the range from 0.87 μmolL^{-1} to 2.34 μmolL^{-1} (R^2 = 0.989) with a detection limit of 250 nmolL^{-1}. To further demonstrate the selectivity and applicability of the method, oxalic acid detection is realized in some food products with excellent analyte concentration recovery.

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A Highly Selective Chemosensor for Detection and Determination of Cyanide Using an Indicator Displacement Assay and PC-ANN and its logic gate behavior

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Cyanide, found in waste of industrial, acts as a drastic poison on the nervous system directly. Moreover, the low concentration of Cyanide absorbed through the skin causes weakness, conking out of the leg and arms [1]. Due to the potential impact of these toxic ions on human health and environment, sensitive methods of their detections are widely sought. Cyanide has been determined by various methods such as titrimetric, electrochemical methods and ion - exchange chromatography that are expensive, require time consuming and have a high detection limit [2]. In this study, we designed a metal complexing indicator displacement assay (M-IDA) as a simple, inexpensive and rapid method [3] based on competition between Zincon and Cyanide for binding with Zn (II) was developed to determine Cyanide. By adding Cyanide, the maximum absorption of the solution was decreased at 618 nm simultaneously with appearance a new absorption peak at 484 nm and the color of solution changed from blue intense color to orange color. This system showed a XNOR logic function using Zn (II) and Cyanide as chemical inputs and the color intensity of Zincon (at 484 nm) as output. Cyanide can be detected with a limit of the detection of 1.92×10^{-7} $molL^{-1}$ (0.005 ppm) in a fast response below 15 sec and no interference of other anionic materials. It is possible to obtain accurate results by compressing the data into scores using principal component analysis-artificial neural network (PC-ANN). Cyanide detection method was applied to determine Cyanide anion in biological samples.

Key words: Chemosensor, Cyanide, Differential Pulse Polarography, Indicator Displacement Assay, Logic gate, PC-ANN, Zinc, Zincon

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A Highly Selective Chemosensor for Detection and Determination of Cyanide Using an Indicator Displacement Assay and PC-ANN and its logic gate behavior

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A highly sensitive modified carbon paste electrode based on NiO/CNT nanocomposite and ionic liquid for voltammetric determination of NADH

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Abstract: Investigation of the redox reactivity of NADH and its oxidized form (NAD^+) are important, because over 300 dehydrogenases use these compounds as cofactor. Due to the great importance of NADH in biology system, its determination has attracted much interest. Most of the detection methods are based on the electrochemical oxidation.[1] Modified electrodes shows a better electrochemical response with lower over-potential and high sensitivity for NADH or other biological samples compared with unmodified carbon paste electrode in physiological condition.[2] In this work, we describe synthesized of NiO/CNT nanocomposite and characterization of it by different methods such as TEM, SEM and XRD. Under the optimized conditions of pH 7.0, the linear range for NADH determination was in the range from 0.08 to 400 μmol L^{-1} and the detection limit was calculated as 0.03 μmol L^{-1}. The proposed sensor was successfully applied for the determination of NADH in real samples.

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A Label-Free DNA electrochemical sensor for determination of quercetin

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Abstract: Flavonoids are a group of polyphenolic compounds widely distributed the medicinal plant, vegetable, fruit juice and variety of beverages. Flavonoids have attracted an increasing interest in electrochemical sensing which are benzo-ɣ-pyrone derivatives with several hydroxyl group attached to the ring structures C_6-C_3-C_6; of which some contain o-dihydroxy catechol (the B ring) such as quercetin, rutin, catechin and luteolin, etc. Quercetin belongs to the flavonol family. It is most abundant among the flavonoid molecules. In this study, a sensitive electrochemical procedure based on Label-free DNA interaction with quercetin at a modified nanocomposite pencil graphite electrode was introduced as a promising tool for determination of it. The decrease in guanine and adenine oxidation peak intensity signals was used as an indicator for the sensitive determination of quercetin. Finally, propose biosensor was applied for the determination of quercetin in food samples with satisfactory results.

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A label-free sensitive electrochemical DNA biosensor for quercetin determination

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Here the voltammetric behavior of quercetin at the surface of a dsDNA-coated electrode was investigated. Quercetin, one of the most abundant natural flavonoids, presents in daily food with the average human daily intake estimated to be 16 mg/person [1]. Quercetin is of interest because of its pharmacological function including the protection of human DNA from oxidative attack [2]. Phenolic compounds are responsible for the brightly colored pigments of many fruits and vegetables, and they protect plants from diseases and ultraviolet light and help prevent damage to seeds until they germinate [3]. The interaction between quercetin and dsDNA was investigated using cyclic voltammetry. It was observed that addition of DNA, decreased the oxidation peak current of quercetin by 11% and shifted the oxidation peak potential to more negative values. In addition, the current of reduction peak reduced while no considerable change was observed in the potential of the peak. So, we used this interaction for the fabrication of a biosensor to determine ultra-trace amounts of quercetin. Chitosan, a biocompatible, biodegradable and non-toxic cationic polymer that forms polyelectrolyte complexes with DNA, was used for DNA immobilization on carbon nano tube paste electrode. A considerable increase (more than three times) was observed in the oxidation signal of quercetin accumulated on the dsDNA-coated electrode compared with the DNA-free electrode, indicating the pre-concentration of quercetin due to the interaction with the surface-confined DNA layer. In order to use the proposed biosensor in real samples, the main experimental parameters affecting its response such as accumulation and stripping pH, accumulation time, time of DNA deposition and accumulation and stripping media were optimized.

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A method for determination of uranium in water samples after dispersive liquid-liquid microextraction

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Abstract

Dispersive liquid-liquid microextraction (DLLME) is a preconcentration method, which used a ternary system of solvents. In this system, an appropriate mixture of a few microliter of extraction and dispersive solvents is rapidly injected into an aqueous solution, resulting in a cloudy state including fine droplets of the extraction organic solvent dispersed in the liquid phase that markedly increases the contact surface between phases and reduce the extraction time with the increasing pre-concentration factors [1-3]. In this study, a simple and rapid dispersive liquid-liquid microextraction (DLLME) was developed for the determination of uranium in water samples prior to high performance liquid chromatography with diode array detection. 1-(2-pyridylazo)-2-naphthol (PAN) was used as complexing agent. The effect of various parameters on the extraction step including type and volume of extraction and dispersive solvents, pH of solution, concentration of PAN, extraction time, sample volume and ionic strength were studied and optimized. Under the optimum conditions, the limit of detection (LOD) and preconcentration factor were 0.3 $\mu gL^{-1}$and 194, respectively. Furthermore, the relative standard deviation of the ten replicate was <2.6%. The developed procedure was then applied to the extraction and determination of uranium in the water samples.

Key Words*:* Uranium, Dispersive liquid-liquid microextraction, HPLC, Water samples

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A molecularly imprinted electrochemical sensor based on polypyrrole, sol-gel, and gold nanoparticles hybrid nanocomposite modified pencil graphite electrode for caffeine determination

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The molecularly imprinted polymer (MIP) technique has been demonstrated as a powerful technique in designing and synthesizing some artificial receptor molecules for the mimicking of biological functions usable in analytical chemistry [1]. In the present study, a novel sensitive and selective nanocomposite imprinted electrochemical sensor for the indirect determination of caffeine has been prepared. The integration between gold nanoparticles, conductive polymers (polypyrrole), and sol-gel technology is a simple approach to improve the conduction of electrons from sol-gel MIPs to the electrode surface with the entrapment of conductive polymers and metal nanoparticles within sol-gel networks [2]. The fabrication process of the sensor was characterized by cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). Several significant parameters controlling the performance of the sensor were optimized. The imprinted sensor has the advantages of high porous surface structure, inexpensive, disposable, ease of preparation, good reproducibility and repeatability, high selectivity and sensitivity. The linear ranges of the MIP sensor were in the range from 2.0 to 50.0 and 50.0 to 1000.0 nmol L^{-1}, with the limit of detection of 0.9 nmol L^{-1}. Furthermore, the proposed method was successfully intended for the determination of caffeine in real samples (urine, plasma, tablet, green tea, energy and soda drink). Satisfactory recoveries for all samples were obtained between 90% and 116% with RSDs of 1.1-6.6%. These results indicate that the complex matrix of the real samples has no significant interference for the determination of the caffeine using this sensor.

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A nanocomposite-based solid-phase extraction procedure followed by flow injection inductively coupled plasma-optical emission spectrometry to determine cobalt ions in fuel ash and soil samples

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Cobalt due to be an essential trace element to man for metabolic processes is an important element, not only for industry but also for biological systems . Although cobalt is essential nutrient, excessive doses result in a variety of adverse responses. Nevertheless, in higher concentrations, Co is toxic to humans and to terrestrial and aquatic animals and plants [1]. Hence, removal, extraction and determination of cobalt ions seems to be essential. Among the preconcentration techniques, solid phase extraction (SPE) technique has increasingly become a popular technique. Activated carbon (AC) has been also widely used in solid phase extraction technique for adsorb metal complexes. It has allowed to very high enrichment factor in various matrix [2]. Unfortunately, activated carbons are notoriously difficult to separate from solution. This problem can be solved by applying magnetic carrier technology (MCT) so magnetic materials can be readily collected from the sample solution by using an external magnetic field placed outside of extraction container.

The present study investigates application of magnetic nanocomposite based on activated carbon (MNC-AC) as an excellent adsorbent for solid phase extraction and determination of trace amount of Co from aquatic samples using flow injection inductively coupled plasma-optical emission spectrometry (ICP-OES). MNC-AC, carrying target ions, was easily separated from the aqueous solution by applying an external magnetic field. So, no filtration or centrifugation was necessary. After extraction and collection of MNC-AC, the analytes were desorbed using acidic propylene carbonate. The desorbed analytes were introduced into the nebulizer of ICP-OES by using flow injection technique. In order to obtain high extraction efficiency, the parameters affecting SPE such as pH of sample solution, chelating agent, amount of adsorbent, extraction time, type of eluent and desorption time on extraction efficiency of the cobalt ions were investigated and optimized. Under the optimized conditions, DL for Co, was 0.008 mg L^{-1}, the EF of the proposed method for the cobalt ions was 334.97 and the RSD (RSDs, C = 0.1 mg L^{-1}, n = 5) was 8.53%. The method had a linear dynamic range within the range of 0.014–0.4 mg L^{-1}. Finally, the applicability of the proposed method was successfully confirmed by extraction and determination of cobalt (II) from fuel, ash and soil samples.

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A New Carbon Paste Electrode Based on Multi-walled Carbon nanotubes and MIPs for Olanzapine Determination

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ZYPREXA (olanzapine) is an atypical antipsychotic that belongs to the thienobenzodiazepine class. It's molecular formula is $C_{17}H_{20}N_4S$ With the chemical designation of 2-methyl-4-(4-methyl-1-piperazinyl)-10H-thieno[2,3-b] [1,5]benzodiazepine. In this work an olanzapine selective nano-composite sensor based on molecularly imprinted polymers (MIPs)as a sensing element، multi-walled carbon nanotubes (MWCNTs)،graphite powderand paraphin oil is constructed.During the recent years, MWCNTs have been used in composition of carbon paste electrodes [1،2] because of their interesting and extraordinary physicochemical properties, such as high surface area, high electrical conductivity and remarkable mechanical strength . Molecularly Imprinted Polymers (MIPs) are polymers that have been processed using the molecular imprinting technique which leaves cavities in polymer matrix with affinity to choose the "template" molecule. The process usually involves initiating the polymerization of monomers in the presence of a template molecule that is extracted afterwards, thus leaving complementary cavities behind. These polymers have affinity for the original molecule and have been used in applications such as chemical separations, catalysis, or molecular sensors [۳]The best results in this work were obtained from the nano-composite sensor with the electrode composition of 5% MWCNTs، 28%MIP،17%paraphin،50%graphite powder.The proposed sensor shows a Nernstian response (28.7mV decade^{-1}) in the range of 1.0×10^{-6}- 0×10^{-2} mol L^{-1}with detection limit of 8.0×10^{-7}. The present sensor can be used over the pH range of 3.5-8 and displayed very good selectivity، response time ، and lifetime. it was successfully applied in analysis of olanzapine in pharmaceutical formulations.

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A New Carbon Paste Selective Electrode for Sensitive and Selective Determination of Erythromycin Ethylsuccinate

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Erythromycin esters such as ethylsuccinate is antibacterially inactive and only becomes effective after it is hydrolyzed to erythromycin in plasma and other body fluids. Erythromycin prevents protein synthesis at bacterial ribosomes, resulting in the death of the organism [1].

Different analytical methods have been developed for the determination of erythromycin ethylsuccinate and its metabolities including chromatography, spectrophotometry and voltammetry [2,3], thus for easy, fast and selective determination of erythromycin ethylsuccinate, In this study a new carbon paste electrode based on the use of ion association complex of erythromycin ethylsuccinate with phosphotungstate is constructed. However, no ISE for this drug was reported. The most advantages of CPEs are ease of preparation, modification and regeneration and specially low cost.

The proposed electrode exhibits a Nernstian slope of 59.0±0.2 mV/decade for erythromycin ethylsuccinate over a concentration range of 7.4×10^{-8}–1.0×10^{-3} M, with a detection limit of 5.0×10^{-8} M. The electrode has a fast response time (<5 s), satisfactory reproducibility and good life time (more than 2 months). The potentiometric response of the electrode was studied in buffered solution. The influence of the temperature on the response of the electrode was investigated and the temperature coefficient of the electrode was calculated by recording the calibration graphs at different temperatures. The electrode was applied in potentiometric titration of erythromycin ethylsuccinate solution also and determination of erythromycin ethylsuccinate in tablet and blood serum samples.

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A New coated wire Electrode for Sensitive and Selective Determination of Clarithromycin

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Clarithromycin is a macrolide antibiotic which prevents bacterial growth by interfering with protein synthesis. It is an acid-stable version of erythromycin and is particularly effective against gram-negative bacteria [1]. Several methods have been reported for the determination of CLA such as high performance liquid chromatography with uV detector, fluorescence, mass spectrometry and electrochemical [2,3]. However, most of these methods are either time-consuming or require expensive and sophisticated instruments, well controlled experimental conditions and some sample pretreatments**.** The introduction of an ISE for fast, simple and selective determinat ion of clarithromycin is an urgent need. However, no ISE for this drug was reported.

In this work**,** a new coated wire PVC membrane electrode based on the use of ion association complex of Clarithromycin with phosphotungstate is constructed. The proposed electrode exhibits a Nernstian slope of 59±0.3 mV/decade for CLA over a concentration range of 2.2×10^{-7}–1.0×10^{-2} M, with a low detection limit of 1.0×10^{-7} M. The electrode has a fast response time (<6 s), satisfactory reproducibility and good life time. The potentiometric response of the electrode was studied in buffered solution. The influence of the temperature on the response of the electrode was investigated and the temperature coefficient of the electrode was calculated by recording the calibration graphs at different temperatures. The electrode was applied in potentiometric titration of CLA solution and determination of CLA in tablet and blood serum samples.

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A new fluorescence sensor based on the graphene quantum dots for the determination of Au^+ ion in water samples

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In this work, a simple fluorescence sensor for the detection of Au^+ ions based on the quenching of the fluorescence of graphene quantum dots (GQDs) has been developed. GQDs as a new member of the graphene family have recently emerged and ignited tremendous research interest. Due to the pronounced quantum confinement and edge effects, GQDs exhibit extraordinary optical and electrical phenomena which are not obtainable in other kinds of quantum dots. Unlike semiconductor quantum dots, GQDs show considerably low toxicity, excellent solubility, high stability, stable photoluminescence, better surface grafting, high electrical conductivity and high thermal conductivity, thus making them promising potential in fields like fluorescent probe, optoelectronic devices, sensors, cell imaging, etc [1,2]. In this work, GQDs were prepared by a simple carbonization method using glucose as a carbon source. The fluorescence characteristics of the prepared GQDs were studied at different conditions. It was found that the fluorescence of GQDs is quenched by Au^+ ions. Based on this phenomenon, a new analytical method was developed for the determination of Au^+ in the concentration range of 1.0-50 µM, with a detection limit of 0.4 µM. The developed method was applied to the determination of Au^+ in real water samples with satisfactory results.

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A new method for the simultaneous determination of aflatoxins B_1, B_2, G_1 and G_2 in pistachio samples by using ultrasound assisted emulsification microextraction coupled with HPLC-UV

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A new method was applied for the simultaneous determination of aflatoxins B_1, B_2, G_1 and G_2 at ppb level in pistachio samples by using ultrasound assisted emulsification microextraction (USAEME) coupled with HPLC-UV. At first aflatoxins were extracted from pistachio matrice by a mixture of methanol and water (80:20:v/v), the sample extracts were purified by silica gel column chromatography, finally analytes were moved from aqueous phase to the small volume of organic phase with USAEME technique (an emulsion consisting of chloroform in water was made with the help of ultrasonic waves). Aflatoxins were measured using HPLC-UV. In the optimized conditions the calibration curves were linear for B_1, B_2, G_1 and G_2 with correlation coefficients within the range 0.9914-0.9991. The limit of detection (LOD) was between 0.32 to 1.35 ng.ml^{-1} ($3S_b$/m, n=3) and the relative standard deviation (RSD) was less than 9.78% (n=3). The recoveries at the two spiked levels obtained in the range 81.2 to 101.6%. The methods proposed were applied to the determination of the target AFs in pistachio samples. The results show that ultrasound assisted emulsification microextraction coupled with HPLC-UV is simple, sensitive, cheap and efficient method for the determination of trace amounts of aflatoxins. Appears to be generalizable these method for measuring aflatoxins in various agricultural and food samples.

A new method for the simultaneous determination of aflatoxins B_1, B_2, G_1 and G_2 in pistachio samples by using ultrasound assisted emulsification microextraction coupled with HPLC-UV

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Aflatoxins are a family of mycotoxins produced as secondary metabolites by the ubiquitous moulds, Aspergillus flavus, A. parasiticus, and others less frequently encountered Aspergillus strains [1]. Some of the most famous include B1, B2, G1 and G2. Here, a new method was applied for the simultaneous determination of aflatoxins B_1, B_2, G_1 and G_2 at ppb level in pistachio samples by using ultrasound assisted emulsification microextraction (USAEME) coupled with HPLC-UV. At first aflatoxins were extracted from pistachio matrice by a mixture of methanol and water (80:20:v/v), the sample extracts were purified by silica gel column chromatography, finally analytes were moved from aqueous phase to the small volume of organic phase with USAEME technique (an emulsion consisting of chloroform in water was made with the help of ultrasonic waves). Aflatoxins were measured using HPLC-UV. In the optimized conditions the calibration curves were linear for B_1, B_2, G_1 and G_2 with correlation coefficients within the range 0.9914-0.9991. The limit of detection (LOD) was between 0.32 to 1.35 $ng.ml^{-1}$ ($3S_b/m$, n=3) and the relative standard deviation (RSD) was less than 9.78% (n=3). The recoveries at the two spiked levels obtained in the range 81.2 to 101.6%. The methods proposed were applied to the determination of the target AFs in pistachio samples. The results show that ultrasound assisted emulsification microextraction coupled with HPLC-UV is simple, sensitive, cheap and efficient method for the determination of trace amounts of aflatoxins. Appears to be generalizable these method for measuring aflatoxins in various agricultural and food samples.

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A new molecularly imprinted polymer for the selective extraction of Fenvalerate from food samples by solid-phase extraction

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The molecularly imprinted technique has considerable potential for applications in the areas of separations, catalysis, biosensors, and the development of biomedical materials [1]. A molecularly imprinted polymer (MIP) for the selective solid phase extraction (SPE) was prepared using fenvalerate (FEN) as template and pyrrole as monomer. Fenvalerate is a highly active contact insecticide that is effective against a wide range of pests, including strains resistant to organ chlorine, organ phosphorus and carbonate insecticides. It is also used on other crops, such as vines, tomatoes, pomes, other fruit and a wide variety of other crops. Polypyrrole was the first of conducting polymers that shows relative high conductivity. Up to now there is less information about using of ppy for chemical synthesis of MIPs. Polymerization occurs readily in the presence of different oxidants, such as $FeCl_3$ and $K_2S_2O_8$ [2]. In this study, the MIP- PPy was synthesized by chemical oxidation of pyrrole with $FeCl_3$ by mixing a solution of pyrrole with an oxidizing solution of $FeCl_3$. For polymer preparation, 1 mmol (223.2 mg) of template and 4 mmol of pyrrole were dissolved in 40 mL of acetonitrile:aceton (9:1 v/v) with an oxidizing solution of $FeCl_3$ (9.2 mmol). The synthesis was allowed to proceed at 5–7 °C. The synthesized was performed without agitation and under nitrogen gas atmosphere. The MIP precipitate was collected by filtration after 24 h, rinsed with distilled water and dried at 25-35 °C. As a reference, a non-imprinted polymer (NIP) was simultaneously prepared in the same way but without the addition of the template. The MISPE experiments were performed in cartridges packed with MIP and NIP in an off-line mode. In this study we use high performance liquid chromatography (HPLC) that is a rapid and sensitive method for routine analysis of fenvalerate in food. For fenvalerate, LOD was 0.19 ng g^{-1} and LOQ was 0.63 ng g^{-1}, respectively. Accuracy was exmined by the determination of the recovery of the fenvalerate. The recovery of fenvalerate was quite good. Relative standard deviation for within laboratory repeatability (%RSDr, n=5) was 4.07.

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A new sensor for highly selective determination of ascorbic acid, epinephrine, and uric acid by differential pulse voltammetry using poly(bromocresol orange) modified glassy carbon electrode

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Abstract

A highly selective electrochemical method was developed for the simultaneous determination of ascorbic acid (AA), epinephrine (EP)[1], and uric acid (UA) using bromocresol orange film modified on glassy carbon electrode. The modified electrode shows excellent electrocatalytic activity toward the oxidation of AA, EP and UA[2]. The calibration curves were obtained with 5.0-1300 µmol L^{-1} AA, 0.2-470 µmol L^{-1} EP, and 1-100 µmol L^{-1} UA. Detection limits of 1.7 µmol L^{-1} AA, 0.08 µmol L^{-1} EP, and 0.11 µmol L^{-1} UA were obtained. The influence of various substances on the selectivity of the sensor was studied[3]. The results confirm the highly selectivity, good sensitivity, and stability of the modified electrode Satisfactory results were achieved for the determination of EP, AA and UA in epinephrine injection solution, vitamin C tablets and human urine samples.

Keywords: Bromocresol orange modified film electrode,Simultaneous determination, Ascorbic acid, Epinephrine and Uric acid, Differential pulse voltammetry.

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A new solid phase extraction method for preconcentration of antimony using impregnated multi-walled carbon nanotubes

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Abstract

Antimony is a toxic element which has received relatively little environmental attention, with only few studies in soils, sediments and biological materials [1]. Several antimony compounds are used as additives to metal coatings and rubber and others are added to textiles as flame retardants [2]. Ion pair-solid phase extraction was applied to the preconcentration of antimony. The ion pair consisting of $SbCl_4^-$ anion and the benzyldimethyltetradecyl ammonium cation was formed on the surface of multi-walled carbon nanotubes, then eluted with nitric acid, and the elements finally quantified by ETAAS. In order to obtain the best recovery and high enrichment factor, the influence of some experimental parameters affecting the ion pair formation and extraction conditions such as furnace temperature program, HCl volume, flow rates, the nature and concentration and volume of effluent, sample volume and temperature were studied and optimized. The adsorption capacity of the impregnated MWCNTs is 27.5 mg g^{-1} for antimony. The following analytical figures of merit were determined: Enrichment factor of 230, assay precision of ±4.8%, linear calibration plot from 13.0 to 190 ng L^{-1}, and detection limit of 3.5 ng L^{-1}. The method was applied to the determination of antimony in synthetic sample and certified reference material: trace elements in water (SRM 1643d).

Keyword: Ion pair, ETAAS, Solid phase extraction, Sb

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A novel and sensitive electrochemical nano sensor for detection of Molybdenum (VI) ions in *Urtica dioica* (Nettle) plant

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In this work we have reported a new strategy for sensitive electrochemical sensing of Molybdenum oxide (Mo (VI)) in the presence of Calcon using a multi-walled carbon nano tube/carbon paste electrode (CNT/CPE), applicable in *Urtica dioica* (Nettle) plant as a natural sample. In the first time, the electrochemical parameters of modified electrode such as electron transfer coefficient (α), apparent charge transfer rate constant (k_s) and the surface coverage of mediator (Γ) have investigated in Fe^{+2}/Fe^{+3} redox probe solutions. The voltammetric results showed that Calcon as ligand is very useful for determination of Mo (VI) and is able to improve sensitivity in detection of Mo (VI). By using differential pulse voltammetry, we obtained two linear segments of 2×10^{-9} – 8.6×10^{-6} M of Mo (VI) at the surface of modified electrode in the 120 s accumulation time of the open circuit potential, which in this condition, the detection limit of Mo (VI) by signal to noise > 3 was found to be 0.62 nM. The combination of Calcon with CNT as mediators in carbon paste electrode shown that this electrode is capable, sensitive and simple to quantify Mo (VI) in *Urtica dioica* (Nettle) plant by average recovery of 97.64% and the influence of several cations and anions on the determination of Mo (VI) was studied.

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A novel approach for determination of xanthine using a sensitive and selective sensor based on a mono layer-molecularly imprinted sol-gel/ MWCNTs

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Abstract

Fish is considered as one of the major sources of the protein for human diet. However, it is one of the most highly perishable food products and just after landing the breakdown of adenosine-5-triphosphate in fish muscle is started if storing under improper condition. This leads to production of different compounds, such as adenosine-5-monophosphate, ionosine-5-monophosphate, inosine, hypoxanthine, xanthine and uric acid. During inappropriate storage conditions the level of xanthine is progressively increased and therefore quantification of this molecule represents a reliable method to predict the time of death and estimate freshness of fish [1,2].

The objective of this research is to develop a sensor using multiwall carbon nanotubes (MWCNTs) and molecularly imprinted sol gel (MIP-sol-gel) that can serve as a mimic enzyme for highly selective measurement of xanthine. A mono layer molecularly imprinted sol-gel (MIP-sol-gel) was electrochemically synthesized and used to modify pencil graphite electrode (PGE) coated with multiwall carbon nanotubes. FE-SEM was used for characterization of the various layers that were formed at the electrode surface. Also the electrochemical response characteristics of the modified electrode after each modification step was studied by cyclic voltammetry and electrochemical impedance spectroscopy in a solution containing $[Fe(CN)_6]^{3-/4-}$ as a redox probe. The results showed that MWCNTs enhanced the conductivity and sensitivity of the electrode and MIP-sol-gel layer was successfully formed on the electrode surface. After incubation of xanthine onto the MIP-sol-gel/MWCNTs modified electrode, the electrode was used for determination of xanthine concentration using differential pulse voltammetry. At the optimum conditions, the calibration curve was linear in two concentration ranges of 0.02–0.10 and 0.10–20.0 µmol L^{-1} with a detection limit of 20 nmol L^{-1}. The study of the effect of interfering substances on the determination of xanthine indicates the high selectivity of the proposed sensor. Finally the developed sensor was used for determination of xanthine in fish samples.

Keywords: Carbon nanotubes; Molecularly imprinted sol gel; Xanthine

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A novel chitosan-polypyrrole magnetic nanocomposite as µ-sorbent for isolation of naproxen

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A novel extracting medium based on chitosan-polypyrrole magnetic nanocomposite was synthesized by chemical polymerization for micro-solid phase extraction. In this work, magnetic nanoparticles, the modified chitosan magnetic nanoparticles and different types of chitosan-polypyrrole magnetic nanocomposites were synthesized. Extraction efficiency of the chitosan-polypyrrole magnetic nanocomposite was compared with the chitosan-Fe_3O_4 nanocomposite and Fe_3O_4 nanoparticles for the determination of naproxen in aqueous samples, via quantification by spectrofluorimetry. The scanning electron microscopy images of the synthesized nanocomposite revealed that they possess a porous structure. Among different synthesized magnetic nanocomposite, chitosan-polypyrrole magnetic nanocomposite showed a prominent efficiency. Influencing parameters on the morphology of chitosan-polypyrrole magnetic nanocomposite such as weight ratio of components was assayed. In addition, effect of different parameters influencing the extraction efficiency of naproxen including desorption solvent, desorption time, amount of sorbent, ionic strength, sample pH and extraction time were investigated and optimized [1-3]. Under the optimum condition, a linear calibration curve in the range of 0.04 - 20 mg L^{-1} with R^2= 0.9986 was obtained. The limits of detection ($3S_b$) and limits of quantification ($10S_b$) of the method were 0.015 and 0.04 mg L^{-1} (*n*=*3*), respectively. The relative standard deviation for water sample spiked with 0.1 mg L^{-1} of naproxen was 3% (*n*= *5*) and the absolute recovery was 92%. The method was applied for the determination of naproxen in tap water, human urine and human plasma samples. The relative recovery percentages for these samples were in the range of 56-99%.

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A novel DNA biosensor for detection of phenazopyridine

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Abstract

Phenazopyridine (PAP) [2,6-diamino-3-(phenylazo)pyridine], is a heterocyclic aromatic azo compound that has been used in the treatment of urinary tract infections since 1930s and is utilized to alleviate the pain in conditions such as cystitis and urethritis [1]. The interaction of phenazopyridine hydrochloride with ds-DNA based on the decreasing of the oxidation signal of guanine and adenine was studied electrochemically at a pencil graphite electrode (PGE) using differential pulse voltammetry. The decrease in the intensity of the guanine and adenine oxidation signals after their interaction with phenazopyridine hydrochloride were used as indicator signals for the sensitive determination of phenazopyridine hydrochloride. This biosensor made of pretreated pencil graphite electrode (PGE) modified with multiwall carbon nanotubes (MWCNTs) and chitosan (CHIT) decorated with the ds-DNA [2]. The difference between adenine and guanine signals of the ds-DNA after and before interaction with phenazopyridine hydrochloride was directly proportional to phenazopyridine hydrochloride concentration, which used for quantitative inspections. Under the optimum conditions, a linear dependence of the guanine and adenine oxidation signals was observed for the phenazopyridine hydrochloride concentration in the range of 0.05–30 g mL−1 with a detection limit of 0.025 gmL−1. This method is successfully applied for the determination of phenazopyridine hydrochloride in human plasma, urine samples, and pharmaceutical.

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A novel DNA biosensor for the detection of naringin based on electrochemical interaction

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Abstract

Recently, The use of electrochemical DNA-based biosensor have been developed for trace amounts of drug determinations due to high rapid, simple, and sensitive analysis. The present paper introduces a sensitive and novel electrochemical DNA-based biosensor for the measurement of naringin using differential pulse voltammetry (DPV). This biosensor was based on double-stranded DNA (ds-DNA)/ poly (diallyl dimethyl ammonium chloride) (PDDA)/ multiwall carbon nanotubes (MWCNTs) modified pencil graphite electrode (PGE) for monitoring the guanine and adenine oxidation signals. The results showed that the chemically modified electrode exhibited considerable sensitivity. The interaction of naringin with ds- DNA caused the decrease in the oxidation signals of the guanine and adenine bases. The decreasing intensity of the guanine and adenine oxidation signals because of naringin interaction was used as an indicator signal for the sensitive determination of naringin [1]. The effects of different parameters, such as concentration of ds-DNA and time of interaction between naringin and ds-DNA/MWCNTs-PDDA/PGE and instrumental parameters on the decreasing response were optimized. The all parameters were optimized in the buffer of 0.5 M acetate containing 0.02 mol L^{-1} NaCl (pH 4.8) [2]. Under the optimized conditions, using differential pulse voltammetry represented two linear segments of calibration; first segment was from 0.0580 to 5.80 mg/L and second one was from 5.80 to 580 mg/L. Detection limit of naringin for oxidation signal of guanine was achieved 0.0103 mg/L. The relative standard deviation of five reported measurements of 5.80 mg/L naringin was 3.78 % and 4.23 % for oxidation signal of guanine and adenine, respectively. The interference investigations of other species were also studied. Finally, this high-stable electrochemical biosensor was applied to analyze the naringin of the various citrus juices.

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A novel high selective and sensitive aminopetrin voltammetric sensor based on a molecularly imprinted polymer- carbon Paste electrode

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ABSTRACT:

The design and construction of a highly selective voltammetric sensor for aminopetrin by using a molecularly imprinted polymer (MIP) as recognition element were introduced. Molecularly imprinted polymers (MIPs) are extensively cross-linked polymers containing specific recognition sites with a predetermined selectivity for analytes. The molecularly imprinted was synthesized by precipitation polymerization, using aminopetrin as a template molecule, methcrylic acid as a functional monomer and ethylene glycol- dimethacrylate as a cross- linking agent. When the imprinted molecule Polymer is constructed, the imprinted polymer with a high affinity for the template molecule is obtained. This affinity is due to the shape and the arrangement of the functional of the monomer units. An aminopetrin selective MIP were synthesized and then in corporate in the carbon paste electrodes (CPEs). The MIP modified Carbon paste (CP) electrode prepared by mixing of graphite powder, paraffin and MIP. The sensor was applied for aminopetrin determination using differential pulse voltammetric method. The MIP- CPE electrode showed very high recognition ability in comparison to CPE. Electrochemical behavior of aminopetrin was investigated using cyclic voltammetry thechnique. Some parameters affecting the sensor response such as polymer components and pH were optimized and then the calibration curve was plotted. A dynamic linear calibration Curve in the range of 1.0×10^{-5} ___ 1.0×10^{-3} mol L^{-1} was obtained. The detection limit of the sensor was calculated as 2.2×10^{-4} mol L^{-1}.

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A Novel Impedimetric Sensor for Low Level Determination of Hydrogen Peroxide Based on Graphene Modified Electrode

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There is an increasing interest on rapid and accurate detection of hydrogen peroxide (H2O2), not only because it exists as a product of the reactions catalyzed by many highly selective oxidases, but also because it is an essential compound in food, pharmaceutical and environmental analyses [1]. A variety of analytical methods have been developed for the assay of H_2O_2 including titrimetry, spectrophotometry, spectrofluorometry, chemiluminescence, high performance liquid chromatography and, especially, electrochemistry [2,3].

A novel impedimetric, stable and sensitive non-enzymatic hydrogen peroxide sensor was developed by electrodepositing metallic gold nanoparticles on graphene sheets modified GC electrode. The electrochemical performance of the GNP-graphene sheets electrode for detection of hydrogen peroxide was investigated by cyclic voltammetry, chronoamperometry and electrochemical impedance spectroscopy.The GNP-graphene sheets electrode displayed a synergistic effect of gold nanoparticles and graphene sheets towards the reduction of hydrogen peroxide in pH 7 buffer solution, showing higher reduction current and positive shift in peak potential. At detection potential of -300 mV, the GNP-graphene electrode sensor presented a wide linear range up to 4.0 mM hydrogen peroxide with a detection limit of 0.5 nM (signal/noise = 3). Furthermore, the GNP-graphene sheets modified electrode exhibits high stability and selectivity to hydrogen peroxide and the poisoning by chloride ion as well as interference from the oxidation of common interfering species (ascorbic, dopamine, uric acid and carbohydrate) are effectively avoided. The GNP-graphene sheets modified electrode allows highly sensitive, stable and fast impedimetric sensing of hydrogen peroxide, which is promising for the development of non-enzymatic hydrogen peroxide sensor.

Keywords: Impedimetric sensor; Hydrogen peroxide, Graphene, Gold nanoparticles.

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A novel nanoporous coating based on carbon nanotubes/ polyaniline-co-poly (o-toluidine) composite for the headspace solid-phase microextraction of BTEX

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Benzene, toluene, ethylbenzene and xylene (BTEX) are a group of harmful volatile organic compounds, which are found in petroleum and its derivatives. They are widely used in the manufacture of paints, synthetic rubbers, adhesives, degreasing agents and agricultural chemicals. BTEX are also emitted as combustion products of wood, gasoline and fuel. The presence of BTEX in the environment is a great concern because of their toxicity. The effects of exposure to these substances comprise changes in the liver and damaging effects on the kidneys, heart, lungs and the nervous system [1]. Polyaniline (PANI) and its derivatives attract considerable attention as these materials are promising candidates for application in many methods. It should be noticed that poly (o-toluidine) (POT) has been found to have an additional advantage over polyaniline. The electrochemical generation of copolymers is a convenient method to prepare new materials with the desired properties which are different from individual homopolymers [2]. Carbon nanotubes have exhibited outstanding chemical stability, thermal conductivity, superior mechanical strength and unique electrical properties. These remarkable properties have made them good ideal additives for structural and functional composites. In recent years, carbon nanotubes (CNTs)/ polymer composites have attracted a great interest because these functional CNT based composites show eminent prospects and opportunities for new applications in a wide variety of areas [3]. In this work, oxidized multiwalled carbon nanotubes/ polyaniline-co-poly (o-toluidine) composite was directly electrodeposited on the surface of a stainless steel wire as a new coating. It was applied in the headspace solid-phase microextraction (HS-SPME) of BTEX from aqueous samples and determination by gas chromatography with mass detection (GC-MS). The characteristics of the fiber coating were studied by the scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) spectroscopy. To evaluate the coating, influence of various parameters on BTEX extraction efficiency by HS-SPME was investigated. At the optimum conditions, the linearity and LODs (S/N=3) were 0.01–50 and 0.001–0.05 ng mL^{-1}, respectively. The intra-day and inter-day relative standard deviations at 0.5 ng mL^{-1} concentration level (n=5) using a single-fiber were from 5.4 to 8.3 and 7.5 to 10.3%, respectively. The fiber-to-fiber RSD% (n=3) was between 8.4 and 12.5%. Finally the development method was applied to the analysis of real samples.

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A novel polypyrrole film containing both organic-inorganic counter ions as a highly efficient extracting medium

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A polypyrrole (PPy) film containing dodecyl benzyl sulfonic acid (SDS) and $H_2PO_3^-$ as organic and inorganic counter ions was electrodeposited on a stainless steel wire. The electropolymerization process was carried out under a constant deposition potential, applied to the corresponding aqueous electrolyte containing pyrrole monomer and mixture of SDS and $H_2PO_3^-$ ions with different concentration [1,2]. The surface characteristic of the PPy–SDS film synthesized at the presence and absence of $H_2PO_3^-$ was investigated using scanning electron microscopy (SEM). The surface structure of the PPy–SDS–$H_2PO_3^-$ film in compared with PPy–SDS was very porous and non-smooth.

The initial feasibility tests revealed that the PPy–SDS–$H_2PO_3^-$ film could be used as a highly efficient extracting medium. The applicability of this unbreakable fiber coating was examined by headspace SPME of some selected chlorobenzenes (CBs), as model compounds, from aqueous samples.

Parameters influencing the morphology of the PPy film such as the type of counter ions and their weight ratios, the applied voltage and the electrodeposition time were optimized. In addition, the extraction parameters including the extraction temperature and time, ionic strength, desorption condition and stirring rate were investigated and optimized. Under optimum conditions and using a gas chromatography-mass spectrometry (GC-MS), the relative standard deviation values for a double distilled water spiked with the selected CBs at 30 ng L^{-1} were 3–8% (n = 3) and the limit of detection for the studied compounds were between 0.5 and 1 ng L^{-1}. The calibration curves of analytes were investigated in the range of 0.8–1000 ng L^{-1} and correlation coefficients (R^2) between 0.9992 and 0.9997 were obtained. Finally the developed method was applied to the analysis of some water samples and the relative recovery values were found to be in the range of 95 to 103%.

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A novel sensor for direct determination of L-phenylalanine based on graphene oxide/L-phenylalanine nanocomposite glassy carbon electrode

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L-Phenylalanine (PhA) is an essential amino acid for human an most livestock. It is used widely as food or feed additive in infusion fluids or for chemical synthesis of pharmaceutically active compounds [1, 2]. Hyperphenylalaninemia (HPA) serves as the most common inherited disorder of amino acid metabolism and was defined as an increased plasma phenylalanine concentration above 120.0 μmol L^{-1} [3]. Although HPA patients show no clinical symptoms during the neonatal period, if undiscovered and untreated during early infancy, the patients will manifest with impaired cognitive development and function leading to mental retardation. Therefore, determination of trace PhA in blood plays an important role in clinical medicine.

Electrochemical detection of PhA was investigated on a grapheme-oxide/L-phenylalanine (GOPhA) nanocomposite coated glassy carbon electrode. PhA itself didn't show any oxidize peak in a wide potential range at a glassy carbon electrode; while, a GOPhA coated glassy carbon electrode can show the electrochemical signal of PhA at 900 mV when applying cyclic voltammetry. In order to optimize the electrochemical response of PhA, investigated the effects of pH on its oxidation in 0.1 mol L^{-1} PBS with various pH values ($4.0 < pH < 8.0$) at the surface of GOPhA-GCE by CV. The effects of pH on both peak currents and peak potentials were assessed. The results showed that the maximum current was obtained at pH 6.0 that was selected as an optimum pH. The effects of scan rate on peak current at the GOPhA-GCE in the presence of 200.0 μmol L^{-1} PhA at pH 6.0 were investigated in the range of 20-150 mVs^{-1} by CV. The results showed that the maximum current was obtained at 50 mVs^{-1} that was selected as an optimum scan rate.

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A novel simultaneous standard addition method for spectrophotometric determination of food dyes (Sunset yellow and Quinoline yellow) in a single step

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Abstract

In recent years there has been an increasing interest in using synthetic additives and food colours especially azo dyes in commercial food products [1]. Moreover, it is necessary to determine food dyes quantitatively and qualitatively in order to comply with regulations in many countries. Recently a novel method for determination of an analyte in the presence of known interferences called the net analyte signal standard addition method (NASSAM) was introduced [2]. Although NASSAM was used for individual standard addition, the novel simultaneous standard addition method i.e. Generalized Net Analyte Signal Standard Addition Method (GNASSAM) was introduced for simultaneous standard addition of analytes in a single step.

Sunset yellow and Quinoline yellow demonstrate spectral overlap with typical application of spectrophotometry technique. GNASSAM was used for simultaneous determination of Sunset yellow and Quinoline yellow in synthetic binary mixtures and real samples. The investigated method was also used to calculate figures of merit; Moreover, good limits of detection (SY=0.13 and QY=0.16mgL^{-1}) and suitable selectivity and sensivity were determined. HPLC method was applied for finding subspace in binary mixtures of real samples. The results of investigated method for real sample were compared with results of HPLC. Obtained results were in a good agreement with those of HPLC method.

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A ratiometric fluorescent cyanide chemosensor based on novel naphthalimide dye

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Although cyanide is well known one of the most toxic materials and extremely harmful to the environment and human health, cyanide and hydrogen cyanide were still widely used in different fields such as gold mining, electroplating, metallurgy, synthetic fibers and resins industry. Numerous chemical methods for the detection of cyanides, based on simple chromophore based on anion-induced changes on the optical properties (either absorption or emission) of receptors are especially attractive.

A novel fluoro substituted 1,8-naphthalimide dye, was synthesized[1-2] as a fluorescence prob for cyanide which enables the measurement of emission intensities at two different wavelengths. For this purpose, its fluorescence spectral properties have been studied via adding cyanide to a 1×10^{-5} mol dm^{-3} in ethylacetate solutions of compound and this compound exhibits considerable emission spectrum shifts with intense blue to yellow fluorescence. emission spectrum shifts to longer wavelengths from 421nm to 530nm and the complete disappearance of the emission spectrum at 421nm and appearance of the emission bond at 530nm was found with a larg $\Delta\lambda_{shift}$=109nm and the linear response of the prob covered the cyanid concentration from 4×10^{-6}M to 2.4×10^{-5}M (excitation wavelength:343nm). The change of the intensity was more obvious and the ratio of emission intensities at 530 and 421nm (I_{421}/I_{530}) decreased from 0.6 to 35, while the concentration of cyanide in the solution changed from 4 to 24µM.

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A resorcinol-formaldehyde xerogel based μ-SPE for determination of pesticides in aqueous sample

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Carbon aerogels (CAs) are novel porous carbon materials that have received considerable attention over the past decade [1]. They are usually utilized as electrode materials and adsorbent materials, due to their high surface area and porosity [2]. These materials can be obtained from the carbonization of organic aerogels, which are prepared from the sol-gel polycondensation of certain organic monomers, such as resorcinol and formaldehyde [3]. In this study, a resorcinol-formaldehyde xerogel based micro solid-phase extraction (μ-SPE) sorbent was employed for preconcentration of some selected pesticides. Three pesticides from aryloxyphenoxypropionate group including clodinafop-propargyl, haloxyfop-etotyl and fenoxaprop-P-ethyl were extracted from aqueous samples by μ-SPE and subsequently determined by GC-MS. Effect of different parameters influencing the extraction efficiency of these pesticides including the sample flow rate, sample volume and extraction time were optimized. Under optimum conditions a linear calibration curve in the range of 0.10-500 ng L^{-1} with R^2> 0.99 were obtained. The RSD% for samples at 50 μg L^{-1}level was lower than 10% (n=5) and detection limits were between 0.05 and 0.20 μg L^{-1}. The optimized method was successfully applied to the determination of Pesticides from Zayende rood and paddy water samples.

Keywords: μ-SPE, Resorcinol-Formaldehyde xerogel, aryloxyphenoxypropionate, GC-MS.

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A Sensitive Determination of Trace Amounts of Silver with A New Modified Carbon Paste Electrode

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Abstract

Silver has been extensively used in different applications such as catalysis, electronics, photonics and photography. For example, silver has the highest electrical and thermal conductivity and reflectivity of all metals. Silver exists in nature in the form of silver sulphide minerals or coexists with other non-ferrous metal sulfide ores [1].It is reported that the concentration of silver in water higher than 1.6 nmol L^{-1} is toxic to fish and micro-organisms. It has also been found that silver is toxic to humans at a concentration as high as 0.9 μmol L^{-1} in drinking water [2]. Thus, the determination of trace amounts of silver ions in various media is necessary. This study introduces the sensitive determination of silver using of modified carbon paste electrode based on multi-walled carbon nanotubes and a new synthesized chelating agent of N-(4-{4-[(anilinocarbothioyl)amino]benzyl}phenyl)-N-phenylthiourea. This electrochemical method is based on accumulation of silver ions at the surface of a new multi-walled carbon nanotube modified paste electrode. After preconcentration, the measurements were carried out in a closed circuit by electrolysis of the accumulated Ag(I) ions by voltammetric scanning from -0.2 to +0.2 V. Under optimized conditions, a linear response range from 0.5 to 270 ng mL^{-1} was obtained. The detection limit and relative standard deviation for seven replicate measurements were 0.079 ng mL^{-1} and ±2.4%, respectively. The procedure was applied to determination of silver(I) in water, tea leaves and certified reference materials with satisfactory results.

Keyword: Anodic stripping voltammetry, Carbon paste electrode, Silver, N-(4-{4-[(anilinocarbothioyl)amino]benzyl}phenyl)-N-phenylthiourea,

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A sensitive nonenzymatic glucose biosensor based on nickel and copper nanoparticles deposited on polypyrrole nanowires

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For diabetic patients, regular measurements of blood glucose levels are required to determine whether the treatments are working effectively. Enzyme-modified electrodes have some disadvantages, such as instability, the high cost of enzymes, complicated immobilisation procedures, and critical operating conditions. Therefore, considerable attention had been paid to developing nonenzymatic electrodes to overcome these problems. The direct electrocatalytic oxidation of glucose at a nonenzymatic electrode would have advantages over enzymatic electrodes. Recently, efforts to develop a practical nonenzymatic glucose sensor have been centered on achieving a breakthrough in electrocatalysis. In this context, different substrates, such as platinum, copper and metal oxidates (ZnO and CuO) [1-3], have been studied. The most important finding in this area to date is that the highly active surface area of the electrode material plays a key role in the electrooxidation of glucose.

Nickel and copper nanoparticles (NPs) electrodeposited on polypyrrole (PPy) matrixes coated on glassy carbon electrode (GCE), by the sequential electrodeposition of these NPs. X-ray diffraction (XRD) analyses reveal that the Ni and Cu NPs were successfully deposited on the PPy/GCE. The electrochemical behavior and electro-catalytic activity of this modified electrode were characterized by cyclic voltammetry and electrochemical impedance spectroscopy. The morphology of electrodes was characterized by scanning electron microscopy. The electrochemical sensor exhibited strong electro-catalytic activity with toward oxidation of glucose. The detection limit linear range was found 5 μM and 10 μM to 1.5 mM, respectively. Moreover, the sensor showed excellent sensitivity, selectivity, and stability.

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A silver nanoparticle plasmon resonance-based sensor for the determination of selenium

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Selenium has been recognized as an essential nutrient for plant, animal, and human body, meanwhile, at higher concentrations it can be toxic. The range between the concentration in which selenium is essential and toxic is quite narrow. Therefore, its determination at trace levels is very important [1,2].

In the present study, we are reporting a simple, sensitive and selective colorimetric sensor for the determination of selenium (IV). The method is based on the surface transformation of silver nanoparticles by Se(IV) in an ammonia buffer solution (pH=9). Citric acid used as the reducer and stabilizer in synthesis of silver nanoparticles could reduce SeO_3^{2-} ions to Se^0 atoms. These atoms could react with the surface silver atoms and form Ag_2Se surrounding Ag nanoparticles which results in a decrease in the plasmon absorption signal of the nanoparticles at 405 nm. The decrease is proportional to the concentration of Se(IV) which therefore can be determined quantitatively. The effect of several parameters including pH, amount of silver nanoparticles, type of buffer and incubation time on the absorbance signal was investigated and optimized. Under the optimum conditions, the calibration graph was linear the range of 50 − 800 nM for selenium with a detection limit of 24 nM. The standard deviation of six replicate determination of 200 nM Se(IV) was 1.57 %. The effect of some potentially interfering species was also studied. The proposed method was applied to various samples including Selen plus A,C,E , selenium sulfide shampoo and nuts with satisfactory results.

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A simple and green method for synthesis of silver nanoparticles using kiwi, spearmint, medlar and savory leaf extract

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Abstract

Silver nanoparticles display new properties depending upon their size, morphology, and distribution. Various approaches by means of plant extract have been used for the synthesis of metal nanoparticles. These approaches have many advantages over chemical, and microbial syntheses. Because there is no need of the enlarged process of culturing the cell, hazardous chemicals, and uneconomical purifications. In this study, the plant extract of leave of kiwi, spearmint, medlar and savory was prepared in water and alcohol and treated with different concentration of silver nitrate at different temperatures to obtain silver nanoparticles. Formation of stable silver nanoparticles at different concentrations of $AgNO_3$ gave mostly spherical particles with a diameter ranging from 5 to 40 nm. The effect of temperature on the synthesis rate of the silver nanoparticles was studied by carrying out the reaction in the water bath at 25-95 ºC . The synthesis of nanoparticles was confirmed by change in color and the formation of Ag-Np and completion of the reaction was monitored (different ranges) with UV-Visible absorption spectroscopy using a double beam spectrphotometer PG T80. The mixture was then filtered and centrifuged at 6000 rpm for 5.0 min and washed with deionized water for several times . Silver nanoparticles were characterized by scanning electron microscopy (SEM). The particles size was found by particle size analyzer. FT-IR spectroscopy was used to determine functional groups on the obtained Ag nanoparticles. Antibacterial activity of obtained silver nanoparticles was studied. This green chemical method has several advantages such as cost-effectiveness, compatibility for environment, fast and easy to apply and also by the functional groups on the obtained silver nanoparticles we can use for analysis process.

Keywords: Green synthesis, Silver nanoparticle, kiwi, Spearmint, Medlar, Savory leaf.

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A simple and rapid method for determination of furfural in transformer oil by single drop microextraction combined with gas chromatography and flame ionization detection

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In the present study, a simple and inexpensive method based on headspace single drop microextraction (SDME) followed by gas chromatography-flame ionization detection has been used successfully for the determination of furfural in transformer oil samples.Single drop microextraction is a liquid phase microextraction technique that uses a few microliter of an organic solvent which is directly immersed in the solution or headspace above the solution, to extract analyte from sample solution. SDME is a simple, fast and low cost procedure that overcomes the drawbacks of solid phase microextraction such as memory effect and fragility of the fibers and also is a powerful technique for extraction of analyte from complex matrices and provides a good clean up system for extraction [1,2]. In this procedure, 2 µL organic solvent was used for extracting analyte from a 3-mL transformer oil sample. The influence of important variables on the extraction efficiency such as type of organic solvent, extraction and equilibrium time, volume of organic solvent, stirring rate and extraction temperature were investigated. The analytical parameters under optimized conditions were obtained. The intra- and inter-day RSDs were less than 4% and 7%, respectively. The method shows good linearity (with squared correlation coefficient of 0.9982) and limit of detection (26 µg/L). The applicability of the method was also examined with different transformer oil samples.

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A solvatochromic flourescenece prob for organic solvents using a novel dye

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Solvatochromic compounds can be used as an analytical fluorescence spectrophotometric probes and, eventually, for water quantification in binary solvent mixtures.. In this case, solvatochromic spectrophotometric probes could become an alternative for fast certification analysis, since they are easier to use than the traditional Karl–Fischer titration method for precise water content determinations. A novel N-Allyl-fluorosubstituted 1,8-naphthalimide dye, was synthesized[1-2] as a fluorescence prob for determining water content in organic solvents. For this purpose, dye was treated by increasing amount of water in a binary mixture of MeCN/H_2O from 0% to 30%(v/v) and the complete disappearance of the emission spectrum at 423nm and appearance of the emission bond at 543nm was found and the linear response of the prob covered the water content from 0% to 5%(v/v). This compound exhibits considerable solvatochromism with intense blue to yellow fluorescence in various solvents. Changing from a non-polar to a polar protic solvent(such as ethylacetate to ethanol), increases the solvent interaction with emission spectrum shifts to longer wavelengths from 419nm to 531nm and absorption spectrum shifts of the compound was found in a range of solvents to be broad and its fluorescence spectral properties have been studied in several polar/non-polar and protic/aprotic solvents, as well as in propanol–acetone mixtures and THF-DMSO mixtures[3]. The results reveal a strong influence of the solvent's polarity and its hydrogen-bond donor (HBD) capability on the photophysical properties of compound.

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A study on two different electrodes for sucrose determination

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Abstract

Two novel sensors were developed for the determination of sucrose: An electrochemical sensor using glassy carbon electrode modified with cupric oxide (CuO)/multiwall carbon nanotubes (MWCNTs) nanocomposite (CuO/MWCNTs/GCE) and an electropolymerized molecularly imprinted polymer modified multiwall carbon nanotubes/glassy carbon electrode (MIP/ MWCNTs/GCE) [1,2]. Every two sensors were successfully applied for determination of sucrose in sugar beet juice samples, exhibiting their potential as fast and accurate sensors. The fabricated sensors are in good agreement with those from the HPLC method for the sucrose detection, demonstrating that the developed sensors have great potential for the practical usage for real sugar beet samples. In comparison, for determination of sucrose MIP/ MWCNTs/GCE is the most selective sensor made so far. This sensor with a limit of detection 3 μmol L^{-1} sucrose, provides an efficient way for eliminating interferences from compounds with similar structures to that of sucrose.

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A Survey Upon Trace Determination of Gatifloxacin in Pharmaceutical and Biological Samples Using Hydrophobic Palladium Nanoparticles-Assisted Ionic Liquid Microextraction Coupled with Spectrofluorimetry

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Gatifloxacin (GFLX), (±)-1-cyclopropyl-6-fluoro-1,4-dihydro-8-methoxy-7-(3-methyl-1-piperazinyl)-4-oxo-3-quinoline carboxylic acid (Figure 1) is the fourth generation of a new class of synthetic antibacterial fluoroquinolone agents. It is a novel extended spectrum fluoroquinolone with an improved Gram positive and anaerobe coverage compared with older agents such as ciprofloxacin. Different analytical methods have been proposed for quantitative analysis of gatifloxacin in real samples. These techniques include spectrofluorimetry, polarography, voltammetry, chemiluminescence, and high performance liquid chromatography (HPLC) [1-3]. A simple and rapid microextraction procedure based on the application of palladium nanoparticles and hydrophobic 1-Hexylpyridinium hexafluorophosphate [Hpy][PF6] ionic liquid (IL) was applied to trace determination of gatifloxacin. A mixture of [Hpy][PF6] and palladium nanoparticles was injected into the sample solution containing the analyte of interest and a cloudy solution was formed. After centrifuging, the sedimented phase containing enriched analyte was determined by spectrofluorimetry. Palladium nanoparticles used in this microextraction procedure significantly improved the extraction recovery which is due to its proper hydrophobicity. [Hpy][PF6] was used as the extraction solvent due to some physicochemical properties such as high hydrophobicity, water immiscibility and quite viscosity. Under optimum experimental conditions, the proposed method provided a limit of detection (LOD) of 0.09 µg L^{-1} and a relative standard deviation (R.S.D.) of 3.0%. The present technique was applied to gatifloxacin determination in pharmaceutical formulations and human urine.

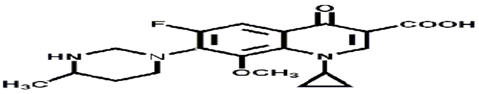

Fig. 1.

Keywords: Ionic liquid, Microextraction, Palladium nanoparticles, Gatifloxacin, Spectrofluorimeter

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A targeted metabolomics approach to understand metabolites variations in Rice under diazinon stress

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Recently, random forest (RF), one of the latest ensemble methods in machine learning, has been recognized as an effective method for classification, especially in the fields of metabolomics [1]. Analytical data acquired from mass spectrometry and chromatography have been extensively employed for classification using RF. RF is an ensemble classifier method that is an extension of decision tree concept based on classification and regression tree (CART). RF combines many trees to form a forest for analysis. An individual tree represents a model describing the characteristics of an input feature that is present in a subset of the whole dataset. Although RF has been widely utilized for classification, it is also capable of quantitative analysis. When a combined learner (forest) composed of multiple models (trees) is used instead of adopting a single model developed using a parametric method (such as PLS), the determination of concentrations would be more robust through the simultaneous estimation of concentrations from many independent trees. Each tree in a forest is developed by employing randomly selected samples in a given dataset, and variables (wavelengths or wave numbers) used to build a tree are also randomly selected, so each tree can be regarded as independent. When many independent trees are combined for analysis, the risks of biased decisions or over fitting would greatly decrease [2]. Metabolomics is an important platform of biologic systems that provides holistic metabolic information of the living system to the clinic and pharmaceutical industry. By the quantitative measurement of the metabolites and their dynamic changes in biological samples, metabolomics has been widely used in many areas, such as drugs discovery and both disease diagnostics and treatment.

In this study, we have explored RF as a potential multivariate method for GC/MS analysis of metabolomics data of rice (Oryza Sativa) under insecticide (diazinon) stress, in this study more than 23 metabolites of amino acides, sugars, fatty acids and etc. were quantitavely determined and time-course metabolic response onto different days after treatment were measured.

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A volatmmetric sensor based on NiO/CNTs nanocomposite ionic liquid carbon paste electrode for simultaneous determination of droxidopa and serotonine

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Abstract: Drug and biological compound analysis have played a vital rolein drug quality control, and such has been found to have a greatimpact on public health [1]. Therefore, a simple, fast, selec-tive, sensitive and accurate method for the determination of activepharmaceutical and biological compounds is very important [2]. Synthesis and application of NiO/CNTs and 1,3-dipropylimidazolium bromide as high sensitive sensors for voltammetric determination of droxidopa (DXP) in the presence of serotonine using carbon paste electrode has been reported for the first time. The synthesized nanocomposites were characterized by different methods such as TEM, SEM and XRD. Under the optimized conditions of pH 7.0, the plot of peak current vs. DXP concentration consisted of two linear segments with detection limit about 0.07 μmol L^{-1}. The proposed method was successfully applied for the determination of DXP in tablet, serum and urine samples.

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AC Impedance Characteristics of a PEM Fuel Cell under Different Gas Feed Modes; H_2/O_2 and H_2/Air

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This study mainly presents the AC impedance characteristics of a PEMFC under H_2/O_2 and H_2/Air gas feed modes. Nondestructive and powerful electrochemical impedance spectroscopy (EIS) method provided useful information regarding different processes that take place in the membrane electrode assembly of PEM fuel cell [1]. AC impedance spectra were recorded at different voltages corresponding to different current density regions. Based on the AC impedance spectra, charge transfer resistance, mass transfer resistance, and ohmic resistance can be extracted using the equivalent circuits set up. The Nyquist plots were fitted by Zview software. Results show that oxidant concentration has significant effects on the AC impedance of a PEM fuel cell. The PEM fuel cell in H_2/air gas mode had only one impedance arc at high frequency, which is corresponded to one relaxation time, (i.e., kinetic arc) [2]. It was observed the diameter of the high frequency arcs decreases with decreasing cell voltage due to the increased driving force at higher overpotentials. AC impedance spectra of H_2/O_2 PEM fuel cell consist of two arcs at low and high frequency so that low frequency arc probably is related to water flooding of cell by water trapped inside the electrodes or flow channels. The diameter of high frequency arcs evidently grows in the H_2/air mode, because oxygen concentration decreases as compared to H_2/O_2 mode.

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Accuracy and Precision of Chemical Quantitative Analysis using Multivariate Curve Resolution Methods

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Multivariate Curve Resolution (MCR) methods seek to decompose a two way datamatrix using a bilinear model into the product of two smaller factor matrices without any priorinformation or using only a limited knowledge about the investigated system. Although the maingoal of SMCR methods has been species resolution and identification from overlappingmultivariate signals, by adopting a proper strategy, these methods can also be applied to obtainquantitative information from complex mixtures especially in presence of unknown interferences.

The main drawback of all MCR methods is however the presence of rotational ambiguities in thesolutions obtained by these methods which usually can be reduced by applying different naturalconstraints. An aspect which is significantly less well documented in the literatures is accuracy ofthe obtained quantitative results in presence of non-eliminated part of rotational ambiguity.

An important task in chemometricsis the quantitative evaluation of the reliability ofthe parameters estimated by MCR methods. In this evaluation,two different but correlated aspects should be considered.The first aspect is the problem of rotational ambiguities andscale freedom of parameter estimations, a ubiquitous problemin all factor analysis two-way data bilinear decompositionmethods. The second aspect is how experimental errorsand noise are propagated from experimental data to parameterestimations when MCR calculationsare performed. When there is no noise, only rotationaland scale ambiguities are present. On the other hand, whennoise levels in experimental data increase, both aspects areintermixed and it is rather difficult to discern between themand differentiate between effect and cause.

In this discussion the effect of the rotational ambiguity on the quantitative results obtained by MCR methods will be investigated. Also precision in the calculated results is considered. Error propagation in feasible solutions of bilinear chemical data for single and multi-set data analysis will explained.

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Acid-induced homogeneous liquid phase microextraction followed by online-phase separation coupled to HPLC for determination of chlorobenzenes

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Abstract

Chlorobenzene compounds are listed as priority pollutants by the United States Environmental Protection Agency (USEPA). A simple homogenous liquid phase microextraction methodology applying octanoic acid as extraction phase was introduced for determination of chlorobenzenes. In this approach, phase separation phenomenon occurred by changing in pH of the solution and an acidic solution was used as phase separation reagent. The extraction phase was separated based on filtration against of centrifugation from emulsion and it introduced to HPLC. Ten milliliter of the sample solution was poured into a glass-vial. Forty five microliter of octanoic acid was solved in water sample by adjusting pH. By rapid addition of 120 µL of concentrated HCl (phase separation reagent), a cloudy solution was obtained. Then, the cloudy solution was passed through an in-line filter located in a suitable holder [1] and separated based on emulsion filtration. By changing the HPLC valve position, filter was laid in the mobile phase pass and extraction phase was eluted by mobile phase and introduced in to the separation column for analysis. This method was used for extraction and determination of five chlorobenzenes in water samples. Experimental design and response surface methodology were used for optimization of various parameters influencing the extraction efficiency of the method [2]. Under optimal conditions, chlorobenzenes were effectively extracted and preconcentration factors of 237- 280 were obtained. The calibration curve was investigated in the concentration range of 1-200 µg L^{-1} and good linearity was achieved with coefficient of determinations better than 0.995. Detection limits were in the range of 0.3 and 0.5 µg L^{-1} and suitable precision with RSDs better than 5.1 % (n = 3) were attained. Finally, the proposed method was applied to determine the concentration of chlorobenzenes in different water samples and acceptable recoveries were gained.

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Acoustic Frequency of the Pyrolysis of Electroactive Species as a Fingerprint Detection System for Selective Determination of Carbon Monoxide by AC Amperometry: Correlation between Music and Electrochemistry

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Abstract

As the frequency of acoustic waves (AW)is considered as a fingerprint probe for selective detection and determination of gas species [1-3], therefore in this study, a novel technique is introduced based on acoustic wave of the pyrolysis of electroactive species inside flame as fingerprint detection system for selective determination of gas pollutants by alternative current (AC) amperometry. Straight correlation is existed between burning frequency of pollutants and that applied to an electrochemical cell for selective detection and determination. To claim this phenomenon, carbon monoxide (CO) is selected as detection system. Burning frequency of CO inside a H_2/air flame is evaluated in an isolated system using a microphone and the sound card of a computer through software. Sound frequencies are then converted into AC electronic signals (waveform) using a programmable function generator. Significant anodic current is evaluated for the oxidation of CO on the surface of glassy carbon electrode in the presence of foreign gases such as O_2, CO_2, H_2S, C_2H_2, H_2 and also vapors of acetone and alcohols. This technique can be considered as a selective and sensitive detection system for CO detection at parts per million (ppm) levels. Linear range is between 35-100 ppm. Detection limit for CO detection is also evaluated to ~18 ppm with response time equal to ~12s. Reproducibility for about four replicate analyses of CO is evaluated to 3.09%. The reliability of this method is finally evaluated via determination of CO in two different industrial gas samples.

Keywords: Acoustic frequency; Pyrolysis; Fingerprint; AC amperometry; CO detection.

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Adsorption isotherms for removal of unsymetrical dimethylhydrazine (UDMH) from military industries wastewater using activated carbon

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1,1-Dimethylhydrazine (unsymmetrical dimethylhydrazine, UDMH) is one of the hydrazine derivatives with the formula $(CH_3)_2NNH_2$ that is mainly used as a high energy fuel in military applications as a rocket propellant and fuel for thrusters and small electrical power generating units. 1,1-Dimethylhydrazine is a hypergolic rocket fuel ingredient, often used as a bipropellant in combination with the oxidizer nitrogen tetroxide and Red Fuming Nitric Acid (RFNA). UDMH has higher stability than hydrazine, especially at elevated temperatures, and can be used as its replacement or together in a mixture [1]. This compound is volatile and toxic, and is readily absorbed by oral, dermal or inhalation routes of exposure. The International Agency for Research on Cancer (IARC) has classified 1,1-dimethylhydrazine in group B2 (human carcinogen) [2]. Its Threshold Limit Value (TLV) in working air is 0.1 mg/m^3; in community air, 0.001 mg/m^3; and in domestic water, 0.02 mg/l [3].This project aim to survey a new method to remove UDMH from wastewater using activated carbon (AC). Furthermore, some effective parameters in removal process including solution pH, temperature, contact time and adsorbent amount has been optimized. In an aqueous solution of UDMH at 25 ^{0}C and pH 4.0, the adsorption capacity was evaluated using both the Langmuir and Freundlich adsorption isotherm models. Equilibrium data fitted very well with Freundlich model with R^2 0.984 and the maximum predicted relative adsorption capacity of adsorbent (K_f) and the constant (1/n) indicated adsorption intensity was found to be 0.8912 $(mg/g)(lit/mg)^{1/n}$ and 1.893 ,respectively. Finally, the removal efficiency was obtained to be 89.9% . The results indicated that the proposed method was successfully applied for removal of UDMH in military industries wastewater.

Key words: Dimetylhydrazine, Removal, Adsorption isotherm, Activated carbon

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Adsorption isotherms for removal of unsymetrical dimethylhydrazine (UDMH) from military industries wastewater using activated carbon

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1,1-Dimethylhydrazine (unsymmetrical dimethylhydrazine, UDMH) is one of the hydrazine derivatives with the formula $(CH_3)_2NNH_2$ that is mainly used as a high energy fuel in military applications as a rocket propellant and fuel for thrusters and small electrical power generating units. 1,1-Dimethylhydrazine is a hypergolic rocket fuel ingredient, often used as a bipropellant in combination with the oxidizer nitrogen tetroxide and Red Fuming Nitric Acid (RFNA). UDMH has higher stability than hydrazine, especially at elevated temperatures, and can be used as its replacement or together in a mixture [1]. This compound is volatile and toxic, and is readily absorbed by oral, dermal or inhalation routes of exposure. The International Agency for Research on Cancer (IARC) has classified 1,1-dimethylhydrazine in group B2 (human carcinogen) [2]. Its Threshold Limit Value (TLV) in working air is 0.1 mg/m^3; in community air, 0.001 mg/m^3; and in domestic water, 0.02 mg/l [3].This project aim to survey a new method to remove UDMH from wastewater using activated carbon (AC). Furthermore, some effective parameters in removal process including solution pH, temperature, contact time and adsorbent amount has been optimized. In an aqueous solution of UDMH at 25 ^{0}C and pH 4.0, the adsorption capacity was evaluated using both the Langmuir and Freundlich adsorption isotherm models. Equilibrium data fitted very well with Freundlich model with R^2 0.984 and the maximum predicted relative adsorption capacity of adsorbent (K_f) and the constant (1/n) indicated adsorption intensity was found to be 0.8912 $(mg/g)(lit/mg)^{1/n}$ and 1.893 ,respectively. Finally, the removal efficiency was obtained to be 89.9% . The results indicated that the proposed method was successfully applied for removal of UDMH in military industries wastewater.

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Adsorption of Acid red 14 dye on modified copper oxide nanoparticles by tetrabutylammonium bromide (TBAB) in aqueous solutions

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Industrial dyestuff comprises one of the largest groups of organic compounds that cause discolouration of water and cause great loss of aquatic life. The removal of these colors and other organic materials is a priority for ensuring a safe and clean environment [1-2]. In this work the utilization possibility of copper oxide nanoparticles-modified trabutylammonium bromide (CONTBAB) has been studied as an adsorbent for the removal of colored pollutants from an aqueous medium in the industrial effluent. The adsorption studies for the evaluation of the modified copper oxide nanoparticles adsorbent for the adsorption of AR 14 from the aqueous solutions were carried out using the batch contact adsorption method. The equilibrium and kinetics study are investigated and the effects of various process parameters such as solution pH, adsorbent dosage, adsorbent size, initial dye concentration, temperature, contact time and salt were examined. The mean size, the surface morphology and stability of the adsorbent were characterized by TEM, SEM, FTIR, XRD and TGA analysis. The maximum percentage removal of AR 14 dye from aqueous solution using 0.2 g of modified adsorbent at pH 4, temperature 25±1°C, initial dye concentration of 50- 120 mg/L, and for a mixing time period of 20 min was more than 98.0%. Furthermore, under same conditions, the maximum adsorption of dye on unmodified copper oxide nanoparticles was 40.0%. Adsorption equilibrium reach within 20 min of contact time and adsorption capacity was found to be 45.0 mg g^{-1}. It was observed that the pseudo-second-order kinetic model fits better than other kinetic models with good correlation coefficient. Equilibrium data were fitted to the Langmuir model. The obtained results showed that the modified nanoparticles can be reused for least 8 adsorption–elution cycles without any decreased in their efficiency. The method was applied to the determination of AR14 in river water, Industrial water samples.

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Adsorption of Brilliant Green from Aqueous Solution by Magnetic Nanoparticles of Fe_3O_4

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Dye and dye stuffs are extensively used in various areas such as textile, plastic, food, cosmetic, carpet and paper industries. Wastewaters of these industries contain dye with metals, salts, and other chemicals which may be toxic to aquatic environment. Hence, the presence of dyes in wastewaters is a major environmental problem as they are generally resistant to degradation by biological treatment methods [1]. Magnetic nanoparticle of Fe_3O_4 was used for adsorption of brilliant green from aqueous solution. The mean size and the surface morphology of the nanoparticles were characterized by TEM and XRD techniques. Adsorption studies of brilliant green were performed under different experimental conditions, such as nanoparticles amount, dye concentration, pH of the solution, ionic strength, and contact time . Experimental results indicated that Fe_3O_4 had adsorbed more than 96% of dye under optimum condition. The isotherm evaluations revealed that the Langmuir model attained better fits to the equilibrium data than Freundlich model. Maximum adsorption capacity of Fe_3O_4 nanoparticles was 117.6 mg g^{-1}. The applicability of two kinetic models including pseudo-first order and pseudo-second order models was estimated. Adsorption process follows to pseudo-second-order kinetic model. Adsorption process was exothermic. The changes of enthalpy (ΔH) was determined to be -483.04 kJ mol^{-1}. The calculated values of ΔG at studied temperatures indicate that the adsorption process is spontaneous [2]. The dye was desorbed from nanoparticles by alkaline solution of 1 mol L^{-1} of NaCl and adsorbent was reused.

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Adsorption of Cu^{2+}, Zn^{2+}, Pb^{2+} and Ca^{2+} ions using chitosan and N-di and trihydroxy benzoyl chitosan nanoparticles

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Chitosan (CS) is a β(1→4) heteropolysaccharide whose chains are formed by 2-acetamido-2-deoxy-β-D-glucopyranose (GlcNAc) and 2-amino-2-deoxy-β-D-glucopyranose (GlcN) units. The free amine function of chitosan gives it a better ability to chelate ions of transition metals than other natural compounds such as cellulose derivatives [1]. It was shown that the chelation process and the stability of the metal–chitosan complex may be influenced by mixing (mechanical or ultrasound) [2]. There are no reports on the ability of chitosan derivatives to chelate ions. Thus, synthesis of chitosan derivatives and chelating properties of its derivatives are attractive and have been examined. In this work, modified chitosans with 3,4-dihydroxy benzoyl groups (CS-DHBA) and 3,4,5-trihydroxy benzoyl groups (CS-THBA) were synthesized and their nanoparticles were prepared via ionic crosslinking by tripolyphosphate (TPP)[3]. Then, chelating properties of nanoparticles for Cu^{2+}, Zn^{2+}, Pb^{2+} and Ca^{2+} were determined by weighing procedure and atomic absorption.Considering the interaction of chitosan with metals the reaction may be described by:

$$\text{Chitosan} + M^{n+} \rightarrow (\text{Complex})^{n+}$$

The affinity of the chitosan for cations may be represented by the sequences at pH= 6.5:

$$Cu^{2+} > Pb^{2+} > Zn^{2+} > Ca^{2+}$$

The adsorption capacities for Cu^{2+}, Zn^{2+}, Pb^{2+} and Ca^{2+} ions onto CS, CS-DHBA, CS-THBA and their nanoparticles under different pH are shown in Table1.

Table 1. Adsorption capacity of CS, CS-DHBA, CS-THBA and their nanoparticles for adsorption of Cu^{2+}, Pb^{2+}, Zn^{2+} and Ca^{2+} at pH=5.5 and pH= 6.5

Samples	mg/g pH= 6.5				mg/g pH= 5.5			
	Cu^{2+}	Pb^{2+}	Zn^{2+}	Ca^{2+}	Cu^{2+}	Pb^{2+}	Zn^{2+}	Ca^{2+}
CS	۶۴,۳۶	۳۶,۱۰	۳۵,۲۵	۹,۸۱	۵۶,۱۸	۳۱,۶۴	۳۲,۹۳	۱۰,۴۷
CS-DHBA	۱۰۴,۷۱	۸۹,۲۴	۵۴,۱۹	۲۳,۷۴	۱۰۳,۳۶	۷۶,۱۴	۵۰,۰۷	۲۲,۷۴
CS-THBB	۱۱۲,۶۶	۹۵,۲۴	۵۳,۷۲	۲۸,۵۰	۱۱۰,۰۴	۹۱,۳۲	۴۹,۰۶	۲۵,۴۱
CSNp	۹۰,۴۱	۳۸,۱۷	۴۰,۲۶	۱۵,۳۷	۸۲,۱۰	۳۴,۸۲	۳۷,۰۷	۱۷,۳۷
CS-DHBANp	۱۱۹,۰۵	۹۶,۳۱	۵۷,۴۸	۳۸,۰۰	۱۱۶,۲۵	۷۰,۴۱	53.18	۲۶,۷۰
CS-THBBNp	۱۲۶,۱۳	۱۱۴,۶۱	۶۱,۰۸	۳۶,۵۰	۱۱۸,۲۱	۱۰۲,۰۵	۵۴,۱۱	۲۹,۴۱

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Adsorption of Geimsa Stain dye onto activated Azarolus pyrene seeds as a natural adsorbents from aqueous solution, and study on kinetic and thermodynamic ofremoval process

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Adsorbents was for removal dyes used include agricultural solid wastes such as: fly ash, coal-burning industries, saw dust, wood, banana pith, pine saw dust, palm seed and Ziziphus nummularia etc[1]. In this of Azarolus pyrene's seed as a good, available and inexpensive adsorbent has been introduced and used for the removal of Geimsa Stain dye from several water solutions successfully. Azarolus pyrene is tree native to southern Mexico, northern Central America and south Iran.

Giemsa stain solution is a DNA stain that allows different chromosomes to be distinguished by their specific pattern [2].

The effect of various parameters such as pH, amount of adsorbent, size of adsorbent particles, and contact time on removal processing was investigated. To investigate the mechanism of adsorption, several kinetic models were tested including Lagergren, pseudosecond-order, particle diffusion, film diffusion, and Elovich models. Adsorption isothermal data could be interpreted by the Langmuir, Freundlich,Tempkin, and Dubinin_Rudushkevich (DR) isothermmodels. Thermodynamic parameters like free energy (ΔG), enthalpy (ΔH),and entropy (ΔS) of the system were calculated. SEM micrographs using a scanning electron microscope were studied. An FT-IR analysis for Azarolus pyrene was performed[3].

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Adsorption of Reactive Red-120 on Fe_3O_4/SiO_2 nanoparticles using (3-aminopropyl)triethoxysilane as modifier

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This work describes adsorption of Reactive Red-120, as a model azo dye, on the surface of silica-coated magnetic nanoparticles functionalized with $NH_2(CH_2)_3Si(OC_2H_5)_3$, 3-aminopropyltriethoxysilane, (Fe_3O_4/SiO_2/APTES)[1]. The mean size and the surface morphology of the nanoparticles were characterized by TEM, FTIR , PSA and SEM techniques. Adsorption of Reactive Red-120 was studied in a batch reactor at different experimental conditions such as nanoparticle dosage, dye concentration, pH of the solution, and contact time between reagents. Experimental results indicated that Fe_3O_4/SiO_2/APTES nanoparticles were adsorbed more than 98% of the dye under the optimum operational conditions of nanoparticle dosage of 15 mg, a pH of 3.0, and a contact time of 7 minutes when dye with initial concentration range of 10-50 mg L^{-1} was used. A comparative attempt among the building constituents of the adsorbent revealed that their adsorption capacities for Reactive Red-120 are in the following order: Fe_3O_4/SiO_2/APTES > Fe_3O_4/APTES > Fe_3O_4/SiO_2 > Fe_3O_4. The maximum adsorption capacity of Fe_3O_4/SiO_2/APTES was 12 mg per gram for Reactive Red-120. The adsorption isotherm fit better to the Langmuir model than the Freundlich model. The Langmuir adsorption constants were 1.71 L mg^{-1}. The dye could be desorbed by using NaCl in acetone solution suggested in literature [2] from the adsorbent surface.

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Albumin– morphine drug interaction and its application for selective determination of morphine in the presence of oxycodone and thebaine drugs: Scattering studies

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Human serum albumin (HSA) is a major protein component of blood plasma and plays an important role in the regulation of colloidal osmotic pressure and the transport of numerous endogenous compounds such as fatty acids, hormones, toxic metabolites, bile acids, amino acids, and metals [1]. HSA also binds a wide variety of drug molecules. Drug molecules in the general circulation are either bound to plasma proteins or exist in unbound form. Drugs that bind to HSA with high affinities usually interact with one or two specific sites on the protein. Changes in protein binding are often discussed based on this binding site concept. Competitive displacement between co-administered drugs which share the same binding site is a typical example. Such knowledge of drug binding sites is one of the important issues for analyzing the mechanism of altered pharmacological effect accompanied with altered protein binding [2].

Resonance Rayleigh scattering (RRS) has become known for its sensitivity and simplicity as an analytical technique which has been mainly developed in recent years. This technique has been applied successfully to study macromolecules and the determination of some metal ions, nonmetals and physicochemical constants, etc. RRS is very sensitive to the interaction caused by weak binding forces such as intermolecular electrostatic attraction, hydrogen bonding, hydrophobic interaction, and aggregation interaction of biological macromolecules [3]. The spectral characteristics and scattering intensity are strongly influenced by the molecular size, shape, conformation, and interfacial properties, which further provide favorable new information for the study of the interaction of biological macromolecules and the molecular recognition.

In this study, the interaction of three drugs (i.e. morphine, thebaine and oxycodone) with two kind albumin (i.e. Human serum albumin and bovine serum albumin) in various conditions followed by albumin-drugs system RRS spectra by synchronous scanning at $\lambda_{ex}=\lambda_{em}$. Various factors that may potentially affect scattering efficiency (i.e. pH, ultrasonication time and buffer composition) were optimized using “one-at-a-time” method. Result showed that morphine drug has highest affinity to human serum albumin at pH 9 and can be determined at levels as low as 60 ng mL^{-1}.

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Aluminum selective electrode using modified clinoptilolitenano-particles by surfactant and Arsenazo(III)

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Abstract

In this work, natural Semnanianclinoptilolite was prepared in nanometer dimensions and refluxed at 70°C for removing soluble impurities. By contacting NCP with alcoholic HDTMABrsolution, HDTMABr modified NCP (NSMZ) was produced[1,2].The obtained NSMZ was contacted withArsenazo(III) solution to obtain NSMZ-Ars as active component for aluminum.All raw and modified samples were characterized by XRD, FTIR and SEM methods.All raw and modified samples were used for the modification of PVC matrix and the obtained membranes were used for potentiometric determination of Al(III).Only, membrane electrode containing NSMZ-Ars showed suitable responsetoaluminum by average slope of19.2 ± 0.7. The working concentration range of the electrode was 8.0×10^{-5} to 2.0×10^{-1} M with a detection limit of 1.3×10^{-7} M aluminum.

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Amperometric detection of NADH at Pt/Graphene/Fe_3O_4 nanohybride-modified glassy carbon electrodes

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We have developed Pt/Fe_3O_4/ reduced grapheme oxide nanohybrides modified glassy carbon (Pt/ Fe_3O_4/ r-GO /GC) electrode as a novel system for the preparation of electrochemical sensing platform.[1-3]. Characterization of as-made nanocomposite using Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), transmission electron microscopy (TEM) and energy-dispersive analysis of X-rays (EDAX) where the Pt, Fe, Si, O and C elements were observed. Due to the synergistic effect between Pt, Fe3O4 and r-GO the nanohybrides exhibited excellent performance toward dihydronicotinamide adenine dinucleotide (NADH) oxidation, with a low detection limit of 6 nM.

Keywords: NADH; Pt/Fe_3O_4/ reduced grapheme oxide; nanocomposite; Chemical modified electrode; Electrocatalytic.

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Amperometric determination of quercetin at a magnetic core/shell Fe_3O_4@ZnO nanoparticles modified glassy carbon electrode

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In this paper, Fe_3O_4@ZnO magnetic nanoparticles (MNPs) with core/shell structure have been synthesized by a simple method, to modify glassy carbon surface and improve its efficiency for determination of quercetin. The synthesized MNPs were characterized by X-ray powder diffraction (XRD), transmission electronic microscope (TEM) and scanning electronic microscope (SEM). The XRD results confirmed the coexistence of Fe_3O_4 and ZnO phases in the nanoparticles (NPs). SEM and TEM results showed that the prepared Fe_3O_4@ZnO MNPs are made of the spherical shape particles with an average size of about 15 nm. The electrochemical behavior of quercetin, as an important biological molecule,at the surface of modified electrode was investigated bycyclic voltammetry, chronoamperometry, amperometry and differential pulse voltammetry. Under the optimal conditions a linearity range of quercetin was 7.9×10^{-7} to 6.1×10^{-5} M with detection limit (S/N=3) and sensitivity of 0.16 μM and 0.04 μA μM^{-1}, respectively. The charge transfer coefficient (α), electron transfer rate constant (k_s) and diffusion coefficient (D) for oxidation of quercetin at proposedelectrodeare determined as 0.7, 9.92 $s^{-1}$and 2.59×10^{-4} cm^2 s^{-1}, respectively. The validated method was applied successfully for determination of quercetin in some foods (honey, onion, green and black tea, pistachio, grape juice, apple, pear) and human breast milk.

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Amperometric hydrogen peroxide sensor based on covalent attachment of thionine onto glassy carbon electrode

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ABSTRACT

In this study a new hydrogen peroxide sensor was fabricated based on covalent immobilization of thionin (TN), a commercially available dye, onto a glassy carbon electrode (GCE) by using the 4 step strategy previously described by our group [1]. The glassy carbon surface functionalization was carried out by Schiff base reaction between NH_2 groups of TN and the surface amine groups with glutaraldehyde (Glu) cross-linker. Cyclic voltammograms of the thionin modified electrode in phosphate buffer solution exhibited a pair of well-defined redox peaks, indicating the preservation the thionin redox activity through immobilization. The modification steps were monitored by cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). The surface concentration of immobilized TN was obtained as 6.21×10^{-11} mole cm^{-2}. The proposed sensor showed high sensitivity to H_2O_2 detection with a significant decrease in overvoltage for hydrogen peroxide reduction, as well as a drastic increase in the peak currents in comparison with the bare GC electrode. The fast amperometric response to H_2O_2 (less than 10 s), Linear response ranges of 5.0×10^{-5}-8.0×10^{-3} M and 8.0×10^{-3}-4.5×10^{-2} M and a detection limit of 1.2 μM (S/N=3) were obtained under optimum conditions. In addition, the selectivity, stability and repeatability of this sensor were evaluated with satisfactory results.

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An artificial neural network-genetic algorithm optimization and voltammetric determination of pantoprazole based on electropolymerized-molecularly imprinted polypyrrole nanotube modified pencil graphite electrode

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Pantoprazole is a highly gastric potent proton pump inhibitor being introduced for the treatment of disorders of gastric acid hypersecretion and etc. Molecularly imprinted polymer (MIP) has been used as electrochemical sensor [1] in recent years. In this work, MIP of pantoprazole was prepared through electropolymerization of pyrrole and as a consequent electrodeposition of polypyrrole on a functionalized carbon nanotube modified pencil graphite electrode [2] in the presence of $NaClO_4.H_2O$ as supporting electrolyte and pantoprazole as template molecule. The ability of the fabricated MIP to act as a sensor of determining pantoprazole was investigated. Molecular modeling and computational methods were applied to select the suitable functional monomer for imprinting process [2]. Preparing of MIP and quantitative measurements were performed by cyclic voltammetry (CV) and differential pulse voltammetry (DPV), respectively. Several important parameters controlling the performance of the pantoprazole-selective polypyrrole film were investigated by Plackett-Burman design (PBD) and were optimized simultaneously using artificial neural network-genetic algorithm (ANN-GA) [3] by the experiments designed by central composite design (CCD). Eight factors were studied by PBD and three significant factors (pH, monomer concentration and uptake time stirring rate) were optimized simultaneously by ANN-GA. The optimal values of the factors i.e. monomer concentration, pH, CV scan rate, number of CV cycles, carbon nanotube amount, uptake time, uptake step stirring rate and template concentration were 4.3×10^{-3}M, 6.8, 200 mV/s, 50, 1g/l, 20 min, 388 rpm and 1×10^{-4}M, respectively. The molecularly imprinted layer exhibited selectivity and sensitivity toward pantoprazole. The calibration curve dynamic range was 5×10^{-6}-7×10^{-4} M with the slope of 13.30 µA/mM.

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An efficient microextraction procedure for trace determination of asulam

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Asulam, methyl sulphanylcarbamate, is a pesticide belonging to the carbamates family, which have seen increasing popularity due to their broad biological activity spectrum, as insecticides, herbicides, miticides, fungicides, nematocides or molluscicides. Asulam is a selective post-emergence herbicide controlling broadleaf plants, perennial grasses, and non-flowering plants. Asulam dissolves moderately in water and has the potential to contaminate ground water. It is very mobile in sand, loamy soil, loam and clay loam soil. The features justify the importance of asulam control in environmental samples and imply the search for efficient analytical methodologies [1].

An efficient new microextraction preconcentration method was introduced for asulam determination at trace levels. Asulam was reacted with nitrite and 1-naphthol to produce an azo dye, and then was enriched by a dispersive liquid-liquid microextraction procedure. In this method, 1-hexyl-3-methylimidazolium hexafluorophosphate was prepared in situ and used as an efficient extractant. The optimum condition was obtained and used for the preparation of a linear calibration graph. Beer's law was obeyed in the range of 1-80 ng mL^{-1} asulam. The recoveries and relative standard deviations as percents for 60 ng mL^{-1} asulam were 99.1 and 1.4, and for 3 ng mL^{-1} asulam were 105.6 and 5.5, respectively. The selectivity of the method on the determination of asulam was evaluated. The introduced procedure was applied for the analysis of different samples.

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An electrochemical approach for grafting functional groups onto mesoporous silica

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Significant attention has been attracted to the utilization of mesoporous silica in various fields. However, their application in the absence of surface modifications and functionalization is limited. By means of modification of these materials, new applications such as medical diagnostics, microelectronics and drug delivery [1,2] are reported for them. More over electrochemical syntheses of organic compounds are green methods that utilize environmentally friendly solvents, which is a great advantage of these methods [3]. In this report, an electrochemical synthesis method was applied for the functionalization of mesoporous silica (MCM-41) with a new functional group, including catechol-benzensulfinic acid, achieve novel applications of these materials. The modification of mesoporous silica, MCM-41, using an electrochemical method has been developed, and the process was monitored by cyclic voltammetery and spectrometric methods. Sulfonylbenzenediols-functionalized MCM-41 mesoporous silica was characterized by low angle X-ray diffraction, Fourier transform infrared spectroscopy, mass spectrometry, thermal analysis, elemental analysis, high resolution transmission electron microscopy, and surface area measurement (S_{BET}). The obtained results of this work indicate that electrochemical modification of mesoporous silica can be used as a new green novel tool for grafting functional groups onto these materials in the room temperature.

Keywords: Electrochemical functionalization, Surface properties, Mesoporous silica

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An electrochemical approach for grafting functional groups onto mesoporous silica

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An electropolymerized aniline-graphene nanocomposite fiber coating for headspace solid phase microextraction and determination of N-methylpyrrolidine in cefepime

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Cefepime is a fourth generation, semisynthetic cephalosporin. Its chemical, pharmacokinetic, and clinical characteristics can be found in literature. Cefepime could degrade into N-methylpyrrolidine (NMP) in the process of preparation and storage. The determination of NMP is crucial because the NMP level has a direct effect on the purity of cefepime and its preparation, and the NMP in cefepime for injection may also be toxic to patients[1]. Graphene (Gr), a two-dimensional-monoatomic thick building block of a carbon allotrope, has emerged as a kind of new generation material of the 21st century. Conducting polymers (CPs) also have been extensively studied and widely applied in various organic devices. Among the CPs, polyaniline (PANI) is the earliest and potentially one of the most useful conducting polymers because of its facile synthesis, environmental stability, and simple acid/base doping/dedoping chemistry. Graphene, as a new member of carbon nanomaterial, has been applied for fabricating the composites with CPs [2]. In this work, an aniline-graphene nanocomposite polymer was electrochemically prepared and applied as a new fiber coating for solid phase microextraction (SPME) of NMP. The aniline-graphene nanocomposite was directly electrodeposited on the platinum wire surface in sulfuric acid solution using cyclic voltammetry (CV) technique. This new coating can be prepared easily in a reproducible manner and the thickness of polymer can be precisely controlled by the number of CV cycles. The morphology of PANI- graphene spme fiber was characterized by scanning electron microscopy (SEM). To enhance the sensitivity of method, the effects of pH, ionic strength, extraction time, extraction temperature and the headspace volume on the extraction efficiency were optimized. Linear range of the analytes was found to be between 10 and 960 μgL^{-1} with regression coefficients (R^2) of 0.94 for NMP. Limits of detection (LODs) were 3 μgL^{-1} NMP. Relative standard deviation (R.S.D.) was found to be 5.0%. The proposed method was successfully applied for the determination of NMP in cefepime as a pharmaceutical preparation.

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An electrospun magnetically-doped polyurethane nanocomposite for a facile approach in micro-scaled analysis

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A magnetic polyurethane (PU) nanocomposite was synthesized by electrospinning technique and applied for isolation and preconcentration of fluoxetine form aquatic and biological samples. The nanocomposite was electrospun using PU polymer solution containing the dispersed magnetic nanoparticles. The magnetic property of iron nanoparticles along with the use of electrospun technique led to the formation of a suitable sorbent toward isolation of fluoxetine. The magnetic PU nanofibers could be subsequently removed from the sample solution by applying a permanent magnet. The scanning electron microscopy (SEM) image of the magnetic PU nanofiber confirms that their diameters are in the range of 68–113 nm. The major parameters influencing the morphology of the magnetic PU nanofibers comprising the weight ratio of iron and PU components, the applied voltage and the coating time were optimized. Moreover, parameters including the eluting solvent, amount of sorbent, extraction time, pH and salinity of aqueous samples were considered for optimization [1-3]. The detection limit of the developed method under optimized conditions and the use of a fluorescence spectrometry was 1 μgL^{-1}. The relative standard deviation (n = 5) at a concentration level of 150 μgL^{-1} was 2 %. The method was linear in the range of 50-5000 μgL^{-1} with a correlation coefficient of 0.9997. The whole procedure showed to be conveniently rapid, efficient and economical to extract fluoxetine from environmental and biological samples. Eventually, the developed method was applied to the analysis of water, urine, milk and plasma samples and relative recoveries of 76 to 99% were achieved.

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An optical sensor for iron determination based on 2,4,6-tri(2-pyridyl)-s-triazine as sensing reagent

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A high selective optical sensor for Fe(II) ions was developed based on entrapment of a sensitive reagent, 2,4,6-tri(2-pyridyl)-s-triazine, in a silica sol–gel thin film coated on a glass substrate. Several sols were prepared in order to obtain the best sensor in terms of sensitivity, stability, leaching and response time. Results indicate that films based on tetraethoxysilane (TEOS) as precursor, sol-gel pH ~ 3, water:alkoxide ratio of 4 and TPTZ concentration of 0.112 mol L^{-1} are most suitable to be used as Fe(II) sensors. The fabricated sensor can be used for determination of Fe (II) with an outstanding high selectivity over a dynamic range of 5- 115 ng mL^{-1} and a detection limit of 1.68 ng mL^{-1}. It also showed reproducible results with relative standard deviation of 3.5 and 1.27% for 10 and 90 ng mL^{-1} of Fe (II), respectively, along with a fast response time of ~180 second. Total iron also was determined after the reduction of Fe (III) to Fe (II) using ascorbic acid as reducing agent. Then, the concentration of Fe (III) was calculated by subtracting the concentration of Fe (II) from the total iron concentration.

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An Ultra Sensitive HS-GC-FID Method for Determination of Paraben in Nail Polish Samples

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The general presumption that the preservative laden cosmetic products may be one of the causative agents for cancer has remained a matter of controversy during this decade [1]. So it is important to review these products to measure and identify the amount of preservatives in them . In this study, we introduce a new method for measuring and identifying paraben preservative in nail polish matrix [2]. A new sensitive and easy operation method was developed for determination of ultra trace amount of paraben in this matrix. An ethanol esterification reaction catalyzed by Au nanoparticles in head space vial followed by gas chromatography with flame ionization detection was applied for the determination of paraben in nail polish samples [3]. The effects of various parameters on the sensitivity of the method were investigated. The best performance was obtained with an analytical head space auto sampler coupled with a gas chromatograph with flame ionization detector. The ethanol esterification reaction was based on Au nano particles as an efficient catalyst with very high surface to volume ratio. The best conditions including a 60 m $_{x}$ 0.32 mm I.D. $_{x}$ 0.25 µm CP-sil 5CB capillary column, nitrogen as carrier gas, 20 min stirring in 73 °C as optimized incubation time and temperature and finally 4% w/w for concentration of Au nanoparticles in nail polish sample. The method validations performed for static HS- GC- FID demonstrated linear calibration ranges of 0.0014-146.34 µg.ml^{-1} (r^2= 0.9976) of ethyl ester paraben of nail polish sample respectively. The limit of detection (LOD) and limit of quantification (LOQ) for paraben were 0.00042 and 0.00140 µg.ml^{-1} respectively. This method has the requisite accuracy, sensitivity, precision and selectivity to assay paraben in nail polish samples.

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An ultrasensitive chemiluminescence biosensor for glucose based on immobilization of enzyme and chitosan- induced Au/Ag alloy nanoparticles in chemiluminescence cell

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Abstract:

A novel glucose biosensor based on the chemiluminescence (CL) detection of enzymatically generated H_2O_2 was constructed by one covalent immobilization of glucose oxidase in glutaraldehyde-functionalized glass cell. At following, chitosan-induced Au/Ag nanoparticles (NPs) were synthesised [1] and immobilized on it. Herein, chitosan molecules acted as both the reducing and stabilizing agent for the preparation of NPs and a coupling agent for glucose oxidase (GOD) and Au/Ag alloy NPs [2]. In addition to catalyzes the luminol CL reaction, Au/Ag alloy NPs offer excellent catalytic activity toward hydrogen peroxide generation in enzymatic reaction between glucose oxidase and glucose. Under the optimum condition, the linear response range of glucose was found to be 1.2×10^{-6} to 6.25×10^{-3} mol L^{-1}, and the detection was 5.0×10^{-7} mol L^{-1}. The present CL biosensor has been used to determine the glucose concentrations in real serum and urine samples with satisfactory results.

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Analysis of peptides derived from protein digestion using trypsin immobilized on magnetic nanoparticles by HPLC with two detectors Fluorescence and electrospray ion mobility spectrometry

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Abstract: The quantitative determination of proteins is extremely important in many areas, such as biochemistry, biotechnology and immunodiagnostics [1]. There are two methods for quantitative analysis of proteins. First, direct analysis of intact protein (without converting to the peptide) and the other method, protein digestion to break down the protein to its constituent peptides and then analyze these peptides. Our group previously analyzed intact protein with electrospray ion mobility spectrometry (ESI-IMS) [2]. In this work, the analysis of HSA and BSA as a test proteins was performed by protein digestion using trypsin immobilized on magnetic nanoparticles (MNPs) followed by high performance liquid chromatography (HPLC) and ESI-IMS as the detector for the first time. One of the main advantages of using trypsin immobilized on MNPs was related to reduce the time of protein digestion [3]. The analytical parameters such as linearity; limit of detection (LOD) and relative standard deviation (RSD) were obtained for the polypeptides with molecular weight above of 10 kDa. The ESI-IMS signal was linear form 1.5 to 150 µM and from 1.5 to 120 µM with limit of detections 0.60 and 0.45 µM for HSA and BSA digestion, respectively. The results were also compared with the fluorescence detector (FLD) basis on native fluorescence of tryptophan and tyrosine. The fluorescence signal was linear form 3 to 150 µM and from 4.5 to 120 µM with limit of detections 1.5 and 2.5 µM for HSA and BSA digestion, respectively. The RSD for three replicates of the ESI-IMS for the analysis of HSA and BSA at 15 µM were 3% and 2.3%, respectively. The RSD (n=3) of the HPLC-FLD for the analysis of HSA and BSA at 15 µM were 1.5% and 2.5%, respectively. The results showed that this method was more sensitive than HPLC-FLD. The ESI-IMS introduces a new technique for the analysis of protein based on polypeptides derived from protein digestion.

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Analysis of trifluralin in environmental water samples using dispersive liquid-liquid microextraction (DLLME) assisted with dispersive μ-soild phase extraction (μ-SPE) followed by negative corona discharge ion mobility spectrometry (N-CD-IMS)

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Abstract: A two-step microextraction method [1] was performed for the extraction of trifluralin as a common herbicide in environmental water samples followed by continuous negative corona discharge ion mobility spectrometry (CD-IMS). Ion mobility spectrometry (IMS) is a well-known technique, which offers low detection limit, fast response, simplicity, and portability.Ionization source is one of the key parts of an IMS instrument. The advantage of corona discharge as an ionization source over ^{63}Ni is a higher total ion current and therefore a lower detection limit and wider dynamic range. The higher current also allows using narrower ion pulse widths, which enhances the resolving power. In addition, there is no need to use radioactive materials . We designed a new detector for gas chromatography based on continuous negative corona discharge ion mobility spectrometry [2].

In this work we used negative corona discharge as an ionization source. At first, n-octyl-triethoxysilane surface-modified magnetic iron oxide nanoparticles (C8MNPs) were synthesized [3]. The DLLME was performed by injecting 1 mL of methanol (disperser) containing10μL of n-hexane (extraction solvent) into 10 mL of water sample. C8MNPs were immediately added to this solution for the retrieval of the extraction solvent and the extracted trifluralin by sonication . Subsequently, a magnet was used to isolate the C8MNPs. Then, the water sample was decanted, the nanoparticles were dried, and the trifluralin was extracted from nanoparticles by sonication with methanol. Variables affecting the performance of both steps were thoroughly optimized. Under the optimized conditions, analytical parameters such as linearity and limit of detection were obtained. A good linearity (1-100ng/ml) and low detection limit (0.3ng/mL) were obtained. The enrichment factor was 50. This method was applied for the determination of trifluralin in well water samples.

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Analysis of trifluralin in environmental water samples using dispersive liquid-liquid microextraction (DLLME) assisted with dispersive µ-soild phase extraction (µ-SPE) followed by negative corona discharge ion mobility spectrometry (N-CD-IMS)

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Abstract: A two-step microextraction method [1] was performed for the extraction of trifluralin as a common herbicide in environmental water samples followed by continuous negative corona discharge ion mobility spectrometry (CD-IMS). Ion mobility spectrometry (IMS) is a well-known technique, which offers low detection limit, fast response, simplicity, and portability.Ionization source is one of the key parts of an IMS instrument. The advantage of corona discharge as an ionization source over ^{63}Ni is a higher total ion current and therefore a lower detection limit and wider dynamic range. The higher current also allows using narrower ion pulse widths, which enhances the resolving power. In addition, there is no need to use radioactive materials . We designed a new detector for gas chromatography based on continuous negative corona discharge ion mobility spectrometry [2].

In this work we used negative corona discharge as an ionization source. At first, n-octyl-triethoxysilane surface-modified magnetic iron oxide nanoparticles (C8MNPs) were synthesized [3]. The DLLME was performed by injecting 1 mL of methanol (disperser) containing10µL of n-hexane (extraction solvent) into 10 mL of water sample. C8MNPs were immediately added to this solution for the retrieval of the extraction solvent and the extracted trifluralin by sonication . Subsequently, a magnet was used to isolate the C8MNPs. Then, the water sample was decanted, the nanoparticles were dried, and the trifluralin was extracted from nanoparticles by sonication with methanol. Variables affecting the performance of both steps were thoroughly optimized. Under the optimized conditions, analytical parameters such as linearity and limit of detection were obtained. A good linearity (1-100ng/ml) and low detection limit (0.3ng/mL) were obtained. The enrichment factor was 50. This method was applied for the determination of trifluralin in well water samples.

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Anodic stripping voltammetric determination of trace copper by using Palladized Aluminum electrode as a novel solid electrode

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Copper is an essential trace element in biological systems and in living organisms. But the copper can be tolerated by human at certain concentration and excessive intake of this element caused certain diseases in humans. Hence it is both vital and toxic to many biological systems, depending on the level of concentration. Thus, the determination of copper in various matrices including pharmaceutical compounds, natural waters, biological samples and so on is very important [1].

Commonly, the analytical methods used for the quantification of copper are based on use of expensive and sophisticated instruments [2]. Electrochemical stripping techniques have received considerable attention for trace analysis of heavy metals including copper, due to their sensitivity, low cost, rapidity, selectivity, portability and low detection limits [1]. The usual working electrode for stripping voltammetry was a mercury electrode. However, due to the toxicity of mercury, future regulations and occupational health considerations severely restrict the use of mercury as an electrode material. Therefore, new alternative solid electrode materials with similar performance are highly desired [3].

In this work, Palladized Aluminum electrode has been used as a novel solid electrode for stripping voltammetric determination of copper at micro molar concentration levels. This electrode has advantages such as simple and rapid preparation, low cost, non toxicity, high stability, good sensitivity and selectivity. The effects of experimental conditions such as electrode preparation, pH, deposition potential, deposition time, type and concentration of supporting electrolyte, etc were investigated. Under the optimum conditions, a linear calibration graph was obtained in the range of 3-60 μM with a correlation coefficient of 0.998. The methodology was applied successfully for determination of copper in multi-vitamin tablets.

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Application dispersive liquid–liquid microextraction method and UV–Vis spectrophotometry for determination of Lanthanum ion and optimization by experimental design methods

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Abstract

In this work, for the first time, dispersive liquid–liquid microextraction (DLLME) combined with UV–Vis spectrophotometry was used for preconcentration and determination of Lanthanum ion in water samples. N-(2-aminoethyl)- salicylaldimine (AESI), Chloroform ($CHCl_3$) and Acetone were used as the chelating agent, extraction and dispersive solvent, respectively. The effective factors of the extraction process such as volume of extraction and dispersive solvent, pH, and concentration of chelating agent were optimized by a full central composite design (CCD). The optimum condition for pH, volume of extraction solvent, volume of dispersive solvent, concentration of chelating agent, were 6.0, 150 ml, 400 ml and 0.01 mg/l respectively. Under the optimum coditions, the enrichment factor of this method for La was reached at 93. In the optimum condition detection limit, linear over the range and concentration factor were 1.2 μg L^{-1}, 2 - 700 μg mL^{-1} and 93 respectively. The proposed method was successfully applied for measuring nickel in Aquent samples.Relative standard deviation for five replicate determinations of La at 25 μg mL^{-1} concentration level was calculated to be 3.8%. The proposed procedure was successfully applied to the determination of La ion in water samples.

Keywords: Experimental design; UV–Vis spectrophotometry; DLLME; Lanthanum

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Application of a new fiber coating based on electrochemically reduced graphene oxide for cold fiber headspace solid phase microextraction of tricyclic antidepressants

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Abstract

Tricyclic antidepressant (TCADs) drugs have been widely prescribed in the treatment of psychiatric disorders such as depression, mainly endogenous major depression [1]. The relatively narrow therapeutic/toxic index of TCADs necessitates clinical monitoring for effective and correct patient treatment and for forensic purposes [2,3]. In this work, a new fiber based on electrochemical reduction of grapheme oxide was prepared on copper wire for solid-phase microextraction (SPME) of TCA◌◌Ds, including amitriptyline, trimipramine and clomipramine in biological samples. The feasibility of direct-immersion (DI) SPME and headspace (HS) SPME for the determination of TCADs was studied. The effects of the four parameters including pH, ionic strength, extraction temperature with and without the cooling the fiber, and extraction time were investigated in DI-SPME and HS-SPME. The comparison showed that headspace cold fiber solid phase microextraction (HS-CF-SPME) results in the best outcome for the extraction of TCADs from biological samples. Under the optimized conditions of this mode, the calibration curves were linear within the range of 1-500 ng mL^{-1} and the detection limits were between 0.08-0.20 ng mL^{-1}. The intra-day and inter-day relative standard deviations obtained at 10 ng mL^{-1} (n=5), using a single fiber, were 5.7-8.2% and 7.0-8.3%, respectively. The fiber to fiber repeatabilities (n=4), expressed as the relative standard deviations (RSD%) were between 10.3-12.7% at a 10 ng mL^{-1} concentration level. The method was successfully applied to the analysis of TCADs in plasma samples.

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Application of ATR-FTIR for a rapid determination of Gemfibrozil in Pharmaceutical formulation

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A simple, fast and accurate Fourier transform mid-infrared spectroscopy method was developed for determining gemfebrozil in different pharmaceutical formulation using attenuated total reflectance. ATR is a method, which simplifies sample handling, avoids the use of organic solvents and thus saves time and chemical reagents [1-2]. For the determination of gemfibrozil, mixed of grinding tablet, starch and standard was used in the form of powdered for acquisition of the FT-IR spectra of standards and samples. The most suitable region was found in the range of 1190 - 1235 cm^{-1}. The standard addition method was used for establishing the calibration line and the excellent coefficient of determination (R^2) was achieved (0.9911). The method provides a limit of detection of 0.006 mg per tablet (n=7), a relative standard deviation (RSD) of 2.65% for 5 independent measurement of standard (at level of 300mgg^{-1}).The recovery values obtained in the analysis of pharmaceuticals are within the 98-104% range.

The analytical method proposed is non-destructive and the cost and time of analysis are very much reduced, allowing for the fast and direct determination of gemfibrozil content.

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Application of cold-induced aggregation microextraction as a fast, simple and organic solvent-free method for the separation and preconcentration of Se (IV) in rice and various water samples

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Selenium is an essential element for plants, animals and humans. However, at high concentrations (upper intake limit of 400 µg/day) [1], it can produce chronic toxicity symptoms, such as liver carcinoma, cirrhosis, loss of teeth, hair and nails, irritation of the eyes and paralysis. Thus, great efforts have been devoted to develop a simple and sensitive method for the determination of selenium in various samples. Recently, cold-induced aggregation microextraction method (CIAME), as a high performance and powerful preconcentration method was demonstrated by Baghdadi and Shemirani [2]. This method is based on using room temperature ionic liquids (RTILs) as extraction solvents. In CIAME, there is no interface between the water and the extractant phase. During the formation of fine droplets of the extractant phase, the hydrophobic species are collected by the extractant molecules, and the extraction process ends after the formation of the droplets. As a result, mass transfer from aqueous phase into separated phase has no significant effect on the extraction time. CIAME avoids the large consumption of toxic solvents. The results demonstrate that CIAME is a simple, rapid, sensitive, and easy to operate method, and it can be very useful in the analysis of pollutants. The developed method is based on CIAME of Se (IV) using the 1-butyl-3-methylimidazolium hexafluorophosphate ionic liquid as an extractant followed by spectrophotometry determination. The extraction of Se (IV) was performed in the presence of dithizone as the complexing agent. In this method, a very small amount of 1-butyl-3-methylimidazolium hexafluorophosphate was added to the sample solution containing Se-dithizone complex. Then, the solution was kept in a thermostated bath at 50 ºC for 4 min. Subsequently, the solution was cooled in an ice bath and a cloudy solution was formed. After centrifuging, the extractant phase was analyzed using a spectrophotometric detection method. Some important parameters that might affect the extraction efficiency were optimized (HCl: 0.6 mol L^{-1}, dithizone: 4.0×10^{-6} mol L^{-1}, ionic liquid: 100 µL). Under the optimum conditions, good linear relationship, sensitivity and reproducibility were obtained. The limit of detection (LOD) ($3S_b/m$) was 1.5 µg L^{-1} and the relative standard deviation (RSD) was 1.2% for 30 µg L^{-1} of Se (IV). The linear range was obtained in the range of 5–60 µg L^{-1}. It was satisfactory to analyze rice and various water samples.

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Application of copper electrode modified with copper sulfideforelectrocatalytic oxidation of glucose

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Abstract

While many electrochemical studies have been done on Au and Ag surfaces, Cu has received smaller attention due to its high sensitivity and reactivity towards oxygen[1,2].Although metals such as Pt, Au, and Ag are very active in the anodic oxidation and cathodic reduction, they are too expensive for practical applications.In this study, copper sulfide modified copper (CSMC) electrode was prepared and used as a new electrode. The efficiency of the (CSMC) electrode for electro-catalytic oxidation of glucose wasinvestigatedand very good results were obtained.Electrochemical behavior of glucose greatly improve at copper sulfied modified copper electrode, indicating that the anodic oxidation of glucose catalyzed at copper sulfide modified copper electrode.Surface physical characteristics of the modified electrode were studied by scanning electron micrographs (SEM). Results showed a good coverage of copper sulfide catalyst particles on the surface of copper electrode.The purpose of the present work is to investigate the electrochemical oxidation of glucoseon modified electrode in alkaline solutions at room temperature. The electro-oxidation of glucose on copper sulfide modified copper electrode have been studied by the methods of cyclic voltammetry, quasi-steady state polarization and chronoamperometry. The rate constants and anodic transfer coefficient (α)and exchange current density (j_0) of the electro-oxidation reactions are reported.

Keywords: copper sulfied, modified copper electrode, glucose, electrochemical behavior.

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Application of curcumin nanoparticles in “Lab on paper” method as a simple, visual and green pH probe

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Abstract

Microfluidic paper-based analytical devices (µPADs) also known as a lab-on-paper are an alternative technology for fabricating simple, cost effective, portable and environmental friendly platforms for clinical diagnosis, chemical and biochemical sensing. The concept of a µPAD is to perform an experiment on a small piece of paper and is well-suited for colorimetric assays [1,2]. The analytical method reported in this paper is to design and fabricate a paper based microfluidic device for the pH sensing using label free curcumin nanoparticle (CURN) as the first green “lab on paper”. Curcumin is a hydrophobic polyphenol compound which has been recognized as the active principle of turmeric. Curcumin has been exhibited the individual properties such as antioxidant, anticarcinogenic, antibacterial. CURN is one of nanomaterials, which it has surface energy transfer property. These properties make CURN extremely versatile compounds [3]. The color of loaded paper based microfluidic device using the CURN changed from yellow to orange and red to brown following the change of pH in the range of 7-13. The mean color intensity change in the relation of pH was recorded and employed as an analytical signal for quantitative sensing of pH. The parameters that affect on this assay were optimized to enhance the selectivity and sensitivity of the method. Under optimum conditions, the extent of mean color intensity was linearly proportional to the pH in the range of 8-13. The relative standard deviation of ten replicates measurements in pH=9 and pH=12 were 2.3% and 1.48% respectively. The assay is a sensitive and precise method for the determination of pH in alkali solutions. The proposed method was successfully applied to the determination of pH in different water samples with satisfactory results. This method possesses many favorable features that including a simple, cost effective, completely user and environmentally friendly and wide dynamic range sensing method.

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Application of Dithiobis (4-methylthiazole) in construction of a new Lu^{3+} Nano-Composite carbon paste electrode

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Abstract

A Modified Nano-Composite carbon paste electrode for Lu^{3+} ions was prepared by employing 2, 2'-Dithiobis(4-methylthiazole) as an ionophore and paraffin oil as a binder. Nano-composite carbon paste electrode was modified by multi-walled carbon nano-tube (MWCNTs) and nano-silica to improvement in their characterizations such as sensitivity and selectivity, especially in term of response time, response stability and lifetime in comparison with typical carbon paste electrodes [1]. The best performance for nano-composite sensor was obtained with electrode composition of 25% paraffin oil, 3% TMT, 3% MWCNTs, 0.5% nano-silica and 40% graphite. The sensors exhibits Nernstian response for the Lu^{3+} ions over the concentration range 5.0×10^{-8}-1.0×10^{-2} M at pH 2.8-9.3 with a lower detection limit of 2.5×10^{-8} M and the slope of 19.9 ± 0.3 mV per decade. The response time was relatively quick in the whole concentration range (~5 s). In this research, potentiometric selectivity coefficients, describing the preference of the electrode for Lutetium ions relative to an interfering ion, were determined by the matched potential method (MPM) [2]. The sensor revealed high selectivity with respect to all common alkali, alkaline earth, transition and heavy metal ions, including members of the lanthanide family other than Lu^{3+}. The Lu^{3+} sensor was successfully applied as an indicator electrode in the potentiometric titration of Lu (III) ions with EDTA [3].

Keywords: ion selective electrode; potentiometric electrode; carbon paste

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

Application of Doehlert Design approach for removal of chromium (VI) and As (V) from aqueous solution using sargassum glaucescens biomass: Modeling, Optimization, and Thermodynamic studies

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Chromium and arsenic are toxic elements, causing adverse effects on human life and environment from surface waters as well as groundwater contamination. Both chromium and arsenic are known to be carcinogen, mutagenic and teratogenic to human health [1].In this paper, response surface methodology (RSM) approach using doehlert design [2] is applied to develop mathematical model and optimize process parameters for Cr (VI) and As(V) removal from aqueous solutions using sargassum glaucescens. The individual and combined effect of three process parameters, i.e. initial pH, biosorbent dose (g l^{-1}) and contact time on Cr (VI) and As (V) adsorption were studied. Analysis of variance (ANOVA) showed the relative significance of process parameters in removal process. Initial solution pH and biosorbent dose were found to be more significant than contact time. The second-order regression model was developed to predict the removal efficiency using design expert software. The results indicated that sargassum glaucescens biomass can be used effectively for the simultaneous removal of Cr (VI) and As (V) even present at high concentrations under the same conditions of pH and biomass dosage. FTIR analysis were conducted to interpret the functional groups involved during the Cr (VI) and As (V) intraction.

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Application of electrochemical impedance spectroscopy for thiourea determination

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Abstract

Thiourea $(NH_2)_2CS$ has the important role in varied industrial process especially in electrorefining process. In cathodic copper refinery units different additives are introduced into the electrolytic bath to maintain cathode quality requirements. These additives, which act mostly as inhibitors, adsorb onto the cathode surface where they take part in the electrochemical crystallization process. Thiourea and gelatin are common organic additives used in modern tank houses to produce smooth, dense and coherent deposits. These additives influence the electrocrystallisation of copper in various ways. Accordingly thiorea determination is very important. The present work describes the application of adsorption ability of thiourea on the surface of platinum electrode for thiourea detection. The results display that thiourea binds to surface and provides a resistance against electron transfer. This resistance is increased linearly with thiourea concentration. The charge transfer resistance (R_{ct}) was measured with the help of electrochemical impedance spectroscopy (EIS) technique. Experimental factors optimized for acquire the best results. The electrode resistance provided a linear response to the thiourea over the concentration range of 0.5-6 ppm with a standard deviation of 6.5% and detection limit of 0.23 ppm.

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Application of Electrochemically Synthesized Hematite/Magnetite Nanocomposite as a New Sorbent to Remove Lead Ions From water

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Water pollution by heavy metal ions such as Pb, As and Cd occur globally [1]. The conventional methods for heavy metal ions removal from water include electrochemical and chemical precipitations, ion exchange, reverse osmosis and adsorption [2]. Among the above mentioned different methods, adsorption of heavy metal ions on the surface of solid nanomaterials is one of the most recommended physicochemical methods is commonly used for heavy metal ions removal from water samples and aqueous solutions [3]. Adsorption is attractive due to its merits of efficiency, cheap and simple operation [4]. In this study, hematite/magnetite nanocomposite is synthesized by the low voltage electrical arc method in the presence of 1.2 Tesla external magnetic field. Scanning electron microscopic (SEM) and transmission electron microscopic (TEM) images show that the synthesized hematite/magnetite nanocomposite includes uniform nanoparticles with 90 nm average diameters. The prepared hematite/magnetite nanocomposite is used as a new nanosorbent to remove lead ions from polluted waters. Experimental data shows that the adsorption of lead ions on the surface of hematite/magnetite nanocomposite is acceptably fitted to the Freundlich isotherm model. Based on the experimental data, a 0.8 mg g^{-1} maximum adsorption capacity is achieved for adsorption of lead ions on the surface of hematite/magnetite nanocomposite. The experimental conditions for the removal of lead ions from waters are optimized by the "one at a time" method. The optimum conditions for lead ions removal includes pH=5, 25 ml sample volume, 25 mg sorbent and 20 min retention time in room temperature. The effects of some ions such as Cu^{2+}, Ni^{2+}, Fe^{2+}, Zn^{2+}, Mg^{2+} and Na^{+} are studied on the removal efficiency of lead ions. The presented method is successfully applied for removal and determination of lead ions from real samples.

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Application of experimental design for removal of sunset yellow by copper sulfide nanoparticles loaded on activated carbon

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In this study a novel and green approach was reported for the synthesis of copper sulfide nanoparticles. This material was characterized using different techniques such as SEM, XRD and BET. The usability of copper nanoparticles loaded on activated carbon (CuS-NP-AC) for the removal of sunset yellow (SY) from aqueous solutions was investigated. The effects of variables such as pH, initial SY concentration, adsorbent dosage and sonication time on its removal were studied and optimized by a central composite design (CCD) combined with response surface methodology (RSM) and desirability function (DF) [1]. A good agreement between experimental and predicted data at optimum value of all variables has been reported. Fitting the experimental equilibrium data to various isotherm models such as Langmuir, Freundlich and Tempkin models showed the suitability and applicability of the Langmuir model [2]. Kinetic models such as pseudo -first order, pseudo-second order, as well as Elovich and intraparticle diffusion models were tested for fitting experimental data. It was found that the second-order equation [3] and intraparticle diffusion models are applicable to explain the kinetic and rate of the adsorption process. The small amount of proposed adsorbent (0.016 g) is applicable for simultaneous removal of SY (RE>95%) with high adsorption capacity (145-153 mg g^{-1}).

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Application of functionalized SBA-15 mesoporous silicas for removal of drugs

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Pharmaceuticals and related products are chemicals of emerging environmental concern. Ciprofloxacin and Ofloxacin are some of antibiotics in Fluoroquinolone group, which are widely used for infections symptoms treatment. Ciprofloxacin can be detected with the wide range of concentration (0.2 ppb - 30 ppm) from several sources, for example, hospital sewage, pharmaceutical production plant, and even in effluent of wastewater treatment plant[1]. Within the large class of Fluoroquinolone carboxylic acids, Ofloxacin is used to treat urinary and respiratory tract infections in humans and animals. Exposures to residues of antibiotics and their transformed products might cause a variety of adverse effects, including acute and chronic toxicity, and microorganism antibiotic resistance) [2].

Removal of pharmaceuticals by adsorption is one of the most promising techniques, due to its convenience once applied into current water treatment processes. Mesoporous silica such as MCM-41, MCM-48, SBA-15 are promising adsorbents owing to their novel structure that comprises uniform ordered structure, high pore volume, and high surface area (up to 1000 m^2/g) [3].

In this communication the preparation and application of SBA-15 mesoporous silica containing sulfonic acid Groups for removal of Ciprofloxacin and Ofloxacin antibiotics from aqueous solutions are reported. The adsorbent was characterized by means of transmission electron microscopy (TEM) and scanning electron microscopy (SEM). UV Spectroscopy was used for antibiotics determination in water samples. The effect of parameters affecting the adsorption process such as aqueous phase pH, contact time, adsorbent dose, temperature and initial antibiotic concentrations were examined. By using 0.02 g of the adsorbents in 20 ml of the water samples containing the studied antibiotics (initial concentration of each 5 mg/l), under optimum conditions; pH=4, contact time=1 h for sorbent, the amount of removed of Ciprofloxacin and Ofloxacin were 92.6 and 67.4%, respectively. The adsorbents were used successfully for removing the target drugs from various real water samples. The analysis of the adsorption data showed that Langmuir and Freundlich isotherms can suitably describe the experimental data obtained by modified mesoporous silica.

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Application of functionalized SBA-15 mesoporous silicasfor removal of drugs

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

Application of GO/ Nd-TiO$_2$/HPC nanocomposite for the removal of Pb^{2+} from aquatic media

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Nowadays, water, soil and air pollution are among the biggest problems of societies, in which heavy metals like lead are the major contributors.

Graphene is a carbon structure made of carbon atoms which are linked together by sp^2 hybridization. Its structure is a monolayer of honeycomb network. Because of its network structure and width of a carbon atom, Graphene has found many versatile applications. One of its most important applications is its use as an adsorbent in removal of some heavy metals including lead and cadmium ions [1, 2]. Organic polymers and titanium dioxide are also able to absorb heavy metal ions, as well. So it's expected that a nanocomposite of these three will be able to have a good adsorption characteristic. Because of having high specific surface area and suitable functional groups, this nanocomposite is considered a useful adsorbent. Also, to transfer the photocatalytic characteristic to the visible area, Nd^{+3}ion is used as a doping agent [3]. To increase the adsorption capability, pH effect, adsorption value and contact time are studied and optimized.

In this research the potential of GO/ Nd-Tio$_2$/HPC nanocomposite to eliminate the Pb^{+2} ions from water was investigated. In order to study the radiation effect on adsorption capability of the nanocomposite and survey the photocatalytic nature, all the experiments were done in presence and absence of the visible light. Effect of influential parameters including pH, removal time, and sorbent amount was optimized. The results proved increase in removal recoveries in presence of light along with appropriate removal recovery.

Key words: Nanocomposite; Graphene; TiO2; Photocatalyst; Heavy metal

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Application of infrared spectromtery for monitoring of mercaptan removal from petroleum refinery products by carbon nano fibers

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Nowadays, environment pollution especially air pollution is one of the main concerns in the world. There are many causes for air pollution. Impurities in the fuels are important source of air pollution and one of them is sulfur [1]. It can be introduced to the environment by oxidative combustion of petroleum based fuels. Mercaptans or thiols (R–SH) are the most common sulfur containing chemicals in petrol based products. Carbon nano fibers (CNF) has been utilized as adsorbent because of their unique physicochemical characteristics, such as their amorphous structure, relatively large surface area, stabilityin acidic/basic environments, being amenable to surface functionalization, and high chemical reactivity because of the exposed edges and unsaturated bonds on their graphene layers[2]. In this work, it was tried to evaluate the role of CNF, in removal of iso-propyl mercaptan (2-propanethiol) from a condensate alkane solution. Samples were collected from the homogenized suspension in different intervals being analyzed by ATR-FTIR spectrometry and the spectral data was processed by MCR-ALS chemometric [3] technique with adequate initial estimation and constraints to evaluate the trend of mercaptane removal. The concentration of the analyte in the same samples was also quantitatively determined according to UOP 163 method. The proposed analytical method demonstrated that overall trend on elimination of mercaptane in such that in the firse hour of the process, about 60% of the mercaptane is adsorbed on CNF. Analysis of samples by standard method confirmed the results of proposed method. The rate of removal was reasonable because the process is conducted in normal condition with no promoter or accelerator while the CNF has not been modified or functionalized, being employed as fabricated.

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Application of inorganic fillers in nanocomposite membranes for gas separation

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Abstract

In recent years, there has been a dramatic increase in the number of research studies carried out on the separation of gases by nanocomposite membranes. Besides, a lot of works have focused on improving the separation performance as well as the mechanical properties of these membranes. Nanocomposite membranes that composed of an intrinsic polymer matrix with inorganic (nano) fillers as dispersed materials, have been investigated too. These membranes have the potential to provide a solution for trade-off problem of polymeric membranes because they possess a combination of proper properties of both the nanoscale inorganic particle and polymer matrix such as good permeability, selectivity, mechanical strength and thermal and chemical stability [1]. This paper first give an outline of the concept and the key advances of nanocomposite membranes with inorganic fillers. Subsequently, recent developments are presented. Furthermore the preparation techniques and structures of nanocomposite membranes with inorganic fillers, their applicability to gas separation are reviewed [2]. The selection of suitable types of inorganic filler such as silica, zeolite, metal oxide nanoparticles and carbon nanotube and the performance of the resulted MMM membranes are discussed and represented the major contribution in this paper [3]. Finally, future direction and perspective in polymer- inorganic nanocomposite membranes research for gas separation was also briefly outlined.

Keywords: Gas separation, Nanocomposite, Membrane, Inorganic fillers.

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Application of L-cystine modified zeolite for preconcentration and determination of trace levels of cadmium by flame atomic absorption spectroscopy

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Abstract

Cadmium is a toxic heavy metal that appears in the environment mainly due to industrial processes. The effect of cadmium poisoning is manifested in a variety of symptoms, including high blood pressure, kidney damage and destruction of red blood cells. Therefore, the determination of trace levels of cadmium in environmental samples is necessary [1]. Amino acids are good metal-binders because they can coordinate with metals through oxygen, nitrogen and sometimes sulfur atoms. Cystine is an amino acid results from cysteine oxidation with the formation of a disulfide bridge [2]. Based on the complexing power of L-cystine toward metal ions and it’s low solubility in neutral aqueous solution, it was applied for the modification of zeolite Y to preconcentrate trace level of cadmium ion.

Using the modified zeolite, a solid phase extraction column and preconcentration method was developed for the determination of cadmium (II) ion in aqueous samples by flame atomic absorption spectrometry. The method is based on sorption of cadmium (II) ion on the modified zeolite. The sorbent exhibited good sorption ability for cadmium (II) at pH= 5. Cadmium ion was eluted from the column by nitric acid. The calibration graph was linear in the range 0.75–80 ng mL^{-1} cadmium in the initial solution. The detection limit and preconcentration factor of this method were found 0.31 ng.mL^{-1} and 100 respectively. The various parameters such as pH, sample and eluent flow rate, volume of the sample, volume and concentration of the eluent solution were optimized. The developed method was applied for the determination of trace amount of cadmium ion in drinking water, river water and wastewater samples.

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Application of liquid phase microextraction based on solidified organic solvent drop for preconcentration of trace amount of copper in environmental samples prior to its determination by flame atomic absorption spectrometry

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Abstract

In this research, a simple and sensitive method of liquid phase microextraction based on solidified organic solvent drop was used for the preconcentration of trace amount of copper prior to its determination by flame atomic absorption spectrometry. In this technique, a free microdrop of the organic solvent which has a melting point near room temperature in the range of 10-30 °C is transferred to the surface of an aqueous sample, while being agitated by a stirring in the bulk of the solution. After the completion of the extraction, a sample vial is cooled and the solidified microdrop is transferred into a conical vial, where it melts immediately. Finally, the analyts can be determined in the extractant. In this method, 2-Hydroxy acetophenoxime, undecanol and ethanol were used as chelating agent, extraction solvent and dilution solvent respectively. Effects of parameters such as pH, type and volume of buffer, ligand concentration, type and volume of extraction solvent, dilution solvent, extraction time, ionic strength, volume of the water samples were investigated. Under the optimized conditions a linear calibration curve in the range of 5.0- 12.0 $\mu g.L^{-1}$, with detection limit of 0.8 $\mu g.L^{-1}$ and preconcentration factor of 66.3 were achieved. The relative standard deviation for six replicate determinations of 0.007, 0.050 and 0.100 $mg.L^{-1}$ of copper (II) were 4.35%, 1.01% and 0.50%, respectively. This method was applied for determination of Cu in real samples.

Keywords: Copper, 2- Hydroxy acetophenoxime, Solidified floating organic drop microextraction, Flame atomic absorption spectrometry

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Application of methyltrioctylammonium chloride as ion pair assisted and disperser agent for dispersive liquid–liquid microextraction combined with microvolume UV–vis spectrophotometric determination of trace bismuth

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Bismuth has been associated with medicine for more than two century, because of their demulcent properties in the gastrointestinal disorders. Bismuth and its compounds are also used in the cosmetic industry, semiconductors, fuel carrier, alloys and metallurgical additives, fabrication of catalysts and in the preparation and recycling of uranium nuclear fuels. As the use of bismuth and its compounds in different areas of life increased, it has spread in the environment and the chance of exposure of organisms to bismuth has increased [1, 2]. Therefore, due to the presence of bismuth in environmental samples at low levels, a fact that claim the use of sensitive, low cost, rapid and reliable analytical methods for the preconcentration and determination of this element at sub µg L^{-1} levels. Dispersive liquid-liquid microextraction (DLLME) is a miniaturized form of liquid–liquid extraction (LLE) that employs microliter volumes of extraction solvent. This technique is based on a ternary component solvent system similar to homogeneous LLE and cloud point extraction. The advantages of the DLLME method are simplicity of operation, rapidity, low cost, high recovery and enrichment factors. This method has been applied for the determination of trace organic pollutants and metal ions in the environmental samples, and also coupled with different detection techniques, such as Uv-vis spectrophotometery [3].

A new dispersive liquid–liquid microextraction (DLLME) method coupled with microvolume UV-vis spectrophotometry was developed for the determination of trace amount of bismuth. The method is based on the complex formation of Bi(III) with iodide (BiI_4^-) and extraction into CCl_4 as an ion pair assisted by methyltrioctylammonium chloride, which also acts as a disperser agent. The effect of important parameters, such as pH, concentration of H_2SO_4 and iodide in the sample solution, amount of methyltrioctylammonium chloride, type and volume of extraction solvent were investigated and optimized. The presented method is capable of determining bismuth in the range of 5 to 400 ng mL^{-1} with a limit of detection 1.66 ng mL^{-1}. The relative standard deviation for eight replicate measurements of Bi(III) at 200 and 75 ng mL^{-1} concentration levels was calculated to be 1.14 and 2.66 %, respectively. The developed method was successfully applied to the determination of bismuth in human plasma, bismuth subcitrate tablet and water samples.

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Application of missing values estimation in multivariate calibration and calibration transfer

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Multivariate data may include missing values for various reasons. Missing values can be estimated using different approached, such as interpolation, NIPALS like procedure and etc [1]. A main goal in the calibration methods such as CLS, ILS, PCR and PLS is the prediction of unknown samples using the estimated model [2]. In this study multivariate calibration purposes is achieved by using missing values estimation strategies. Consequently, multivariate prediction of unknown samples is possible with no need to construction and application of calibration models .Standard error of prediction (SEP) set as the criterion for evaluation of the proposed method .For this purpose NIPALS, Kiere, Wold, and iterative imputation methods were examined. Finally the SEP of calibration methods were compared with the SEPs from the proposed method. Among the calibration models, PLS resulted in lowest SEP values.

The methods of handling missing values were successfully used as for calibration transfer purposes [3], as well . Calibration transfer is utilization of calibration model from one condition to analysis the data of a second condition. Transfer of calibration from one instrument to another is an example. A novel point in this approach is the possibility of calibration transfer in the absence of transfer samples, which is an advantage to common calibration transfer techniques.

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Application of modified magnetic nanoparticles as nano adsorbent for determination of morphine in human hair samples by high performance liquid chromatographic with photo-diode array detection

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Morphine, a phenolic compound is an alkaloid, which can cause disruption in the central nervous system, is frequently used to relieve severe pain in patients, specially that undergoing surgical procedure [1]. Accurate analysis of hair for identifying ingestion of drugs of abuse is dependent on excluding from the measurement drugs present from sources other than ingestion, particularly contaminating drug from the environment [2]. In recent years, a new procedure for SPE, based on the use of magnetic or magnetically modified adsorbents called magnetic solid-phase extraction (MSPE), has been developed [3]. The aim of the present project was to determination of morphine using magnetic solid phase extraction based surface modified magnetic nanoparticles in human hair samples by high-performance liquid chromatography with UV/Vis detection. In this study, after synthesis of magnetic nanoparticles and modification with KH570, magnetic nanoparticles in used as sorbent to extraction of morphine from hair samples. For chromatographic conditions, maximum wavelength 292 nm, flow rate 1 ml/min and mobile phase acetonitrile-phosphate buffer at pH 3 (60-40%) were optimized. The main factors influencing the extraction efficiency including the amount of the magnetic nanoparticles, the extraction time, the pH of sample solution, volume of sample, and desorption conditions such as volume of solvent, time of desorption were studied and optimized. Under the optimized experimental conditions, a good linearity was observed in the range 1-800 μg L^{-1} for hair sample solution with the correlation coefficients (R^2) 0.99 were obtained. The preconcentration factor of 208.69 was achieved in this method. The limits of detection of the method was 1 μg L^{-1} bases S/N=3 and Good reproducibility with the relative standard deviations (n = 5) 2.59 % were obtained. In order to evaluate reliability and applicability of the proposed method, extraction and preconcentration of morphine using modified magnetic nanoparticles for hair samples were evaluated and relative recovery obtained was 87.62%.

Keywords : Morphine, Nano adsorbent, Magnetic nanoparticles, HPLC, Human hair samples

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Application of modified multi wall carbon nano tube with diphenyl carbazone for extraction and determination of trace amounts of cadmium ions in food and natural water samples

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Abstract

The first study on the high efficiency of multi wall carbon nano tube (MWCNT) coated with diphenyl carbazone as a new sorbent solid phase extraction (SPE) has been reported. Sorbent modified multi wall carbon nano tube was used to preconcentration and separation Cd in food and water samples. Nano sorbent was synthesized by the reaction of diphenyl carbazone with functionalized multi walled carbon nanotube. Initially, the surface of the multi-walled carbon nanotube oxidized by a mixture of nitric and sulfuric acid and then functionalized using thionyl chloride. The diphenyl carbazone has been attached to the multi-walled carbon nanotubes in a somewhat shorter time and lower temperature than previous reported methods [1-2].The properties of sorbent were characterized by scanning electron microscope (SEM) and FT-IR. A microsample introduction system was employed for the nebulization micro-volume of diluted solution into FAAS. Some effective parameters on extraction and complex formation, such as pH, concentration and volume of the chelating agent, adsorbent dosage, eluent concentration and volume, extraction time, salt effect and sample volume have been optimized. The calibration graph was linear range between 70-1000 ng mL^{-1} with detection limit of 22.4 ng mL^{-1}. Relative standard deviation (RSD) for Cd ions were 1.3%. The proposed method has been applied to the determination of Cadmium ions at ng mL^{-1} levels in real samples sush as tomatoes, potato, wheat flour, red beans, oat, tap water, river water and sea water with satisfactory results.

Keywords: Solid phase extraction, Preconcentration, Cadmium, Separation.

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Application of modified multi wall carbon nano tube with diphenyl carbazone as sorbent for solid phase extraction for preconcentration of copper ions in food and natural water samples

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Abstract

A simple solid phase extraction methodology based on the application of multi wall carbon nano tube (MWCNT) coated with diphenyl carbazone as a new sorbent was proposed for the preconcentration of trace levels of copper in food and water samples. In this method copper ions was complexed with diphenyl carbazone and extracted into an solid phase. After phase separation, the enriched analyte in the final solution is determined by flame atomic absorption spectrometry (FAAS). Nano sorbent was synthesized by the reaction of diphenyl carbazone with functionalized multi walled carbon nanotube. Initially, the surface of the multi-walled carbon nanotube oxidized by a mixture of nitric and sulfuric acid and then functionalized using thionyl chloride. The diphenyl carbazone has been attached to the multi-walled carbon nanotubes in a somewhat shorter time and lower temperature than previous reported methods [1-2].The properties of sorbent were characterized by scanning electron microscope (SEM) and FT-IR. Some effective parameters on extraction and complex formation, such as pH, concentration and volume of the chelating agent, adsorbent dosage, eluent concentration and volume, extraction time, salt effect and sample volume have been optimized. The calibration graph was linear range between 30-500 ng mL^{-1} with detection limit of 22.4 ng mL^{-1}. Relative standard deviation (RSD) for Cu ions were 3.6%. The proposed method has been applied to the determination of copper ions at ng mL^{-1} levels in real samples sush as tomatoes, potato, wheat flour, red beans, oat, tap water, river water and sea water with satisfactory results.

Keywords: Solid phase extraction, Preconcentration, Copper, Separation.

Reference

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Application of Modified ZnO Nanosheet by S-substituted Thiouracil Derivative for Removal of Pb^{2+}, Cu^{2+} and Ag^{+} Ions

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Abstract

Heavy metal pollution has become one of the most serious environmental problems today. The treatment of heavy metals is very important due to their recalcitrance and persistence in the environment. In recent years, various methods for heavy metal removal from wastewater have been extensively studied. Heavy metals removal methods include chemical precipitation, ion exchange, adsorption, membrane filtration and electrochemical methods. The most important method is adsorption, because it is very flexible, efficience, low-cost and simple in use [1]. ZnO nanosheet is one of nanomaterials that have considerable applications in chemistry. Extraction of mineral ions aiding nano adsorbents is one of the interesting subjects for research. Recently, ZnO nanosheet has been used for extraction of some mineral ions.

In this study, ZnO nanosheet was modified by S-substituted thiouracil derivative. For this purpose, firstly a little methanol solvent was poured on ligand in order to dissolving the ligand in solvent completely. Then, ZnO nanosheet was incorporated there to and left to remove the solvent. This modified adsorbent was used for removal of Pb^{2+}, Cu^{2+} and Ag^{+} ions. Ions concentration was measured by flame atomic absorption spectroscopy (FAAS). The adsorbent values, extraction time and solution pH were optimized. Removal of Pb^{2+}, Cu^{2+} and Ag^{+} ions was done after 30min in 25 ml of 2 mgL^{-1} solution of these three ions by using 15mg of modified adsorbent. In this research, Langmuir, Freundlich and Temkin isotherms were analyzed. It was concluded that absorption process follows Langmuir isotherm model. Finally, adsorption capacity of adsorbent was calculated about 500 µg, 142 µg and 333 µg of Pb^{2+}, Cu^{2+} and Ag^{+} ions per mg of adsorbent, respectively.

Keywords: ZnO nanosheet, S-substituted thiouracil derivative, Removal of Pb^{2+}, Cu^{2+} and Ag^{+} Ions

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Application of MS Office Picture Manager soft-ware in compression of 2D spectral data

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Nowadays, according to the increasing amount of data produced by analytical instruments, proper compression in shortest possible calculation time, in maximum possible compression level, and without loss of information is desired. Large volume of data applies considerable pressure on the supporting systems for data storage. Also the high volume of data results in low processing efficiency and long time of data analysis and processing. According to the above descriptions, the importance of an efficient, rapid data compression technique is obvious [1, 2]. In the previous works, data compression was often based on discrete wavelet transform (DWT) to compress large data sets [1-3]. The main limitation of the method is its complexity to use and the other is this method cannot compress higher order data and it is limited to vectors (1^{st} order data), whereas, in this report a matrix (2^{nd} order data) was considered to be compressed. Also the simplicity of the proposed compression procedure, using the easily available MS Office Picture Manager is an advantage. To show the performance of data, we used some two-way simulated and experimental data. Two dimensional data sets (e.g. HPLC- DAD, or excitation emission fluorescence) were simulated in MATLAB workspace and compressed to a considered level in MS Office Picture Manager. Acceptability of the compressed data sets from the first to nth levels was then examined. The goal is to find the minimum size for data after proper compression. The acceptable level of compression is when the structure of data and its main information retained. The criterion is using estimated Singular Values (SVs) from a data matrix and comparing it with SVs of compressed data. Median Absolute Deviation (MAD) from the correlation between original SVs and SVs of compressed data was utilized as a simple statistical test for determination of the optimum level of compression [1]. In this way, the optimum level of compression is the level for which the value of MAD is not rejected. In this study, the effect of various factors such as different level of noise, peak width, overlap, and the number of components were investigated. According to the obtained results from those simulations it can be concluded that the number of acceptable levels of compression decreased with increase in noise level. The narrower peak width, the lower acceptable level of compression was observed. The number of components and the extent of peaks overlap, affected the number of compression levels. The method was applied for compression of a number of real 2D NMR spectral data sets and results from simulations were confirmed.

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Application of Multivariate Curve Resolution- Alternating Least Square (MCR-ALS) to kinetic spectral data for simultaneous determination of some aliphatic amines

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A kinetic spectrophotometric method based on the reaction of 1,2- naphthoquinone-4-sulphonate (NQS) with amines coupled with multivariate curve resolution-alternating least squares (MCR-ALS) has been proposed for simultaneous determination of ethylamine, propylamine and butylamine. The MCR-ALS methodology adequately exploits the second-order advantage [1] which enables quantitation of analyte in the presence of unknown and uncalibrated interferences. Ethylamine, propylamine and butylamine react with NQS at pH 9.5 to produce spectrophotometrically detectable derivatives. The derivatives show spectra with considerable overlapping. These amines are difficult to be determined on the basis of their respective absorption spectra of their derivatives with NQS. Therefore, determination of these three aliphatic amines has been carried out due to the difference between their reaction rates. Quantitative determination of an analyte in the mixture is performed using a synthetic solution as standard containing only the analyte of interest. Initial estimates of concentration profiles of different analyte were built using evolving factor analysis (EFA) approach. MCR-ALS algorithm was applied to the experimental data under the non-negativity, unimodality and closure constraints. The quantitative determination of these compounds in different synthetic mixtures and some real samples such as river water, well water, tap water and soil has been performed and satisfactory results have been obtained. Using the proposed method, accurate quantitation is possible even in the presence of rank deficiency. Good recoveries were obtained in the range of 88.41-114.96% for all the compounds.

Keywords: Kinetic spectrophotometry, MCR-ALS, aliphatic amines, 1,2-naphthoquinone-4-sulphonate

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Application of multivariate curve resolution approaches to improve analytical separation of Iranian rice volatiles by HS-GC-MS

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Multivariate curve resolution (MCR) methods [1,2] are proposed to improve the analysis of volatile components of Iranian rice sample (Domsiah) by headspace gas chromatography–mass spectrometry. The results of primary analysis indicate the existence of 31 components in aroma volatiles of the selected Iranian rice cultivars. Then extended multivariate curve resolution-alternating least square (MCR-ALS) [3] is used by applying proper constraints to obtain pure elution and mass spectral profiles for the components present in each peak cluster. The resolved components were identified by similarity searches using the NIST mass database and their percentage were calculated for qualitative and quantitative analysis, respectively. The number of identified components was extended to 85 by applying the MCR-ALS techniques. The main identified volatile chemicals that were found in Iranian rice sample (Domsiah) were; Nonanal (18.22%), Hexanal (14.33%), 2-benzoyl-6,7-dimethoxy-4-methylidene-2H-1,3-benzothiazine (7.81%), epilophodione (7.023%) and 1-methyl-4-(1-methylethenyl)-Cyclohexene (6.61%). The percentage values of *LOF* and r^2 resulted for these analyses were to be 18.42 % and 93.26%, respectively, which are acceptable. Results obtained indicate the applicability of combinations of SHS-GC-MS techniques with chemomtrics tools to improvement of analysis of complex samples.

Keywords: Gas chromatography–mass spectrometry, Multivariate curve resolution, Rice, flavor volatiles, Static headspace sampling.

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Application of multivariate image analysis in QSPR study of ^{13}C chemical shifts of β-naphthalene derivatives

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Abstract

Multivariate image analysis-Quantitative structure property relationship (MIA-QSPR) approach has been used to predict ^{13}C NMR chemical shifts of β-naphthalene derivatives. In this method, descriptors correlating with dependent variable are pixels (binaries) of 2D chemical structures. Variant pixels in the structures (substitutes) account for explained variance in the property (chemical shifts). A case study is carried out in order to predict ^{13}C NMR chemical shifts of 10 carbon positions of 24 mono substituted β-naphthalenes. The resulted descriptors were subjected to principal component analysis (PCA) and the most significant principal components (PCs) were extracted. Then, MIA-QSPR modeling was done by means of principal component regression (PCR) and principal component –artificial neural network (PC-ANN) methods. A correlation ranking procedure is proposed here to select the most relevant set of PCs as inputs for PCR and PC-ANN modeling methods. Here, the ^{13}C chemical shifts of studied compounds were predicted using density functional theory (DFT) calculations, too. The widely applied method of gauge included atomic orbital (GIAO) B3LYP/6-311++ G have been used. The performance of the GIAO was also compared with PCR and PC-ANN models. Results showed the superiority of the PC-ANN over GIAO and PCR models.

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Application of multiwalled carbon nanotubes modified for selective solid phase extraction of trace amounts of Au(III)

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From the discovery of carbon nanotubes in 1991, single and multi-walled carbon nanotubes due to their high adsorption and desorption capacities have been employed as sorption substrates in solid-phase extraction for the preconcentration of metal species from diverse matrices. Looking for successive improvements in sensitivity and selectivity, in the past few years, carbon nanotubes have been utilized as sorbents for solid phase extraction in three different ways: like as-grown, oxidized and functionalized nanotubes [1].

A novel sorbent for separation of gold ions was prepared by functionalizing multiwall carbon nanotubes with 5-(4-(dimethylamino) benzylidene)-dihydro-2-thioxopyrimidine-4,6(1H,5H)-dione and underutilized to develop a solid-phase extraction method to separate and concentrate trace amounts of gold ions from some real samples by flame atomic absorption spectrophotometry measurements. The optimum experimental conditions such as pH, flow rates, type and the smallest amount of eluent for elution of gold ions, break through volume and effect of coexisting ions on the separation and determination of gold ions were evaluated. An implementation, it was found that the sorption is quantitative in the pH range 4–8, whereas quantitative desorption occurs with mixture of 5.0 mL of 1.0 mol L^{-1} HCl and thiourea 1.0 mol L^{-1}. Preconcentration factor was 100. Relative standard deviation for nine replicate determinations of 2.0 μg mL^{-1} of gold in the final solution was found 1.83%. The columns were used repeatedly after washing with distilled water. The new sorbent was successfully applied for separation, preconcentration and determination of gold in different samples.

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Application of multiwalled carbon nanotubes modified for selective solid phase extraction of trace amounts of Au(III)

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Application of nanoparticles in pathogen detection

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Nanoparticle-based detection strategy can provide an easy, convenient, and rapid sensing method for a wide range of bacterial pathogens. The rapid and sensitive detection of bacteria is extremely important in the fields of biotechnology, medical diagnosis, and food safety. In recent years, biosensors have been explored for their application in the detection of pathogenic bacteria [1]. Gold nanoparticles (AuNPs) are among one of the most extensively studied nanomaterials. Gold nanoparticles has been widely used to improve the detection sensitivity in various analytical application. Gold nanoparticles exhibit many unique and interesting physical and optical properties such as surface plasmon resonance (SPR). The surface plasmon resonance of gold nanoparticles is an optical property that holds promising potentials in biosensing, molecular imaging, and photothermal treatment of diseases [2]. The combination of spectroscopy detection methods and AuNPs provides a promising platform for the development of highly sensitive and convenient biosensors. We herein present in our study of using UV–vis absorption spectroscopy and transmission electron microscopy (TEM) to determine the interaction between AuNPs with bacteria. AuNPs were applied to detection of some pathogenic and non-pathogenic microorganisms. Various bacterial genus such as Staphylococcus aureus ATCC 25123, Escherichia coli ATCC 25922, Pseudomonas aeruginosa ATCC 27853, Serratia marcescens, Salmonella typhi, Klebsiella pneumonia, Shigella, Bacillus licheniformis, Proteus vulgaris, and Bacillus subtilis were cultured and their interactions with the synthesized AuNPs were evaluated. When bacterial samples were mixed with different synthesized AuNPs, the color of AuNPs were changed from red to violet that can be seen by the naked eye. The aggregation phenomena were investigated by UV–vis spectroscopy and.TEM. This method has proved to be applicable for colorimetric sensing of some pathogen bacteria**.**

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Application of nanostructural poly (o-aminophenol) as a new sorbent for solid phase microextraction of bisphenol-A in leaching from drinking and baby bottle

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Abstract

Conductive polymers due to their multi-functionality, ease of synthesis and their stability, have taken more attractions to be applied as efficient sorbents for the sample preparation purposes especially for solid phase microextraction. Poly (o-aminophenol) (POAP) is a conductive and electroactive polymer which has most usage for construction of electrochemical sensors [1]. Conductivity of POAP is very low but thermal stability of polymer is higher than most of conductive polymers like polypyrole. On the other hands, Bisphenol-A was used by manufacturers as an intermediate in producing epoxy resins, polycarbonate, flame retardants and other specialty products [2]. It has also been shown that bisphenol A leached from lacquer-coated cans and baby feeding bottles [3] due to the hydrolysis of the polymer during thermal treatment. Bisphenol-A has been demonstrated to exhibit estrogenic activity [4]. Most countries have classified it as an endocrine disrupter. In this research nanostructural poly (o-aminophenol) was synthesized by electropolymerization and used for solid phase microextraction. Thin film of Poly (o-aminophenol) (4 µm thickness) was shaped by sweep potential for 45 min on the surface of stainless steel wire. Polymer was synthesized by potentiostat procedure too. Prepared polymer by sweep potential procedure showed nanostructures on the serface. Acetic anhydride was used for derivatization of bisphenol-A and analysis of acetylated bisphenol-A was utilized by gas chromatography-flame ionization detector. Affecting parameters on derivatization and extraction such as amount of acetic anhydride, stirring rate, temperature, ionic strength and extraction time were optimized. Limit of detection and RSD were achieved 0.6 µg/L and 4% respectively under optimized condition. The correlation coefficient (0.9981) indicated a good linearity between 2 and 500 µg L^{-1}. To examine the feasibility of the method, new SPME fiber was applied for analysis of bisphenol-A released from milk and drinking water bottle. All the leachate samples were collected from the containers that had been filled with 50mL of 100 °C hot water. Bisphenol-A was detected in the range 5–15 µg L^{-1} in leachate from plastic baby bottle and was below the LOD for drinking water bottle.

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Application of Nd-TiO_2/PEG/GO nanocomposite for the removal of Pb^{2+} from aquatic media

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Nowadays, water, soil and air pollution are among the biggest problems of societies, in which heavy metals like lead are the major contributors.

Graphene is a carbon structure made of carbon atoms which are linked together by sp^2 hybridization. Its structure is a monolayer of honeycomb network. Because of its network structure and width of a carbon atom, Graphene has found many versatile applications. One of its most important applications is its use as an adsorbent in removal of some heavy metals including lead and cadmium ions [1, 2]. Organic polymers and titanium dioxide are also able to absorb heavy metal ions, as well. So it's expected that a nanocomposite of these three will be able to have a good adsorption characteristic. Because of having high specific surface area and suitable functional groups, this nanocomposite is considered a useful adsorbent. Also, to transfer the photocatalytic characteristic to the visible area, Nd^{+3}ion is used as a doping agent [3]. To increase the adsorption capability, pH effect, adsorption value and contact time are studied and optimized.

In this research the potential of Nd-Tio_2/PEG/GO nanocomposite to eliminate the Pb^{+2} ions from water was investigated. In order to study the radiation effect on adsorption capability of the nanocomposite and survey the photocatalytic nature, all the experiments were done in presence and absence of the visible light. Effect of influential parameters including pH, removal time, and sorbent amount was optimized. The results proved increase in removal recoveries in presence of light along with appropriate removal recovery.

Key words: Nanocomposite; Graphene; TiO2; Photocatalyst; Heavy metal

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Application of Polymer Monolithic Column for Demonstration of Modulation Principles in Comprehensive Two-dimensional Liquid Chromatography (LC×LC)

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A new modulation process was introduced for two-dimensional liquid chromatography (LC×LC) system. Hydrophilic interaction chromatography (HILIC) as first dimension and size exclusion chromatography (SEC) as second dimension columns employed for this work. Monolithic separation column was used as solid phase interface aiming to focus and concentrate analyte bands in the monolithic sorbent from the first dimension effluent prior to injection in the second dimension. Poly ethylene glycol (PEG) was applied as a template for optimization of system. Compared to the non-modulated signal, the presented modulator yielded narrow peaks, on the other hands a concentration enhancement factor up to 16 was achieved. Some chromatographic conditions such as mobile phase composition, flow rate, time programming, kind of first and second dimension column and the preparation of a polymer monolithic separation body were investigated. Water and acetonitrile were used as first dimension mobile phase with a flow rate of 0.080 ml/min and 0.020 ml/min respectively and tetrahydrofuran (THF) was used as second dimension mobile phase with a flow rate of 1.000 ml/min. Time programming organized to switching every 30 second between 2 loops. A monolith synthesized inside a polyether ether ketone (PEEK) tube was selected as solid phase for trapping. This monolithic solid phase has a large through-pore structure to allow the mobile phase to flow through the solid phase at very low backpressure. The butyl methacrylate (BUMA)-based monolithic columns were prepared by in situ polymerization within the confines of 0.8-mm-i.d. and a 2.3-mm-long PEEK tubing. Practically useful monolithic columns were successfully obtained using a polymerization mixture comprsing 20.7% butyl methacrylate (BUMA), 27.5% ethylene dimethacrylate (EDMA), 20.6% 1-propanol, and 30.9% 1,4-butandiol. Modulated LC separation showed that the trapped and concentrated molecules can be released periodically. The modulating interface can both serve as a preconcentration device and injector as the same time for the second dimension column of an LC×LC setup which was better than other systems were reported by other authors [1, 2].

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Application of sawdust nanoparticles as a cheap and non toxic solid sorbent for the removal of lead ions from polluted waters

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Sawdust nanoparticles are prepared by an innovative vibratory ball mill (Spex). The innovated spex consists a stainless steel cylindrical tank with 10 cm inner diameter and 20 cm inner height which is vibrated by an inverter-controlled electromotor. A 100 g sawdust powder is filled in the tank where includes 200 hard chromium electroplated balls. The spex is vibrated by 1200 rpm for 10 h. Scanning electron microscopic (SEM) and transmission electron microscopic (TEM) images show that the milled powder includes spherical nanoparticles with 40 nm average diameters. The prepared sawdust nanopowder is used as a new nanosorbent for removal of lead ions from polluted waters. Experimental data shows that the adsorption of lead ions on the surface of sawdust nanoparticles is acceptably fitted to Langmuir isotherm model. Fourier transform infrared spectroscopy (FTIR) is used to describe the adsorption mechanism of lead ions on the surface of sawdust nanoparticles. The experimental conditions to remove lead ions from waters are optimized by the "one factor at a time" method. The optimum conditions for lead ions removal includes pH=5, 50 ml sample volume, 500 mg sorbent and 20 min retention time in room temperature. The adsorbed ions are easily eluted by 1 ml 1.5 M HNO_3 solution. Based on the experimental data, a preconcentration factor of 500 is achieved. The effects of some ions such as Cu^{2+}, Ni^{2+}, Fe^{2+}, Zn^{2+}, Mg^{2+} and Na^{+} on the removal efficiency of lead ions. The prepared sawdust nanoparticles can be used as an innovative green nanosorbent for removal and determination of lead ions from real samples.

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Application of solvent impregnated microporous silica nanoflakes for microextraction of chlorophenols from wastewater samples followed by gas chromatography-electron capture detection

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Abstract

Chlorophenols (CPs) can be formed by the human industrial activities and can also be generated by the chlorination process of tap water containing aromatic impurities [1]. CPs and several other phenolic compounds, considered as the most toxic and carcinogenic pollutants, have been issued by the US Environmental Protection Agency (EPA) in a list of highly polluting materials [2]. Gas chromatography with electron capture detection (GC-ECD) is a sensitive technique for quantification of CPs. Derivatization of phenols to corresponding acetyl derivatives are usually carried out before the analysis by gas chromatography to obtain sharper peaks and better separation [3]. In the present study, solvent impregnated microporous silica nanoflakes was used as a new mode of liquid phase microextraction, for extraction of CPs from wastewater samples. Microporous silica nanoflakes on the surface of stainless steel wire was prepared by modified hydrothermal precipitation process inside the soda glass tube as precursor. Before the extraction, CPs were derivatized at room temperature by acetic anhydride. For extraction step, the conditioned fiber was immersed into the organic solvent (typically hexyl acetate). The fiber was then inserted into the derivatized sample solution for extraction. Effective extraction and derivatization parameters were studied and optimized. Under optimal conditions, LOD values were between 1 and 530 ng/L and RSDs were from 3.9 to 9.8%. Finally, the method was employed for the analysis of wastewater samples. Two types of wastewaters from Mobarakeh steel company were evaluated as real sample and recoveries were found to be better than 83%.

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Application of spectroelectrochemistry for determination of Uric Acid by Trilinear decomposition methods

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Abstract

Uric acid (UA) is the principal end product of purine’s metabolism; therefor, its determination serves as a marker for the detection of disorders associated with purine metabolism such as Gout and Lesch-Nyhan syndrome. Diagnosis of these deseases is confirmed by monitoring the amount of UA in serum or urine, that requires simple and rapid detection and quantitative methods. Spectroelectrochemistry is a hybrid technique that provides simultaneously electrochemical and spectroscopic information about a system. Spectroelectrochemistry is not only a very good technique to understand reaction mechanisms, but also is intrinsically trilinear technique that can be used for multi-way data analysis. Trilinear decomposition methods can be used for analysis multi-way data, their important is because of their applicability for quantitative analysis of complex multi-component samples even in the presence of unknown interferences; this is commonly called the second order advantages. In this work UV-Vis spectra of uric acid were recorded every second during 65 second and simultaneously cyclic voltamograms were recorded on graphit mesh electrode modified with carbon nanotubes (CNT). Modification by CNT was caused to decrease over voltage of uric acid oxidation up to 300mV. These results indicate that the proposed method can be an alternative for the determination of uric acid in biological samples.

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Abstract

Uric acid (UA) is the principal end product of purine's metabolism; therefor, its determination serves as a marker for the detection of disorders associated with purine metabolism such as Gout and Lesch-Nyhan syndrome. Diagnosis of these deseases is confirmed by monitoring the amount of UA in serum or urine, that requires simple and rapid detection and quantitative methods. Spectroelectrochemistry is a hybrid technique that provides simultaneously electrochemical and spectroscopic information about a system. Spectroelectrochemistry is not only a very good technique to understand reaction mechanisms, but also is intrinsically trilinear technique that can be used for multi-way data analysis. Trilinear decomposition methods can be used for analysis multi-way data, their important is because of their applicability for quantitative analysis of complex multi-component samples even in the presence of unknown interferences; this is commonly called the second order advantages. In this work UV-Vis spectra of uric acid were recorded every second during 65 second and simultaneously cyclic voltamograms were recorded on graphit mesh electrode modified with carbon nanotubes (CNT). Modification by CNT was caused to decrease over voltage of uric acid oxidation up to 300mV. These results indicate that the proposed method can be an alternative for the determination of uric acid in biological samples.

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Application of TiO_2/UV-C Photocatalytical Processes for the Removal of Reactive Red 198

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Abstract

Background: Dyes have complicated structure, are usually toxic and resistant to biological treatment which entere into environment by industrial waste streams [1]. The aim of this study was the removal of reactive red 198 dye (RR 198) by photolysis (UV) and photocatalytical (TiO_2/UVC) processes. This study was conducted to investigate the effects of dye concentration, pH, TiO_2 dose, and contact time.

Methods: The study was carried out by drawing on batch reactor. Synthetic wastewater was prepared by solving of RR198 in tap water. Dye concentration was determined with spectrophotometer on 518 nm.

Results: The results of this study show that Tio_2/UV process is more effective than UV-C process alone. The results of the study also show that constant rate of photochemical degradation in acidic pH and during photocatalytic degradation process was 0.0019 and o.0381 min^{-1}, respectively. Photolysis and photocatalytic degradation of considered dye follow the first order kinetic model. Increasing 0f Tio_2 dose from 0.4 to 10 gr led to Increasing of dye removal efficiency from 97.8 to 100%. Increasing of dye concentration from 100 to 250 mg/l and pH from 4 to 10 led to decreasing of dye degradation efficiency from 100 to 97.66 and from 100 to 94.6, respectively. The best efficiency was at pH=4.

Conclusion: Removal of RR 198 with photolysis and photocatalitical processes has the best efficiency in acidic condition (pH=4) among which the TiO_2/UVC process is better than the UVC.

Keywords: Photocatalytical degradation, UV ray, TiO_2, Reactive Red 198 dye.

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Application of ultrasound-assisted emulsification microextraction for the determination of amlodipine and nifedipine in plasma samples

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Amlodipine besylate (AMB) and nifedipine (NIF) are a dihydropyridine type calcium channel blocking agents. AMB is used in the treatment of hypertension, chronic stable and vasospastic angina [1] and NIF widely used in the management of hypertension in the prophylaxis of angina pectoris and in the treatment of Raynaud’s syndrome [2]. NIF is a lipid soluble drug that is rapidly and completely absorbed from the gastrointestinal tract after oral administration. After oral administration of therapeutic doses of AMB absorption produces peak plasma concentration between 6 and 12 h and >95% is bound to plasma proteins.

A microextraction technique for aqueous samples, known as ultrasound-assisted emulsification–microextraction (USAEME) has been proposed [3]. USAEME procedure combines microextraction system and ultrasonic radiation in one step. Hence, ultrasonic radiation used to assist emulsification the water-immiscible organic solvent.

This study aims to develop an in-syringe USAEME to carry out the extraction, emulsification and phase separation procedures, expanding the automation of USAEME and also simultaneous determination of AMB and NIF in plasma samples. To the best of our knowledge, there is no report on the usability of the USAEME procedure using low-density organic solvents for the determination of AMB and NIF. Central composite design (CCD) combined with desirability function (DF) was applied for optimization purposes. The optimization procedure of in-syringe USAEME (CCD combined with DF) can be used as a new approach to obtain the optimized values of variables in experimental design.

The optimized method was used for determination of these drugs in plasma samples. The results demonstrated that our proposed method had wide dynamic linear range (2 – 1200 ng mL^{-1}) with a good linearity ($R^2 > 0.9991$) and detection limits of 0.170 ng mL^{-1} for AMB and 0.151 ng mL^{-1} for NIF. Enrichment factors were achieved 59 for AMB and 48 for NIF. The established USAEME-HPLC-UV method has been successfully applied for the determination of these drugs in spiked plasma samples. Satisfactory recovery results showed that the matrices under consideration do not significantly affect the extraction process.

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Application of Walnut Hull Waste as Netural Adsorbent for Removal of Heavy Metals from Environmental Samples

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The efficacy of walnut hull waste prepared from agricultural waste was investigated in this study as a novel adsorbent for the elimination of lead (II) from contaminated streams. Removal of lead (II) as a model for heavy metals by walnut hull waste from aqueous solution was studied under different experimental conditions.The selected parameters were solution pH (5), walnut hull dosage (0.5–2 g), lead (II) nitrate concentrations (25–100 mg/L), contact time (15–240 min), and solution temperature (30–50 ˚C). The experimental results indicated that the maximum lead (II) removal could be attained at a solution pH of 5. The dosage of walnut hull was also found to be an important variable influencing the lead (II) removal percentage [1,2,3]. The kinetic analysis showed that the pseudo-second-order model had the best fit to the experimental data.The Dubinin–Radushkevich equation provided the best fit for the experimental data of the equilibrium adsorption of lead (II) onto walnut hull waste at different temperatures.The thermodynamic evaluation of lead adsorption on walnut hull waste revealed that the adsorption phenomenon under the selected conditions was a spontaneous physical process. Accordingly, walnut hull waste was shown to be a very efficient and low-cost adsorbent, and a promising alternative for eliminating lead from enviromental sampels.

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Application of γ-Fe_2O_3@Ag nanocomposites for the removal of sulfide from water samples

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Sulfur is with a content of about 0.048% the 15th most abundant element of our earth's crust. It is an essential element for plants and animals; it makes part of various amino acids, co-enzymes and vitamins[1]. Sulfide contamination in water remains a serious environmental problem and presents a threat to living systems,especially to human health.Consequently,the detection of sulfide has gained great importance. The sulfide in water usually represents the sum of H_2S, HS^- and S^{2-} that remain in equilibrium in aqueous solution[2]. In this work we found γ-Fe_2O_3@Ag, provide high surface area and specific affinity for sulfid adsorption from aqueous systems. monodisperse γ-Fe_2O_3 (maghemite) nanoparticles were used. Nanocomposites were prepared via in situ chemical reduction of silver ions by maltose in the presence of particular magnetic phase and molecules of polyacrylate serving as a spacer among iron oxide and silver nanoparticles[3]. Influence of pH on the adsorption process was studied over arange of 3.0–10.0, adjusted with 0.01 M NaOH or 0.1 M HNO_3. The optimum pH for removing of sulfid from water solutions was found to be 6.0. Effect of other parameters,such as stirring time and weight of nanocomposites on the removal of sulfide were studied and optimum conditions were established.

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Application of γ-MnO_2 nanoclusters for preconcentration and determination of metal ion of pb^{2+} in aqueous samples

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Recently solid-phase extraction (SPE) with nansized adsorbents has been used for preconcentration methods due to advantages such as high surface area, high enrichment factor, high recovery, rapid phase separation and etc[1]. A novel solid-phase extraction (SPE) method application of γ-MnO_2 nanocluster is as adsorbent.

In this study a new, fast, sensitive and simple method with nanocluster manganese dioxde of as γ-MnO_2 have been used for separation and preconcentration of lead in water sample. γ-MnO_2 nanoclusters were synthesized by low temperature hydrothermal method as the previous report[2]. In the presented procedure, γ-MnO_2 is precipitated by chemical oxidation of manganese(II) ions by ammonium persulfate solution. The synthesized sample was characterized by Scanning Electron Microscopy(SEM) and x-ray Diffraction Spectroscopy(XRD). A major step in the chemical understanding of γ-MnO_2 was achieved when Ruetschi and Ruetschi and giovanoli demonstrated that its chemical composition and density could be explained by assuming a Cation vacancy model namely that non-stoichiometry is found only on the Cationic lattice[3]. The proposed SPE method includes two preconcentration and elution steps which their effective parameters including sample volume, preconcentration and elution temperatures, sample pH, eluent composition and weight of solid phase are investigated and optimized by ,,one at a time,, method. The extracted ion are determined by atomic absorption spectrometric(AAS). The Optimal experimental conditions are pH=5, 50mg solid phase for 250ml sample, preconcentration and elution at room temperature, eluent solution including 3 M HCL and 8%wt thiourea and 1.5ml eluent volume. At the optimized conditions, pb^{2+} ion can be extracted by efficiency %94. The presented method showed detection limits of 0.10 ppb

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Applying second-order calibration for quantitative determination of 10-deacetylbaccatin III in *Taxus Baccata* L.

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Abstract

Researches on the *Paclitaxel* (*Taxol)* and its analogs are one of the most stimulating subjects in cancer therapeutic studies. The powerful effects of this compound on breast, ovarian and lung cancer have been proven, however, it is extremely rare and expensive [1]. In the present work, methanolic extract of *Taxus Baccata* L. after treatment with diaion resin and cleaning up by hydrophilic interaction solid phase extraction was analyzed using high-performance liquid chromatography-diode array detection (HPLC-DAD). Due to co-elution and the matrix effect, the classical univariate method for determination of analytes based on the peak areas would fail, and the “second-order advantage” has to be used to solve this problem. Therefore, two second-order calibration methods, multivariate curve resolution-alternating least squares (MCR-ALS) [2] and parallel factor analysis 2 (PARAFAC2) [3] have been used to resolve and quantify 10-deacetylbaccatin III (10-DAB III) in *Taxus Baccata* L. in presence of the matrix interferences. To assess the ability of these methods their results are compared with those of conventional HPLC-DAD in both complete and incomplete separation conditions. The predicted amount of 10-DAB III according to the calibration curves in the mixture obtained by PARAFAC2, MCR-ALS with and without tri-linearity constraint and non-assisted HPLC-DAD were 86.37, 81.08, 84.37 and 20.68 µg/mL, respectively. Quantitative results exhibited that the predicted amount of 10-DAB III with direct analysis in incomplete separation condition is nearly four times less than that obtained using Chemometrics. However, by consuming a relatively long time and solvent and putting some efforts, when the peak of 10-DAB III is completely separated from its co-elutions its amount was found to be 75 µg/mL which is in agreement with the results of HPLC-DAD assisted with Chemometrics. Based on these results, we believe that the proposed methodology can be considered as a faster, cheaper and more accurate alternative method to quantify 10-DAB III in complex mixtures in the presence of co-elution interferences.

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Aptamer coated on magnetic nanoparticles for extraction of adenosine from urine samples followed by electrospray ion mobility spectrometry

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Abstract

Magnetic nanoparticles (MNPs) coated with aptamer was developed for the extraction of adenosine in urine samples followed by electrospray ionization-ion mobility spectrometry (ESI-IMS). MNPs were synthesized by co-precipitating Fe^{2+} and Fe^{3+} in an ammonia solution. Magnetite NPs coated with 3-aminopropyltriethoxysilane were prepared by silanization reaction. After activated with glutaraldehyde, amino-aptamer was covalently immobilized on these MNPs [1]. The ion mobility spectrum of adenosine showed individual adenosine peaks at low concentrations and individual and dimer of adenosine peaks at high concentrations. However, the ion mobility of eluent at low adenosine concentrations showed only peaks, related to dimer of adenosine. In other words, aptamer captured two adenosine molecules between the top G-quartet and the two short stems, where they bonded to each other [2]. The mass spectrometry of the eluent at low adenosine concentrations also validated the presence of dimer (m/z 535.95). The effect of parameters on extraction efficiency was investigated. To ensure the specific interactions between aptamer and adenosine, the same experiments were performed on activated and scrambled magnetic nanoparticles [3]. Scrambled oligonucleotides have the same base composition but in random order. Nearly, 85% of adenosine was separated from the surface of blank and scrambled MNPs after the sample percolation and washing steps and almost 90% of analyte could be retained by aptamer after washing stages. Under the optimized conditions, the linear dynamic range was found to be 0.05-5.00 µg/mL with detection limit of 0.02 µg/mL. The extraction efficiency was 0.94 % and the relative standard deviation was 4 % for three replicates measurements of adenosine at 0.25 µg /mL in urine samples. The method was applied for the determination of adenosine in urine samples of patients with lung cancer. The results demonstrated the capability of the method.

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Aptazyme-Based electrochemical biosensor for the amplified adenosine triphosphate and lead ion detection

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“DNA aptamers and DNA enzymes (DNAzymes or deoxyribozymes) are single-stranded DNA molecules with ligand-binding and catalytic capabilities, respectively. Allosteric DNA enzymes (aptazymes) are deoxyribozymes whose activity can be regulated by the binding state of an appended aptamer domain.”[1]

In the present study, a simple strategy for the rational design of allosteric DNAzyme-based biosensor for electrochemical detection of lead ion has been developed. This design strategy involves the use of two synthetic oligonucleotides, one of which contains an adenosine triphosphate aptamer linked to a DNA enzyme element (8-17 DNAzyme which acts as a nuclease in the presence of Pb^{2+}) and the other of which acts to regulate the catalytic activity of the linked aptamer–deoxyribozyme. This latter molecule, denoted regulatory oligonucleotide (RON), has a sequence complementary to the nucleotides spanning the aptamer and DNAzyme junction.

Here, in the first step, a layer of Allosteric DNAenzymes was self-assembled on the surface of CDtrode. Subsequently, RON was immobilized onto the gold nanoparticles (GNP). In this strategy, RON acts as an “inhibitor”. Immobilized Inhibitors on the GNP surface (INH-GNP) hybridized with aptazyme molecules that self-assembled on the gold electrode surface and they can enhance the sensitivity of the aptazyme-based biosensor [2]. In the absence of the aptamer’s cognate ligand, INH-GNP suppresses the catalytic ability of the aptazyme by forming a stable DNA–DNA duplex. In the presence of the ligand (ATP), the formation of a ligand–aptamer complex alleviates the suppressive effect of the INH-GNP and restores the catalytic activity of the aptazyme. Now, the aptazyme can interact with the substrate,a short oligonucleotide containing a single ribo-adenine (rA), and aptazyme activity was thereby induced cleaving the substrate at its ribonucleotide site in the presence of Pb^{2+}. The changes in the charge transfer resistance have been monitored using the voltammetric and electrochemical impedance spectroscopic (EIS) techniques.

This electrochemical biosensor was proved to have a wide dynamic range from 1mM to 1μM for ATP and capability of lead ion detection at sub-nanomolar scales.

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Assembling and characterization of efficient dye sensitized solar cells with dense/porous titania thin nano film photoanodes and fast redox mediator systems

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Dye-sensitized solar cells (DSCs) were fabricated by using a compact TiO_2 ultra thin layer film (~70 nm) prepared by sol-gel method (TBL-SG) onto a fluorinated tin oxide (FTO) conducting substrate and porous titania films as a photoanode (prepared by sol-gel method) showed enhancement of photovoltaic performance in DSCs with cobalt and ferrocene based mediators in iodide-free electrolytes were developed by selecting a suitable combination of cobalt polypyridine and ferrocene complex and an organic sensitizer. An asymmetry cobalt complex was used as a mediator for first time in dye-sensitized solar cells and it improved the photocurrent of cells. One-electron outer-sphere electron transfer from d orbitals of cobalt is more favorable in LS state and lower symmetry. Thus, the asymmetric cobalt(II) complex ($[Co(dtb)_2bpy]^{2+}$) can perform faster electron regeneration reaction with the oxidized dye in a DSC. All the fabricated DSCs showed best performance with the titania blocking layer.

By use of cyclic voltammetry technique, electrochemical behavior of the mediator complexes investigated in acetonitrile solutions. SEM images results showed the thickness of titania mesoporous layer about 7.9 μm. Anatase particles size of titania thin layer film was about 15 nm and this surface showed a homogenous and very compact morphology. The thickness of titania blocking layer measured about 86.5 nm. Three-dimension J-V measurement plots of the DSCs with titania working electrode and different redox mediators under AM1.5G and dark conditions showed the DSC parameters. Redox couples in DSCs typically transfer an electron to the photooxidized dye on the microsecond time scale. Transient absorption spectroscopy is useful to study the kinetics of this regeneration process [1]. We investigated the regeneration of D35 by a cobalt-based redox mediator via monitoring the decay of dye cation absorption and compared the results with the regeneration of D35 by ferrocene.

Keywords: Dye Sensitized Solar Cell; Sol-Gel; Blocking Layer; Fast Redox Mediator; Asymmetrical Cobalt Complex; LASER Transient Absorption

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Binding Studies probed by experimental and chemometrics methods

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Bone metastases are common in patients with many types of cancer, especially breast and prostate cancer, to these strictly palliative interventions, bisphosphonates have emerged in recent years as a highly effective therapeutic option for the prevention of skeletal complications secondary to bone metastases. The interaction of DNA with four aliphatic chain α-aminobisphosphonate were investigated in a tris–hydrogen chloride buffer (pH 7.4) in the presence and absence of spectroscopic probes (neutral red dye and Hoechest) spectroscopic methods, electrochemical techniques and viscometry. The spectroscopic and voltammetric studies showed that the groove binding mode of interaction is predominant in the solution containing DNA and α-aminobisphosphonates. Furthermore, the results indicated that the binding strength of the DNA - α-aminobisphosphonate complexes are dependent on alkyl moiety presented in the structure of α-aminoalkylphosphonic acids. The principal component analysis (PCA) and theoretical quantum mechanical and molecular mechanics (QM-DFT/B3LYP/6-31+G* and MM-SYBYL) methods were also applied to determine the number of chemical components presented in complexation equilibrium and identify the structure complexes of DNA with α-aminobisphosphonates, respectively.

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Biocompatible Magnetic Iron Oxide Nanoparticles: Synthesis and Physicochemical Characterizations

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Abstract

In this work, magnetite (Fe_3O_4) nano-particles with a size range of 8–12 nm were prepared via the modified controlled chemical co-precipitation method from the solution of ferrous/ferric mixed salt solution in basic normal. In the practice, two types of surfactant includes maleic acid and polyethylene glycol were studied. Maleic acid was chosen as the suitable surfactant to reach ultrafine, nearly spherical and well-dispersed Fe_3O_4 nano-particles, which had well magnetic properties. The size distribution of nanoparticles were determined by particle size analyzer. The magnetite nano-particles was characterized by X-ray powder diffraction analysis (XRD), high resolution transmission electron microscopy (HR-TEM), atomic force microscopy (AFM), fourier transform infrared spectrometer (FT-IR), field emission scanning electron microscopy (FE-SEM) and vibrating sample magnetometer (VSM). The XRD patterns suggested that the Fe_3O_4 nano-particles were pure Fe_3O_4 with a spinel structure and that the immobilizing process did not result in the phase variation of Fe_3O_4. Furthermore the effect of many factors on the Fe_3O_4 nanoparticles was studied, such as reaction temperature, pH of the solution, stirring rate and concentration of maleic acid. And the 3-(4, 5-dimethylthiazol-2-yl)-2, 5-diphenyltetrazolium bromide (MTT) test was done to evaluate the biocompatibility of magnetite nano-particles. The results showed that Fe_3O_4 nano-particles can be produced in the sizes range from 8 to 12 nm by changing the operational factors (for example, concentration of maleic acid, reaction temperature, solution pH, and stirring rate). The saturation magnetization (Ms) of the Fe_3O_4 nano-particles increase from 48.20 to 54.12 emu/g when the sizes of magnetite increase from 8 to 12 nm, which shows that the magnetic properties of the Fe_3O_4 nano-particles are preferable.

Key words

Biocompatible, Magnetite Nanoparticles; Maleic Acid; Polyethylene Glycol; cytotoxicity.

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Biological activities of pyridazine derivatives as novel anti-HIV-1 agent by combining of density function theory and Quantitative structure-activity relationship results

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Abstract

Quantitative structure-activity relationship (QSAR) method could predict the activities of compounds using their molecular and electronic properties. Those properties can be obtained from the calculation in quantum mechanical methods [1,2]. Recently , a new series of diarylpyridazine (DAPD) derivatives have been synthesized with anti-HIV-1 (human immunodeficiency virus type 1) activity [3]. In this study, pyridazine derivatives were investigated by quantitative structure–activity relationship (QSAR) analysis with G09 using density functional theory (DFT) based descriptors. Quantum chemical calculations at the DFT/B3LYP theory level, with the 6-311G* basis set, was employed to calculate a set of molecular properties of 24 pyridazine compounds with anti-HIV activity. The correlation between biological activity and structural properties was obtained by using the multiple linear regression (MLR) method. The QSAR model indicates that the quantum chemical descriptors such as dipole moment, polarizability, stabilization energy , frequency , refractivity, entropy, enthalpy, log P play an important role in the anti-HIV activity of pyridazine derivatives. The results of the present study may be useful on the designing of more potent pyridazine analogues as anti-HIV agents. The best model based on both internal and external validation characteristics was an equation with 6 descriptors and the R square of prediction for the best MLR model was equal to 0.895.

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Biomedical evaluation of blood serum samples by infrared spectrometry utilizing Fisher criterion feature selection and SVM

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Quantitative analysis of bio-clinical parameters is one of the most important aspects of medical diagnosis and treatment. Total protein, triglycerides, glucose and urea contents in blood are determined as metabolic panels for evaluation of liver and kidney disease or diabetes, among other metabolic and nutritional disorders. The routine strategies used in medical laboratories are complicated methods and use expensive reagents together with some tedious analyses. A favorable method for determination of clinical parameters must be easy, fast and accurate one. For this reason many researchers all around the world try to develop the methods which comprise the above mentioned factors. FTIR spectroscopy is one of the techniques that have been applied in biomedical analysis [1]. But it needs to application of some strategies to increase the quality of information that is achieved, especially in biomedical samples which have complicated structures. Chemometrics and FTIR spectroscopy have been utilized for detection of serum samples with normal values of some biomolecules. Fisher criterion [2] based feture selection as a powerful approach was employed to extract the most informative spectral features of infrared spectra obtained from serum samples. Selected variables were introduced to a support vector machines [3] model to build a predictive machine based on bio components' concentration. Combined pattern recognition model was capable of utilization for direct classification of serum samples and illness pattern diagnosis. For samples classified as healthy, the percentage of misclassified samples was found to be 12%, 6 %, 15%, 9%, 15%, 10% and 5% for cholesterol, creatinine, glucose, HDL, LDL, urea repectively.

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biosensor based DNA for study of Enhancers and Inhibitors Antitumor Activities of Vitamin C and K3 Combinations

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Vitamin K3 (menadione, 2-methyl-1,4-naphthoquinone) is a synthetic derivative of vitamin K1, which exhibits antitumor activity against liver, cervix, nasopharynx, colon, lung, stomach, breast, leukemia and lymphoma cell lines[1]. The molecular mechanisms of menadione cytotoxicity are comprised of two main components:

a) covalent binding to macromolecules (protein, DNA) via Michael addition

b) formation of ROS through redox cycling, resulting in oxidative stress that can oxidize lipid, protein and DNA.

Vitamin K3 is reduced intracellularly via one or two-electron transfer. The two electron reduction of quinone to hydroquinone can form nontoxic conjugates or slowly autoxidize to reform quinone. After the single-electron reduction of the quinone to semiquinone, the semiquinone reduces oxygen to the superoxide radical and regenerates the quinone. As a result, redox cycling can ensue and produce large amounts of superoxide, which can dismutate via superoxide dismutase to form H_2O_2 and O_2 or take part in metal-catalyzed reactions to form more toxic species of active oxygen[2].

A biosensor based on interaction of menadione and damage of H_2O_2 and other ROS at a salmon sperm ds-DNA–modified pencil graphite electrode and PDDA-MWCNTS (DNA-PDDA MWCNTs–PGE) was introduced as a promising tool for Survey behavior of menadione. The mechanism of the interaction and damage was investigated and confirmed by differential pulse voltammetry (DPV) and Electrochemical impedance spectroscopy (EIS). The changes in the current of the oxidation signals of guanine and adenine were obtained before and after interaction and damage with menadione. The damage of dsDNA by damaging reagents decreases the charge transfer rate by reducing the negative charge on the electrode surface.

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Biosensor based on ds-DNA decorated chitosan modified multiwall carbon nanotubes for voltammetricbiodetection of Carbenicillin

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Abstract

Voltammetric methods were used to probe the interaction of antibiotic drug Carbenicillin with calf thymus DNA at bare and modified pencil electrode. Binding constants (K) and binding site sizes (s) were determined from the voltammetric data. Carbenicillin showed an oxidation peak at 0.739 V at a bare pencil graphite electrode (PGE). When ds-DNA was added into the Carbenicillin solution, the peak current of Carbenicillin decreased and the peak potential underwent a shift. UV–vis spectra showed that the absorption intensity of the ds-DNA at 260 nm decreased with increasing Carbenicillin concentration, proving the interaction between Carbenicillin and the ds-DNA. The results also showed that Carbenicillin could interact with the ds-DNA molecules via the intercalative binding mode. Finally, a pretreated pencil graphite electrode (PGE) modified with multiwall carbon nanotubes (MWCNTs) and chitosan (CHIT) decorated with the ds-DNA were tested in order to determine Carbenicillin content in solution. Electrochemical oxidation of Carbenicillin bonded on DNA/MWCNTs–CHIT/PGE was used to obtain an analytical signal. A linear dependence was observed to exist between the peak current and 0.03–6.5 ng mL^{-1}Carbenicillin with a detection limit of 0.011 ng mL^{-1}. The sensor showed a good selectivity and precision for the determination of Carbenicillin. Finally, applicability of the biosensor was evaluated by measuring the analyte in urine and drugs samples with good selectivity.

Keywords: Carbenicillin, DNA biosensor Intercalative binding, Chitosan-modified multiwall carbon nanotubes, Voltammetry.

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Biosensor based on ds-DNA decorated chitosan modified multiwall carbon nanotubes for voltammetric biodetection of Doxorubicin

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Abstract

Voltammetric methods were used to probe the interaction of anticancer drug Doxorubicin with calf thymus DNA at bare and modified pencil electrode. Binding constants (K) and binding site sizes (s) were determined from the voltammetric data. Doxorubicin showed an oxidation peak at 0.518 V at a bare pencil graphite electrode (PGE). When ds-DNA was added into the Doxorubicin solution, the peak current of Doxorubicin decreased and the peak potential underwent a shift. UV–vis spectra showed that the absorption intensity of the ds-DNA at 260 nm decreased with increasing Doxorubicin concentration, proving the interaction between Doxorubicin and the ds-DNA. The results also showed that Doxorubicin could interact with the ds-DNA molecules via the intercalative binding mode. Finally, a pretreated pencil graphite electrode (PGE) modified with multiwall carbon nanotubes (MWCNTs) and chitosan (CHIT) decorated with the ds-DNA were tested in order to determine Doxorubicin content in solution. Electrochemical oxidation of Doxorubicin bonded on DNA/MWCNTs–CHIT/PGE was used to obtain an analytical signal. A linear dependence was observed to exist between the peak current and 0.03–6.5 ng mL^{-1} Doxorubicin with a detection limit of 0.011 ng mL^{-1}. The sensor showed a good selectivity and precision for the determination of Doxorubicin. Finally, applicability of the biosensor was evaluated by measuring the analyte in urine and drugs samples with good selectivity.

Keywords: Doxorubicin, DNA biosensor Intercalative binding, Chitosan-modified multiwall carbon nanotubes, Voltammetry.

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Biosensor based on ds-DNA For detection DNA damage induced by Fe(III) and Cu(II) mediated oxidation of dopamine and studied influence antioxidant glutathione and ascorbic acid

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Abstract

There is increasing evidence supporting a causal role of oxidatively damaged DNA in neurodegeneration during the natural aging process and neurodegenerative diseases such as Parkinson’s and Alzheimer’s [1]. The presence of dopamine hydrochloride (DA), a kind of redox-active catecholamine neurotransmitters, coupled with catalytic ferric ions or copper could induce oxidatively DNA damage. DNA damage involves oxidation of guanine to 8- oxoguanine (8-oxoG). It proceeds through the reactive oxygen species (ROS) generation[2]. This paper proposed an electrochemical biosensor have investigated the role of Fe(III) and Cu(II)-catalyzed oxidation of dopamine to induce oxidatively generated DNA damage and its prevention. Incubation of DNA-modified electrode with DA solution containing Cu(II) or Fe(III) has been shown change in the electrochemical properties and the oxidative damage to the DN. It was found that presence of Cu(II) and Fe(III) in solution caused damage to DNA. The inhibitory effect of glutathione and ascorbic acid on the DA-mediated DNA damage has also been investigated using the biosensor. In The present work, a pretreated pencil graphite electrode (PGE) modified with multiwall carbon nanotubes (MWCNTs) and chitosan (CHIT) decorated with the ds-DNA were prepared as a sensor [3]. DNA damage was detected by using differential pulse voltammetry and electrochemical impedance spectroscopy using K3[Fe(CN)6]/K4[Fe(CN)6]. The change in the charge transfer resistance (Rp) and decrease in the intensity of the guanine and adenine oxidation signals after incubation of the DNA biosensor in the damaging solution for a certain time was used as indicators of DNA damage.

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Biosorption of lead (II) ions on *Sargassum ilicifolium*: Application of response surface methodology

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Abstract

In this study, the biosorption of lead (II) ion on *Sargassum ilicifolium*, brown seaweed, was investigated in a batch system. Lead is commonly present in different types of industrial effluents and responsible for environmental pollution, toxicity and heavy metal contamination [1]. Thus, the removal of lead from water is essential. Marine algae are abundant, renewable and could be obtained from the oceans [2, 3]. Recent investigation by various groups have shown that selected species of seaweeds possess impressive adsorption capacities for a range of heavy metal ions and dye anions but no information are available for lead (II) ion from aqueous solutions by *Sargassum ilicifolium*. The effects of operating parameters such as initial pH, initial metal ion concentration and biosorbent concentration on the lead (II) biosorption were studied using response surface methodology (RSM). The optimum biosorption conditions were determined as initial pH 3.7, biosorbent concentration 0.2 g l^{-1} and initial lead (II) ion concentration 200 mg l^{-1}. The maximum uptake capacity of *Sargassum ilicifolium* for lead (II) ions was founded to be 195 mg g^{-1} at optimum conditions. The Langmuir and Freundlich isotherm models were also applied to the equilibrium of system and data were better fitted with the Freundlich isotherm.

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Biosorption of malachite green from aqueous solution by pinus needle leaf

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Malachite green (MG) is an N-methylated diaminotriphenyl- methane dye widely used in the fish and dye industries [1]. Aquaculture industries have been using malachite green extensively as a topical treatment by bath or flush methods without paying any attention to the fact that topically applied theropeutants might also be absorbed systemically and produce significant internal effects [2]. Many physicochemical methods such as coagulation, precipitation, filtration, and oxidation have been attempted for treatment of effluent containing dyes. But, they are not cost effective. The adsorption process has been found to be effective compared to other methods.

In this work, *pinus needle leaf* has been used as a new low cost adsorbent to remove the MG from aqueous solutions. Without any chemical treatment, the leaves were washed, dried and grounded into powder (mesh size 40-70) for use in the experiments. Optimal experimental conditions including pH, adsorbent dosage, temperature, salt concentration and contact time have been established. The experiments showed that the adsorption process was quick and the equilibrium was attained within 15 min using 0.1 g of the adsorbent. The proper pH for removal of MG was about 7.4-9.5. The maximum equilibrium capacity of the pinus needle leaf was calculated from Langmuir model that was 114.9 mg g^{-1}. It was seen that the increase in the salt concentration resulted in a decrease of MG adsorption onto biosorbent and percent removal efficiency. Using the equilibrium concentration constants obtained at different temperatures, various thermodynamic parameters, such as ΔG^0, ΔH^0 and ΔS^0 have been calculated. The results illustrate that the pinus needle leaf is expected to be an effective and economically adsorbent for removal of MG from aqueous systems.

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Biosorption of Zn^{2+}, Cu^{2+} and Ni^{2+} in single and ternary metal systems by *Sargassum ilicifolium*

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Abstract

The biosorption process for removal of Zn^{2+}, Cu^{2+} and Ni^{2+} by *Sargassum ilicifolium* was studied in single component and ternary systems. Heavy metals are persistent environmental contaminants since they cannot be degraded or destroyed. Heavy metal pollution represents an important problem due to its toxic effect and accumulation throughout the food chain leading to serious ecological and health problems [1]. Biosorption, known as the sorption of heavy metals onto biological materials, is becoming a potential alternative for toxic metals removal from waters [2, 3] Experimental parameters such as biosorbent dose, pH and time were analyzed using response surface methodology (RSM). It was found that the overall biosorption data were best described by the pseudo second-order kinetic model. The biosorption process was slightly slower in the ternary system comparing with monometallic system which was related to competition phenomena between metal cations in solution. In monometallic and ternary systems, the Dubinin – Redushkevich (D-R) isotherm model showed a better fit with the following sorption order: $Zn^{2+} > Ni^{2+} > Cu^{2+}$. FT-IR analysis revealed that chemical function groups such as carboxyl, hydroxyl and amine on *S. ilicifolium* would be the active binding sites for biosorption of Zn^{2+}, Cu^{2+} and Ni^{2+}. In addition, the effectiveness of the biomass was investigated in several sorption-desorption cycles using HCl.

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Calibration transfer in presence of unknown interference

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Abstract

Multivariate calibration is a very useful tool for extracting chemical information from spectroscopic signals [1]. However, some problems have been identified by investigators trying to use the above procedure [2]. They all involve a calibration on a primary instrument and an attempt to use the calibration model on a secondary instrument that produces responses that differ in some way. In these cases applying multivariate calibration methods leads to erroneous results [1-3]. A solution to this problem is to apply chemometrics techniques to correct for instrumental and environmental differences, thereby making the model transferable and avoiding full recalibration. These methods are named as calibration transfer methods. Various methods for calibration transfer exist in the literature [1-3]. All of these methods are applicable when there is not any unknown interference. In this studya new method has been proposed to evaluate the performance of calibration transfer approach in presence of unknown interference. The results show the potential of proposed method in order to correct the spectral differences of involving components in presence of unknown interference. It was shown that in presence of unknown interference, ignoring the interference and or applying a classical two way calibration transfer method will lead to inaccurate results. A methodology based on second-order data (excitation emission matrices) with methods for analyzing bilinear data, multivariate curve resolution–alternating least-squares (MCR–ALS), combined with transference of calibration is proposed to predict the analyte concentration in the presence of unknown interference.

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Calibration transfer in rank deficient systems

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Abstract

A common solution to problem of rank deficiencies and complete resolving of it is column augmentation [1]. If there is not any difference between pure spectra of components in different data matrices, then analysis of augmented data gives the parameters and correct spectral profiles. However in the case of individual data analysis, only the parameter can be estimated correctly and not spectral profiles [2-3]. In presence of spectral differences it is not possible to augment the data. Calibration transfer makes it possible to augment the data. In this study, first, calibration transfer method was used as an additional step in a hard modeling procedure to correct the spectral differences between the two data matrices [3]. However in first step, the rank deficient equilibrium system cannot be resolved completely and only the parameters can be estimated correctly and linear combinations of pure spectral profiles are obtained.

In the next step, after calculation of correct parameters, a new method was proposed to calculate the pure spectral profiles of the considered rank deficient system. The proposed new method is based on piece wise direct standardization (PDS) method. However, this step is another new application of calibration transfer in calculation of spectral profiles. This method gives a range of acceptable pure spectral profiles and not unique spectral profiles. In order to investigate the potential of proposed method, three different simulated equilibrium systems were used.

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CA-Membrane Modified with 1-(2-Pyridylazo)-2-naphthol for Preconcentration and Determination of Ag(I) in Water Samples

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The quick determination of minute quantities of ionic species by simple methods is of special interest in analytical chemistry [1]. Silver can enter the environment via industrial water because it is often an impurity in copper, arsenic and antimony industries and silver has been recognized as a toxic element in marine environments [2]. The development of 1-(2-pyridylazo)-2-naphthol (PAN)-modified polymeric membranes (Fig. 1) for the effective batch preconcentration and determination of Ag(I) traces with flame atomic absorption spectrometry (FAAS) is described. The method is based on the chemical bonding of the metal species with a suitable ligand, which has been immobilized into a water-insoluble cellulose acetate (CA) membrane followed by simple rinsing of the chelating agent–metal complex with an acidified methanolic solution. The latter is directly aspirated to the nebulizer of a FAA spectrophotometer without any other treatment. As an analytical demonstration, trace concentrations of Ag(I) were successfully detected in real samples, such as seawater, river and lake water, wastewater as well as in a reference material, without any laborious and time-consuming treatment. Several working parameters were investigated. A pre-concentration factor of 125 was achieved by simple immersing of a circular piece of the CA–PAN membrane (0.6 cm diameter) in the tested samples for 30 min at room temperature. The analytical curve was rectilinear up to 40 µg L^{-1} Ag(I) with detection limit of 0.9 µg L^{-1}, a quantitation limit of 2.9 µg L^{-1} and a relative standard deviation lower than 3.3%.

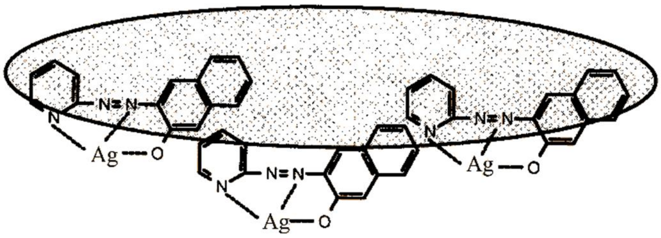

Figure 1. Schematic representation of the proposed extraction

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Captopril detection in pharmaceutical and biological samples using a modified carbon paste electrode in the presence of *para*-aminobenzoic acid as a mediator

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Abstract

Captopril (CAP), (S)-1-(3-mercapto-2-methyl-1-oxopro-pyl)-l-proline, is the first discovered orally active angiotensin converting enzyme (ACE) inhibitor. The CAP is used for the treatment of hypertension, congestive heart failure, and left ventricular dysfunction after myocardial infarction. It contains a sulphydryl group and binds readily to albumin and other plasma proteins [1]. Para-aminobenzoic acid (PABA) is a non-protein amino acid that is widely distributed in nature. Since a small amount of it is present in Vitamin B-complex, it is included as a member of vitamins. Since it improves the protein used in the body and is also related to red blood cell formation as well as for assisting the manufacture of folic acid in the intestines, it is biologically important [2]. Carbon nanotubes (CNTs) have emerged as a novel class of nanomaterials and consequently, receive considerable interest in a plethora of areas [3], none more so than in electrochemistry and electrocatalysis where their use has escalated over the last decade owing to the reported electrocatalytic properties of carbon nanotube modified electrodes. In the present work, we determined CAP using multiwall carbon nanotubes modified carbon paste electrode (MWCNT/CPE) in the presence of PABA as a mediator in an aqueous buffer solution. The characterization of modified electrode was investigated by cyclic voltammetry (CV) and electrochemical impedance spectroscopy techniques. Then the electrochemical behavior of CAP was studied in the presence of PABA at the surface of modified electrode by differential pulse voltammetry (DPV) technique. The voltammograms showed a higher current and more defined voltammetric behavior than that in the absence of PABA. Other voltammetric techniques such as CV, linear sweep voltammetry (LSV), Chronocoulometry and chronoamperometry of CAP were studied. Using these techniques, electron transfer coefficient, exchange current density and diffusion coefficient were calculated. Also some of analytical parameters such as repeatability, linear dynamic range and detection limit for CAP were obtained. Finally, the proposed method was successfully applied for determination of CAP in pharmaceutical and biological samples.

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Carbon nanotubes/ poly-ortho-aminophenol composite as a new coating for the headspace solid-phase microextraction of polycyclic aromatic hydrocarbons

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Ortho aminophenol (oAP) is an interesting member of the class of substituted anilines which is known as nonconducting in its polymeric state. The electrosynthesis of nonconducting polymers with high resistivity, such as poly-o-aminophenol (PoAP), is self-limiting and, therefore, its continuous electrosynthesis is challenging. Carbon nanotubes (CNTs) have been extensively used in a variety of applications based on their unusual physical and chemical properties such as, highly accessible surface area, excellent electrical conductivity, high mechanical strength and good chemical stability. It has been shown experimentally that the introduction of CNTs into a polymer matrix improves the electric conductivity as well as mechanical properties of the original polymer matrix [2]. Despite its self-limiting character, electrodeposition of PoAP has been continued by the addition of oxidized carbon nanotubes into the aqueous monomer solution without any other supporting electrolyte [3]. In this work, oxidized multiwalled carbon nanotubes/ poly-ortho-aminophenol (MWCNTs/ PoAP) composite was directly electrodeposited on the surface of a stainless steel wire as a new coating. It was applied in the headspace solid-phase microextraction (HS-SPME) of a group of polycyclic aromatic hydrocarbons (PAHs) from aqueous samples and determination by gas chromatography with mass detection (GC-MS). The Characteristics of the fiber coating was studied by the scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) spectroscopy. To evaluate the coating, influence of various parameters on PAHs extraction efficiency by HS-SPME was investigated. At the optimum conditions, the linearity and LODs (S/N=3) of the method based on the MWCNTs/ PoAP fiber were 0.01–20 and 0.002–0.01 ng mL^{-1}, respectively. The intra-day and inter-day relative standard deviations at 0.5 ng mL^{-1} concentration level (n=5) using a single-fiber were from 4.7 to 9.3 and 6.4 to 10.1%, respectively. The fiber-to-fiber RSD% (n=3) was between 7.1 and 12.5%. Finally the development method was applied to the analysis of real samples.

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Carbon paste electrode modified with Ag nanoparticles- Polyaniline- Heteropoly acid for voltammetric determination of paracetamol

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Abstract

In this research,carbon paste electrode (CPE) was modified with nano Ag poly aniline heteropoly acid(Ag pani keggin) and the modified electrode was used for voltammetric determination of paracetamol.In the present work differential pulse voltammetry was studied in KCl as electrolyte at the surface of modified electrode,Also cyclic voltammetric result showed a one well defined oxidation peak .The proposed differential pulse voltammetric method was successfully applied to paracetamol in the potential range from 0.5 to 1 V ,under different pH condition but the best result were obtained at PH 2.0 .In addition the result revealed that the modified electrode shows an electrocatalytic activity toward the oxidation of paracetamol under the optimum condition.the anodic peak current showed a linear relation versus paracetamol concentration in the range of 6.62×10^{-7} to 9.92×10^{-5} M,with a regression equation of Y= $2 \times 10^{-5}x - 1 \times 10^{-6}$, ($R^2 = 0.9999$),and detection limit of 0.016 µM (signal to noise=3).Moreover the modified electrode demonstrated good reproducibility(RSD=2.6%, n=10). This method has been applied to the demonstration of paracetamol in waste water and also paracetamol tablet samples.

Refrences:

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[2] Voltammetric Determination of Acetaminophen in the Presence of Codeine and Ascorbic Acid at Layer-by-Layer MWCNT/Hydroquinone Sulfonic Acid-Overoxidized Polypyrrole Modified Glassy Carbon Electrode

[3] S.M.Ghoreishi, M. Behpour , S.Sadeghzadeh, M. Golestaneh . Electrochemical Determination of Acetaminophen in Different Pharmaceutical Forms with GoldNanoparticles Carbon Paste Electrode, Acta Chim. Slov.58,2011,69-74

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Carboxylic derivative multiwalled carbon nanotubes as efficient adsorbent for removal of bromothymol blue

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Abstract

In the present work, an efficient adsorption system for removal of bromothymol blue (BTB) from wastewater using oxidized multiwalled carbon nanotubes (MWCNTs-COOH) has been described. The influence of effective variables such as solution pH, contact time, initial BTB concentration, and amount of (MWCNTs-COOH) and temperature on the adsorption efficiency in batch system was examined. The BTB content was determined by UV-Vis spectrophotometer. Among different kinetic models (pseudo-first-order, pseudo-second-order, intra-particle diffusion and Elovich model), experimental data of removal process followed the pseudo-second-order kinetic model with high correlation coefficient. Evaluating the fitness of experimental equilibrium data by various conventional isotherm models including Langmuir, Freundlich and Tempkin models (based on considering R^2 value as a criterion) show the applicability of Langmuir model for interpretation of experimental data with maximum adsorption capacity of 55.3 mg g^{-1} of adsorbent. Thermodynamic parameters (Gibb's free energy, entropy and enthalpy) of adsorption show that adsorption process is according to general procedure to feasible with endothermic nature.

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CdTe quantum dots as novel and highly efficient nonmaterials for removal of Methylene Blue dye under visible light

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Abstract: The industrial wastewater usually contains a variety of hazardous compounds and toxic substances, and water pollution has become a serious environmental problem facing humans. Thus, there is a considerable need to treat these colored effluents before discharging them to various water bodies [1]. The degradation of pollutants in water has attracted much interest in the last several decades due to the large-scale production of synthetic dyes, slow biodegradation, low decoloration and high toxicity [2]. The annual more than 10,000 different synthetic dyes are produced worldwide production of dyes is more than 7×10^5 tons, and it has been estimated that nearly 5–15% of these lost dyes are continuously entering the water systems from various industries [3]. In this paper the use of QD CdTe has been investigated to degradation the Methylene Blue dye. The QD size was controlled by adding TGA as a capping agent. The QD CdTe was characterized by using X-ray diffraction (XRD) and Transmission Electron Microscopy (TEM), PL and UV-Vis. The average size of Quantum Dot CdTe was found to be 3 nm. The effect of dosage of Q.D, pH, temperature, time and initial dye concentration on the degradation efficiency of dyes were studied. The efficiency of dye degradation for Methylene Blue were nearly 86% .The optimum conditions were obtained in pH=3, T=65 ^{0}C and 30 min for Methylene Blue.

Keywords: CdTe quantum dots, Degradation, Methylene Blue.

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CdTe quantum dots as novel and highly efficient nonmaterials for removal of Thionine and Celestine blue dyes under visible light

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Abstract: The industrial wastewater usually contains a variety of hazardous compounds and toxic substances, and water pollution has become a serious environmental problem facing humans. Currently, synthetic dyes are the main pollutant groups of wastewater . The presence of even very small amounts of dyes in water can cause serious environmental problems, for instance, growth of aquatic bacteria can be reduced light penetration into water by organic dye molecules [1]. Thus, there is a considerable need to treat these colored effluents before discharging them to various water bodies. The degradation of pollutants in water has attracted much interest in the last several decades due to the large-scale production of synthetic dyes, slow biodegradation, low decoloration and high toxicity [2]. The annual more than 10,000 different synthetic dyes are produced worldwide production of dyes is more than 7×10^5 tons, and it has been estimated that nearly 5–15% of these lost dyes are continuously entering the water systems from various industries [3]. In this paper, the use of CdTe quantom dot has been investigated to degradation the celestine blue and thionine dyes. The QD size was controlled by adding thioglycolic acid(TGA) as a capping agent. The quantom CdTe was characteized by using X-ray diffraction (XRD) and Transmission Electron Microscopy (TEM), PL and UV-Vis. The average size of CdTe quantom dot was found to be 3 nm. The effect of dosage of Q.D, pH, time and initial dye concentration on the degradation effeciency of dyes were studied. A modified Langmuir- Hinshelwood model with a pseudo-steady state approachis used for kinetic analysis.

Keywords: CdTe quantum dots, Degradation, Thionine , Celestine blue.

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Central Composite Design Applied for the Optimization of Fatty Acids Esterification Using Hydrochloric acid-Methanol Reagent Followed by Gas Chromatography Determination

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The analysis of fatty acids has become increasingly important, because of their biological functions, nutritional and health implications. Because of this, a method for analyzing fatty acids that provides both rapid and reliable results is of great value. Many methods are currently used to analyze fatty acids. These methods, however, are not necessarily convenient and often must be optimized for reaction conditions, including the catalyst and the temperature [1, 2].

Fatty acids were esterified by Hydrochloric acid-methanol and then determined by gas chromatography. GC analysis was carried out using a BPX70 120 length column. A central composite design was used to optimize the experimental parameters, considering the effects of different temperatures, incubation time of reaction on recovery of esterification. Both temperature and incubation time were found to be significant. The optimum condition obtained 100^{0}C for temperature and 20 minute for incubation time of reaction.

Key words: *Gas chromatography, Fatty acids, Derivatization methods, Response surface method*

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Central Composite design for the optimization of Salting-out cloud point extraction of Cu(II) using anionic surfactant

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ABSTRACT:

In this paper a new and simple cloud point extraction (CPE) method has been developed for the preconcentration and spectrophotometric determination of Cu(II) in water samples. Dithizone was used as the chelating agent and an anionic surfactant [1-2], sodium dodecyl sulfate (SDS), was used as extracting agent at room temperature. Central composite design (CCD) based response surface method (RSM) was applied for the optimization of main experimental parameters such as pH of solutions, concentration of the chelating agent, surfactant and salt. Under the optimum conditions the analytical characteristics of the method such as limit of detection, linear range, relative standard deviation (R.S.D.) and relative standard error (R.S.E.) were calculated. Calibration graphs were linear in the range of 10-100 ng mL^{-1} with detection limits of 3.8 ng mL^{-1} Cu(II). The interference effect of some anions and cations was also tested. The method was successfully applied to the simultaneous determination of Cu(II) in water samples.

***Keywords*:** Cloud point extraction; Cu (II); dithizone; SDS; Experimental design;

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Ceramic/carbon nanoparticle nanocomposite as a solid-phase extraction adsorbent for the determination of the organophosphorus pesticides in aquatic samples

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Solid-phase extraction (SPE) is the most popular sample preparation method and is very actively used in the field of separation science. Many SPE sorbents have been developed, such as silica-based materials, carbon-based sorbents, ion-pair and ion-exchange sorbents, immunoaffinity extraction sorbents, molecularly imprinted polymers, metal-loaded sorbents, and mixed-mode sorbents [1]. Ceramic carbon materials (CCMs) are silica-carbon composite materials that are prepared by mixing carbon powder with sol-gel-derived ceramic binder. Compared with other silica-based materials, CCMs have the advantages of conductivity, stability, macroporosity, heterogeneity, and versatility.

The recent years have witnessed the burst of the nanoscience and nanotechnology in almost all scientific areas. The reason behind such enormous impact is that, on the nanometer scale, the properties of matter can differ from those in the micro and macroworld and even depend not only on the chemical composition and phase but also on the size of the given materials [2]. Organophosphates are the basis of many pesticides and chemical warfare agents. Organophosphorus pesticides (OPPs) are a class of chemicals that generally act as cholinesterase inhibitors and have been widely used in agriculture due to their high efficiency as insecticides [3]

In this study, we reported ceramic/carbon nanoparticles nanocomposite (C/C NPs), for first time, as a new type of SPE adsorbent. Scanning electron microscopy was applied to characterization of C/C NPs. The applicability of this novel SPE adsorbent was evaluated by extracting and determining, by HPLC-UV, four OPPs in water samples. The results demonstrated that our proposed method had wide dynamic linear range (0.1 – 400 ng mL^{-1}) with a good linearity ($R^2 > 0.9972$) and low detection limits (9.2 – 75.0 pg mL^{-1}). Extraction recoveries were achieved ranging from 70 to 99.5%. The established SPE-HPLC-UV method has been successfully applied for the determination of the OPPs in spiked water samples (well, tap, sea and mineral). Satisfactory recovery results showed that the matrices under consideration do not significantly affect the extraction process.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ۱۳۹۲

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Abstract:

An optical sensor membrane based on quinidine red (an acridinium derivative) was constructed and has been developed for the determination of Cr(VI) ion that displays excellent performance over a wide linear dynamic range. The sensing membrane is capable of determining Cr(VI) with an outstanding selectivity over the dynamic range between 3.6×10^{-7} to 1.3×10^{-2} M with a limit of detection of 1.2×10^{-7} M [1]. The sensor shows a fast response time (less than 1 min) and can be used for more than 4 months without observing any major deviation. The optode revealed very good selectivity with respect to many cations including alkali, alkaline earth, transition and heavy metal ions. The proposed sensor could be used in an acidic pH of 5.5 which prevents the interference from many other interfering ions. It was applied to the direct photometric determination of Cr(VI) in black tea, and in water samples. The data given by the proposed optode was also checked with those form atomic absorption spectrometry [2,3]. The results were really reasonable from the precision and accuracy points of view.

Keywords: Cr(VI), Photometry, Quinidine red, Optical sensor

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Chemical Analysis constituent of essential oil of *Pulicaria gnaphalodes*(vent.) Boiss. from Iran

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Abstract: The genus Pulicaria, family Asteraceae, is represented by ca. 100 species, more than 5 of which grow wild in Iran [1-2]. The Pulicaria species proved various activities such as anti-inflammatory, antilukemic, potential cancer chemopreventive and cytotoxic agents [2]. In this investigation the essential oil of *Pulicaria gnaphalodes*(vent.) Boiss. obtained and analysis the plant material collected from Ferdows in July 2013. (Ferdows is a city in land the capital of Ferdows County, located in the northwest of South Khorasan Province in Iran. It is about 345 kilometers south of Mashhad and 200 kilometers northwest of Birjand). The collected plant materials were dried in the shade and suitable air condition. The essential oil obtained by hydrodistillation using a Clevenger-type apparatus from aerial parts of *Pulicaria gnaphalodes*. The composition of the volatile essential oil from aerial parts of *Pulicaria gnaphalodes* was investigated by GC-FID and GC-MS. Kovat's retention indices were calculated using co-chromatographed standards hydrocarbons. The individual compounds were identify by MS and their identity was confirmed by comparing their retention indices relatives to C8 –C32 n-alkanes and by comparing their mass spectra and retention times with those of authentic samples or with data already available in the NIST library and literature Adams [3]. β-citronellol(20.03%) was the main constituent in the oil from aerial parts of *Pulicaria gnaphalodes*. The other major constituent the aerial parts oil were 1,8-cineole (13.48%), α-pinene (6.16%), terpinen-4-ol (4.96%), geraniol (4.67%), α-terpineol (4.51%), citronellyl acetate (4.35%), myrtenol (4.28%) and chrysanthenone (3.98%) [3]. The main component in the isolated oil of *Pulicaria gnaphalodes* were great amount of monoterpenes (80.52%), and smaller amounts of sesquiterpenes (3.24%) from total compound were identified in the plant oil (83.76%).

Keyword index

Pulicaria gnaphalodes, composition, β-citronellol, 1,8-cineole, α-pinene, terpinen-4-ol, geraniol, α-terpineol, citronellyl acetate, myrtenol, chrysanthenone, monoterpenes, sesquiterpenes.

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Chemical Analysis constituent of essential oil of *Pulicaria gnaphalodes*(vent.) Boiss. from Iran

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Abstract: The genus Pulicaria, family Asteraceae, is represented by ca. 100 species, more than 5 of which grow wild in Iran [1-2]. The Pulicaria species proved various activities such as anti-inflammatory, antilukemic, potential cancer chemopreventive and cytotoxic agents [2]. In this investigation the essential oil of *Pulicaria gnaphalodes*(vent.) Boiss. obtained and analysis the plant material collected from Ferdows in July 2013. (Ferdows is a city in land the capital of Ferdows County, located in the northwest of South Khorasan Province in Iran. It is about 345 kilometers south of Mashhad and 200 kilometers northwest of Birjand). The collected plant materials were dried in the shade and suitable air condition. The essential oil obtained by hydrodistillation using a Clevenger-type apparatus from aerial parts of *Pulicaria gnaphalodes*. The composition of the volatile essential oil from aerial parts of *Pulicaria gnaphalodes* was investigated by GC-FID and GC-MS. Kovat's retention indices were calculated using co-chromatographed standards hydrocarbons. The individual compounds were identify by MS and their identity was confirmed by comparing their retention indices relatives to C8 –C32 n-alkanes and by comparing their mass spectra and retention times with those of authentic samples or with data already available in the NIST library and literature Adams [3]. β-citronellol(20.03%) was the main constituent in the oil from aerial parts of *Pulicaria gnaphalodes*. The other major constituent the aerial parts oil were 1,8-cineole (13.48%), α-pinene (6.16%), terpinen-4-ol (4.96%), geraniol (4.67%), α-terpineol (4.51%), citronellyl acetate (4.35%), myrtenol (4.28%) and chrysanthenone (3.98%) [3]. The main component in the isolated oil of *Pulicaria gnaphalodes* were great amount of monoterpenes (80.52%), and smaller amounts of sesquiterpenes (3.24%) from total compound were identified in the plant oil (83.76%).

Keyword index

Pulicaria gnaphalodes, composition, β-citronellol, 1,8-cineole, α-pinene, terpinen-4-ol, geraniol, α-terpineol, citronellyl acetate, myrtenol, chrysanthenone, monoterpenes, sesquiterpenes.

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Chemical Analysis constituent of essential oil of *Pulicaria gnaphalodes*(vent.) Boiss. from Iran

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Chemical composition and evaluation of antioxidant essential oil from laurus nobilis

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Abstract:

Laurus nobilis is a perennial herb that belongs to the lauraceae family. This plant is cultivated in many temperate and warm parts of the world, particularly in Greece, Spain, Portugal, Morocco and Turkey[1]as well Iran. Traditional use of laurus nobilis leaves in the food industry is connected not only with the flavoring but also probably with the preservative capability of the substance present in the leaves. Herbs and spices are important natural antioxidants. Their antioxidant activity has been attributed to the presence of alkaloid and phenolic compounds[2]. In this work The leaves of the laurus nobilis plant gathered from Babolsar in the north of Iran. The essential oils from air-dried leaves of laurus nobilis isolated by Hydro distillation (HD) using a Clevenger-type apparatus and analyzed by gas chromatography-mass spectrometry (GC-MS). The yield of the volatile fraction obtained was 1.4%. The components were identified in the oil with 1,8-cineol,α-terpinyl acetate, methyl eugenol, sabinene , α-pinene, β-pinene , linalool, eugenol as main components. Antioxidant activity and total phenolic content were determined spectrophotometrically(Uv-Vis). Antioxidant activity of the essential oil extracted was evaluated by phosphomolybdenum method. It was expressed as ascorbic acid equivalents. The total phenolic content of laurus nobilis extracts was determined according to the folin-ciocalteu method.

Key word: laurus nobilis, essential oil, antioxidant activity

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Chemical Composition of the essential oil of Ferula Ovina (Boiss) Boiss. From Iran, obtained by hydrodistillation and HS-SPME and comparison result.

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The flora of Iran comprises 30 species of ferula, including 15 endemics[1]. Its several Species are Commercial Sources of asafoetida , an oleo – gum resin used as a flavoring material , a condiment and in medicine . *F. communi*s was reported to be highly toxic to animals and humans. *F. assa-foetida* has several medicinal properties including antispasmodic , aromatic , carminative , digestive , expectorant , laxative , sedative , nervine , analgesic , an the Lminitic , aphrodisiac and antisepic properties . F . gummosa shows several Phramacological activities , including antispasmodic , expextorant , anticonvulsant , anticatarah and antinociceptive . in this investigation composition of the essential oil of *Ferula Ovina* is reported[2].

The plant material was collected from shahrood, province Semnan, Iran. plant material drayed in the shed presences suitable air condition. The essential oil obtained by head space-solid phase microextraction (HS-SPME) instrument and hydrodistillation. Constituent of the oil analyses perform with GC and GC/MS[3].

β-Myrcene (10.57%) , Limonene (13.13%) , Camphen (18.31%) , α-pinene (50.94%)

were the main component in the oil obtained by Head- Space Solid Phase Microextraction (HS-SPME). β-Pinene (5.23%) , Camphene(9.08%), β-Myrcen(10.93%), Limonene (13.29%) , α-pinene 21.45% were the main constituent in essential oil obtained by hydrodistillation.

These results imply that although the use of HS-SPME has the advantages of simplicity, solvent-free sampling and lowering the amount of sample needed, it can also affect the results of the essential oils analysis.

Key words: *Ferula Ovina*, Head Space Solid Phase Microextraction, essential oil, α-pinene Camphen, Limonen.

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Chemical composition of the essential oil of Haplophyllum acutifolium From Iran. Obtained by Hydrodistillation and HS-SPME and comparison result.

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Haplophyllum is the largest genus of Rutaceae in Iran. It has 30 species in Iran that 14 taxa are endemic to Iran. Species of the family generally have flowers that divide into four or five parts, usually with strong scents. They range in form and size from herbs to shrubs and small trees [1]. The Haplophyllum genus has diverse medical properties involving carminative, sedative, antiseptic, appetizing, anti-hiccup, tonic, and anti-convulsion agent[2].

In this work Haplophyllum acutifolium, collected from razavi Khorasan Province, Iran. The plant essential oil obtained by hydrodistillation using a Clevenger–type apparatus. The composition of the volatile oil from aerial parts of salvia chloroleuca was investigated by GC-FID and GC-MS. Kovat's retention indices were calculated using co-chromatographed standards hydrocarbons. The individual compounds were identify by MS and their identity was confirmed by comparing their retention indices relatives to C8 –C32 n-alkanes and by comparing their mass spectra and retention times with those of authentic samples or with data already available in the NIST library and literature Adams[3].Twenty six components, representing 99.26% of the total components were identified. The major compounds were Elemol (20.27%), Sabinene (17.15%) and Trans- Caryophllen (16.07%). The other main constituent of the oil were α-Pinene (8.18%), Limonene (4.72%) and Terpinen-4-ol (5.39%).

Also Essential oil obtained with HS-SPME and analyzed by GC and GC/MS[3]. Sabinene with 39.42%, 72.04%, 43.41% was the main constituents in the leaf, flower and stem oils respectively of Haplophyllum acutifolium. The other main constituent of the oil were α-Pinene in the leaf (32.23%), flower (17.55%) and stem (19.19%), Limonene in the leaf (10.31%) and stem (9.52%), Trans – Caryophllen in the leaf (10.59%) and stem (5.99%). The other notable compound was 1,8- cineole (4.84%).

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Chemical Composition of the essential oil of Prangus Ferulacea(L.)Lindl. From Iran, obtained by hydrodistillation and HS-SPME and comparison result.

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The genus of Prangos with the common Persian name of "Jashir" include 15 species which

, are growing wildly in many regions of Iran. Some species are distributed in Anatolia

Central Asia and Caucasian. Five species delicated to Iran include: *P. gaubae*, *P alligonoides*,*P. cheilantifolia*, *P. tuberculata and P. crossoptera* which are distributed in the north and central provinces of Iran [1]. some of *Prang*os species have been traditionally used as carminative , antifungal , antibacterial , antioxidant[2] .

the plant material was collected from Shahrood, Semnan Province, Iran. the plant material drayed in the shed presences suitable air condition. The essential oil by hydrodistillation and HS-SPME. The obtained essential oil analyzed With GC and GC/MS instrument[3].

the majoor components of the oil obtained by hydrodistillation were beta-phelandren (16.21%), alpha-pinene (14.36%) sabinene(9.90%), ocimen-beta-E (9.35%), terpinen-gamma (4.84%), alpha-phellandren (10.86%), myrcene (6.32%).

Chemical Composition of the Essential Oil from Flowers, Leaves and Stems of *Prangus Ferulacea* by Using Head Space Solid Phase Microextraction showed that The major components of the stems oil were α- pinene (36.13٪) and sabinene (14.94٪). The main component of flower oil were α-pinene(42.18٪), β-phelandrene(18.69%), α-phelandrene(12.83%) and sabinene(12.46٪**)**. The predominant components of the leaf oil were α-pinene(49.49%), ß-phlandrene(17.28%), E-β-ocimene(11.4%) and sabinene(7.91%).

Key words: *Prangus Ferulacea*, Head Space Solid Phase Microextraction, essential oil, α-pinene, phelandrene, sabinene.

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Chemical composition of the essential oil of *salvia chloroleuca* Rech.f and Aell. From Iran.

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The salvia of Iran comprises 58 species, including 17 endemics [1]. The known antioxidant, aromatic and antimicrobial properties of different members of the genus Salvia made it popular in the cosmetic industry, medicine and as food flavouring and preservation products since ancient times. The genus Salvia has widely used in traditional folk medicine [2].

In this investigation *salvia chloroleuca* Rech.f and Aell., Collected from razavi Khorasan Province, Iran. The plant material drayed in the shed and suitable air condition. The plant essential oil obtained by hydrodistillation using a Clevenger–type apparatus. The composition of the volatile oil from aerial parts of *salvia chloroleuca* was investigated by GC-FID and GC-MS. Kovat’s retention indices were calculated using co-chromatographed standards hydrocarbons. The individual compounds were identify by MS and their identity was confirmed by comparing their retention indices relatives to C8 –C32 n-alkanes and by comparing their mass spectra and retention times with those of authentic samples or with data already available in the NIST library and literature Adams[3]. Thirty five components, representing of the total components were identified ٩٤,٣٩%. Sabinene with 14.35% was the main constituents in the oils of salvia chloroleuca. The othr main constituent of the oil were Germacrene-D, α-Pinene, trans-Caryophllene, β- Pinene, 1,8-Cineole, Bicyclogermacrene (١١,٥٩%, ٧,٢٣%, ٧,٧٦%, ٦,٩٧%, ٨,٤٥% respectively). A comparison of the chemical composition of volatiles of *salvia chloroleuca* from this study with previous studies on the same genus showed variation of the major components.

Keyword index:
Salvia chloroleuca, labiatae, Sabinene, trans- Caryophllene, α-pinene, 1,8- cineole, Germacrene-D.

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Chemical composition of the essential oil of *Tanacetum polycephalum* Obtaind by HS-SPME, from Iran

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Tanacetum is one of the largest genera of the family Asteraceae[1]. Various remedies containing *Tanacetum* species are used in the treatment of arthritis, fever, migraine, menstrual disorders, stomach-ache, toothache, and insect bites. *Tanacetum polycephalum* is used in folk medicine to treat many disorders [2].

The present report deals with the analysis and comparison of composition of the volatiles obtained by head space-solid phase microextraction (HS-SPME) procedures from the crushed dry of *Tanacetum polycephalum*. The plant material was collected from Shahrood, semnan Province, Iran[1]. The SPME headspace volatiles were collected using a fiber coated with polydimethylsiloxane (PDMS). procedures from the crushed dry plant of *Tanacetum polycephalum*. The composition of the volatile essential oils was investigated by GC-FID and GC-MS. Kovat's retention indices were calculated using co-chromatographed standards hydrocarbons. The individual compounds were identify by MS and their identity was confirmed by comparing their retention indices relatives to C8 –C32 n-alkanes and by comparing their mass spectra and retention times with those of authentic samples or with data already available in the NIST library and literature Adams[3].

The major components being 1,8-cineole(23.63%), santolina triene(15.45%), α-pinene (14.61%), β -pinene(10.66%), camphor(9.70%). These results imply that although the use of HS-SPME has the advantages of simplicity, solvent-free sampling and lowering the amount of sample needed, it can also affect the results of the essential oils analysis.

Keyword index

tanacetum polycephalum, 1,8-cineol, α-pinene, β -pinene. Chrysanthenyl acetate, camphor

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Chemical composition of the essential oil of *Tanacetum polycephalum* from Iran

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The genus *Tanacetum* is represented by 26 species in the flora of Iran, 12 of them are endemic. *tanacetum polycephalum* growing wild in Iran[1]. *Tanactum* species are reported to possess anthelminic, carminative, antispasmodic and anti-migraine properties having a repellent effect against the Colorado potato beetle. *Tanacetum parthenium* has been used since ancient time for a variety of medicinal purposes, and recently has gained considerable prominence due to its ability to alleviate the symptoms of migraine, arthritis and psoriasis, and to inhibit blood platelet aggregation. As a herbal remedy, *tanacetum* has traditionally been used in balsams, cosmetics, dyes, insecticides, medicines, and preservatives [2].

The plant material was collected from Shahrood, semnan Province, Iran[1]. the chemical composition of the essential oil obtained by hydrodistillation from *tanacetum polycephalum* was analysed by GC and GC-MS . Kovat's retention indices were calculated using co-chromatographed standards hydrocarbons. The individual compounds were identify by MS and their identity was confirmed by comparing their retention indices relatives to C8 –C32 n- alkanes and by comparing their mass spectra and retention times with those of authentic samples or with data already available in the NIST library and literature Adams[3]. The major components being camphor (12.07%), 1,8-cineol(10.66%), trans-Chrysanthenyl acetate (9.57%), α -pinene(8.01%).

Keyword index

Tanacetum, *tanacetum polycephalum*, camphor, 1,8-cineol, α-pinene, Chrysanthenyl acetate

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Chemical composition of the essential oil *Rheum ribs* L. from Iran. Using head space solid phase micro extraction instrument.

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The flora of Iran comprises three species of Rheum of which one of them is endemic [1]. Reheum Ribas use as vegetable products since ancient times in Iran. Rheum ribs is a medicinal plant with various therapeutic effects such as: lowering serum lipids, urea and Creatinin; inhibiting glumerolar mesantial cells, increasing the interval of dialysis in patients with renal failure. Investigation on effects of aqueous extract of Rheum ribs on rat showed that aqueous extract of Rheum ribs only decreased serum cholesterol with no effects on other measured factors in blod[2,3].

In this investigation This plant The plant material was collected from Sbsevar region, Razavi Ghorasan provinces, Iran. the plant material drayed in the suitable air condition. The SPME headspace volatiles were collected using a fiber coated with polydimethylsiloxane (PDMS). The composition of the volatile essential oils was investigated by GC-FID and GC-MS. Kovat's retention indices were calculated using co-chromatographed standards hydrocarbons. The individual compounds were identify by MS and their identity was confirmed by comparing their retention indices relatives to C8 –C32 n- alkanes and by comparing their mass spectra and retention times with those of authentic samples or with data already available in the NIST library and literature Adams [4].the main component in the essential oil were α-pinen with 78.58 percent. The other notable component in the essential oil were Camphene and Limonene (7.22% and 5.53%, respectively).

Key words: *Rheum ribs*, essential oil, α-pinen, Camphene, Limonene

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In this investigation This plant The plant material was collected from Sbsevar region, Razavi Ghorasan provinces, Iran. the plant material drayed in the suitable air condition. The SPME headspace volatiles were collected using a fiber coated with polydimethylsiloxane (PDMS). The composition of the volatile essential oils was investigated by GC-FID and GC-MS. Kovat's retention indices were calculated using co-chromatographed standards hydrocarbons. The individual compounds were identify by MS and their identity was confirmed by comparing their retention indices relatives to C8 –C32 n- alkanes and by comparing their mass spectra and retention times with those of authentic samples or with data already available in the NIST library and literature Adams [4].the main component in the essential oil were α-pinen with 78.58 percent. The other notable component in the essential oil were Camphene and Limonene (7.22% and 5.53%, respectively).

Key words: *Rheum ribs*, essential oil, α-pinen, Camphene, Limonene

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Chemical composition of the essential oil *Rheum ribs* L. from Iran. Using head space solid phase micro extraction instrument.

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Chemiluminescence of graphene quantum dots induced by direct chemical oxidation and its analytical application

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We report on the first observation of chemiluminescence (CL) of graphene quantum dots (GQDs) induced by direct chemical oxidation. GQDs are graphene sheets smaller than 100 nm that exhibit numerous unique physicochemical properties due to the pronounced quantum confinement and edge effects. Compared to semiconductor QDs, GQDs show considerably low toxicity, excellent solubility, chemical inertia and stable photoluminescence [1,2]. In this work, GQDs were prepared by a simple carbonization method and characterized by X- ray diffraction, Fourier transform infrared spectroscopy and transmission electron microscopy. It was found that Ce(IV) could oxidize GQDs to produce a relatively intense CL emission. The mechanism of CL generation was investigated based on the fluorescence and CL emission spectra. It was attributed to the radiative recombination of oxidant-injected holes and thermally excited electrons in the GQDs. In order to investigate the analytical application potential of GQDs-Ce(IV) CL system, it was applied to the determination of uric acid. Under the optimized conditions, the proposed CL system exhibited excellent analytical performance for determination of uric acid in the range of 1.0×10^{-6} M to 5.0×10^{-4} M with a limit of detection of 5.0×10^{-7} M. The method was applied to the determination of uric acid in human plasma and urine samples, with satisfactory results.

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Cloud point extraction and HPLC determination of pantoprazole sodium in Pharmaceutical and Biological Samples

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Abstract

Pantoprazole is a selective and irreversible proton pump inhibitor used in medicine as an antiulcerative agent. It is used in the treatment of gastroesophageal reflux disease (GERD), reflux esophagitis, gastric ulcers, duodenal ulcers and Zollinger-Ellison Syndrome [1]. Cloud point extraction (CPE) is based on the phase behavior of non-ionic surfactants in aqueous solution، which exhibit phase separation after an increase in temperature or the addition of a salting out agent. Separation and perconcentration based on CPE are becoming an important and practical application of surfactants in analytical chemistry [2, 3]. A simple, rapid and low cost method for determination of pantoprazole in pharmaceutical and human plasma samples was investigated. The method is based on the cloud point extraction of pantoprazole and the subsequent determination by HPLC-UV. The use of mixture ammonium acetate buffer (pH adjusted to 6.5 with sodium hydroxide)/acetonitrile (52:48 %v/v) as mobile phase at a flow rate of 1 ml/min and UV detection set at 290 nm was found to serve as the optimum mobile phase composition. Effect of parameters such as the percentage of non-ionic surfactant Triton X-114 (0.0-0.3% v/v), pH (3-8), ionic strength (0.0-0.1 M NaCl) and temperature (25-70 ºC) on recovery and enrichment factor were investigated and optimized. Under the optimal conditions, i.e. 0.15% (v/v) of Triton X-114 at pH=5, ionic strength of 0.15 %v/v and temperature 55 ºC, 5 mL of human plasma containing of Triton X-114 was centrifuged. Good enrichment factor and recovery were observed for pantoprazole. Recovery, enrichment factor, LOD and relative standard deviations (RSD%) for pantoprazole was >90%, 92, 0.7µg/ml and less than 6%, respectively. The proposed method was successfully used for determination of pantoprazole concentration in human plasma and pharmaceutical samples.

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Cloud Point Extraction and Preconcentration of Hg(II) in Natural Samples with New Schiff Base Ligand and Determination it by Cold Vapor Atomic Absorption Spectrometry (CVAAS)

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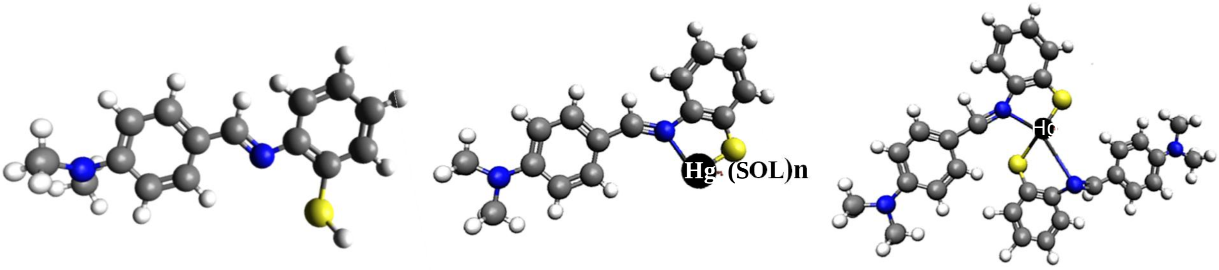
The determination of trace Hg(II) in biological samples is particularly difficult because of the complex matrix and the usually low concentration of Hg(II), which requires sensitive instrumental techniques and frequently a preconcentration step. Cloud point extraction (CPE) is an upcoming technology to preconcentrate and separate many of the trace elements from different chemical and biological systems. The system is sustainable as it involves benign extractants like surfactants and that too at low concentrations at slightly elevated temperatures to form clouds that separate out from the bulk solution. In addition, the extraction behavior of many elements depends on its chemical species [1]. In the first part of this research new thiolic ligand of (E)-2-[4-(dimethylamino) benzylideneimino]thiophenol (DABT) was prepared from reaction of ortho-aminothiophenol and para-dimethylaminobenzaldehyde at labratory temperature in MeOH as a solvent. The purity of the ligand and its complexes were checked by FT-IR, UV-Vis spectroscopy, elemental analysis (CHN) and conductivity measurement. The structure of ligand is presented in scheme 1.



Scheme 1. A) the structure of ligand B) the structure of ML(Sol= solvent) C) The structure of ML$_2$

In the second part of this research, we have developed a procedure for the determination and preconcentration of Hg(II) in the real samples at pH 6 via complex it with the (E)-2-[4-(dimethylamino)benzylideneimino] thiophenol was investigated as a new schiff base Ligand in the CPE with non-ionic surfactant Triton X-114 prior to it determination by CVAAS. Analytical parameters including concentrations for DABT, Triton X-114 and HNO$_3$, bath temperature, centrifugation rate and time were optimized [2]. The influences of the matrix ions on the recoveries of Hg(II) ions were investigated. The detection limits (3SD$_b$/m, n=5) of 1.6 ng L^{-1} along with enrichment factors of 60 for Hg(II) were achieved. The proposed procedure was applied to the analysis of environmental samples.

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Cloud point extraction for spectrophotometric determination of nanosized copper oxide in food samples and using Taguchi orthogonal design to multivariate optimization

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Triton X-1۰۰ based cloud point extraction has been demonstrated to be an advantageous approach for the recovery of nanosized copper oxide[1-3] (NCO) from rice. Nano Copper Oxide in the initial aqueous solution was complexed with sodium diethyldithiocarbamat (NaDDC) . Dilution of the surfactant-rich phase with acidified methanol was performed after phase separation and nano Copper Oxide contents were measured by UV-Visible spectrophotometric technique. The variables affecting the cloud-point extraction were optimized using Tauguchi orthogonal array design. The effective factors including pH, extraction time, temperature extraction ,amount of the triton X- 100 , concentration of salt and concentration of the ligand were studied and optimized . Under the optimized condition , the calibration graph was linear in the range of 1-30μg.L^{-1} for Nano Copper Oxide. The limit of detection (LOD) was 0.93 μg.L^{-1}. The relative standard deviation (RSD % n=7) for the determination of 10 μg.L^{-1} was 4.2% . The proposed method has been applied successfully to determine the trace amount of nano copper oxide in some of rice samples.

Keywords : Cloud point extraction; Nano Copper Oxide; Triton X-100; Food sample

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CNT/ZnO composite coating on the copper wire as Novel fiber for solid phase microextraction

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Abstract

A highly porous fiber coated carbon nano tube (CNT)/ZnO on cupper fiber materials were prepared for solid-phase microextraction (SPME) methods. The CNT/ZnO ceramic was synthesized by spray pyrolysis technique [1]. The prepared spray solution was sprayed on cupper wire for fabrication of the SPME fiber. The fiber was evaluated for the extraction of some polycyclic aromatic hydrocarbons (PAHs) from aqueous sample solutions in combination with gas chromatography–flame Ionization detector (GC–FID). In optimum conditions (CNT/Zinc Acetate ratio1:1, extraction temperature 45 °C, extraction time 30 min, ionic strength 30% (W V^{-1}), stirring rate 900 rpm, desorption temperature 290 °C, desorption time 2 min) the repeatability for one fiber (n = 3), expressed as relative standard deviation (R.S.D. %), was between 3.0% and 7.3% for the tested compounds. The limit of quantization the studied compounds were between 5.1 and 20.2 ng L^{-1}. The life time of fiber and stability of the CNT/ZnO fibers are very good, and it can be used more than 120 times at 290 °C without any decreasing at the extraction efficiency. The developed method offers the advantage of being very simple to use, with shorter analysis times, lower cost of equipment, good thermal stability of fiber and high relative recovery in comparison to conventional methods of analysis.

Keywords: CNT/ZnO composite, PAHs, SPME

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Cobalt ions removal from waste waters in south pars gas complex by glycine-coated magnetic Co_3O_4 nanoparticles

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Co_3O_4 nanoparticle were successfully synthesized by solvothermal process[1] and were functionalized with glycine, by a costeffective and environmentally friendly procedure, as an alternative route to typical aminefunctionalized polymeric coatings, aiming to highly efficient removal of Cobalt ions from waste water. The as-synthesized products were characterized by X-ray diffraction ,transmission electron microscopy, scanning electronic microscopy and FT-IR spectroscopy techniques.The amino-functionalized Co_3O_4 nanoadsorbent exhibited high adsorption affinity for aqueous Cobalt ions, resulting from complexation of the metal ions by surface amino groups.The metallo aded glycine - Co_3O_4 nanoparticles could be recovered readily from aqueous solution by magnetic separation and regenerated easily by acid treatment.Factors influencing the uptake of cobalt ions such as solution pH, initial metal ion concentration and contact time were investigated systematically by batch experiments. Findings of the present work highlight the potential for using glycine-functionalized Co_3O_4 magnetic nanoparticles as an effective and recyclable adsorbent for the removal of cobalt ions in water and wastewater treatment. The nanoadsorbent was able to remove over 98% of Ccobalt ions in 50 mL of solution containing 5 mg/L ions at optimized conditions.Furthermore, these nanoadsorbents can be used as highly efficient separable and reusable materials for removal of toxic cobalt ions from waste waters.

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Coil Galvanizing Based on Passive Chromium(III)-Based Materials

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Chrome plating can be done as chemical and electrochemical methods on the metals surface and metalic coated materials in a solution media of chromic acid, chromate, and dichromate. This operation can cause a passive coating on the metals by the chromium(III) and chromium(VI) compounds. Although the chromium (VI)-based compounds represent harmful effects on the employee and user health, galvanizing plant at Steel Company of Mobarakeh had been used these materials. This problem can be increased when people employ the product without any safety tools. Recently, using of these materials in the manufacturing process are limited or forbidden due to these health problems. Thus, at the galvanizing plant the less harmful materials such as the chromium(III)-based materials are used instead of chromium(VI)-based ones. To get this objective pretreatment of the coils is necessary as follows:

First, the coils was cleaned using alkaline solutions. Then, the coils was washed using demineralized water and dried. After pretreatment of the coils, two approaches can be done to protect the coils: 1- Spraying and dewatering; in this method the solution is sprayed on the both sides of moving sheets and dewatering is done by the appropriate rolls. The remaining wet can be dried by blowing the warm air (this method is used at galvanizing plant of steel company of Mobarakeh). 2- Floating and dewatering; in this method the coils are moving at the baths filled of chromium solution and dewatering is done the same as item 1.

The appropriate materials for this objective have several characteristics including: a) the mater should be sprayable (without any excess washing), and colorless. b) It should be usable at operation temperature. c) It should be passive rather than the salt spray to 48 hours. d) The coating should be stabile for a long time storing

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Keywords: Galvanizing, Passivation, Chromium(III)

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Colorimetric determination of mercury (II) ions in presence of thiourea based on cloud point extraction of silver ananoparticles

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Mercury is identified as a highly toxic element because of its accumulative and persistent character in the environment and living organisms. Mercury contamination can occur from industrial wastes, as well as from mineral deposits [1]. Mercury hazards to living organisms such as marine samples are due to the accumulation and biomagnification character of this toxic element that can influence the entire food chain and humans who consume marine food [2]. Therefore, routine monitoring and control of mercury are becoming increasingly important, especially in water systems. Cloud point extraction using micelles is a popular methodology for preconcentration and separation of metal ions and organic compounds [3]. In this research a cloud point extraction method using silver nanoparticles (AgNPs) and non-ionic surfactant Triton X-100 for the spectrophotometric determination of mercury (II) in the presence of thiourea is described. Label free AgNPs are enriched into the non-ionic surfactant phase and show strong surface plasmon resonance (SPR) intensity. In the presence of thiourea the intensity of SPR is increased and then adding mercury (II) to this system leads to decreasing of SPR intensity. This decrease in the absorbance of surfactant rich phase was used as an analytical signal for the determination of mercury (II). The effect of chemical variables such as pH of the sample solution, electrolyte, concentration of thiourea, and concentration of AgNPs on the cloud point extraction was studied. The SPR absorption intensity was linearly proportional to the concentration of mercury (II) in the range of 3 -150 ng mL^{-1} under optimum conditions. The detection limit based on $3S_b$ was 1.4 ng mL^{-1} and the relative standard deviation for ten replicate measurements of 20 and 130 ng mL^{-1} of mercury (II) was 3.6 and 0.6%, respectively. The proposed method was successfully applied to the determination of mercury (II) in water samples with satisfactory results.

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Mercury is identified as a highly toxic element because of its accumulative and persistent character in the environment and living organisms. Mercury contamination can occur from industrial wastes, as well as from mineral deposits [1]. Mercury hazards to living organisms such as marine samples are due to the accumulation and biomagnification character of this toxic element that can influence the entire food chain and humans who consume marine food [2]. Therefore, routine monitoring and control of mercury are becoming increasingly important, especially in water systems. Cloud point extraction using micelles is a popular methodology for preconcentration and separation of metal ions and organic compounds [3]. In this research a cloud point extraction method using silver nanoparticles (AgNPs) and non-ionic surfactant Triton X-100 for the spectrophotometric determination of mercury (II) in the presence of thiourea is described. Label free AgNPs are enriched into the non-ionic surfactant phase and show strong surface plasmon resonance (SPR) intensity. In the presence of thiourea the intensity of SPR is increased and then adding mercury (II) to this system leads to decreasing of SPR intensity. This decrease in the absorbance of surfactant rich phase was used as an analytical signal for the determination of mercury (II). The effect of chemical variables such as pH of the sample solution, electrolyte, concentration of thiourea, and concentration of AgNPs on the cloud point extraction was studied. The SPR absorption intensity was linearly proportional to the concentration of mercury (II) in the range of 3 -150 ng mL^{-1} under optimum conditions. The detection limit based on 3S$_b$ was 1.4 ng mL^{-1} and the relative standard deviation for ten replicate measurements of 20 and 130 ng mL^{-1} of mercury (II) was 3.6 and 0.6%, respectively. The proposed method was successfully applied to the determination of mercury (II) in water samples with satisfactory results.

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Colorimetric sensing of nitrite based on Surface Plasmon Resonance of gold nanoparticle and nanoparticle size effect

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Nitrite (NO_2^-), as a inorganic contaminants in drinking water, have proven to be of great risk to human health [1]. Thus it can be attractive topic for research in analytical chemistry and developing a rapid, highly sensitive, and selective detection of NO_2^- is extremely essential for human life safety. Here, we offer a simple method to NO_2^- recognition based on its ability to etching gold nanoparticle (Au NPs, synthesized with citrate reduction) surface [1]. This effect results in a notable decrease in NPs Surface Plasmon Resonance (SPR) which is linearly proportional to NO_2^- concentration. On the other hand, NPs size can have a remarkable effect on this phenomenon. Based on this effect, NO_2^- determination was tested by using different sizes of Au NPs and it was showed that Au NPs with size of 8 nm in diameter caused more sensitive detection compared with other sizes. The diminution effect of NO_2^- on these NPs is linearly altered over the concentration ranges of 8–120 μmol L^{-1}. The limit of detection was 2.4 μmol L^{-1}, and other common anions nearly have no notable influence. The method was successfully applied to the determination of NO_2^- in water samples.

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Colorimetric sensing of Zinc based on cloud point microextraction: an approach for separation/preconcentration of samples with the dispersion of TiO_2 nanopowders in micellar phase

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Analytical techniques do not present sufficient sensitivity for the trace determination. Therefore the development of preconcentration procedures prior to the analyte determination has been extensively explored in recent decades. Different preconcentration techniques such as solvent extraction, coprecipitation [1], cloud point extraction [2] and solid-phase extraction [3] have been utilized for analyte enrichment. In the present study, TiO_2 nanoparticles (TiO_2-NPs) were applied in cloud point microextraction for efficient preconcentration/separation and analysis of Zn^{2+} in various samples. In this approach, Zn^{2+} ions are adsorbed on TiO_2-NPs and transferred into surfactant rich phase. Subsequently the adsorbed Zn^{2+} ions are desorbed from TiO_2-NPs with dithizone solution by forming a colored complex which could be detected colorimetrically. The influence of chemical variables such as pH of the sample solution, electrolyte type and concentration, amount of NPs, type and volume of eluent on the microextraction system was studied. The calibration graph was linear in the range of 0.5-90.0 ng mL^{-1} of Zn^{2+} (correlation coefficient of 0.9996). A preconcentration factor of 100 was achieved and the detection limit for Zn^{2+} was 0.0083 ng mL^{-1}. The relative standard deviation (RSD) for eight replicate measurements of 10 ng mL^{-1} and 60 ng mL^{-1} of Zn^{2+} was 1.5% and 1.8% respectively. The proposed method was successfully applied to the quantitative determination of Zn^{2+} in tap water, powder milk and Zinc sulfate tablet with satisfactory results.

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Combining Radial Basis Function Neural Network with Genetic Algorithm to QSAR Modeling of Adsorption on Nano-material Surface

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Construction of radial basis function networks (RBFNs) usually involves selection of radial basis function (RBF) centroid locations, in addition to the number of RBF units in the hidden layer. Methods such as genetic algorithm (GA) [1], and *K*-means [2] and cluster analysis are among center selection methods. In all reported casses centers are rows of **X** matrix.

A combination of RBF networks and GA is introduced for better description of quantitative structure-activity relationship (QSAR) models. In this method centers are not exactly rows of descriptor matrix and can be located in any point of samples space. In the proposed approach, centers are randomly selected from calibration set, then GA changes the value of the selected centers and choose the centers from whole space of score matrics to obtain best center values in order to increase the prediction ability. This method was applied for prediction the adsorption coefficients (logk), of 40 small molecules on multi-walled carbon nanotube (MWCNT) surface. The data consists of five solute descriptors [R, π, α, β, V] of these molecules. Prediction ability of RBFN-GA was compared to RBFN and multiple linear regression (MLR) analysis [1]. The Q^2 values for RBFN-GA, RBFN when GA is used to center selection from rows of X matrix, and MLR were calcualted as 0.95, 0.85, and 0.78, respectively, that showed the merit of RBFN-GA.

The method also was applied on activity of a series of 1-[2-hydroxyethoxy-methyl]-6-(phenylthio)thymine] (HEPT) derivatives, as non-nucloside reverse transcriptase inhibitors (NNRTIs). The data included 107 derivatives of HEPT anti-HIV molecules. Prediction ability of RBFN-GA was compared to combinations of cluster analysis (CA) and *K*-means clustering with RBFN (RBFN-CA and RBFN-*K*-means). The Q^2 values for RBFN-GA, RBFN when GA is used to center selection from rows of X matrix, RBFN-*K*-means, and RBFN-CA were calcualted as 0.91, 0.82, 0.80, and 0.71, respectively.

From these results, it can be said that the proposed method is effective to QSAR studies and improve Q^2 value.

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Comparative investigation of petroleum source by infrared spectroscopy and chemometrics

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Petroleum industry demands robust powerful analytical approached which could support the quality inspection, structure investigation, forensic analysis and even economic evaluation [1]. Petroleum samples are different in chemical composition, based on their origins, transformation, unexpected events like adulteration, etc. Difference in chemical composition affects their refining procedure, quality parameters and finally marketing [2]. Considering the useful capability of infrared (IR) spectroscopy as a rapid, economic and non-destructive method for identification of chemical samples, a practical approach was investigated for analysis of petroleum samples based on their informative spectral features in mid-IR region. In this work, totally 85 samples from 3 different sources: 2 petroleum reservoirs and one production for export were compared in binary models, by Fourier transform infrared (FTIR) spectroscopy and least square support vector machines (LS-SVM). Variable selection based on genetic algorithm (GA) was utilized as preprocessing method to improve the reliability of proposed method. Results of binary comparisons were obtained from conducted classifications. More test accuracy values steams from more differences and reversely less test accuracy values refers to more similarities between different fields. The accuracy values for binary classification were between 91.3-97.1 %

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Computational design and synthesis of a high selective molecularly imprinted terpolymer for voltammetric sensing of minoxidil in real samples

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Abstract

Thin film of a molecular imprinted polymer (MIP) based on electropolymerizationmethod with sensitive and selective binding sites for minoxidil (MX) were developed. Thisfilm was cast on glassy electrode by electrochemical polymerization in solution of o-phenylenediamine(o-PD), gallic acid(GA), p-aminobenzoic acid(ABA) and template MX viacyclic voltammetry scans and further deposition of Ag nanoparticles (NPs) [1]. Densityfunctional theory (DFT) approach was developed to study the intermolecular interactions inthe polymerization mixture and to find suitable functional monomers in MIP preparation [2,3]. There is big number of hydrogen bonding for interaction of minoxidil (MX) with threefunctional monomers ABA, GA, o-PD in comparison with two and one functional monomer insolution and gas phase including basis set supper position error. DFT results withinpolarizable continuum model are agreement with experimental electrochemical result in allcases. The surface feature of the electrode modified with molecularly imprinted polymer wascharacterized by cyclic voltammetry (CV), scanning electron microscopic (SEM) andelectrochemical impedance spectroscopy (EIS). Several important parameters controlling theperformance of the Ag NP - MIP/GCE were investigated and optimized. Under optimumconditions the oxidation peak current was proportional to MX concentration over the range0.03-500 µM. The detection limit (DL) was found to be 0.01µM. This sensor has beensuccessfully applied for the determination of MX in real samples.

Keywords: Molecular imprinted polymer; Electropolymerization; Computational design; DFT, Hydrogen bonding, Silver nanoparticles

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Computational studies on the interactions of magnolol and honokiol with human serum albumin

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Magnolia Balk Extract (MBE) used in Asian traditional medicine for the treatment of anxiety, sleeping disorders, and allergic diseases [1]. Magnolol (MG) and honokiol (HK) are the two primery active phenolic constituent of MBE. Since bioactive components of MBE are potential herbal drugs, it was of interest to obtain insight in to their binding affinities with the carrier proteins such as Human Serum Albumin (HSA) using experimental techniques and molecular modeling.

Human serum albumin (HSA) is plentiful plasma protein that has a greatly transport capacity of blood plasma and ability of binding for many of substance. Its single polypeptide chain consist of 585 amino acids with three α-helical domains (I, II, and III) and each domain contains two subdomain A and B. three binding regions of HSA are located in subdomain IIA, IIIA, and IB that are well known site1, site2, and site3 respectively[2].

In the present work binding modes, binding free energies and inhibition constants, (KI), between of HSA-MG and HSA-HK systems have been investigated by using molecular docking method. Molecular docking results indicated that the main active binding site for MG is site1. also the main active binding site for HK is site2. The results are listed in Table 1.

Table1: Amino acid residues involved in HSA-MG and HSA-HK systems interaction with the free binding energy for the best selected docking positions.

Component	Binding energy (kcal/mol)	KI (µM)	Residue of HSA which is responsible for binding
Magnolol	-7.43	3.57	Phe 149, Tyr 150, Glu 153, Ser 192, Ser 193, Lys 195, Gln 196, Lys 199, Leu 238, His 242, Arg 257, Ala 291, Glu 292
Honokiol	-6.79	10.52	Leu 387, Ile 388, Asn 391, Cys 392, Phe 403, Leu 407, Tyr 411, Leu 430,Val 433, Gly 434, Cys 438, Leu 453, Leu 457, Arg 485

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Computer-aided sensor design to analysis of triamterene in pharmaceutical and biological samples using electropolymerized molecularly imprinted polymer based on multivariate optimization methods

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Molecularly imprinting technology gets increasingly interesting for the preparation of useful materials with predetermined selectivity for application in several areas of analytical chemistry [1]. In this study, a computational approach was used for screening functional monomers and polymerization solvent in the rational design of molecularly imprinted polymers (MIPs) [2]. On the basis of computational results, pyrrole (PY) and dimethyl sulfoxide (DMSO) were selected as functional monomer and polymerization solvent, respectively. A sensitive MIP-based electrochemical sensor was fabricated successively by electrodepositing carboxyl functionalized multi-walled carbon nanotubes (MWNTs-COOH) and electropolymerizing pyrrole in the presence of triamterene (Tr) onto a pencil graphite electrode (PGE) surface. The MIP-MWCNT electrode showed high recognition ability in comparison with NIP-MWCNT. The influences of several parameters on response of sensor were investigated using multivariate methods [3]. A Plackett–Burman experimental design for screening has been used to evaluate the influence of nine parameters (concentration of MWCNT, deposition time of MWCNTs onto the bare PGE surface, concentration of PY, concentration of Tr, number of cycles in electropolymerization, scan rate of cyclic voltammetry technique, stirring rate of solution, electrode loading time and pH of buffer solution) on the analytical response. Then, the significant parameters have been simultaneously optimized using a central composite design (CCD). The optimum values for the three significant parameters i.e. concentration of MWCNT, deposition time of MWCNTs onto the bare PGE surface and number of cycles in electropolymerization were calculated to be 0.47 g/l, 2000 s and 15, respectively. Then the calibration curve was plotted. A dynamic linear range of 0.08–265 µM was obtained. The detection limit of the sensor for Tr was obtained 3.35 nM ($3s_b$, n=7). The prepared sensor showed a good reproducibility (RSD% of 3.99, n=3) and regeneration capacity. The selectivity of MIP electrode in this work was evaluated in the presence of different interfering molecules. This sensor was successfully used for Tr determination in serum and pharmaceutical samples.

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Conducting Polypyrrole Film Doped with Anionic Chromotropic Acid as a Solid State Potentiometric Sensor for Cr(VI)

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Development of novel chemical sensors is closely related to the development of new materials for both molecular recognition and signal transduction. Potentiometric ion sensors based on conducting polymers are relatively new types of chemical sensors. Conjugated polymers are useful as transducers in chemical sensors. One of the key issues is to combine conjugated polymer transducers with selective molecular recognition in order to obtain durable chemical Sensors [1]. Present research describes the preparation and the performance of polypyrrole film doped with anionic chromotropic acid as a solid state potentiometric sensor for Cr(VI). Polypyrrole (PPY) films were obtained on different substrates by anodic electropolymerization using chromotropic acid disodium salt (CHR) as an anionic dopant. The deposition of PPY film has been performed by the means of cyclic voltammetry. The effect of polymerization condition on the characteristics of potential response of the sensor towards Cr(VI) was investigated. The concentration of pyrrole and CHR, the electrode substrate, the number of voltammetric cycles and pH were optimized. Under the optimized conditions, the proposed sensor works satisfactory in the concentration range of 10^{-4} to 10^{-1} mol L^{-1} of chromate ion with a slope of 29.8 mV $decade^{-1}$ and a fast response time of 30 s. The developed sensor has been applied for the potentiometric determination of chromate in industrial wastewater samples.

Keywords: Chromotropic acid disodium salt, Polypyrrole, Chromate, Ion selective electrode.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ۱۳۹۲

Conversion of Manganese Oxides in the presence of cerium (IV) ammonium nitrate: Catalyst evolution

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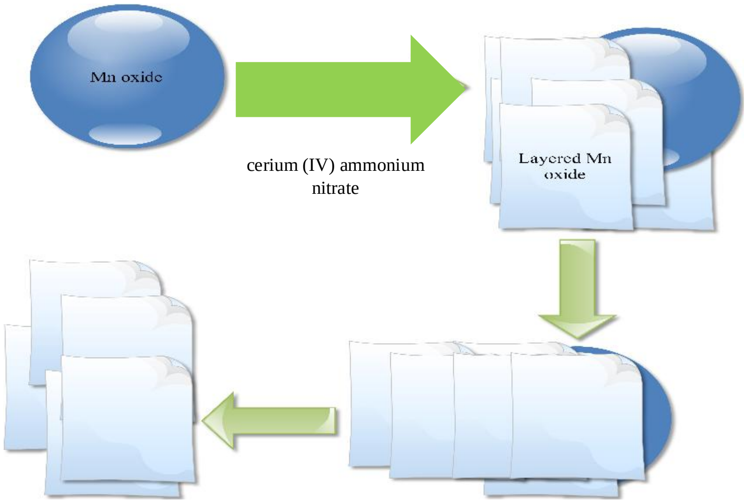
Hydrogen is a promising material that can replace fossil fuel. A simple way to produce hydrogen is electrolysis of water. Water electrolysis includes two half-reaction: water oxidation occurs at the anode, and water reduction occurs at the cathode. Water electrolysis in industrial scale is limited by water oxidation process [1]. Thus, we need an effective and stable catalyst for water oxidation.

Mn oxides have been reported as heterogeneous catalysts for water oxidation by some groups [2]. In 2011, in a different approach and aimed at simulating the Mn_4CaO_5 cluster in PSII, layered Mn oxides was introduced as a structural and functional analogs to the WOC in PSII found so far [3].

Here, we found that Mn_3O_4, α-Mn_2O_3, β-MnO_2, $CaMnO_3$, $Ca_2Mn_3O_8$, $CaMn_3O_6$ and $CaMn_4O_8$ in the presence of cerium (IV) ammonium nitrate (Ce(IV)) convert to layered Mn oxide. The converted compounds were characterized by SEM and TEM.

According to Pourbaix diagram, we can conclude that many Mn oxides can convert to nanolayered Mn oxide. We also studied oxygen evolution of these oxides before and after conversion. The new results show that converted oxides are more efficient catalysts toward water oxidation.



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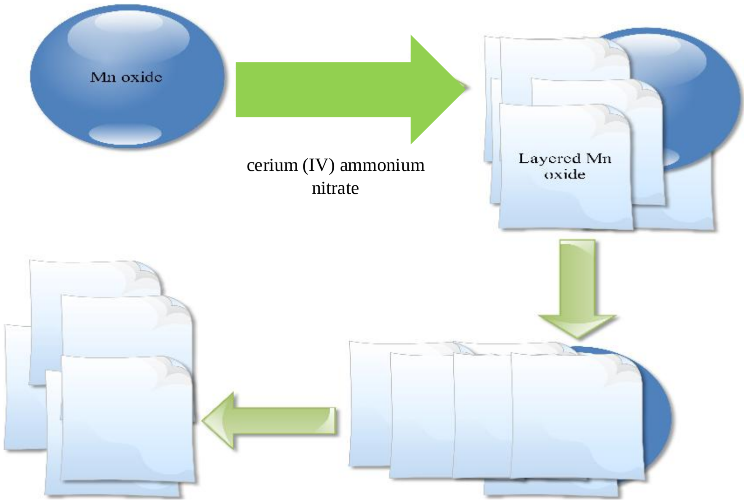

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Copper (II) ions solvent extraction frome chlorid solution by oxime ligands

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Abstract

Oximes are also known as a class of powerful acidic extractants [1], applied in various metal separation and purification investigations [2-3].3-tert-butyl-2-hydroxy-5-methyl benzaldehyde oxime (**HL^1**) and 3-tert-butyl-2-hydroxy-5-methoxy benzaldehyde oxime (**HL^2**) are synthesized and characterized by conventional spectroscopic methods. Distribution study of the prepared ligands between sodium chloride aqueous phase and dichloromethane shows their suitability for using in solvent extraction process. Liquid-liquid extraction experiments were performed for illustrating extractive performance of (**HL^1**) and (**HL^2**) ligands towards copper(II) ions. The selectivity of the studied oximes was checked by performing a competitive extraction experiments of Cu^{2+}, Cd^{2+}, Zn^{2+}, Co^{2+}, Ni^{2+} and Pb^{2+} ions (each 1×10^{-4} M) from aqueous solution in different pH into dichloromethane solution of the ligands (0.005 M). The results shown that the extraction percentage of the Cu^{2+} ions by **HL^1** and **HL^2** in pH 6.5 and 6 respectivly is about 100% wherever extraction percentage of other ions except Co^{2+} with **HL^1** is under 10%. The observed results show a high selectivity of the ligands towards copper ions. Both ligands transfer copper ions into the dichloromethane organic phase by a cation exchange mechanism Conventional log-log analysis show a 1:2 (metal: ligand) stoichiometry for the copper extracted species by both studied extractants. Both oximes were successfully applied to the extraction, recovery and detection of copper in different real samples.

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Coprecipitation for preconcentration trace amounts of manganese, nickel and cobalt prior to its FAAS determination

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The monitoring of the levels of heavy metal ions in environmental samples is an important part of analytical chemistry due to their positive or negative influences on the human body [1]. Atomic absorption spectrometer (AAS) is generally main instrument of analytical chemistry laboratories for the determination of traces heavy metal ions, due to its relatively low cost. However the main two problems for the determination of heavy metal ions by AAS are low levels of metal ions and interferic influences of main components of samples. Therefore, it is usually necessary to carry out a preconcentration/or separation step prior to the analysis. Coprecipitation is one of the efficient separation/preconcentration techniques for traces of heavy metal ions. The coprecipitation technique has some advantages: simple, fast method and several analyte ions can be preconcentrated and separated from the matrix simultaneously.

In the present work, we developed a carrier element-free coprecipitation [2], by using an organic coprecipitant, 2-[4-(N,N-dimethyl) ohenyl] 5(4H)-oxazolone for separation and preconcentration of manganese, nickel and cobalt ions. In our study, we have performed an insoluble form (in water) of organic compound, used as a coprecipitating agent, and adsorbed the heavy metals on the precipitate. It is a great advantage due to there is no contamination and interference risk for analytes from a carrier element. The influences of analytical parameters such as amount of coprecipitant, sample volume, diverse ions, etc. were investigated.

Under optimized conditions, calibration curves were constructed for determination of the analyte ions. Linearity for the analyte ions in the original solution (35 mL) were maintained between 0.005 and 1.0 μg mL^{-1} for manganese, 0.005 and 1.5 μg mL^{-1} for nickel and 0.01-2.0 μg mL^{-1} for cobalt. The detection limits of the method based on $3S_b/m$ (n =7) for manganese, nickel and cobalt ions were 1.5, 1.8 and 3.2 ng mL^{-1} in the original solution, respectively. Seven replicate determinations of a mixture of 0.2 μg mL^{-1} manganese, nickel and cobalt ions in the original solution gave relative standard deviations of 2.1%, 1.7 and 2.0%, respectively. Finally, the developed method has been successfully applied for the determination of the analyte ions in the real water samples.

Key words: Coprecipitation; preconcentration manganese, nickel and cobalt determination

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Coprecipitation of trace amounts of silicon with aluminum hydroxide and the determination by flame atomic absorption spectrometry

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Abstract: Coprecipitation has been widely used for the preconcentration of trace amounts of analyte [1]. According to the literature survey, coprecipitation of silicon with $CaCO_3$ [2], and $Al(OH)_3$ [3] have been reported in geology and polymer literatures but no analytical research has been performed for preconcentration of silicon through coprecipitation. In the presented research a simple preconcentration method of silicon based on coprecipitation of it with aluminum hydroxide prior to its flame atomic absorption (FAAS) determination is established. The recovery values of analyte ions were higher than 95%. The parameters including types of hydroxide ion source for precipitation, acid type for dissolution step, amount of aluminum ion as collector, pH, temperature, standing and centrifuge time, and sample volume were optimized for the quantitative recoveries of the analyte. The influences of matrix ions were also examined. The relative standard deviation was found to be 3.2%. The limit of detection was calculated as (0.1 mg L^{-1}).The preconcentration factor is 100 for (200 mL) solution. The proposed method was successfully applied for the determination of silicon in natural water and alloy samples.

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Core/shell Fe_3O_4@Ag magnetic nanoparticles modified carbon paste electrode for enhanced electrochemical detection of olanzapine in schizophrenia patients

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In this work,carbon paste electrode modified with Fe_3O_4@Ag magnetic nanoparticles (MNPs) was used to investigate the electrochemical behavior of olanzapine. Olanzapine is an antipsychotic drug that is used in treatment of patients with schizophrenia. At first, magnetic nanoparticles of Fe_3O_4 as a core were synthesized by co-precipitation of Fe(II) and Fe(III) ions in deionized water under N_2 gas protection.Then silver nanoparticles weresynthesized, as shell, by adding solution of $AgNO_3$(6.4×10^{-2}M) and $NaBH_4$ was utilized as a reductant agent [1]. The synthesized Fe_3O_4@Ag core/shell magnetic nanoparticles were characterized by X-ray powder diffraction (XRD), transmission electron microscopes (TEM) and scanning electron microscopy (SEM). Fe_3O_4@Ag core/shell MNPsmodified carbon paste electrode enhanced the electron transfer rate and showed good electrocatalytic behavior for the oxidation of olanzapine [2, 3]. Utilizing these MNPsled to an enhancement in the surface area of carbon paste electrode, which is calculated to be 0.076 cm^2. The effect ofsome experimental parameters such as pH of solution, scan rate, amount of modifier, accumulation time (t_{acc}) and potential (E_{acc}) were calculated. The electrode process was controlled by adsorption and t_{acc} and $E_{acc}$was calculated to be 150 s and 0.25 V, respectively. The lowest difference between anodic and cathodic peak potentials and the highest currentwas observed in acetate buffer (pH 4.3) and the best ratio of carbon paste to modifier was7:1. The proposed electrode was used for determination of olanzapine in blood serum and plasma of human with Schizophrenia.

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Correlation solubility of Beclometasone Dipropionate in supercritical carbon dioxide (CO_2)

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Abstract

The corticosteroid Beclometasone Dipropionate (BDP) which was selected as the model drug in this study is a common anti-inflammatory used in the treatment of asthma and chronic obstructive pulmonary disease [1].

BDP is slightly water soluble. So the low solubility of BDP in water limits the rate of drug absorption, which resulted in an increase in the dose of drug usage. In this direction, Understanding the solubility of a solute in a supercritical fluid, which must be experimentally determined or theoretically modeled, the first and most important step in the development of supercritical fluid technology.

Experimental solubility of BDP in supercritical CO_2 were correlated with Peng-Robinson equation of state (PR-EOS) along with the van der Waals mixing rule and three semi-empirical density based models (Chrastil, Mendez-Santiago and Teja (M-T) and kumar and Johnston (K-J) models). The results show that the three density based models and PR-EOS accuracy correlate the experimental solubility of BDP in supercritical CO_2 with maximum average absolute relative deviation (AARD %) of 11.42%. The obtained AARD values were 7.5%, 7.2%, 7% and 11.42% for the Chrastil, the M-T, and the K-J models and PR-EOS, respectively. So, among the four models, the K-J model gave the best result for correlation of the experimental data.

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Corrosion control of mild steel using Hydrogel on mild steel in in normal hydrochloric acid medium

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Abstract:

The use of corrosion inhibitor is one of the most effective measures for protecting metal surfaces against corrosion in acid environments. The corrosion of iron and mild steel (MS) is a fundamental academic and industrial concern that has received a considerable amount of attention. To control corrosion, organic inhibitors are used to protect the metal from corrosion by forming a barrier film on the metal surface[1]. Organic compounds containingelectronegative functional groups and p-electrons in conjugated double or triple bonds generally exhibit good inhibitive properties by supplying electrons via p orbitals. Specific interaction between functional groups and the metal surface and heteroatoms like N, O, and S play an important role in inhibition due to the free electron pairs which they possess[2]. When both of these features combine, increased inhibition can be observed The aim of this work is to investigate the inhibiting influence of hydrogel on mild steel corrosion in 1.0M hydrochloric acid solution. This has been studied by potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) techniques. Tests were conducted in 1.0M HCl solution with different concentrations of inhibitor. Electrochemicalexperiments were carried out using an Autolab (PGSTAT 12). A three-electrodearrangementwas used for electrochemical studies.Workingelectrodewas preparedfrom a mild steel sheet, mounted in polyester so that the area exposed to corrosivesolution was $1 cm^2$. A saturated calomel electrode (SCE) and graphite rodwere used as the reference and counter electrodes, respectively. Potentiodynamic polarization curves were obtained with a scan rate of $0.5\ mV\ s^{-1}$ in the potential range from−150 to +150 mV relative to the corrosion potential. Corrosion current density valueswere obtained by Tafel extrapolation method.The immersion time before eachmeasurementwas 60 min to access an equilibriumpotential. The impedance measurements were carried out at the open circuit potential (E_{ocp}) and FRA software. The alternating current frequency range was extended from 100 kHz to 0.1 Hz with a signal amplitude perturbation of 5 mV .The corrosion parameters as well as inhibition efficiencies were obtained for different concentrations of inhibitor.The inhibition efficiencies showed that the hydrogel acts as a good corrosion inhibitor for mild steel in hydrochloric acid.The inhibition efficiency slightly decreased in the temperaturerange 30–60 °C. Hydrogel is a mixed safe corrosion inhibitor for carbon steel in medium acid solutions that acts both over the cathodic process of hydrogen evolution and the anodic process of metal dissolution. Thermodynamic adsorption and activation parameters were discussed. Adsorption of the synthesized inhibitor was found to follow the Langmuir adsorption isotherm.

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Coupling of resorcinol-formaldehyde xerogel based µ-SPE and HS-SDME for determination of BTEX in aqueous sample

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Recently, a new form of carbon adsorbent, carbon aerogel (CA) has appeared [1]. CAs are novel mesoporous carbon materials with many interesting properties, such as low mass densities, continuous porosities, high surface areas, great mesopore volume and high electrical conductivity [2]. Generally, CAs result from the reaction of resorcinol (R) with formaldehyde (F) in slightly basic condition [3]. In this study, a micro solid phase extraction (µ-SPE) method in combination with headspace single drop microextraction (HS-SDME) along with gas chromatography- mass spectrometry detection (GC–MS) was developed for the analysis of BTEX in aqueous samples. These two different extraction methods have been coupled to boost the advantage of each. A new RF- xerogel was synthesized based on sol-gel technology and employed as a sorbent for µ-SPE. Influential parameters such as sample flow rate, extraction volume, extraction time, extraction temperature and drop hanging time were optimized. Under the optimized conditions, a detection limit of 0.03-0.1 µg L^{-1} was obtained. The linear range was obtained in the range of 0.1 to 100 µg L^{-1} with correlation coefficient of more than 0.99. The RSD% value at three concentration levels was lower than 8%. The enrichment factor (EF) of the coupled system was found to be higher than HS-SDME. The optimized method was applied to the determination of BTEX in Zayenderood and paddy water samples while accuracy and matrix effect were also studied.

Keywords: Micro solid phase extraction, Headspace single drop microextraction, Sol-gel technology, Resorcinol-Formaldehyde xerogel, BTEX, Gas chromatography-mass spectrometry.

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Crude assay by infrared spectrometry: analytical chemistry for geochemical economics

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Considering the important role of energy in the life stream of the society, crude oil could be mainly assumed as a valuable natural resource which is primarily consumed for production of fuels, such as diesel, gasoline, kerosene, jet fuel, etc. [1].Quality monitoring and characteristics control of crude oil is of high importance for different reasons. In order to evaluate the potential applicability of a crude oil resource, and also to explore its economic justification level, one needs to characterize the extracted samples, comparing a multivariate model of exploring cost, quality, geographic location and application.One of the important tasks in petroleum industry is to distinguish the characteristics of crude oils, based on their geological origin. It is well-known that the chemical characteristics of a crude oil are dependent on its origin [2]. Thus it is of high importance for the petroleum industry to use a method by which the origin of new sample could be predicted or verified in a fast way without performing several tests.Crude oil samples from different Iranian oilreservoirs in both, raw and combination forms have been characterized by attenuated total reflectance mid infrared spectroscopy. Obtained spectra were classified by chemometric techniques to propose a method for geological based classification of the crude oil samples. Row mean centering and principal component analysis (PCA) supported - leverage value based outlier detection were used as preprocessing strategies. PCA, cluster analysis and soft independent modeling of class analogy (SIMCA) were utilized to classify the spectra. Obtained results confirmed that SIMCA is a robust chemometric technique for origin classification of crude oil samples based on their IR spectra, while the mixture samples were also classified satisfactory in some cases. Root mean square error, method precision and regression coefficient for the prediction of origin of an independent validation set of samples were 1.41%, 96.7% and 0.957 respectively.

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Degradation of Picric Acid in Water Using ZnO Nanoparticles

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The presence of ordnance compounds in environments has been identified and has generated questions on the potential of nitramines and nitroaromatic compounds to cause adverse effects to life. Conventional treatment techniques to remove organic matters from water samples including coagulation, flocculation, precipitation, oxidation, adsorption, and reverse osmosis have been reported [1,2]. However, it is very difficult both to obtain satisfactory treatment efficiency and to reduce the cost associated with infrastructure construction, energy consumption and frequent use of additional chemicals [3]. Recently, the use of semiconductor nanoparticles for the degradation of contaminants has been attended with great interest. The primary waste products from explosives production and handling were TNT and RDX with smaller scale production of other nitro-organic compounds like picric acid (2,4,6-trinitrophenol).

Picric acid is frequently found in forensic laboratories for use in the Christmas Tree stain and for urine detection [2,3]. In this present study, we report the synthesis of semiconductor ZnO nanostructure using a chemical precipitation method. The morphology and structure of prepared nanoparticles were characterized by SEM and XRD analysis. The optical properties were investigated by UV–vis technique. The absorption spectra exhibit a maximum pick at 384 nm .The activity of the prepared ZnO nanoparticles has been investigated for the degradation of Picric acid in aqueous solutions. The effects of some experimental parameters such as the amount of ZnO nanoparticles, pH, ionic strength, time and temperature on the degradation efficiency of Picric acid were studied. The obtained results showed that up to 60% of Picric acid was decomposed under optimum conditions. The proposed method is simple, rapid and cost effective. This approach was successfully used for the treatment of different water samples.

Keywords: Picric acid, Water, ZnO nanoparticles, Degradation

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Dependence of the Energy Conversion Efficiency of Dye-sensitized solar cell (DSSC) to the Thickness of TiO_2 Nanoparticulate Electrode

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Abstract

Dye-sensitized solar cell (DSSC) consists of a TiO_2 nanostructured film of the photo electrode, dye molecules on the surface of the TiO_2 film, an electrolyte layer and a counter electrode [1,2]. Among these, the nanoporous TiO_2 film plays an important role as the photo electrode in DSC because it adsorbs a large number of the dye molecules which provide electrons. Therefore, the conditions of the TiO_2 film affect the cell performance such as current density (J_{SC}), open circuit voltage (V_{OC}) and fill factor (FF) [3]. The thickness of TiO_2 film is one of these conditions. Its variation influences on the internal impedances of DSC related to the electron transport. As a result, it was observed that the variation of the TiO_2 thickness has an effect on the electron transport in the DSSC. The best cell showed an outstanding current density of 10mA with TiO_2 film thickness of 12.0 µm. The further correlation between the TiO_2 films thickness and photoelectron chemical properties of DSSCs fabricated from TiO_2 was probed.

Keywords: Nanoporous; TiO_2; DSSC; Film Thickness

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Derivatization and Determination of phenol in groundwater by Gas Chromatography- Mass Spectrometry

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Gas chromatography (GC) is a popular technique for the analysis of phenol compounds. However, because of their high polarity, these analytes tend to give broad, tailed peaks, and these effects increase as the chromatographic column ages. To avoid these drawback, several derivatization reactions have been proposed to transform phenols in less polar compounds, with better chromatographic characteristics. Phenol acetylation with acetic anhydride in presence of carbonate or hydrogencarbonate, is one of the most common derivatization procedures. Reaction can be performed in aqueous samples in a few minutes with high efficiency. Phenyl acetate was extracted by several methods [1-3].

In this research, we compared solid-phase extraction (SPE) with conventional reversed phase adsorbent such as C_{18} and liquid-liquid extraction (LLE) with n-Hexan. According to results, extraction efficiency in SPE procedure is lower than LLE and time consuming. The extracted samples was analyzed by gas chromatography with mass spectrometry detection (GC–MS). Experiments, were performed to check the effect of sample concentration and determination of efficiency of recovery, Ultrapure water was spiked by three concentration levels of phenol. The mean recovery and relative standard deviation is 85 ± 7.8%. Calibration curve was linear in the concentration range of 10 ng mL^{-1} to 2 µg mL^{-1} in the original solution and correlation coefficient is 0.9998. The developed method has been used to monitor phenol contamination in river water samples collected from Zayanderoud River in Isfahan province, Iran.

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Desalination of brackish water using carbon nanotubes, with nano technology new Methods

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In this research an intensive aspects, using the carbon nanotubes and CNTs applications in desalination of seawaters and chemical agents were investigated for potentially simplification in reverse flow transmission of water and elimination of salts [1]. Single wall carbon nanotubes (SWCNTs) were functionalized by the hummer's method to attach the carboxylic groups. otherwise the processing parameters on sodium and potassium elimination efficiency of seawater, surface morphology Also, embed-nanotubes. of disposed factor absorbance an as were reported of the functionalized SWCNTs were seen by scanning electron microscopy (SEM), and X-ray diffraction system was used to characterization of the crystallite structures [2,3]. Functionalized SWCNTs were characterized by FT-IR. The initial concentration of mentioned agents before desalination, were indicated 25000 ppm and 365 ppm, by flame emission detector (photometer films), respectively. In this project three parameters as solution pH, absorber concentration and temperature were examined to achieve the optimized values at batch system. Decreasing of sodium and potassium at 6, 7 and 8 pH values, then at flow rates of 10, 20, 40, 60, 80, 100 and $120\frac{\mathrm{ml}}{\mathrm{min}}$, were examined by using vacuum pump set and SPE cartridge, consequently the concentration decreasing demonstrated with flame emission set. Results show that, the high elimination (about 90%) of the sodium was happened at the neutral pH and at the 80ml/min. flow rate, and 75˚C. In the other hand, amount of the potassium was decreased about 40% at the neutral pH and 100ml/min. and at the room temperature.

Key words: desalination, functionalized, carbon nanotubes.

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Design and construction of a label-free nanobiosensor for rapid detection of prolactin

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Abstaract:

Due to improving the sensitivity and performance of nanobiosensors by nanoparticles, the nanotechnology have been found enormous applications in this field of bio-analytical chemistry. Low toxicity of gold nanoparticles cause that these particles have been used more and more in biological systems[1]. Prolactin is a hormone produced by the pituitary gland in the brain and plays an important role in the reproductive health of both women and men. however its main role, is to stimulate the production of milk in women after childbirth, High level of prolactin (hyper-prolactinemia), can cause liver disease, kidney disease, and hypothyroidism. Localized surface plasmon resonance (LSPR) of gold nanoparticles (GNPs) can be used as the basis for detection of hormones[2]. In this work a procedure for synthesis of colloidal solution of naked Au nanoparticles have been described. The GNPs was about 20 nm in size[3]. The GNPs were modified by antiporolactin antibodies. The shift in localized surface plasmon resonance of bioconjucated GNPS in the presence of prolactin can be used as selective and label-free biosensor.

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Design and construction of a novel optical sensor for determination of trace amounts of Lanthanum ion

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Abstract

In this work, for the first time, a highly selective and sensitive Lanthanum (La) optical sensor [1] based on immobilization of N-(2-aminoethyl)- salicylaldimine (AESI) on a triacetyl cellulose membrane was reported [2]. This optode exhibits a linear range of 30 to 150 ppb of the La (III) ion concentration with a detection limit of 14.38 ppb. Response time of the newly designed optode was with in 30-60 s, depending on the La(III) ion concentration. Response of the sensor is independent of the pH of the solution in the range of 3.0–5. The proposed optode shows low detection limit, fast response time, and most significantly, very good selectivity with respect to a number of transition metals ions (Cu^{2+}, Ce^{3+}, Cd^{2+}, Cr^{3+}, Zn^{2+}, Hg^{2+}, and Fe^{2+} ions) [3]. The sensor was successfully regenerated with a thiourea solution and its response was reversible and reproducible (R.S.D. less than 1.48%). This optode was applied to the determination of La(III) in spiked sample.

Keywords: N-(2-aminoethyl)- salicylaldimine, Lanthanum, Optical Sensor, Triacetylcellulose

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Design and Fabrication of Magnetic Nano-particles for Drug Delivery

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Abstract

Magnetic nanoparticles (MNPs) are a major class of nanoscale materials with the potential to revolutionize current clinical diagnostic and therapeutic techniques. Due to their unique physical properties and ability to function at the cellular and molecular level of biological interactions, MNPs are being actively as carriers for targeted drug delivery [1,2].Nanocarriers are generally less than 100 nm in size and have theability to transport and release drugs to a target site with high efficiency and limited side effects[3,4]. Application of magnetic nano-particles depends largely on the preparation processes to select optimal conditions and type of modification. In this study iron oxide nano-particles (Fe_3O_4) were prepared by the co-precipitation method and then modified with 3-mercaptopropyltri methoxysilane in dioxane. Then functionalized sorbent was provided by coupling of brilliant crystal blue. The obtained nano-adsorbent was characterized by Fourier transform infra-red spectroscopy, scanning electron microscopy and X-ray Diffraction. Then the effect of various parameters such as pH, and contact time were studied. Also, the adsorption isotherms kinetics and capacity at optimum conditions were investigated. The nano-adsorbent was used to measure for drug release in human biologic fluid. The concentration of drug was measured by conductometry technique.

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Design and fabrication of modified electrochemiluminescence sensor based on CeO_2 nanoparticles for detection of tramadol

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Abstract:

Tramadol are generally used as haemostatic, analgesic and local anesthetic in surgery[1]. A novel method for rapid, inexpensive, sensitive and selective determination of tramadol was proposed by electrochemiluminescence (ECL)[2]. An electrochemiluminescence sensor was fabricated by employing ruthenium complex $[Ru(bpy)_3]^{2+}$ and nafion polymer and paraffin as binding reagent for the determination of tramadol. The ECL sensor was prepared by thoroughly mixing ruthenium complex as luminophor with nafion polymer, graphite powder and CeO_2 nanoparticle which exhibit increased chemical activity due to their large surface to volume ratios, which shows good electrochemical and ECL behaviors. The modified electrodes showed an electrocatalytic response to the oxidation of tramadol, producing a sensitive ECL signal. ECL intensity of the fabricated sensor was greatly enhanced in the presence of tramadol and a sensitive ECL method was proposed for the determination of tramadol based on the enhancement ECL. The ECL intensity was linear with the concentration of tramadol in the range from 2.0×10^{-9} mol/L to 2.0×10^{-7} mol/L and the detection limit was 2×10^{-11} mol/L. The ECL sensor showed good selectivity and long-term stability. Its surface could be renewed quickly and reproducibly by a simple polish step[3]. tramadol was valuable in clinical and biochemical laboratories for monitoring these drugs for various purposes.

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3. Yafeng Zhuang and Jintao Zhang. Luminescence.25(2010), 4, 343-8,

Design and fabrication of modified electrochemiluminescence sensor based on CeO_2 nanoparticles for detection of tramadol

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Abstract:

Tramadol are generally used as haemostatic, analgesic and local anesthetic in surgery[1]. A novel method for rapid, inexpensive, sensitive and selective determination of tramadol was proposed by electrochemiluminescence (ECL)[2]. An electrochemiluminescence sensor was fabricated by employing ruthenium complex $[Ru(bpy)_3]^{2+}$ and nafion polymer and paraffin as binding reagent for the determination of tramadol. The ECL sensor was prepared by thoroughly mixing ruthenium complex as luminophor with nafion polymer, graphite powder and CeO_2 nanoparticle which exhibit increased chemical activity due to their large surface to volume ratios, which shows good electrochemical and ECL behaviors. The modified electrodes showed an electrocatalytic response to the oxidation of tramadol, producing a sensitive ECL signal. ECL intensity of the fabricated sensor was greatly enhanced in the presence of tramadol and a sensitive ECL method was proposed for the determination of tramadol based on the enhancement ECL. The ECL intensity was linear with the concentration of tramadol in the range from 2.0×10^{-9} mol/L to 2.0×10^{-7} mol/L and the detection limit was 2×10^{-11} mol/L. The ECL sensor showed good selectivity and long-term stability. Its surface could be renewed quickly and reproducibly by a simple polish step[3]. tramadol was valuable in clinical and biochemical laboratories for monitoring these drugs for various purposes.

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Design and preparation of new nanocomposite-solid phase microextraction for extraction of chlorophenols derivatives coupled with high performance liquid chromatography

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Almost all commercially available SPME fibers are based on fused-silica fiber. Adsorptions of compounds depend on the agent groups on the stationary phase and surface area. High surface area increases the sensitivity of analysis and decreases the limit of detection. In this way, metal oxides such as Al_2O_3, ZnO, ZrO_2, and nanostructured PbO_2 prepared by electro-oxidizing or electrodeposition techniques have been used as SPME coatings. Alumina and titania based hydroxyl-terminated silicon oil sol-gel coating have been used for SPME of polar compounds. Metal oxide-based SPME coating made of a mixture of AL_2O_3 on a polyvinylchloride matrix dispersed in tetrahydrofurane and Nb_2O_5 coating using a metallo-organic decomposition technique have also been successfully used [1]. Recently, SPME fiber fabricated with nanostrucured titania covered on the titanium wire at low temperature [2]. By developing 1-D nano structures such as nano-rods and nano-plates of metal oxides, a higher surface area can be achieved. The results of the innovative study on a new stationary phase with high efficiency based on nano composite of ZnO-CuO nano plates coating on fused silica are reported in this paper. These composite of nanoplates with a thickness in the range of 30–60 nm and the length of about 100 nm, have been grown on fused silica fibers using a hydrothermal process. The extraction properties of the fiber were investigated using direct solid-phase microextraction mode coupled with high performance liquid chromatography for four chlorophenols derivatives. Determination of chlorophenols derivatives were performed in the water. The calibration curves were linear at the µg l−1 range with low detection limits for mentioned compounds. The RSD for Single fiber and fiber-to-fiber were less than 7.57 and 15.67%, respectively. Promising recoveries (88–97.6%) were obtained in biological sample analysis by applying the proposed technique

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Design of a New Flame-Containing Molecular Emission Cavity for determination of phosphor in Wastewater: Catalytic Behavior of Hydrogen Radical

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Abstract

Determination of inorganic phosphor is of very high importance in environmental and health care applications. Hence knowledge about suitable analytical techniques for phosphor sensing purposes becomes essential. In this study, a new flame-containing molecular emission cavity analysis (MECA) system [1, 2] was designed for determination phosphor in wastewater based on the catalytic properties of hydrogen radicals as shown in Fig. 1. For phosphor determination, parameters having important effect on the excited phosphor emission are: type and amount of acid at which phosphorous sample in introduced, pH and temperature of the solution, effect of sonication on sample introduction, and finally flow rates of H_2 and air. For this purpose simplex and one at a time methods were selected for optimization of the effective factors. According to the results, partially the same correlation was evaluated during optimization of the parameter with simplex and one at a time methods. For instance, luminescence emission of excited phosphor was detected at different pH values via testing different acid solutions such as HCl, $HClO_4$, CH_3COOH, H_3BO_3, etc. ranging between 2-12. The results showed maximum sensitivity at 2.58 pH for $HClO_4$ solution. According to the results, linear dynamic range was between 1.0-20.0 parts per millions (ppm), with detection limit lower than 100 parts per billion (ppb) levels. Except ~500 folds excess of anions such as SO_3^{2-} and NO_3^-, no significant interferences were detected during phosphor analyses. Finally the reliability of the proposed method was evaluated via analyses of phosphor in wastewater samples.

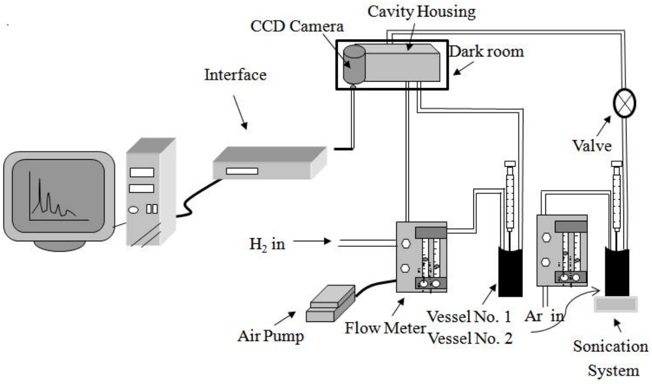

Fig. 1. Schematic of designed cavity system.

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Design of a novel optical sensor for determination of trace amounts of copper by spectrophotometry UV/VIS in the real samples

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Copper is one of the heavy metals which is an essential element not only for life in mammals but also for plants, and plays an important role in carbohydrate and lipid metabolism. Increases in copper concentration in waters and plants have resulted from industrial and domestic waste discharge, refineries, disposal of mining washing, and the use of copper as a base compound for antifouling paints. Therefore, the trace copper content in water and food must be controlled on a daily basis [1]. In recent years, there has been an increasing interest in the development of optical chemical sensors for heavy metal ions. In this context, a sensor is described as a device which is capable of responding to the presence of a heavy metal ion in a reversible and continuous manner [2,3]. An optical sensor was established to determine of copper (II) based on the immobilization of 3-(2-methyl-2,3-dihydro-1,3benzothiezol-2-yl)-2H-chromen-2-one on a triacetylcellulose membrane. Copper (II) ions react with the immobilized ligand and cause an increase in the absorbance of the membrane at 349 nm. This sensing phase had a dynamic linear range of 10^{-6} – 10^{-4} mol/l of copper ions with a limit of detection of 10^{-6} mol/L. The response time of the optode was within 3 min depending on the concentration of Cu^{+2} ions. Response of the sensor is independent of solution pH in the range of 2.0–9.0. The selectivity of the optode to Cu (II) ion in the presence of other metal ions including Pb (II), Mg (II), Ca (II), Ba (II), Zn (II), Ni (II), Co (III), Mn (II), Cd (II), Bi (III), Pd (II) and Al (III) is high. The sensor was successfully regenerated with ethylenediamine solutions and its response was reversible and reproducible (R.S.D. less than 1.4%). The proposed optode was applied successfully for the determination of copper (II) in various samples.

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Design Optical Chemical Sensor by Immobilization of 4-(2-Pyridylazo)resorcinol on a Triacetycellulose Membrane for Determination Cobalt(II) in Various Samples

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Cobalt is one of the most important metal ions, which appears in many real and synthetic samples. Cobalt is an essential element in trace amounts for human and animal, for an example, it is part of vitamin B12 and plays a key role in the body's synthesis of this essential vitamin. There are several techniques for the determination of cobalt such as flame and graphite furnace atomic absorption spectroscopy (AAS), molecular absorption and fluorescence spectroscopy, voltammetry, energy dispersive X-ray fluorescence spectroscopy (EDXRF) and reversed-phase capillary high performance liquid chromatography (RP-Capillary-HPLC).These methods require expensive equipments, skilled operator and sample pretreatment. Therefore, alternative methods such as optical chemical sensors which are simple to use and require less expensive instrument have been developed [1]. A chemical sensor can be defined as a device which responds to a particular analyte in a selective way through a chemical reaction. Chemical sensors contain two basic functional units which are a receptor part and a transducer part.(Figure 1). The optical propertie of the sensor are changed when the receptor part detects an analyte. A spectroscopic measurement is usually associated with a chemical reaction and conventional spectrometric method including absorption, fluorescence, and reflectancespectroscopy can be applied.

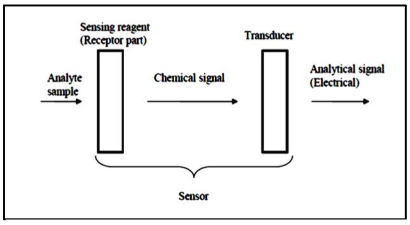
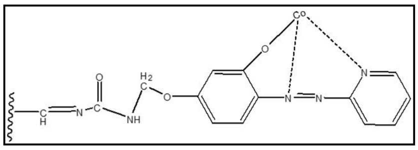


Figure 1. A simplified diagram of the components of a chemical sensor.



Scheme 1. Possible schemes of reaction between PAR with activated

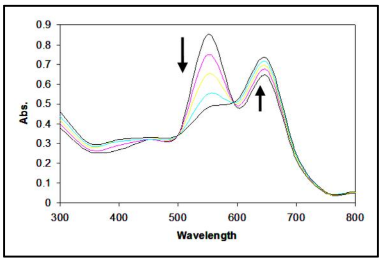


Figure 2. Absorption spectra of optode film in the presence of various concentration of Co (II)

Membrane based optical chemical sensor (optode) for Co (II) was developed by the immobilization of a dye 4-(2-Pyridylazo)resorcinol (PAR) on the triacetylcellulose [2]. Co (II) ions react with the immobilized PAR and cause a decrease in the absorbance of the membrane at 555 nm. The response time of the optode was 11–16 min depending on the concentration of Co (II) ions. This sensing phase had a dynamic linear range of 0.5–5.5 µg ml^{-1} Co (II) ions with a limit of detection of 0.15 µg ml^{-1}. The selectivity of the optode to Co (II) ions at pH 5.0 was good. The coefficients of variation (CV) of the sensor response for 2 µg mL^{-1} Co (II) was، 2.8% and the CV between seven membranes was 3.2%. The sensor could readily be regenerated with a hydrochloric acid solution (0.01 M) [1]in a reversible manner and its response was reproducible (RSD = 3.5%). The method was applied to the determination of Co (II) content of a variety of samples which gave satisfactory results.

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Designing ion sensitive field effect transistor (ISFET) pH sensor using a commercial field effect transistor

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In this study, for the first time, we introduce a simple and low cost pH sensor based on a commercial field effect transistor (FET). The transistor was mechanically treated, isolated and used as an ion sensitive field effect transistor (ISFET) [1] for pH detection, after electrodeposition of the sensing membrane on its surface. Polypyrrole, as a suitable sensing membrane, was electrodeposited on the of transistor from a solution containing pyrrole monomer, hydroquinone monosulfonate [2] as a proper ligand and sodium salicylate for avoiding the substrate oxidation, under a two-step deposition conditions. The prepared sensor showed a near-Nernstian response of 52.3 mV pH^{-1} over a linear pH range of 2.75-12.20, an ultra low hysteresis of 0.56 mV, a very low drift of 0.14 mV h^{-1} and a low response time of less than 8 s.

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Detection limit enhancement of antiarrhythic drugs in capillary electrophoresis with dispersive liquid–liquid microextraction and on-line sample stacking

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Abstract

In clinical studies, there is an increasing demand for an efficient analytical method that permits simultaneous separation and quantification of drugs in a complex matrix with less laboratory work [1]. Assays need to be free of interferences, not only from endogenous substances and other concomitantly administered drugs, but also must distinguish between parent drug and its metabolites [2]. Antiarrhythmics are used to treat disorders of the heart's rhythm. Most of the currently available antiarrhythmics have a narrow therapeutic index, necessitating careful dose adjustement [3]. In this study, a new method was described for the enrichment of antiarrhythmic drugs (propranolol, metoprolol, diltiazem, and verapamil) in plasma samples via a dispersive liquid–liquid microextraction (DLLME) combined with on-column stacking in capillary electrophoresis. Two steps were employed for biological samples clean-up and sensitivity enhancement in capillary electrophoresis. The factors affecting the on-line sample stacking and microextraction procedure were optimized. This assay enhanced sensitivity157-314fold for the studied drugs. The baseline separation was achieved within 18 min. During method validation, the calibration curves were linear over a range of 20−800 ng/mL ($r^2 \geq 0.997$). The relative standard deviations (RSDs) and relative errors (REs) of intra- and inter-day assays were below 20%, which showed good precision and accuracy. Their detection limits ranged between 2.5 to 4.7 ng/mL (S/N = 3). The validated method is successfully applied to determine propranolol, metoprolol, diltiazem, and verapamil in human plasma samples obtained from the patients whose received these drugs.

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Detection of a DNA sequence related to Hepatitis B virus with a sensitive electrochemical DNA biosensor constructed with functionalizing of magnetite nanoparticles by 4-nitrobenzenediazonium

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Abstract

In recent years, the interest for DNA-based diagnostic tests has been growing. The development of systems allowing DNA detec- tion is motivated by applications in many fields: DNA diagnostics, gene analysis, fast detection of biological warfare agents, and forensic applications. Detection of genetic mutations at the molecular level opens up the possibility of performing reliable diagnostics even before any symptom of a disease appears. Optical [1], electrochemical [2], and micro-gravimetrical [3] DNA transductions have been widely studied. An in situ method for direct attachment of 4-nitrobenzenediazonium onto the surface of magnetite nanoparticles to develop an efficient guide for construction of DNA electrochemical biosensor was proposed. For functionalization of magnetic nanoparticles, firstly azo salt of nitroaniline (4-nitrobenzenediazonium) was prepared and was reacted directly with magnetite nanoparticles in basic solution followed by electrochemical reduction of the terminal Nitro group to amine. This NH_2 group can interact with an activated phosphate group of non-labeled ss-DNA related to Hepatitis B virus. Activated non-labeled ss-DNA was prepared using *N*-(3 dimethylaminopropyl)-*N*-ethyl-carbodiimide hydrochloride (EDC) and *N*-hydroxy-succinimide (NHS). The DNA hybridization events were monitored by differential pulse voltammetry (DPV) measurement of the intercalated Methylene Blue (MB) as a well-known electrochemical indicator. Under optimal conditions, the reduction peak currents were linearly decreased with increasing the concentration of HBV target DNA from 7.8 (±0.2) × 10^{-13} mol L^{-1} to 8.8 (±0.3) × 10^{-9} mol L^{-1} with a detection limit of 3.3 (±0.2) × 10^{-13} mol L^{-1}. This DNA biosensor exhibited excellent selectivity, reproducibility, and stability in the HBV DNA hybridization assay.

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Detection of amino acid phenylketonuria disorder by monolithic silica C_{18} column-HPLC analysis using photo diode array without and with derivatization by orthophthaldehyde

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Monolithic silica is one of the interested monolithic materials due to macropores and mesopores with average diameter of 2 µm and 18 nm in its skeleton, respectively. Using of these materials as a high performance liquid chromatography (HPLC) column has been started since 1996 [1]. Monolithic silica C_{18} compared to particle-packed silica C_{18} columns have a low flow resistance, which leads to a low column backpressure and fast analysis and also a sufficient surface area for an efficient separation [2].

Amino acids are the primary components of proteins, and they are involved in several cellular metabolic pathways. The inborn errors of amino acid metabolism are a group of genetic disorders in which an enzyme deficiency results in the accumulation of amino acids. Phenylketonuria (PKU), as a metabolic disorder that resulted in mental retardation, can be detected by simultaneously determination of free levels of phenylalanine (Phe) and tyrosine (Tyr) in physiological samples [3].

In this work, a fast HPLC method based on photo diode array (PDA) detection with monolithic silica C_{18} column (Chromolith RP 18e 100 × 4.6 mm I.D.) has been used for separation and quantization of Phe and Tyr with two method: (a) without derivatization and following condition: the mobile phase containing 5% v/v acetonitrile in water, flow rate of 1.5 mL min^{-1}, column temperature of 30°C and wavelength detection of 210 nm, (b) after derivatization by orthophthaldialdehyde (OPA) in the presence of 2-mercaptoethanol (2-MCE) and following condition: the mobile phase was mixture of acetate buffer (pH = 6) and methanol (50:50 v/v), column temperature, flow rate and wavelength detection were 30°C, 1 mL min^{-1} and 330 nm, respectively.

Finally, rapid separation of Phe ($t_{R(a)}$ = 1.56 min, $t_{R(b)}$ = 5.68 min) and Tyr ($t_{R(a)}$ = 1.2 min, $t_{R(b)}$ = 2.25 min) has been achieved for two mentioned methods. Based on the obtained results, method (a) provides a rapid and an inexpensive analysis compared to method (b) and other methods of HPLC with pre- or post-column derivatization. Eventually it is suitable for diagnosing of PKU in newborns children.

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Detection of artificial yellow colorant Tartrazine in saffron using magnetic carbon nanotubes and UV-Vis spectroscopy

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Abstract

A sensitive, accurate and economic method for the determination of coloring adulteration of tartrazine (Yellow 5) in saffron have been developed and validated. Tartrazine is a synthetic lemon yellow azo dye primarily used as a food coloring. It can cause allergic and intolerance reactions in people who are asthmatics or have an existing allergy to aspirin. The FDA requires that the presence of tartrazine be declared on food and drug product [1, 2]. Because of its high price, saffron is often the subject of adulteration. Thus determination of the coloring adulteration such as tartrazine is crucial. This research was developed a simple method using magnetic carbon nanotubes as the sorbent for pre-concentration and extraction of tartrazine in saffron. Carbon nanotubes (CNTs) decorated with magnetite nanoparticles on their external surface has been fabricated. The Fe_3O_4–CNT composites display good ferromagnetic property at room temperature. Some important parameters that could influence extraction efficiency were optimized such as the extraction mode, sample volume:15ml, amounts of adsorbent: 0.03g, pH of sample solution: 9, extraction time: 20 minute, amount of salting: 0.005g, rate of stirring: 600rpm, desorption solvent: 3.0 mL methanol-NH3(95:5 v/v) and desorption time: 15 minute. The proposed method gave good validation results and compared with those obtained by reported methods. The proposed method has been applied for determination of trace amounts of tartrazine in the saffron samples available in the market and satisfactory results were obtained.

Keywords: Saffron, Tartrazine, UV-Vis sprctroscopy, Carbon nanotube, Nanomagnetite.

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Detection of Single Nucleotide Polymorphism using Graphene Oxide-based Nanoporous Gold Electrode Platform

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Abstract

In particular, methods for the rapid identification of base mutations or single nucleotide polymorphisms (SNPs) would prove useful for the diagnosis of many genetic diseases and in clinical, forensic and pharmaceutical application [1]. While different methods for detection of SNPs, generally, have been developed, but it is still a major challenge to develop new technologies with improved simplicity, selectivity, and sensitivity of DNA hybridization detection. These technologies should not require complicated fabrication, instrumentation, and additional reagents. Graphene oxide (GO), an insulating and graphene (GN) because of its excellent electrical, mechanical, and chemical performances, has been used in various electrochemical applications [2]. To achieve sensitivity and selectivity of the SNPs, a highly sensitive transducer of the signal with high surface area is required.

In this study, a new label free electrochemical biosensing platform using nanoporous gold electrode (NPGE) [3] by taking advantage of soluble graphene oxide (GO) as an indicator and its unique GO/ssDNA interaction for the rapid and sensitive detection of SNP was reported. Because of less effective hybridization of mutant targets in comparison with complementary targets, further GO could adsorb in the presence of SNPs via $\pi-\pi$ interaction and resulted in a large increase of the charge transfer resistance (R_{ct}) of the electrode. The protocol provides a great potential with high sensing, cost-effective, low-cost and fast for discrimination of different SNPs. Furthermore, The DNA biosensor exhibited a high sensitive response to detection of thermodynamically stable SNP (G-T mismatches) in the range of 20-1600 pM with the detection down to 20 pM.

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Determination and modeling the solubility of pioglitazone in aqueous binary mixtures of PEGs 200, 400 and 600 at 298.2 K, 303.2 K and 308.2 K

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About 40% of invented pharmaceutical candidates have poor solubility in water. Therefore, in order to expand the usage of such compounds for various applications it is necessary to establish a technique for increasing their solubility and bio-distributions [1]. The cosolvency method has been used in this research [2].

The binary solvent mixtures were prepared by weighing the solvents. Then the excess amounts of pioglitazone were added in the prepared solvents at 25 °C, 30 °C and 35 °C. They were stored in a shaker, placed for a period of 72 hours in an incubator equipped with a temperature controlling system maintained constant within ± 0.2 °C. The saturated solutions of the drug were centrifuged at 13000 rpm for 15 min, and subsequently diluted with methanol. The solutions were then assayed at 269 nm using a UV-Vis spectrophotometer.

Three cosolvency models were used in the modeling part. The log-linear model of Yalkowsky, Jouyban−Acree model and the combined version of the Jouyban-Acree model and the van't Hoff approach were used to fit solubility data of pioglitazone in the binary solvents, and the overall mean relative deviations (OMRDs) were 9.6 %, 2.5 % and 13.8 % respectively. The density of the solute-free solvent mixtures is measured and employed to calculate the constants of the Jouyban–Acree model and then the density of the saturated solutions and the predicting OMRD was 1.8 %. All three models fit well to the experimental solubilities at all solvents fraction ranges and this has been supported by the small MRDs.

Keywords: Solubility; Pioglitazone; Modeling; Polyethylene Glycols

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Determination and modeling the solubility of pioglitazone in aqueous binary mixtures of PEGs 200, 400 and 600 at 298.2 K, 303.2 K and 308.2 K

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About 40% of invented pharmaceutical candidates have poor solubility in water. Therefore, in order to expand the usage of such compounds for various applications it is necessary to establish a technique for increasing their solubility and bio-distributions [1]. The cosolvency method has been used in this research [2].

The binary solvent mixtures were prepared by weighing the solvents. Then the excess amounts of pioglitazone were added in the prepared solvents at 25 °C, 30 °C and 35 °C. They were stored in a shaker, placed for a period of 72 hours in an incubator equipped with a temperature controlling system maintained constant within ± 0.2 °C. The saturated solutions of the drug were centrifuged at 13000 rpm for 15 min, and subsequently diluted with methanol. The solutions were then assayed at 269 nm using a UV-Vis spectrophotometer.

Three cosolvency models were used in the modeling part. The log-linear model of Yalkowsky, Jouyban−Acree model and the combined version of the Jouyban-Acree model and the van't Hoff approach were used to fit solubility data of pioglitazone in the binary solvents, and the overall mean relative deviations (OMRDs) were 9.6 %, 2.5 % and 13.8 % respectively. The density of the solute-free solvent mixtures is measured and employed to calculate the constants of the Jouyban–Acree model and then the density of the saturated solutions and the predicting OMRD was 1.8 %. All three models fit well to the experimental solubilities at all solvents fraction ranges and this has been supported by the small MRDs.

Keywords: Solubility; Pioglitazone; Modeling; Polyethylene Glycols

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Determination and Separation of Sub-Micro-Molar Amounts of Cadmium (Cd^{2+}) by Floatation -Spectrophotometric Using Bromothymol blue and CTAB

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Abstract

In the present study a new technique and simple floatation-spectrophotometric extraction method was developed to recovery of Cadmium from aqueous samples prior to determination by UV spectrophotometric detection.The method is based on the formation of a complex between Cadmiumand Bromothymol blue in basic media(KH_2PO_4+Na_2HPO_4,pH=9). The complex is floated in the interface of n-hexane and aqueous phase. After removing the aqueous phase the floated particles are dissolved in methanol and the absorbance is measured at 555 nm. Different parameters of the floatation-spectrophotometric technique such as reagent concentration, pH, surfactant(CTAB and SDS), standing time and interfering ions were studied and optimized to obtain the best extraction results with the minimum interference from other compounds. Under these conditions, the extraction of the target compound was almost complete (mean values of recovery more than 96.8%) in a short time (6 min). The optimized method demonstrated good linearity ($r > 0.9989$) in the range of 15-350 ng/ml, sensitivity (limit of detection for Cadmium, 1.7 ng/ml), accuracy (0.25–5.35%) and precision (2.86% ,4.32%). The applicability of the proposed method was demonstrated by extraction of Cadmium from different water samples.

Keywords: floatation-spectrophotometric, Cadmium, Bromothymol blue, water samples.

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Determination Kinetic of Formaldehyde in Industrial Wastewater by Nano Photo Catalysis Method

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Abstract

One of the most important of dangerous chemical compound that is found in the wastewater of different chemical factory is formaldehyde. We need the way for more efficient and less costly method of water and wastewater refinement in a way that again is used in nature and industry.
In the recent work, It is tried to decomposed this substance using nano photocatalysis decomposition method with UV. Using TiO_2 as nano photocatalysis, because it has the most efficient photo activity, the highest stability and the lowest cost. [1]
After absorption of solar radiation (UV) by the nanoparticles, electrons with UV energy moving out of their orbits, which result is causing cavities that are capable of very high oxidation. However, electrons have a strong redox properties, after contact with H_2O and O_2 in the air can cause free oxygen and hydroxide radicals . These free radicals have high activity in oxidation and will be able to decompose pollutants and harmful bacteria into harmless substance such as H_2O and O_2.
In this work, the input is contained 35 ppm of formaldehyde and based on this method the output of reaction reach to 1-2 ppm. This reaction based on the research, [2] , in different PH , temperature and time was done. Using the nano particle help to increase the rate of reaction ,the maximum efficiency was between 95 % – 98 %.

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Determination of acetaminophen using a modified carbon ceramic microelectrode by natural nanozeolite

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Among different ion exchangers, nanozeolites have higher ion exchange ability, faster exchange kinetic, higher surface area compared to micron size zeolites. However, in spite of these advantages, the most important challenge with nanozeolites is their separation from the medium [1]. Acetaminophen (AC) is a long-established substance being one of the most extensively employed drugs in the world. It is an antipyretic and analgesic drug commonly used against mild to moderate pain or for reduction of fevers [2]. Lev and co-workers have been reported the preparation of sol-gel derived carbon-ceramic electrodes (CCE) [3]. This new kind of electrode had been largely utilized for the design of electrochemical devices whose surfaces could be renewed by a simple polishing step.

In the present study, a modified carbon ceramic electrode (MCCE) was developed with natural nanozeolite for the electrochmecal determination of AC. Electrochemical behavior of modified electrode was investigated by cyclic voltammetry and chronoamperometry techniques in a phosphate buffer solution (pH 1.0). When compared to CCE, a CCE containing a nozeolite, the proposed MCCE not only shifted the oxidation potential of AC towards a less-positive potential but also enhanced its oxidation peak current. Further, the oxidation of AC was highly stable at the modified paste electrode. The recorded calibration curves were linear up to 1000 μM for AC. The limits of detection were also obtained as 100 n*M*. The present modified paste electrode was also successfully used for the determination of AC in the presence of common interference compounds. The result demonstrated that the proposed modified electrode exhibited enhanced electrocatalytic activity and good stability for the detection of AC. The modified electrode was successfully applied for the electrochemical determination of AC in real samples.

Keywords: Nano zeolite, Carbon ceramic electrode, acetaminophen, Electrochemical determination, Real samples.

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Determination of acidic dissociation constants of caffeic acid by rank annihilation factor analysis(Rafa)

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Annihilation of the contribution of one chemical component from the original data matrix is a general method in rank annihilation factor analysis (RAFA). In this work, a rank annihilation factor analysis (RAFA) method was proposed for determination of the acidity constants of diprotic acids. After recording the electronic absorbance spectra of the acids at different pH, the contributions of both H2A and A2− were annihilated from the absorbance data, which made feasible the determination of two successive acidity constants. The method was validated by analysis of simulated data and its application to the determination of the acidity constants of caffeic acid, as a reference compound. A close agreement was obtained between the resulted values by RAFA and the declared values. Indeed, the method was used for determination of the acidity constants of caffeic acid in binary solvents mixtures of ethanol and water. The acidity constants of organic reagents play a very fundamental role in many analytical procedures such as acid–base titration, solvent extraction and complex formation, and also transport behavior, binding to receptors and mechanism of action of certain pharmaceutical preparation.

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Determination of acidity constants of some dyes by spectrophotometric method using HypSpec Program

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The accurate determination of acidity and stability constants values are fundamental to understanding the behavior of ligands and their interaction with metal ions in aqueous solution. Such an understanding is important in a wide variety of areas, such as metal ions in biology, in biomedical applications, metal ions in the environment, extraction metallurgy, food chemistry and metal ions in many industrial processes [1]. Various methods for the determination of dissociation constants, such as potentiometric, spectrophotometric, conductometric, and spectroscopic methods, have been reported [2].

Spectrophotometric method is applicable to the compounds whose concentrations or extinction coefficients are not determined. It provides more exact dissociation constants than other methods, even for polyprotic compounds with almost equal pK_a, values and compounds with extremely acidic or extremely basic properties. The most widely used programs and algorithms for the determination of acidity constants from spectrophotometric data are SQUAD, SPECFIT, DATAN and KINFIT.

In this investigation, spectrophotometric data was used for the determination of dissociation constants of some dyes including Eosin Y, Fast Green FCF, 4-(2-pyridylazo)resorcinol (PAR) and Amino hydroxy naphthalenesulfinic acid in aqueous solution by means of HypSpec program as a new version of PHab program [3]. Also the distribution diagrams were depicted by Hyss2009. The obtained results show the good agreement with those from literature.

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Determination of acyclovir in human serum and urine by high-performance liquid chromatography using DLLME and study of effective parameters by Box-Behenken method

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A simple, rapid and sensitive method is proposed for determination of trace amounts of acyclovir using dispersive liquid-liquid microextarction (DLLME)[1] and high performance liquid chromatography (HPLC) that was proposed by using $CHCl_3$ and methanol as extraction solvent and disperser solvent, respectively. Dispersive liquid-liquid microextraction (DLLME) is a novel environmentally benign sample-preparation technique, possessing obvious advantages of simple operation with a high enrichment factor, low cost, and low consumption of organic solvent[2]. The effect of parameters in extraction and separation such as volume of extractant, disperser solvent, pH, type of solvent and concentration of salt were optimized with leave-one-out and Box-Benken[3] methods.

Under the optimal condition, the limit of detection of acyclovir was (1.4 ng L-1) with enhancement factors of 318. The analytical curve absorbance was linear over the range of 0.05-7 ppm acyclovir. The proposed method was applied to the determination of acyclovir in biological fluids such as human plasma with satisfactory analytical results.

parameters	Analytical amounts
Linear range after con.	0.05-7mg/l
Line equation after con.	y=180.5x+102.2
Line equation before con.	y=0.567x+178.3
(R^2)	0.998
RSD	Urine 0.05 Plasma 0.06
Limit of Detection	1.4ng/ml
Enhancement factors	318

Amounts for real sample .

sample	added AC (μ g.L- 1)	Measured AC (μ g.L- 1)	Turn over (%)
urine	0.2	0.196	98.0
plasma	0.15	0.148	98.6

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Determination of Association Constant between Protein and Racemic solution of Chiral Drugs Using Spectrophotometry and HS-NAS

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Background: The enantiomers of a chiral compound share the same physicochemical properties and behavior in achiral environment. However, generally, in biological systems, biochemical receptors and enzymes distinguish chirality, and the activity, metabolism and toxicity of each enantiomer may show significant differences. Increasing evidence suggests that enantioselectivity may occur in the environmental processes and different enantiomers of a chiral compound possess different biological activity and bioavailability [1] , to achieve formation constant between enantiomer and protein need to have a pure of them, Methods currently available for the determination of enantiomeric purity are based either on separation5techniques such as HPLC,CE and GC[2].

Material and methods: totally five data matrices is obtained for simulation and experimental part, which involve the same constant concentration of BSA each separately is titrated with the same various total concentration of propranolol. All measurements is performed with injecting of propranolol to 1.1 cc of BSA in Tris buffer (pH=7.4) in a cuvette. In each inject the solution is shaken and after 3 min the UV-Vis absorption spectra of them is recorded, A thermostatic bath (Peco water) and 1cm quartz cuvette (double layer cell) is used to record spectra at 25°C.

Result: Association constant of S-propranolol with protein and R-propranolol with protein in different mole ratio of enantiomer is achieved by HS-NAS, In comparison with association constant of 100% S-propranolol with protein and 100%R propranolol with protein there is not any significant different. We checked formation constant for other different mole ratio of enantiomer with protein but there was no significant different between them so our system was valid and reproducible to achieve formation constant of racemic solution of chiral drug with BSA which have a small difference in association constant.

Enantiomer mole ratios	0%S	50%S	100%S
K_S	-	1.18×10^4	1.2×10^4
K_R	1×10^4	1.12×10^4	-

K_R, K_S: Association constant between R-propranolol, S-propranolol and BSA

Conclusion: In this work association constants between R-S propranolol and Bovine Serum Albumin is achieved by hard-soft model using UV-Vis spectrophotometry without need any separation method. It can be fast, easy, low cost without any expensive and time consuming separation procedure.

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Determination of Au^+ in water samples by carbon dots- $K_3Fe(CN)_6$ as a new chemiluminescence system

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In this work, a novel chemiluminescence (CL) system has been introduced for the determination of Au^+. This system includes direct chemical oxidation of water-soluble fluorescent carbon dots (C-dots), as luminophor. One of the novel classes of highly luminescent nanomaterials is C-dots. They are generally oxygen-containing carbonaceous nanoparticles with quasi-spherical shapes and with sizes below 10 nm. They typically display high quantum yield fluorescence with interesting size- and excitation wavelength-dependent behavior [1-3]. C-dots were prepared by solvothermal method and characterized by fluorescence spectra and transmission electron microscopy. It was found that $K_3Fe(CN)_6$ could directly oxidize C-dots to produce a relatively intense CL emission. The mechanism of CL generation was investigated based on the CL emission and fluorescence spectra and effect of radical scavengers on the CL intensity. Furthermore, we found that Au^+ has an inhibitory effect on the CL system. Based on this phenomenon, a new analytical method was developed for the determination of Au^+ in the concentration range of 0.05 to 10 mg L^{-1}, with a limit of detection (3s) of 0.02 mg L^{-1}. The developed method was applied to the determination of Au^+ in real water samples with satisfactory results.

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Determination of Ceftazidime in water and biological samples using dispersive liquid–liquid microextraction and UV-VIS spectrophotometer with the aid of response surface methodology and experimental design

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Ceftazidime have been considered to be the drugs of choice for serious infections caused by Klebsiella, Entero-bacter, Proteus, Providencia, Serratia and Haemophylus species [1]. Detect very small amounts in the body fluids, especially in order to identify its toxic effects is important. In this research, a simple and sensitive method for the extraction and determination of ceftizidime in real samples has been developed using dispersive liquid-liquid microextraction (DLLME) prior to UV-VIS spectrophotometer. The appropriate mixture of extraction and dispersive solvents were injected rapidly into the aqueous phase. Then the mixture was centrifuged and the extraction solvent sedimented on the bottom of the conical test tube. The sedimented phase was withdrawn by the microsyringe and measurement using UV-VIS spectrophotometer for analysis. Simplicity of the operation, rapidity, low sample volume, lowcost, high recovery and high enrichment factor are some advantages of DLLME. The experimental design techniques was used to reduce the number of experiments. As a result, costs are reduced and factors involved in the experiment can be optimized in less time. Then, the effective parameters of dispersive liquid-liquid microextraction such as volume of extraction and disperser solvents, sample volume, salt effect, extraction time, centrifuge time and rate were inspected by a centeral composite design (CCD) to identify important parameters and their interactions [2]. UV detection was carried out at 255 nm. The centeral composite design improved the recovery of microextraction.

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Determination of diazinon in apple juice and underground water using dispersive liquid-liquid microextraction coupled with corona discharge ion mobility spectroscopy

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Abstract

In this work, dispersive liquid - liquid microextraction coupled with corona discharge ion mobility spectroscopy (DLLME-CD-IMS) was applied for the extraction and determination of diazinon in apple juice and underground water samples. For the extraction, an appropriate mixture of methanol as dispersive solvent and CCl_4 as extraction solvent was injected rapidly into a glass tube with conical bottom containing aqueous solution of diazinon. This injection led to a cloudy water solution, caused by the fine droplets dispersion of the immiscible extraction solvent (CCl_4) in the aqueous sample. The result of this phenomenon was the generation of a high contact area between the aqueous phase and the extraction solvent. The final step of the microextraction procedure was centrifugation (5 min at 3000 rpm) to collect the dispersed tinny CCl_4 droplets in the bottom of the test tube. Afterward, 5µL of the solution was directly injected into the IMS, for analysis of the analyte. In order to obtaining optimum condition of the extraction, several parameters affecting on the extraction such as: extraction solvent type, dispersive solvent type, extraction and dispersive solvent volume, salt addition and pH were studied. Under optimum condition (pH 7, 60 µL extraction solvent, 1 mL dispersive solvent and without salt addition), enrichment factor 60 and the limit of detection of 0.2 µg/L were obtained. Good dynamic range was obtained in the range of 0.6 - 200 µg/L and 0.8 - 200 µg/L for spiked apple juice and spiked underground water samples, respectively. The relative standard deviations for analysis of 20 µg/L of diazinon in standard solutions, was 5% (n=3). The obtained recovery for spiked samples was above 89%, indicating the capability of DLLME as rapid and convenience method for real sample analysis.

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Determination of erythrosine in food samples by CPE-Scanometry as a new technique for and comparison with spectrophotometric results

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In this study, the trace amounts of erythrosine, as a food dye [1,2] was determined by CPE-scanometry [3] as a new, facile, fast, sensitive, and inexpensive method. The method is based on the CPE of analyte from aqueous solution, diluting the extracted surfactant-rich phase with suitable solvent to 500 µL and transfer of proper volume of it to Plexiglas®cell. The cells containing the analyte solution were scanned with a scanner and then the color of each cell was analyzed with software written in visual basic (VB 6) media to red, green and blue values. The cells were built by creating holes in the Plexiglas®sheet. The proposed method was used to determine erythrosine, as an anionic dye. Effective parameters on the extraction efficiency such as pH of the system, the concentration of the dye and surfactant, equilibration temperature and time, were investigated and optimized. The results and the figure of merits of the proposed method were comparable with those of CPE-Spectrophotometry. Under the optimal conditions, the calibration curve was linear in the range of 0.067-5.33 µg mL-1 for CPE-Scanometry and 0.03-3.0 µg mL-1for CPE-Spectrophotometry. Preconcentration and enhancement factors for the proposed method are 30.00 and 10.45 respectively while for CPE-Spectrophotometry 15 and 3.49 are obtained. The detection limit of two methods was compared. In addition, the effects of some foreign species including cations, anions and dyes were investigated. The method was successfully applied to the determination of erythrosine in food samples.

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Determination of ethanol as a major constituent in samples contain surfactants, fragrances and other additives using adsorbent and by refractometric method

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Ethanol uses as a major constituent in the solvent-based cleaners and increases the cleaning power of the product. Also, it causes more stability of non-polar components of the cleaner such as essences and fatty alcohols in the aqueous medium. Therefore, determination of ethanol contents in the solvent-based cleaners is important for evaluating quality and detecting adulteration [1]. The common analytical methods for measuring of ethanol in the solvent-based cleaners are gas chromatography and alcoholometry [2].

In this study, the ethanol contents in a specific solvent-based cleaner were detected by refractometric method using a simple instrument. The samples containing different concentration of ethanol (between 5-20 Wt%), 0.35 Wt% of surfactants, 0.1 Wt% essence and other additives passed through the column packed with activated carbon large particles that was saturated with ethanol. So, all components of the cleaner except from ethanol adsorbed by activated carbon. Finally, the ethanol concentration in the sample was determined by comparing refractive index of output ethanol from the column with calibrated curve which is refractive index of ethanol on the basis of Wt% of ethanol at 25 ˚c (fig. 1). 10 cm^3 of adsorbent and flow rate of 1.2 ml/min for passing the sample used as optimized conditions for column. The results from all three methods (gas chromatography, alcoholometry and refractometry) are critically evaluated and their impact for studies of authenticity is discussed. The refractometric method is simpler than gas chromatography and has proper accuracy in comparison with alcoholometry.

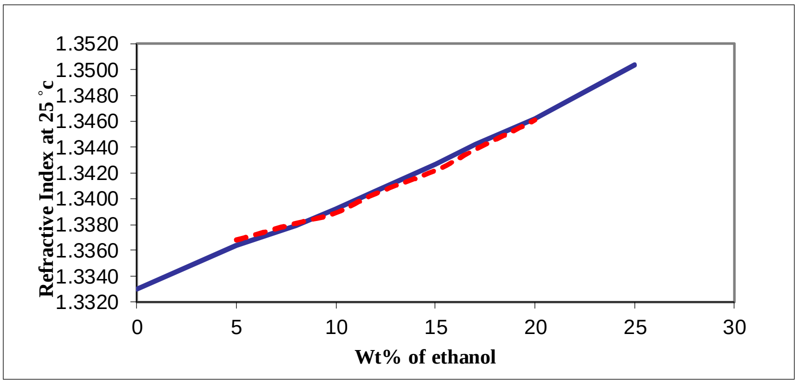

Fig. 1. —— Calibrated curve for ethanol – – – Measured ethanol in the cleaner by refractometry

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Determination of ethanol as a major constituent in samples contain surfactants, fragrances and other additives using adsorbent and by refractometric method

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Ethanol or ethyl alcohol uses as a major constituent in the solvent-based cleaners and increases the cleaning power of the product. Also, it causes more stability of non-polar components of the cleaner such as essences and fatty alcohols in the aqueous medium [1]. Therefore, determination of ethanol contents in the solvent-based cleaners is important for evaluating quality and detecting adulteration. The common analytical methods for measuring of ethanol in the solvent-based cleaners are gas chromatography and alcoholometry [2].

In this study, the ethanol contents in a specific solvent-based cleaner were detected by refractometric method using a simple instrument. The samples containing different concentration of ethanol (between 5-20 Wt%), 2 Wt% of surfactants, 0.5 Wt% essence and other additives passed through the column packed with activated carbon large particles that was saturated with ethanol. So, all components of the cleaner except from ethanol adsorbed by activated carbon. Finally, the ethanol concentration in the sample was determined by comparing refractive index of output ethanol from the column with calibrated curve which is refractive index of ethanol on the basis of Wt% of ethanol at 25 ˚c (fig. 1). 10 cm^3 of adsorbent and flow rate of 1.2 ml/min for passing the sample used as optimized conditions for column. The results from all three methods (gas chromatography, alcoholometry and refractometry) are critically evaluated and their impact for studies of authenticity is discussed. The refractometric method is simpler than gas chromatography and has proper accuracy in comparison with alcoholometry.

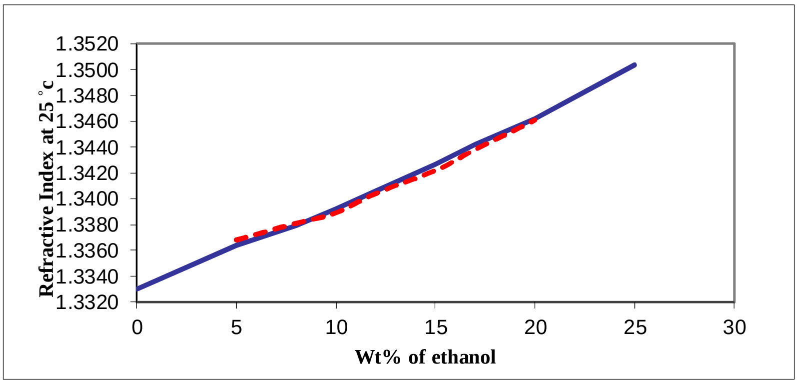

Fig. 1. —— Calibrated curve for ethanol – – – Curve of output solution from column

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Determination of Fe(II) in the iron ore pellet by Magnetic Moment Method

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Measuring the total magnetic moment of a sample in a saturating magnetic field is a quick accurate and reliable method of measuring the magnetic material content of the sample. It is much faster than chemical methods and far more accurate than measurement based on the susceptibility of the material. The principle behind the measuring is to measure the force acting on the sample in a magnetic field with a spatial gradient. The magnetic field is strong enough to saturate the magnetic component in the sample [1].

Accurate analysis of ferromagnetic compounds of iron (especially in iron ore), such as magnetite, is extremely difficult and time-consuming by chemical methods. These difficulties can be overcome by utilizing the magnetic properties of the material. The design and principle of the operation method assure a high degree of precision, combined with a measurement procedure that is quick and simple.

The operation of the measuring is based on measurement of the magnetic moment after the magnetic component in the sample has been magnetized for saturation [2]. The total magnetic moment is: $m = V.\ M_{sat}$
V = volume of the magnetic component in the sample
M_{sat} = saturation magnetization of the magnetic component
In the method, the magnetic moment is determined by measuring the force acting on the sample in a non-homogeneous magnetic field (A field having a vertical gradient of (dH/dz) and comparing it with the gravitational force acting on the sample).
The relative standard deviation for the analysis of three to five replicate measurements of sample containing 0.292, 0.302, 0.440 and 0.598 FeO% were 0.87, 0.81, 1.8 and 0.62 respectively.
Our studies showed that the new magnetic method is capable to analysis of FeO in iron ore sample without need to dissolve the sample. The new method is fast, low cost, precise, accurate and environmental friendly.

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Determination of glucose using of an optimized novel luminal- CuO nanoparticles-H_2O_2 chemiluminescence method by Box–Behnken design

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Abstract

Quantitative determination of glucose is very important in biochemistry, clinical chemistry, food processing and fermentation. Enzymatic method yields maximum specificity for glucose estimation. Glucose can be measured by its reaction with glucose oxidase (GOD), in which gluconic acid and hydrogen peroxide are formed [1]. The catalyzed luminol chemiluminescent reaction has received, for more than 30 years, a great amount of attention because of its high sensitivity and low background signal which make the reaction as an attractive analytical chemistry tool [2]. Recently, nanoparticles reveal remarkable catalytic qualities for a variety of chemical reactions, depending upon their high surface areas, good adsorption characteristics, high activity, and high selectivity. Cupper oxide (CuO), a p-type semiconductor with a narrow band gap (1.2 eV), has been studied intensely because of its interesting properties and widespread applications in various fields. Furthermore, these CuO NPs are considerably more stable and possess an almost unchanged catalytic activity over a wide range of pH and temperatures and exhibited significant peroxidase-like activity [3]. In this paper, we reported a simple and fast CL method for the determination of glucose in plasma. The optimization of variables influencing the chemiluminescence response of the method has been carried out by using experimental design (Box-Behnken design). Under the optimal conditions,there is good linear relationship between the luminal-CuO NPs -H_2O_2 relative chemiluminescence intensity and the concentration of glucose over the range of 1.0×10^{-6} - 1.0×10^{-3}M (R^2 =0.995) with a 3σ detection limit of 2.9×10^{-7}M and the relative standard deviation (RSD) < 3.3% was obtained.

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Determination of haloacetic acids in water samples by acidic methanol esterification following headspace solid-phase microextraction and gas chromatography–mass spectrometry

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Water chlorination is an efficient and low cost technique for proven it from growing malicious microorganisms. However, it reacts with natural organic matter in the raw water via halogen substitution and oxidation reactions, forming a wide range of compounds known as disinfection by-products (DBPs). Two major groups of DBPs are trihalomethanes (THMs) and haloacetic acids (HAAs) that have adverse effect on human health and the environment [1]. The US Environmental Protection Agency (EPA) has established maximum contaminants levels (MCL) for the sum of five HAAs (HAA5) 60 μgl^{-1} [2].

In this study a simple, fast and efficient technique using headspace solid-phase microextraction (HS-SPME), in conjunction with gas chromatography-Mass spectrometry (GC-MS) has been developed for the determination of haloacetic acids (HAAs) in water. The analytical procedure involves liquid- liquid extraction (LLE), evaporation of extraction solvent to dryness, derivatization of HAAs into their methyl esters with acidic methanol, HS-SPME, and GC-MS determination. Four kind of SPME fibers (PDMS, End-capped PDMS, PEG and UCON) were made by sol-gel technique [3] and applied for extraction. The PDMS fiber has shown the highest extraction efficiency for six HAAs. The influential parameters on derivatization and extraction such as derivatization and extraction temperature and time, desorption condition and sample ionic strength were investigated and optimized. The calibration curves were linear in concentration ranging from 0.05 to 500 $\mu gl^{-1}$with correlation coefficients (R^2) of greater than 0.98. Good precision (RSD lower than 14%) and detection limits in the range of 10 -100 ngl^{-1} were obtained for the analytes.

Keywords: Haloacetic acids, Headspace solid-phase microextraction, , Disinfection by-product, GC-MS.

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Determination of megestrol acetate and levonorgestrel in blood samples by supercritical fluid extraction followed by nanostructured supramolecular solvent microextraction

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Levonorgestrel (LeV) is categorized as the second generation of synthetic progestogen widely used as an active ingredient in some hormonal contraceptives. Megestrol acetate (MA) is a progesterone-like compound used to combat malnutrition associated with AIDS and cancer [1]. Suparmolecular solvent microextraction (SUPRAS-ME) is an extraction method that consumes less organic solvent and very high enrichment factors can be obtained by using this method. SUPRASs are nano-structured liquids generated from amphiphiles through a sequential, self-assembly process occurring on two scales, molecular and nano [2]. The present work describes a sensitive procedure for extraction and determination of two drugs (LeV and MA) in blood samples using supercritical fluid extraction (SFE) coupled with SUPRAS-ME. SUPRAS made up of decanoic acid (DeA) assemblies dispersed in THF and water was proposed. The Taguchi orthogonal array (OAD) experimental design with an OA_{16} (4^5) matrix was employed to optimize the SFE conditions. In SFE-SUPRAS-ME procedure, the mixture of DeA and collecting solvent of SFE (THF) were added to water for SUPRAS formation. The effective parameters on the SUPRAS-ME efficiency were studied and optimized utilizing face center design (FCD). Under the optimum conditions linear dynamic ranges (LDRs) varied in the range of 0.7-7 mg kg^{-1}. R^2 ranged from 0.9978 to 0.9983. The Intraday (n = 5) and interday standard deviations were calculated by extracting the drugs from blood samples. Interday RSDs% lower than 7.9%, and intraday RSDs% lower than 6.8% were obtained. The limit of detections (LODs), based on S/N was 0.2 µg g^{-1}.

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Determination of Mercury at the Rotating Gold Electrode by Anodic Stripping Voltammetry

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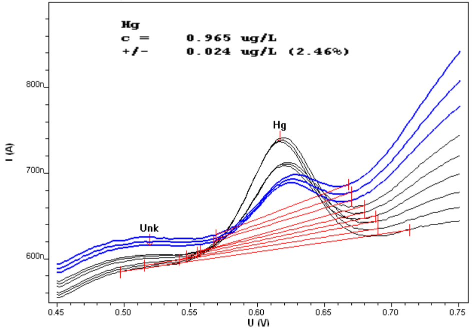
Voltammetry is a category of electrochemical methods used in analytical chemistry and various industrial processes. In voltammetry, information about an analyte is obtained by measuring the current as the potential is varied [1,2]. Anodic stripping voltammetry, a quantitative analytical method for trace analysis of metal cations. The analyte is deposited onto the working electrode during a deposition step, and then oxidized during the sweep potential step. The current is measured during the sweep potential.

Mercury is a toxic heavy metal, and even low concentration of that has harmful effects on human health. Mercury compounds can cause allergic reactions or problems in brain and neurons, so measuring the ultra trace of mercury is very important[3]. Mercury can determine by anodic stripping voltammetry (ASV) at the surface rotating gold electrode. Mercury ions are deposited on the gold electrode by reduction to Hg0 in stripping step and then in sweep potential step the deposited Hg0 is oxidized to mercury ions. But this method is only suitable for samples with a low chloride concentration (Cl^-< 0.01 mol/L or 350ppm). For samples with a higher chloride concentration or in the presence of other halides the positive potential range of the method must be limited, as otherwise the gold electrode will be irreversibly damaged by oxidation. This work describes a modified gold electrode for the determination of mercury by anodic stripping voltammetry (ASV) at the surface of rotating electrode. The calibration curve is linear from 0.4 µg/L to 15 µg/L with a detection limit of 0.2 µg/L. The method has primarily been drawn up for determination of trace amounts of Hg(II) indifferent water and wastewater samples. After appropriate digestion the determination of mercury is possible even in wastewater with a high load of organic substances.



voltammogram of Hg(1ppb)

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Determination of methanol based on fluorescence quenching of TGA-capped CdS quantum dots in plant extracts.

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Abstract

In this paper we report a simple and very sensitive method for determination of trace amounts of methanol in drinks based on fluorescence quenching of TGA-capped CdS quantum dots .the quenching mechanism was attributed to the non-radiative recombination due to the esterification reaction that occurs between methanol and carboxylic group of TGA. Under optimum conditions a wide linear dynamic rang was obtained between 0-12%(v/v). The proposed method manifested several advantages such as high sensitivity, short analysis time, low cost and ease of operation.It is well known that methanol & ethanol are the most common toxic substance consumed by humans.It is not only cell toxic but excessive intake adversely affects human relationships. The determination of this substance is an important task due to their presence in many fields of the food industry either a desired or unwanted product. Also it is important for quality control of the products.methanol is routinely measured during wine production, using well-established methods that usually involve the separation of the analyte from the sample matrix by distillation before chemical or physical measurements, or then using chromatographic methods[1].Chromatographic techniques lack portability and need long analysis time[2] . Biosensor has advantages in terms of selectivity and portability but life-time of the sensor is not long and fabrication of the sensor is rather complicated and expensive.Flow methodology is also proposed in order to increase degrees of automation of the method [3].In this work,CdS quntum dots (QDs) that have been synthesized using thioglycolic acid (TGA) as capping agent through a one step process wich have good stability and excellent water solubility and have strong affinity for investigated analyt.this probe is based on fluorescence quenching of functionalized CdS QDs.factors affecting methanol detection investigated and the optimum experimental conditions were determind.under the optimum conditions the maximum excitation and emission wavelengths of CdS QDs were 427 and 555 nm,respectively the calibration curves (stern-volmer plots) were linearly proportional to the concentratios of methanol over a dynamic range from 0-12% (v/v) with the correlation coefficient of o.999.relative standard deviation 2.9% obtained (n=5).the proposed method is simple,rapid,inexpensive,sensitive and can be used in determination of methanol in plants extract in real sampels.

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Determination of Mn(II) in diethanolamine as 2-(5′-bromo-2′-pyridylazo)-5-diethylaminophenol complex by voltammetry at modified glassy carbon electrode

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One of the most effective and efficient methods for the identification and determination of Mn(II) in diethanolamine (DEA) is an electrochemical method using modified electrode. For this purpose, glassy carbon electrode surface was modified using 2-(5′-bromo-2′-pyridylazo)-5-diethylaminophenol (5-Br-PADAP). The GCE modification was performed by successive cyclic sweeps between -0.2 and +1.1 V (*vs.* Ag/AgCl / 3M KCl) at sweep rate 0.03 Vs^{-1} and the cleaned GCE immersed on the phosphate buffer solution including 0.01 mol l^{-1} phosphoric acid and 0.11 mol l^{-1} NaOH (pH 8), and 0.5 mmol l^{-1} of (5-Br-PADAP). During this process, (5-Br-PADAP) was attached to the GCE surface. This is a new modified electrode for determination of Mn(II) in DEA on the GCE. Mn(II) was determined by adsorptive stripping voltammetry on the modified electrode. Adsorptive accumulation was performed at the potential between 0.8 and 0.9 V (*vs.* Ag/AgCl / 3M KCl) and after equilibrium time 60 s, anodic stripping was carried out by differential pulse voltammetry (DPV). At the optimized conditions including pulse amplitude 0.100 V and potential step 0.009 V, the linear range between 2×10^{-9} and 3×10^{-6} mol l^{-1}, detection limit 2×10^{-9} mol l^{-1} was obtained for Mn(II). This method was successfully applied for determination of manganese in industrial samples including DEA media.

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Determination of oleuropein using an electrochemical DNA biosensor

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Oleuropein, a natural product of the secoiridoid group, is the major bioactive compound in olive leaves [1]. For its wide range of pharmacological properties such as antioxidant [2] and anti-cancer [3], oleuropein is commercially available as food supplement in Mediterranean countries.

We have developed an electrochemical DNA biosensor based on the immobilization of double-stranded deoxyribonucleic acid (dsDNA) on the surface of a chitosane-modified carbon paste electrode for the rapid, sensitive and selective determination of oleuropein. A considerable increase was observed in the oxidation signal of Ole at the DNA-coated electrode compared with the DNA-free electrode, indicating the pre-concentration of Ole due to the interaction with the surface-confined DNA layer. After optimizing different experimental conditions, a linear concentration range of 0.30-12.0 µM with a detection limit of 0.090 µM was obtained. The proposed biosensor was successfully applied to the determination of Ole in olive leaf extract and human serum samples.

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Determination of oxalic acid and citric acid and their mixtures using chemiluminescence method

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A novel kinetic chemiluminescent method has been investigated for the rapid and sensitive determination of oxalic acid (OA) and citric acid (CA) in their mixtures. The method is based on a tris(1,10-phen)ruthenium(III) (Ru(phen)$_3^{3+}$) chemiluminescence (CL) reaction. Ru(phen)33+ was generated in the mixing chamber by oxidizing Ru(phen)$_3^{2+}$ with cerium(IV). After selecting the best operating parameters, calibration graphs were obtained over the concentration ranges 2.0×10^{-7}-2.0×10^{-4} mol L^{-1}and 4.0×10^{-8}-1×10^{-5} mol L^{-1} for CA and OA, respectively. The limits of detection were 1.0×10^{-7} mol L^{-1} for CA and 2.0×10^{-8} mol L^{-1} for OA. Relative standard deviation (RSD) of the method for 11 times determination of 3.2×10^{-6} mol L^{-1} of CA was 2.9%. Based on the differential rate of the chemiluminescent reaction corresponding to CA and OA, a very simple kinetic procedure was developed for the simultaneous determination of both compounds. The CA system gives the highest chemiluminescence intensity at 6 s and the peak is broad, whereas the OA system gives its most intense chemiluminescence emission at 1 s and the peak is sharp [1]. Mixtures of CA and OA in ratios between 30:1 and 3:1 were satisfactorily resolved. The proposed method was successfully applied to the determination of mixtures in pharmaceutical formulations.

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Determination of parabens in different samples using simultaneous air-assisted liquid-liquid microextraction and derivatization followed by GC-FID

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Abstract

In this study, a simple, rapid, and economical method has been proposed for the extraction, preconcentration, and determination of parabens in different samples based on derivatization by acetic anhydride and preconcentration using air-assisted liquid-liquid microextraction (AALLME) in a single step. AALME method was proposed in 2012 [1]. In this study, 1,1,2,2-tetrachloroethane (extraction solvent) containing μL-level of acetic anhydride (derivatization agent) is injected into aqueous sample containing parabens and picoline (catalyst). The mixture of aqueous and organic phases is withdrawn into a glass syringe and then injected into the test tube for several times till a turbid solution is obtained. After centrifuging 1 μL of the sedimented phase is injected into gas chromatography-flame ionization detector (GC-FID). On the extraction efficiency is investigated. Under the optimal conditions, the enrichment factors (EFs) and enhancement factors (EnFs) for the parabens were obtained in the ranges of 310-353 and 2484-5298, respectively. Limits of detection (LODs) were between 0.18 and 8.5 μg L^{-1}. Linear range were between 10–10000 μg L^{-1} ($R^2 >$ 0.999). The obtained extraction recoveries (ERs) ranged from 69 to 107 % and precision, expressed as relative standard deviation (RSD %), was within the range of 4.0-13%.

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Determination of pesticides in Zayandeh Roud River by Gas chromatography-Ion mobility spectrometery

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The use of pesticides in agriculture may lead to contamination of surface and ground water by drift, runoff, drainage and leaching. Surface water contamination usually depends on the agricultural season and does not last long, while ground water contamination has a strong inertia, which may cause a continuous human exposure [1]. Diazinon is an organophosphorus insecticide used as a pesticide for tomatoes, potatoes, grapes and rice. Trifluraline is one of the most common herbicides used to control grasses and weeds in a wide variety of agricultural crops. As a result of its widespread use, trifluralin has been frequently detected in environmental waters. Pesticides are detected in aqueous samples mostly by chromatographic techniques, e.g., gas chromatography coupled with mass spectrometry (GC–MS) [2], electron capture or nitrogen phosphorus selective detection [30, 36]. Ion mobility spectrometry (IMS) is a vanguard analytical technique that provides analytical information in a simple, rapid and, inexpensive manner. For example, an ion mobility spectrometer can be used to extract global information about the volatile profile of a sample. In recent years it was used as a novel detector in GC [3]. The aim of this work has been the development of a gas chromatographic– ion mobility spectrometry (IMS) technique which designed and constructed in institute of materials and energy of space agency, Detector department, preceded by a solid-phase extraction step optimized for the analysis of pesticides selected as the ones most probably found in the Zayanderoud River (Isfahan, Iran).
The limit of detection (LOD) for diazinon and trifluralin were 28 and 37.4 ng µL−1, respectively. Results revealed that the performance of GC-IMS in identification and quantification of pesticides is better than IMS alone.

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Determination of Polycyclic Aromatic Hydrocarbons by Stir-Bar Sorptive Extraction and High Performance Liquid Chromatography Using Polypyrrole doped with chlorosulfonic acid as Sorbent

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Abstract

Polycyclic aromatic hydrocarbons (PAHs) enter the environment through various human activities such as burning fossil fuels and biomass, industrial processes, and oil spills. These organic compounds have two or more aromatic rings and some of them are carcinogenic [1]. Therefore, the determination of these compounds in the environment is of considerable importance.

In this work, we used a stainless steel stir-bar electrochemically coated with polypyrrole and doped with chlorosulfonic acid to extract PAHs from water samples. Analytes are extracted by a Stir-Bar Sorptive Extraction (SBSE) method and determination is carried out by high performance liquid chromatography (HPLC) and UV detection.

A one-at-a-time optimization strategy was applied for optimizing important extraction parameters such as coating time, coating potential, chlorosulfonic acid concentration, pyrrole concentration, extraction time, extraction temperature, ionic strength, solution pH, desorption solvent volume and desorption time.

Under optimal condition, the linear range of the method was between 0.1-100 ng mL^{-1} for biphenyl and anthracene, and 0.1-500 ng mL^{-1} for fluorene, fluoranthene, phenanthrene and pyrene. The detection limits found were between 0.01 and 0.19 ng mL^{-1} for the compounds under study. The recovery of PAHs from spiked water samples were between 86 and 118%. The relative standard deviation varied between 2.5 and 7.4 %. The relative standard deviation of the coating process was between 3.8 and 13.0%.

The proposed SBSE-HPLC-UV method was successfully applied to the analysis of the above PAHs in Zarand Coal Processing Industries wastewater, a well water sample collected from the vicinity of the coal industry and a well water sample from Shahid Bahonar University campus.

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Determination of potassium ion by using nickel (II) hexacyanoferrate nanoparticles (NiHCF-NPs) electrodeposited on Junction Field Effect Transistor (JFET) as ion sensitive field effect transistor (ISFET)

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A simple and low cost potassium sensor based on a commercial junction field effect transistor (JFET) is introduced. The transistor was mechanically treated, isolated and used as an ion sensitive field effect transistor (ISFET) [1] for potassium ion detection. Nickel (II) hexacyanoferrate (NiHCF), as the suitable sensing membrane, was electrodeposited on the copper metal gate (an oxidisable metal) of the transistor under two-step deposition conditions. First, surface of copper metal gate was completely covered with polypyrrole (PPy) using a solution containing pyrrole monomer including sodium salicylate in order to avoiding the substrate oxidation [2]. Then, NiHCF was electrodeposited on ISFET/PPy electrode by using Cyclic Voltammetry mode and used as the sensing membrane toward potassium ion after conditioning of ISFET/PPy/NiHCF electrode. The nanostructure surface of the constructed sensor was characterized by scanning electron microscopy technique.

The prepared sensor showed a near-Nernstian response of 52.3 mV pK^{-1} over a linear wide range of 1.96×10^{-6} – 5.50×10^{-2} M, a detection limit of 1.343×10^{-6} M, a drift of 0.02 mV h^{-1}, and a response time of about 120 sec.

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Determination of promazin using DNA interaction at a surface of nanocomposite modified pencil graphite electrode

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Abstract: Promazine (Sparine) is a medication that belongs to the phenothiazine class of antipsychotics. An older medication used to treat schizophrenia, it is still prescribed, alongside newer agents such as olanzapine and quetiapine [1]. In this study, a sensitive electrochemical procedure based on DNA interaction with promazin at a modified nanocomposite pencil graphite electrode was introduced as a promising tool for determination of promazin. The decrease in guanine and adenine oxidation peak intensity signals was used as an indicator for the sensitive determination of promazin. Under the optimum conditions, a linear dependence of oxidation signals was observed to the promazin concentration with a detection limit of 0.02 μmol L^{-1}. The relative standard deviation of five replicate measurements of 5.0 μmol L^{-1} promazin concentrations was 2.5%, respectively. The influence of potential interfering substances on the promazin determination was studied. Finally, propose biosensor was applied for the determination of promazin in tablets and urine samples with satisfactory results.

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Determination of Simvastatin Content in Powdered pharmaceutical Formulations using Fourier Transform Infrared Spectroscopy

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A quick and reliable analytical method for the quantitative assessment of simvastatin in pharmaceutical formulations was developed using Fourier-transform infrared (FT-IR) transmission spectroscopy for routine quality control. For the determination of simvastatin, conventional KBr was used in the form of pellets for acquisition of the FT-IR spectra of standards and samples [1]. The sample preparation was avoided except grinding for pellet formation and eliminated the use of toxic solvents. The calibration model was followed based on simple Beer's law using the FTIR carbonyl region (C=O) from 1640-1780 cm^{-1}. Direct determination was used in the real sample through peak area measurement and using a baseline correction between 1650-1770 cm^{-1}. For standardization an external calibration line was established from standards of simvastatin in KBr. The excellent coefficient of determination (R^2) was achieved (0.9911) with 1.09% relative standard deviation (RSD%) for 5 independent measurements.

This work clearly revealed the capability of the transmission FT-IR spectroscopy and extended its application for determining the exact quantity of simvastatin to control the processing formulation and quality of finished product with the analysis time of less than 3 min using the neat solid samples.

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Determination of Six Nutrient Mineral Contents of Three Common Vegetables After Being Affected by Freezing and Drying

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Today people apply different ways to keep their food for a long time and vegetable is one of those foods which is considered as a very important nutrient source. For this reason, considering a good way to keep vegetables for a long time is found a very important and challenging matter. Freezing and drying are two common ways of storing vegetables. Six mineral concentrations of three kinds of vegetables involving spinach, leek and parsley have been determined using Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES). The concentrations of Fe, K, P, Na, Ca, and Mg have been compared between dried and freezed vegetables. The vegetables have been digested by the usage of wet digestion method including nitric acid, sulphric acid and by the help of hydrogen peroxide.[1] This way of digestion is considered as one of the best ways leading materials to complete digestion. To make sure about the precision of work all solutions were prepared three times and to find the accuracy, the datas were also investigated using atomic absorption spectroscopy. The results have indicated that there is no way to find one way of storing better than the other. The reason for this claim is the data showed some nutrients lose their concentration while freezing and some others for the case of drying. As a result people should get to know about their body needs before they decide to choose a way to keep their vegetables.

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Determination of sodium benzoate and potassium sorbate preservatives in dairy products using quick, easy, cheap, effective, rugged and safe (QuEChERS) extraction followed by high performance liquid chromatography analysis

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QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) is one of the popular sample preparation methods. It was introduced by Anastassiades in 2003. Most QuEChERS method have been used for analysis of pesticide residues in fruits and vegetables, but recently, the analysis of veterinary drugs, PAH, PCB, PBDE, alkaloids, acrylamides has also been used. Generally, the procedure involves the single-phase extraction of analytes from sample with organic solvent followed by the liquid–liquid partitioning with sodium chloride (NaCl) and anhydrous magnesium sulphate ($MgSO_4$). After simple vortex mixing and centrifugation, clean-up is performed by adsorbents. This step is called dispersive solid-phase extraction (DSPE). Dispersing adsorbent most frequently used is PSA (primary–secondary amine) for remove of compounds such as fatty acids, organic acids, and various sugars, C_{18} for remove of non polar compounds such as lipids and sterols, GCB (Graphite Carbon Black) can also be used for remove of pigments [1, 2].

In this study, sodium benzoate and potassium sorbate as chemical preservatives in dairy products were determined using QuEChERS method followed by high performance liquid chromatographic (HPLC) analysis. These compounds inhibit the growth of bacteria and fungi and yeasts thus increasing the shelf life of food, but causing symptoms such as such as hives, asthma, burning eyes, seizures and genetic damages on consumers [3].

Several factors such as type of QuEChERS (buffering or non-buffering), type of extraction solvent (methanol, acetone, ethyl acetate, acetonitrile, n-hexane/ethyl acetate), sample pH (3, 4, 5) and extraction time (1-10 min) were individually optimized. Experimental design methodology was also used for optimization of PSA and C_{18} amounts (58.57-341.42 mg). As a result, non-buffering QuEChERS methodology was selected and the optimum levels were obtained acetone, 58.6 mg, 3 minutes for the extraction solvent, PSA and C_{18} amounts and extraction time respectively.

At the optimal values of effective factors, calibration curve was plotted in range of 1-500 mg L^{-1} and recovery for sodium benzoate and potassium sorbate were obtained 83 ± 2.9 and 99 ± 1.5, respectively. Finally, milk, yogurt, Doogh as a real samples were chosen .

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Determination of sodium hypochlorite in various samples based on anodic voltammetry using a modified carbon paste electrode

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Abstract

Hypochlorite is a powerful oxidizing agent and it is used for various purposes such as disinfection, bleaching and manufacturing processes. It is an efficient and inexpensive oxidant and is available as sodium hypochlorite in alkaline solutions, with a pH of approximately[1]. It is recognized that chlorine is highly toxic, and provides a strong and persistent odor even at low concentrations Chlorine applied to water in its molecular or hypochlorite form initially undergoes hydrolysis to form free chlorine consisting of aqueous molecular chlorine, hypochlorous acid (HClO), and hypochlorite ions (ClO). Various techniques are used to monitor free chlorine, such as iodometric titration, many colorimetric methods based on reaction of hypochlorite with organic reagents as methyl orange, chemiluminiscent methods such as that based on fluorescein test strip, and voltammetric methods[2]. In this work, a new chemically modified carbon paste electrode was constructed and used for rapid, simple, accurate, selective and highly sensitive determination of sodium hypochlorite using differential pulse voltammetry. The carbon paste electrode was modified by powder Al_2O_3. Compared with carbon paste electrode, the oxidation current had a significant increase at the modified electrode. The influence of pH and potential sweep rate on the electrode response were inestigated. Operational parameters such as deposition potential, deposition time and pulse amplitude were optimized for in order to determine traces amount of NaClO. At the optimum conditions the sensor has a linear response in the 10.0–300.0 μ mol L^{-1} concentration range. In addition, the modified electrode showed a good reproducibility and selectivity, making suitable for the determination of NaClO in real sample such as natural water, tap water and milk.

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Determination of tetracycline by spectrophotometeric technique based on silver nanoparticles surface palsmon resonance

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Tetracyclines (TCs) are broad-spectrum antibiotics that are active against both gram-positive and gram-negative bacteria [1]. It has been widely used as animal feed additives to promote growth, veterinary medicines and current therapy owing to their extensively antibacterial activity in human because of its good oral absorption ,relatively low toxicity and low cost. These substances remain as residues in divers sample such as water, soil, stream sediments agricultural products such as fruits, vegetables , milk and honey [2]. During recent years Because of harmful effects of this drug on human health various methods have been used for determination of tetracycline in different samples. In this work, we have developed a simple and sensitive method for determination of trace levels of tetracycline. The method is based on the redox reaction between silver nitrate and tetracycline in alkaline medium containing ammonia and sodium hydroxide at 65°C. Under this conditions silver nanoparticles (Ag NPs) is formed. The NPs are stabilized in solution by using poly(vinyl pyrrolidone) as a capping agent. The yellow color of the solution that results from the localized surface plasmon resonance of Ag NPs can be observed by the bare eye. The absorbance at 411 nm is proportional to the concentration of tetracycline.The calibration curve is linear in the concentration range from 0.05-5 mg L^{-1}, with a limit of detection of 0.0133 mg L^{-1}. The method was successfully applied to the determination of tetracycline in pharmaceutical forms.

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Determination of the acidity constant of methyl orange by scanometry method and comparison with spectrophotometric result

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Dissociation constants (i.e. pK_a values) can be a key parameter for understanding and quantifying chemical phenomena such as reaction rates, biological activity, biological uptake, biological transport, and environmental fate. Numerous analytical procedures including potentiometric, spectrophotometric, conductometric and spectroscopic have been reported for the determination of acidity constants of organic compounds [1].

The scanometry technique has been introduced recently [2]. In this method commercially available flatbed-scanners for obtaining the images of color solutions was used. The obtained images have been transferred to computer for analyzing and determining the intensity of color solutions. The method is based on the reaction in solution phase, in Plexiglas® cells. The solution was scanned and finally the RGB (red, green and blue) color model was used in color monitor. We used for determination of the acidity constant, pK_a, of methyl red indicator, at first time. The comparison between the current and traditional UV-Vis spectrophotometry methods was studied and the results revealed similar trends in both methods.

For evaluation pK_a of the indicator using spectrophotometric data was down by the Hypspec program. The calculated pK_a values of the cited indicator by scanometry and spectrophotometric methods are 3.22 and 3.29 respectively. The corresponding distribution diagrams were depicted by Hyss2009 program as a new version of the older one [3].

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Determination of Trace Amounts of Ag (I) by Ultrasound Assisted Emulsification Microextraction (USAEME) Combined with FAAS Using 1,4,8,۱۱-Tetra aza cyclo tetra decane (cyclam)

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The silver content of environmental samples is increased with the increasing use of silver compounds in industry and medicine [1]. The separation, concentration and sensitive determination of Ag(I) ions are of increasing interest. Regueiro *et al*. [2] applied a miniaturized approach to ultrasound-assisted radiation in LLE methods (USALLE) by using a micro volume of organic phase to provide the advantage of both dispersive liquid-liquid microextraction (DLLME) and (USALLE) [2]. High extraction efficiency in a short period of time is the main advantage of USALLE. They have successfully applied ultrasound-assisted emulsification micro extraction (USAEME) to determine some emergent contaminants and pesticides in environmental waters. Fontana *et al*. [3] applied this method for determination of poly brominated flame retardants in water samples. They demonstrated that USAEME is an efficient, simple, rapid and cheap extraction technique prior to GC analysis.

In this paper USAEME method combined with the flame atomic absorption spectrometry (FAAS) was proposed for the determination of trace of silver ions using 1, 4, 8, 11- tetra aza cyclo tetra decane (cyclam) as chelating agent. Several factors which affect on the micro extraction efficiency of silver, such as pH, extraction solvent type, volume, concentration of the chelating agent,and extraction time were investigated. and the optimized experimental conditions were established. In the optimum conditions, the enrichment factor was 9.8. The detection limit and the relative standard deviation (RSD) for fifteen determinations of 0.5 μg mL^{-1} silver ions were 6.79 ng mL^{-1}and 5.56 %, respectively. In addition, the calibration graph using the preconcentration system for silver was linear from 0.055 to 1.5 μg mL^{-1} with a correlation coefficient of 0.997. The results for the determination of silver ions in well water and radiology film have demonstrated the accuracy, recovery and applicability of the proposed method.

Keywords: Emulsification microexraction, Ultrasonic, Cyclam, Silver, Radiology film

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Determination of trace amounts of Nickel in potable water by Dispersive liquid-liquid microextraction and Ultraviolet-Visible spectrophotometry

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Nickel is one of toxic elements in nature, so exploring and determination of this element is one of important research areas [1]. Depending of the nature of samples several methods has been developed for the determination of Nickel such as solid phase extraction of saline samples and determination with ICP-AES. Or Cloud point Extraction in water samples and determination by FAAS [2]. Dispersive liquid-liquid micro extraction (DLLME) is based on dispersion of tiny droplets of organic solvent in water based samples is a simple, rapid and inexpensive method. The application of DLLME was shown in extraction of different organic and inorganic compounds from environmental samples [3].

In the present study determination of trace amount of Nickel ions in aqueous samples was performed by the application of DLLME and determination with Uv-Vis spectrophotometry. Nickel ions were complexed by dimethylglyoxime and extracted in a few microlitres of CCl_4 at the presence of dispersing solvent Acetonitrile. Determinations were conducted in 334 nm (absorption maxima of dissolves complex in CCl_4).

Four parameters was optimized using orthogonal array design Taguchi method [4]. pH, ligand to metal ratio (L/M), extraction solvent volume (V_E) and dispersing solvent volume (V_D). The optimized condition for extraction of 5 mL portion of samples was determined as: pH=10, L/M=25, V_E=75 μL and V_D=2 mL. Linear dynamic range was determined by calibration curve from 0.2 ppm to 12 ppm of Ni (R^2=0.996). Detection limit was determined 0.05 ppm Ni (from three times standard deviation of seven replicate of blank divided by the calibration curve slope). And limit of quantification was determined 0.17 ppm Ni (from ten times standard deviation of seven replicate of blank divided by the calibration curve slope). The proposed method was successfully applied to spiked water samples and results were comparable with Flame Atomic Absorption Spectrometry (FAAS).

As a short result, the proposed method is inexpensive and has a performance comparable with the FAAS.

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Determination of trace amounts of Nickel in potable water by Dispersive liquid-liquid microextraction and Ultraviolet-Visible spectrophotometry

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Determination of trace water in various organic solvents using headspace extraction and gas chromatography

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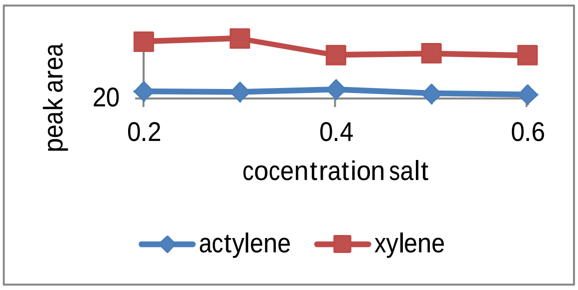
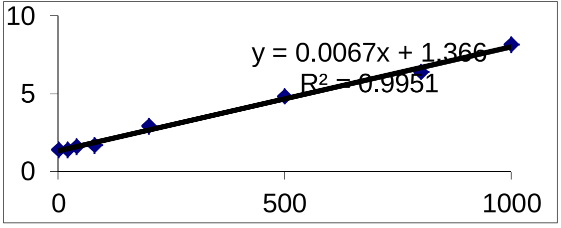
Abstract:

In science and industries, Determination of trace amounts of water in organic solvents is very important. Trace water in organic reactions can effect on type, products and efficiency of reaction[1]. Trace water can also be very harmful in analysis of materials with gas chromatography with ECD and Mass detector.

Since, several methods have been used for the determination of water in solvents such as azeotropic distillation, drying in the oven, using light reflectance infrared spectroscopy, fluorescence and Karl Fischer method[3]. These methods have some disadvantages like slow response, low accuracy, long time process, toxic substances and complexity.

In this study, trace amounts of water in organic solvents have been determined using the reaction of water with calcium carbide. Released acetylene from this reaction can be extracted using headspace extraction method and analyzed by gas chromatography.





The effect of parameters such as temperature, incorporation rate, sample size and the effect of salt on the prepared samples were investigated. Optimal extraction conditions of acetylene ware prepared at a temperature of 39 ˚C, rotation rate 8 r/s. With adding 0.07 mg calcium carbide, and adding salt had no effect on release of acetylene by adding carbide to the solvent content. Standard deviation 12% and detection limit 56 mg/lit of this method are respectively.

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Determination of triazine pesticides residue in edible oils using air-assisted liquid-liquid microextraction followed by gas chromatography-flame ionization detection

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The increasing application of pesticides for agricultural purpose was caused a noticeable pollution of the environment, and is threat to health [1]. So the use of pesticides to control pests is clearly inevitable. These chemicals can reach planet tissues, leaving residues that can be detected in products such as food of animal origin, oils, cereals, and oil seeds [2]. One of the best new generations of pesticides recently introduced in viticulture practice is the triazine class. These pesticides are widely used for a variety of fruits, vegetables and grain crops. The common determination method for pesticides in edible oils is gas chromatography (GC) due to its high separation efficiency and variety of selective detection methods. Although many technological breakthroughs in analytical methodology and instrumentation have been witnessed in the past few decades, a sample preparation step is commonly involved before instrumental analysis especially for those samples which contain high amount of fat or oil. Due to their inherent complexity of matrix, the analysis of chemical contaminants with low concentrations is yet a challenging issue even with advanced hyphenated techniques based on mass spectrometry. In the present study, a rapid, simple, and highly efficient sample preparation method based on air-assisted liquid-liquid microextraction followed by gas chromatography-flame ionization detection was developed for extraction, preconcentration, and determination of five triazine pesticides (ametrine, terbutryn, and atrazine) in edible oils. Initially, the oil samples were diluted with hexane and a few µL of a less soluble organic solvent (extraction solvent) in hexane was added. In order to form fine and dispersed extraction solvent droplets, the mixture of sample solution and extraction solvent is repeatedly aspirated and dispersed with a syringe. Under optimum extraction conditions, the method showed low limits of detection and quantification between 2.4-5.2 and 8-17 µg L^{-1}, respectively. Enrichment factors and extraction recoveries were in the ranges of 74-78 and 74-78 %, respectively. Finally edible oil samples were successfully analyzed using the proposed method and atrazine was found in grapeseed oil.

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Determination of ultra trace amount of Allopurinol by adsorptive cathodic stripping voltammetry using copper (II) as an intermediate

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Abstract

In this work, a simple and sensitive electroanalytical method was developed for the determination of allopurinol (ALO) by adsorptive cathodic stripping voltammetry (ADSV) using Cu(II) as a suitable probe. The complex of copper(II) with ALO was accumulated at the surface of a hanging mercury drop electrode at −0.08V for 60 s. Then, the preconcentrated complex was reduced and the peak current was measured using square wave voltammetry (SWV). The optimization of experimental variables was conducted by experimental design and support vector machine (SVM) modeling. The model was used to find optimized values for the factors such as pH, Cu(II) concentration and accumulation potential. Under the optimized conditions, the peak current at −0.250V is proportional to the concentration of ALO over the range of 17.0–110.0 nmol L^{-1} with a detection limit of 0.43 nmol L^{-1}. The influence of potential interfering substances on the determination of ALO was examined. The method was successfully applied to determination of ALO in plasma and pharmaceutical samples. Recently, an electrochemical method for the determination of AlO in pharmaceutical formulation and dog urine was found based on adsorptive stripping voltammetry [1]. However,the reported method has not been applied for analysis of ALO in plasma samples due to its negative peak potential (−0.90 V vs. Ag/AgCl electrode), whereas the peak potential of the proposed method is shifted to more positive value.

Keywords: Support vector machine, Allopurinol, Adsorptive cathodic stripping voltammetry.

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Determination of ultra trace amount of Allopurinol by adsorptive cathodic stripping voltammetry using copper (II) as an intermediate

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Abstract

In this work, a simple and sensitive electroanalytical method was developed for the determination of allopurinol (ALO) by adsorptive cathodic stripping voltammetry (ADSV) using Cu(II) as a suitable probe. The complex of copper(II) with ALO was accumulated at the surface of a hanging mercury drop electrode at −0.08V for 60 s. Then, the preconcentrated complex was reduced and the peak current was measured using square wave voltammetry (SWV). The optimization of experimental variables was conducted by experimental design and support vector machine (SVM) modeling. The model was used to find optimized values for the factors such as pH, Cu(II) concentration and accumulation potential. Under the optimized conditions, the peak current at −0.250V is proportional to the concentration of ALO over the range of 17.0–110.0 nmol L^{-1} with a detection limit of 0.43 nmol L^{-1}. The influence of potential interfering substances on the determination of ALO was examined. The method was successfully applied to determination of ALO in plasma and pharmaceutical samples. Recently, an electrochemical method for the determination of AlO in pharmaceutical formulation and dog urine was found based on adsorptive stripping voltammetry [1]. However,the reported method has not been applied for analysis of ALO in plasma samples due to its negative peak potential (−0.90 V vs. Ag/AgCl electrode), whereas the peak potential of the proposed method is shifted to more positive value.

Keywords: Support vector machine, Allopurinol, Adsorptive cathodic stripping voltammetry.

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Determination of ultra-trace amounts of tellurium ions using ultrasound-assisted microextraction based on task-specific ionic liquid

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The aim of the present work is to combine the advantages of ionic liquids, ultrasounds and liquid phase microextraction (LPME), in order to develop a fast and selective LPME method based on task-specific ionic liquids for the preconcentration of metal ions. The covalent tethering of a functional group to one or both of the ions of an otherwise ordinary ionic liquid can imbue the resulting salt with a capacity to interact with dissolved substrates in specific ways. These low melting salts, called task-specific ionic liquids (TSILs), are finding an increasing number of applications in synthesis, separations, catalysis, and electrochemistry [1]. Metal ions tend to stay in the aqueous phase because they are hydrated. Therefore, in order to remove metal ions from the aqueous phase into hydrophobic ionic liquid, analytes are usually needed to form complexes thereby increasing the metal's hydrophobicity [2]. A TSIL acts as the hydrophobic solvent and the chelating agent at the same time reducing the chance for IL loss during the aqueous phase. The TSIL used in the present work, was an imidazolium-based IL with a thiol-functionalized anion and a thiourea-functionalized cathion. In this method, tellurium ions were extracted into the fine droplets of the TSIL after chelate formation with its function groups. The fine droplets were made and dispersed as a cloud in the aqueous sample with the help of ultrasonic waves. Ultrasonic radiation is a powerful means for acceleration of various steps in analytical procedure for both solid and liquid samples [3]. This type of energy has also great help in liquid-liquid extraction (ultrasound-assisted LLE) because it facilitates the emulsification phenomenon and accelerates the mass-transfer process between two immiscible phases. Several variable factors that influence the extraction and complex formation, such as pH, TSIL amount, sonication time, centrifugation time and salt addition were optimized. The proposed method was successfully applied to the extraction and preconcentration of tellurium from real samples prior to electro-thermal atomic absorption spectroscopy. According to the obtained results, it was concluded that the proposed method is rapid, simple, sensitive, selective, low cost, volatile organic solvent-free, and efficient for the separation of trace amounts of tellurium ions.

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Determination of venlafaxine in urine and plasma by combining the headspace solid-phase microextraction with electrospray ionization ion mobility spectrometry

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Abstract

In this research, headspace solid phase microextraction technique coupled with electrospray ionization ion mobility spectrometry (ESI-IMS), for the first time. The proposed method has been applied for the extraction and determination of venlafaxine in the biological samples such as urine and plasma. A desorption chamber, as interface, was constructed for online connection of SPME technique to ESI-IMS. Analyte was desorbed by electrospray solvent and entered into the electrospray needle. The solid-phase microextraction (SPME) fiber coated with polypyrrole/sol–gel composite was prepared using a three-electrode electrochemical system and directly deposited on a stainless steel wire by applying a constant potential (1.2 V for 1000 s) [1]. The effect of different parameters including extraction time, temperature, pH and ionic strength were investigated and optimized. Under the optimized conditions, calibration curves were plotted indicating the linear dynamic range of 1-50, 1-85, and 20-1000 μg/L, for standard water, spiked urine and spiked plasma samples, respectively. The relative standard deviation for the drug was less than 11% and the detection limits 0.5, 0.6, 10 μg/L, was obtained for the drug extracted from water, spiked urine and spiked plasma, respectively. The relative recoveries obtained were 80% and 93% for spiked urine and plasma samples, respectively. Finally, some real samples of urine and plasma were analyzed and the obtained results reveal the capability of the proposed method for venlafaxine analysis in biological matrices.

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Determination of Vitamin B_6 using an optimized novel TCPO-Indolizine-H_2O_2 chemiluminescence system

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Abstract

Indolizine derivatives have been used as a key scaffold in the pharmaceutical industry due to the broad spectrum of biological activities associated with this privileged structure. [1]. The chemiluminescence detection has been successfully applied in analytical chemistry because it possesses the most common advantages such as the relatively simple instrumentation, the very low detection limit and wide dynamic range. Indolizine derivatives are a great interest as fluorescent emitters for peroxyoxalate chemiluminescence. Reaction of peroxyoxalates such as bis-(2,4,6-trichloro-phenyl) oxalate (TCPO) with H_2O_2 can transfer energy to fluorescer via formation of dioxetanedione intermediate [2]. Four indolizine derivatives used as the novel fluorescers in the chemiluminescence systems in this study. The relationship between the chemiluminescence intensity and concentrations of fluorescer, peroxyoxalate, sodium salicylate and hydrogen peroxide was investigated. The optimum conditions were obtained for four fluorescers and it was found that the indolizine can be used as an efficient green fluorescent emitter. Vitamin B_6 (Pyridoxine) deficiency may lead to sideroblastic anaemia, dermatitis, cheilosis and neurological symptoms such as peripheral neuritis and convulsions [3]. Vitamin B_6 can induce a sharp decrease in the chemiluminescence intensity of TCPO-indolizine-hydrogen peroxide-sodium salicylate system. A simple, rapid and sensitive CL method for the determination of vitamin B_6 pharmaceutical product and plant foods has been developed. The Results showed a linear relationship between vitamin B_6 concentration and peroxyoxalate chemiluminescence intensity in the range of 7.0×10^{-8} –1.0×10^{-4}. Detection limit of 2.3×10^{-8} M and the relative standard deviation (RSD) < 3.8% was obtained.

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Determination of Zn^{2+} ion by Scanometry method, using Alizarin red s indicator

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Zinc is a heavy metal extensively used in industrial. It also has medicinal properties and widely used for hair and nail health. Because of this significance, multiple methods of measurement for this metal has been reported, such as spectrophotometric methods, various electrochemical techniques, such as differential pulse polarography and using optical sensors [1].

In this study a simple, fast, sensitive and inexpensive method for determination of Zn^{2+} ion by using Alizarin red S reagent described. The results show that Zn^{2+} ion is complexed with alizarin red S in *p*H 9 – 9.5 and made yellow product. In this method, the cells containing the sample solution were scanned with a scanner, and then the color of each cell was analyzed [3] with photoshop software. The software calculates the values of red, green and blue color. The cells have been built by creating holes in the Plexiglas® sheet, using laser. The results showed that the reaction has two linear range 0.005-0.100 and 0.5-25.0 μg L^{-1} of Zn^{2+} ion. The detection limit of the proposed method was 4.16 μg L^{-1}.The best reaction time and the solution pH for this system was obtained 40 min and 9-9.5 respectively. The method was successfully applied to the determination of the Zn^{2+} rat blood and zinc oxide tablet samples.

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Developing of random forest models for predicting of retention times of volatiles in rice

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A new set of 65 experimentally identified aroma volatiles of an Iranian rice cultivar by static headspace coupled to gas chromatography-mass spectrometry were used for the development of the quantitative structure-property relationship (QSPR) models. Data splitting was performed according to random selection that is created best splitting. The training set, having 48 members, was used for model development, while test set, having 17 members, was reserved to evaluate the prediction power of the generated models. The best-selected descriptors that appear in the models can encode different aspects of solute that influenced the solute-stationary phase interactions such as; polarizibilities, electronegativies, hydrophobicity and information about molecular shape, size, and atom distribution. Linear and nonlinear models were developed using multiple linear regressions (MLR) [1] and random forest (RF) [2,3] methods. The statistical results of MLR and RF models are R = 0.935, SE= 2.75 and R = 0.962, SE = 0.57, respectively. Robustness and reliability of the constructed RF model was evaluated by using the leave-many-out cross-validation method which produces the statistics of Q^2=0.86 and RMSE = 0.30. Chance correlation among data matrix was investigated by y-scrambling procedure, which gave the R^2=0.098. Furthermore, the chemical applicability domains of these models were determined via leverage approach. The results of developed models revealed that the statistical parameters of RF model are better than the MLR model, which reveals the capability of RF based on QSRR model in prediction of molecular retention of rice volatiles in SHS-GC-MS.

Keywords: Molecular descriptors, Quantitative structure-property relationship, Retention times, rice, Random forest

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Development and Application of liquid-phase microextraction based hollow fiber (HF-LPME) using high performance liquid chromatography coupled with a diode-array detector (HPLC/PDA) for the determination of sufentanil in water samples

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In this study, development and application of liquid-phase microextraction based hollow fiber (HF-LPME) using high performance liquid chromatography coupled with a diode-array detector (HPLC/PDA) for the determination of sufentanil in water samples was investigated. Sufentanil (N-{4-(methoxymethyl)-1-[2-(2-thienyl))ethyl]-4-piperidyl}-propionanilide) is a synthetic opioid analgesic widely used in clinical anesthesia and analgesia. Sufentanil is the most potent of the synthetic opioids and it is about five to ten times stronger than fentanyl [1]. HF-LPME, introduced by Pedersen-Bjergaard and Rasmussen in 1999, has been widely used in the last decade [2]. HF-LPME technique has many attractive advantages. First, unlike classical liquid–liquid extraction, HF-LPME uses very little organic solvent which is placed in a hollow fiber, and then the analytes in the aqueous sample can be extracted into the organic solvent (acceptor phase). Second, the hollow fiber also plays a role as a filter, because large molecules cannot permeate through the pore in the hollow fiber. Third, HF-LPME device is simple and cost-efficient [3]. The main factors influencing the extraction efficiency of HF-LPME including the extraction time, temperature and speed of stirrer were investigated and optimized. The mobile phase consisted of acetonitrile–phosphate buffer (38:62, v/v) at pH 3 and under isocratic conditions. The flow rate of the mobile phase was 1.0 mL/min. The PDA detector was set at the wavelength 237 nm. The calibration curve was linear over the range 0.05–0.5 mg/L, with the correlation coefficients (R^2)>0.99. The limits of detection (LOD) and limits of quantification (LOQ) of the method were 26 and 78 µg/L, respectively. Good reproducibility was obtained with the relative standard deviations (RSD) within-day 3.1% and between-day 5.7% (n=5). The method was successfully applied to determination of a trace amount of sufentanil in the water samples.

Keywords: Sufentanil, Liquid-phase microextraction, Hollow fiber, HPLC/PDA

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Development and application of solvent-assisted dispersive solid phase extraction for separation, preconcentration and spectrophotometric determination of trace amounts of Hg (II) in natural water samples

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The monitoring of mercury (Hg) in natural waters is very important due to its high toxicity and very high bioaccumulation factor (up to 10^6) in the food chain [1]. Its measurement is a challenging task by its trace level presence in complicated matrices and the interference of other elements. Therefore, in order to determine the trace level mercury ions and to improve the detection sensitivities, high efficiency preconcentration methods and high selective and sensitive analysis techniques are urgently required [2]. Recently, our group introduced a new extraction method that was named solvent-assisted dispersive solid phase extraction (SADSPE) [3]. The method was based on the dispersion of the sorbent into the sample to maximize the contact surface. The advantages of SADSPE method are simplicity of operation, rapidity, low cost, good recovery, and enrichment factor.

In this research, the SADSPE method was developed for separation, preconcentration and spectrophotometric determination of trace amounts of Hg (II) in natural water samples. Dithizone was used as complexing agent. The method was based on the dispersion of benzophenone (sorbent) into the aqueous sample. Thereby, a cloudy solution formed. This cloudy state resulted from the dispersion of fine particles of benzophenone in the bulk aqueous sample. Then, the mixture was centrifuged. Accordingly, the dispersed fine particles of benzophenone were sedimented. The aqueous phase was then separated completely by a syringe. Later, the sedimented phase was dissolved by adding ethanol. The absorption of the resultant solution was measured by spectrophotometer. Some key parameters for SADSPE, such as sorbent selection and amount, type and volume of dispersive solvent, pH, chelating agent concentration, and salt concentration, were investigated. Under the most favorable conditions, good limit of detection (as low as 4.0 μg L^{-1}) and repeatability of extraction (RSD below 3.6%, n=10) was obtained. The calibration curve was linear within the range of 15–250 μg L^{-1} of Hg (II) with $R^2 = 0.9957$. Finally, the method was successfully applied for determination of low levels of Hg (II) ions in various water samples.

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Many phenolic compounds and their conversion products are hazardous to human health. Therefore the US Environmental Protection Agency regulated 11 phenols among the main environmental pollutants [1]. In addition to being highly toxic and potential carcinogen, these compound causes, even at very low concentration, adverse effects on the central nervous system, cardiovascular system, lungs, kidney, and liver [2]. So finding a rapid and simple analytical method as well as high accuracy in the identification and quantitation of the phenolic compounds is necessary to monitor and regulate their usage for ensuring food quality and safety. Several sample preparation methods have been developed for the extraction and preconcentration of phenols such as liquid–liquid extraction (LLE), solid-phase extraction, supercritical-fluid extraction, microwave-assisted extraction, and dispersive liquid–liquid microextraction prior to their determination by analytical instruments.

Recently a new sample preparation technique namely air–assisted liquid-liquid microextraction (AALLME) has been introduced by Farajzadeh [3]. In this study, AALLME method combined with LLE has been developed as a new approach for simultaneous derivatization, extraction and preconcentration of some phenolic compounds in vegetable oils prior to GC-FID and GC-MS determinations. The proposed method consists of two steps: (i) extraction of phenols from vegetable oils by LLE; and (ii) simultaneous derivatization and enrichment of the phenols by AALLME. In the first step, phenolic compounds are extracted into NaOH (0.5 M) followed by AALLME (second step). Under the optimum conditions, the method showed wide linear ranges (2.7-10000 µg L^{-1}). Enrichment factors were in the ranges of 568 to 1128. Relative standard deviations for the extraction of 200 µg L^{-1} of each selected phenols were between 1.7- 4.3%. The method was successfully applied to analytes determination in olive oil, sunflower oil, and kolza oil.

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Development and validation of rapid and sensitive gas chromatographyic method for the simultaneous derivatization, extraction and preconcentration of phenols from vegetable oils

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Many phenolic compounds and their conversion products are hazardous to human health. Therefore the US Environmental Protection Agency regulated 11 phenols among the main environmental pollutants [1]. In addition to being highly toxic and potential carcinogen, these compound causes, even at very low concentration, adverse effects on the central nervous system, cardiovascular system, lungs, kidney, and liver [2]. So finding a rapid and simple analytical method as well as high accuracy in the identification and quantitation of the phenolic compounds is necessary to monitor and regulate their usage for ensuring food quality and safety. Several sample preparation methods have been developed for the extraction and preconcentration of phenols such as liquid–liquid extraction (LLE), solid-phase extraction, supercritical-fluid extraction, microwave-assisted extraction, and dispersive liquid–liquid microextraction prior to their determination by analytical instruments.

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Development of a Cloud Point Extraction and Preconcentration Method for Determination of Trace Amounts of Zinc by Flame Atomic Absorption Spectrometry

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Abstract

Cloud point extraction (CPE) is an attractive technique that reduces the consumption of and exposure to a solvent, disposal costs and extraction time [1]. Cloud point methodology has been used for the extraction and preconcentrations of metal ions after the formation of sparingly water soluble complexes. Compared with traditional solvent extraction, CPE uses low toxic, environmental friendly surfactants and avoids the use of large amounts of toxic and flammable organic solvents [2, 3]. In this work a new cloud point extraction (CPE) method has been developed for the preconcentration of trace zinc prior to its determination by flame atomic absorption spectrometry (FAAS). The analyte in the initial aqueous solution was complexed with Aurintricarboxylic acid (5, 5′-((3-carboxy-4-oxocyclohexa-2, 5-dienylidene) methylene) bis (2-hydroxybenzoic acid)) and Triton X-114 was added as surfactant. After phase separation, based on the cloud point separation of the mixture, the surfactant rich phase was diluted with 0.1mL of 1 mol L^{-1} HNO_3 in methanol and the analyte determined in the enriched solution by flame atomic absorption spectrometry. The main factors affecting CPE efficiency, such as pH of sample solution, concentration of ATA and Triton X-114 were investigated in detail. Under the optimum conditions the calibration graph using the preconcentration method was highly linear with a correlation coefficient of 0.9949 in range of 5-240 μgL^{-1}. The proposed method was applied successfully for determination of trace amounts of zinc in water samples.

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Development of a cloud-point extraction method for rhodium determination in environmental samples without chelating agent

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The release of rhodium from Vehicle Exhaust Catalysts represents a source of highly dispersed environmental contamination, and this metal has been identified in dusts, soils and waters. Since the introduction of catalytic converters in modern automobiles, the determination of rhodium has received increased attention due to their release into the environment and allergic properties [1]. Although numerous research studies have been carried out on developing reliable analytical methods for accurate determination of traces of rhodium in different materials, their reliable determination are still a difficult task due to low environmental concentrations, and significant interaction with sample matrix components. In order to solve these problems, preconcentration-separation procedures have been continuously done by the several researchers. Separation methods based on cloud point extraction (CPE) are practical application of surfactants in analytical chemistry and have become as an alternative to liquid-liquid extraction [2]. In this study, a sensitive CPE procedure for the preconcentration of trace rhodium prior to its determination by flame atomic absorption spectrometry (FAAS) has been developed. Aliquots of 10.0 mL water samples or standard solutions were pipetted to centrifuge tubes. Then, 1 mL of 0.2 mol L^{-1} phosphate buffer (pH 9), 1.0 mL of 0.5% Triton X-114 and 1 mL NaCl 3.5% (w/v) were sequentially added and completely mixed with the sample or standard solutions. The centrifuge tubes containing the mixed solutions were heated in a thermostatic water bath at 45 $^{\circ}$C for 10 min. After cooled in ice bath, the surfactant-rich phase became viscous, and supernatant aqueous waste in the tubes was removed with a pipette. Then, 1.0 mL of 1.0 mol L^{-1} HNO_3 in ethanol was added to it. The final solution was aspirated directly into the flame of AAS against the blank. The main factors affecting CPE efficiency, such as pH of sample solution, concentration of Triton X-114, equilibration temperature and time, were investigated in detail. Under the optimal conditions, the calibration curve was linear in the range of 20.0 ng mL^{-1}– 2.5 μg $mL^{-1}$of rhodium with $R^2 = 0.995$ (n = 8). Based on three times the standard deviation of the blank ($3S_b$), detection limit was 1.8 ng mL^{-1}. Eight replicate determination of 0.5 μg mL^{-1} rhodium gave a relative standard deviation 1.9%. The proposed method has been applied for determination of trace amount of rhodium in biological and water samples with satisfactory results.

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Development of a solid-based disperser liquid-liquid microextraction method for preconcentration of phthalate esters and di-(2-ethylhexyl) adipate followed by gas chromatography-flame ionization detection and gas chromatography-mass spectrometry

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Current trends in analytical chemistry are extensively focused on introduction of new technological developments and, especially, miniaturization, simplification and automation of the whole analytical procedure. So, microextraction techniques have quickly grabbed the attention of researchers and have been developed as alternatives for classic extraction and sample preparation procedures. These methods use only several µL of organic solvent and thus comply with the requirements of green analytical chemistry. The introduction of solid-phase microextraction by Pawliszyn and co-workers [1] initiated interest in microextraction techniques. Since then, several other techniques have been developed. One of the latest popular methods is dispersive liquid-liquid microextraction (DLLME) that was proposed by Assadi et al. [2] in 2006. The purpose of this work is to develop a new version of DLLME, named solid-based disperser liquid-liquid microextraction (SB-DLLME), in which the disperser solvent is replaced by a sugar cube as a solid disperser and the consumption of toxic organic solvents is minimized to propose a green analytical method. In this method, 25 µL 1,2-dibromoethane as extraction solvent was added onto a 0.5-g sugar cube. Then, it rapidly penetrated into the pores of the sugar cube. The cube was left into the sample solution. After manual shaking, the sugar dissolved in the aqueous sample of analytes gradually and played the role of a disperser agent for the dispersion of extraction solvent into the aqueous sample. After formation of a cloudy solution and extraction of the analytes into the fine droplets of extractant, the mixture was centrifuged for 3 min at 5000 rpm and the fine droplets of 1,2-dibromoethane were sedimented at the bottom of the conical test tube. Finally, 1 µL of the collected organic phase was injected into a GC–FID or GC–MS for analysis. Parameters affecting performance of the method were investigated and optimized. Under the optimized conditions, the proposed method showed wide linear range (0.5–2000 µg L^{-1}), good precision (relative standard deviation < 4.5 %) and high enrichment factors (266-556). Moreover, limits of detection were in the ranges of 0.09-0.25 µg L^{-1}. No need for a disperser solvent and higher enrichment factors in comparison with those of conventional DLLME and low cost and short sample preparation time are other advantages of the proposed method.

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Development of an electrochemical method for the determination of antioxidant activity

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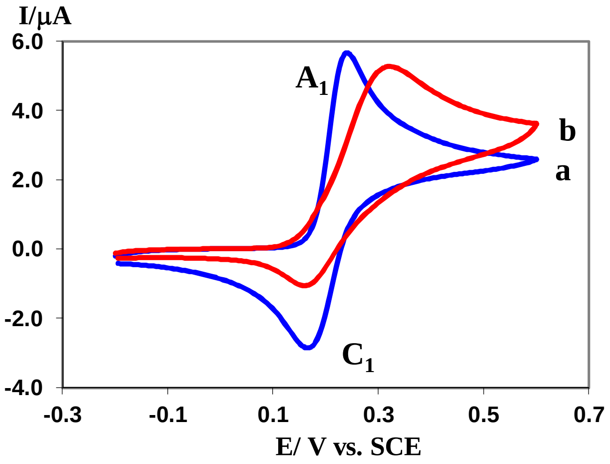
Oxidation is one of the most important processes of food deterioration since it may affect food safety, colour, flavour and texture. Antioxidants may protect food quality by preventing oxidative deterioration. Nowadays, there is considerable interest in finding out about antioxidants [1-2]. It is known that polyphenols are involved in reducing the risk of diseases associated with oxidative stress. The aim of present work is to investigate the applicability of cyclic voltammetry on the determination of antioxidant activity of the phenolic compounds. A new method, rapid and simple to apply, has been developed to measure antioxidant activity. It is based on the electrochemical oxidation of catechols in presence of green tea and black tea and gives reliable results. The proposed method is very easy in operation, without special needs for sample pretreatment. The obtained results are well reproducible and accurate. It has been compared with the total antioxidant status test and gives similar but more repeatable results [3]. This method has been applied to green tea and black tea. It has been confirmed that the antioxidant activity of these products is highly correlated with their polyphenolic content. Cyclic voltammetry at a glassy carbon electrode was employed to elucidate the antioxidant capacity of tested teas. This study confirms the usefulness and complementarity of rapid electrochemical techniques in elucidation of the antioxidant potential of food products.



Cyclic voltammograms of 1 mM catechol: (a) in the absence; (b) in the presence of green tea, at a glassy carbon electrode, in phosphate buffer solution (c = 0.2 M, pH 7.0); scan rate: 10 mV s^{-1}; t = 25 ± 1 °C.

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Development of dispersive liquid-liquid microextraction technique using ternary solvent mixture followed by heating for the rapid and sensitive analysis of phthalate esters and di(2-ethylhexyl) adipate in foodstuffs

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Phthalate esters (PEs), along with di(2-ethylhexyl) adipate (DEHA) are chemical compounds that are widely used as plasticizers because they improve the softness and flexibility of plastics. PEs and DEHA are not chemically bound to plastics, and therefore they can be released from the plastic into the environment [1]. Due to the widespread use of the mentioned compounds, they are considered as ubiquitous environmental pollutants. In order to determine trace levels of PEs and DEHA in food samples, an extraction and preconcentration step is often required prior to their analysis by gas chromatography (GC) or liquid chromatography [2]. Several sample preparation methods have been developed for the analysis of target analytes such as liquid–liquid extraction, cloud point extraction, solid-phase extraction, liquid-phase microextraction, and dispersive liquid–liquid microextraction (DLLME) [3].

In this study, DLLME technique using a ternary solvent mixture is reported for the first time. Selecting a suitable extraction solvent is critical in all solvent extraction methods. The polarities of the analytes selected in this study and most other cases are not the same. Therefore a single extraction solvent with a known polarity cannot extract all analytes efficiently. In order to improve the extraction ability of the extraction solvent towards all interested components, three solvents with different polarities (dichloromethane, $\varepsilon = 8.93$; chloroform, $\varepsilon = 4.81$; and carbon tetrachloride, $\varepsilon = 2.24$) were chosen and solvent selectivity triangle method was used to select an optimal ternary solvent mixture. After centrifuging, the obtained sedimented phase was heated in a water bath in the presence of a few µL dimethylformamide to evaporate the main portion of the extraction solvents in order to obtain further concentration. Finally, 2 µL of the remained phase was injected into the GC–FID system. Under the optimum conditions, the method showed wide linear ranges and low limits of detection and quantification between 0.03-0.15 and 0.09-0.55 µg L^{-1}, respectively. Enrichment factors and extraction recoveries were in the ranges of 980 to 4500 and 20 to 90%, respectively. The method was successfully applied in determination of the target analytes in mineral water, soda, lemon juice, vinegar, dough, and yogurt packed in plastic packages.

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Development of molecularly imprinted polymers via pre-grafting functionalized group based on magnetic nanoparticles for selective determination of 4-nitrophenol in aqueous media

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In this study, a novel approach was developed for preparation of magnetic imprinted polymers by pre grafted functional groups on the surface of magnetic substrate. The amino functionalized magnetic surface provided the proper association to retain template molecules on the surface of nanoparticles. Therefore, the amount of formed imprinting sites was increased for target analyte rather than the conventional methods which were interacted between the functional monomers and template molecules[1]. The resulted selective sorbent was synthesized by 4-nitrophenol (4-NP) and ethylene glycol dimethacrylate (EGDMA) as template and cross-linker agent, respectively. It exhibited proper magnetic behavior and were easily separated from the aqueous solution by applying an external magnetic field[2]. In order to improvement of response for target analyte, the various parameters such as pH effect, type and volume of removal solvent were optimized. The prepared magnetic molecularly imprinted polymer (MMIP) showed high adsorption capacity and fast kinetic binding for the template molecule. The adsorption equilibrium of MMIP was achieved within 90 min and the maximum adsorption capacity reached 129.1 mg g^{-1}. The adsorption curve of MMIP was also fitted with the Freundlich isotherm equation. The experiment exhibited a linear range of 10-3000 µg L^{-1} for 4-NP with the correlation coefficient (R^2) of 0.997. The results of sample analysis confirms the applicability of the proposed MMIP to quantitative analysis of 4-NP in the aqueous media.

Keywords: Molecularly imprinted polymer, 4-nitrophenol, Magnetic nanoparticles, Aqueous media

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Diagnosis of heart defect by infrared spectrometric analysis of serum samples

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Heart defect is main cause of death in Iran, according to the report of ministry of health. Early diagnosis of heart disease may help in prevention of heart attack and reduce the rate of death due to these types of health problems. Currently there are many methods for early diagnosis of heart disease while some of these methods are tedious, invasive and expensive and some would provide low accuracy [1]. However, novel comfortable and more accurate methods are required. In this work an infrared spectroscopic method has been proposed for diagnosis of heart disease by quantitative determination of choline as an important correlated biochemical in blood samples. Choline is a biomarker which can be determined and evaluated as indicator of normal biology, pathogenic process, or pharmacological response to therapeutic intervention [2]. ATR-FTIR spectrometry was used for determination of choline in 82 blood serum samples. Preprocessing methods such as standard normal variate (SNV), multiplicative scatter correction (MSC) and orthogonal signal correction (OSC) were performed and the calibration model was formed based on partial least squares. Effect of three processing methods on final quantitative evaluation of spectral data was compared. Root mean squared error of prediction (RMSEP) for the MSC-PLS model, SNV-PLS model and OSC-PLS model were 62.89%, 42.27% and 0.25% respectively. The most robust calibration model was obtained by OSC-PLS. Data classification was also conducted to diagnose the illness pattern. Obtained results demonstrate that ATR-FTIR spectrometry is feasible as a reliable tool for rapid and simple preliminary diagnosis of heart defects.

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Digital Videometric Analysis Combined with H-point Standard Addition Method for Kinetic Determination of Ascorbic acid and Cysteine

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In recent years, an increasing interest has been focused on the application of image analysis to different research areas.In this method, not only experimental data, but also images of supporting substrate or sample solutions (e.g., the colorfulcomplexes) can be saved in a computer followed by analysis with upcoming more powerful programs. With this approach, the cost and time of analysis could be reduced dramatically compared to UV-Vis spectroscopy [1]. Accordingly, in our group we have employed video analysis combined with chemometrics methods for simultaneous kinetic determinations. In the present work, we analyzed the digital videometric kinetic data by classical H-point standard addition method [2] as an alternative to UV-Vis spectroscopy. As analytical application, the method was employed for simultaneous determination of L-Cysteine and ascorbic acid. The analytes reduced Fe (III) to Fe (II) and the latter produced a colored complex in the presence of phenanthroline (phen). So, the formation of colorful Fe (II)-Phen complex was monitored by a digital video over time.

Videos of each solution were recorded and using home-made software in MATLAB, there were converted to images and then to RGB color values. Plotting each color values of R, G or B as function of time resembled the kinetic profile of the reaction system. The data was then used to derive individual calibration curves (fixed-time kinetic method) as well as H-point standard addition method curves.

Under the optimum conditions the calibration curves were linear in the ranges (0.75 – 7.2 ppm) for Cys and (0.45-7.2ppm) for ASC.

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Direct electron transfer of cytochrome c at graphen modified glassy carbon electrode and its application to reduction of Hydrogen peroxide

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The direct electron transfer between redox proteins (particularly redox enzymes, which are nonconductive) and the electrode surface has been extensively studied because of its potential application in the investigation of bio-molecules. Among the various redox proteins, Cytochrome c (Cyt c) is an important, stable and water-soluble heme-containing metallorotein, which exists at inner mitochondrial membrane [1]. It is an efficient biological electron transporter and it is important to study the electrochemical behavior of it. graphene is an ideal candidate for accommodating proteins and facilitating electron transfer between protein and electrode surface due to its unique surface properties such as one-atom thickness, higher surface area which can afford a higher loading capacity for protein, excellent conductivity can facilitate the electrons transfer from the biomolecules and irreversible protein adsorption ability due to strong hydrophobic interaction [2]. Hydrogen peroxide (H_2O_2) determination is of great importance because of its essential role in food, industry, biology, pharmaceutical, and environmental research [3]. In this work, the dispersed graphene was immobilized on the surface of glassy carbon (GC) electrode to form a graphene modified electrode. Then, Cyt c was immobilized on the surface of graphene modified electrode. The direct electron transfer of the immobilized Cyt c was investigated and electrocatalytic activity of this electrod toward reduction of H_2O_2 was investigated by cyclic voltammetry and chronoamperometry.

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Direct Electron Transfer of Cytochrome c on Silver Nanoparticles Modified Carbon Paste Electrode

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Abstract

In recent years, direct electrochemistry of metalloproteins and metalloenzymes is a subject of immense interest because of its potential application in the study of the redox and electron transfer (ET) properties of biomolecules [1,2].Study on the electrochemical behavior of heme proteins is important for the fundamental understanding of their biological activity.

Cytochrome c has been the subject of extensive studies because of its essential function as the electron carrier respiration systems of higher organisms.Nanoparticles have been prepared by a chemical reduction method. The nanosized silver colloids have been characterized using UV-Vis spectroscopy.

Colloidal silver modified carbon paste electrodes were prepared by mixing 43-nm-diameter colloidal Ag particles with carbon paste. The modified electrodes displayed a low charging current and a favorable electrochemical response of hexacyanoferrate (III).

The direct electrochemical behavior of a horse-heart cytochrome c (cyt.c) adsorbed on this electrode surface is described. It showed a surface-controlled electrode process .

Direct electrochemistry of cytochrome c in this paste electrode was easily achieved, and a pair of well-defined quasireversible redox peaks of a heme Fe (III)/Fe (II) couple appeared in pH 7.0 and10.0 mM phosphate buffer solution (PBS). The fabricated modified bioelectrode showed good electrocatalytic ability for reduction of H_2O_2. The preparation process of the proposed biosensor was convenient, and the resulting biosensor showed high sensitivity, low detection limit, and good stability.

Keywords: Cytochrome c, electrochemistry, Silver Nanoparticles

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Directly suspended droplet microextraction technique for determination of trace amounts of iron and copper in vegetable, fruit and environmental samples

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Abstract

Heavy metal composition of foods is of interest because of their essential or toxic nature. For example, iron, zinc, copper, chromium, cobalt and manganese are essential, while lead, cadmium, nickel and mercury are toxic at certain levels. Iron and copper are essential for human life at low concentrations, but they can also be toxic at high concentrations [1]. However due to matrix effect and low concentration of metal ions, efficient separation and preconcentration steps are essential prior to analytical measurements [2]. In the present study a miniaturized liquid-phase extraction procedure based on directly suspended droplet microextraction for determination of trace amounts of iron and copper in vegetable, fruit and environmental samples was developed. The method is based on the extraction of the iron and copper complexes with 2-mercaptopyridine n- oxide onto a micro drop of methyl isobutyl ketone (MIBK) as extraction solvent and subsequent flame atomic spectrometric determination. The factors influencing the complex formation and extraction were optimized. Under optimum conditions, enrichment factors of ~ 25 are obtained from only 6.5 mL of aqueous phase for iron and copper respectively. The analytical curves were linear between 30-800 $\mu g\ L^{-1}$ and 20- 1200 $\mu g\ L^{-1}$ for iron and copper, respectively. Based on three standard deviation of the blank, the detection limits were 3.76 $\mu g\ L^{-1}$ and 1.84 $\mu g\ L^{-1}$ for iron and copper respectively.

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Discrete Fourier Transform as Applicable Technique for Comparative investigation of some Physical Properties of Nanocarons for Chemical Immobilization on Glass Support during studying mode-locking process in Nd:YAG laser at 1064 nm

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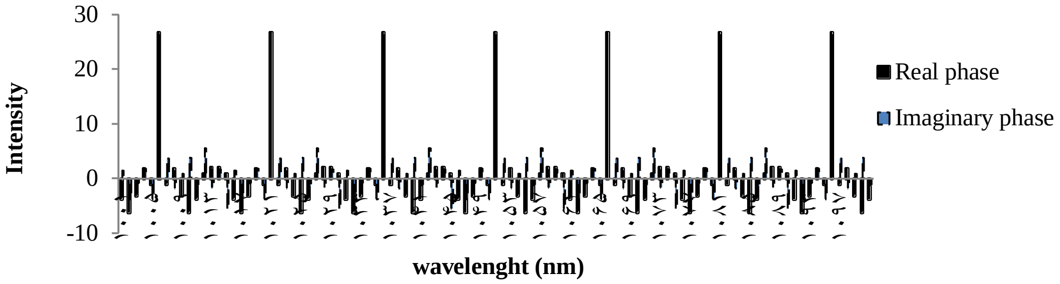
Mode-locking is a technique in optics by which a laser can be made to produce pulses of light of extremely short duration, on the order of picoseconds (10^{-12} s) or femtoseconds (10^{-15} s) [1]. This process is occurred during the interaction between laser and organic/inorganic materials. In this study, “Discrete Fourier Transform” (DFT) was considered as suitable technique during evaluation of the mode-locking properties of various forms activated nanocarbons. For this purpose, different forms of nanocarbons such as graphene and carbon nanodots were activated to via refluxing in H_2SO_4/HNO_3 acid (0.1 M, 1:1, v/v), to form carboxylic function groups (-COOH, ~5.0%), followed by homogeneous and chemically immobilization on the surface of glass supports via formation of ester. The basis of the DFT technique is to induce a fixed phase relationship between the longitudinal modes of the laser's resonant cavity, which the laser is then said to be phase-locked or mode-locked. To test this phenomenon, experimentally, a nanocarbons-modified glass support is situated into a Nd:YAG pulsed laser resonator and for experimentally measuring and evaluating pulses of this laser. The properties of the laser outputs for different samples with different absorption spectra can be theoretically predicted by the DFT algorithm. For this purpose, using an oscilloscope the trace (intensity-time diagram) related to the intensity of the mode-locked laser is measured. This trace is therefore considered as *I(t)* function (time domain) that is converted to *I’(v)* function (frequency dopamine, Fig. 1) during using DFT algorithm via separation of real and imaginary phases. According to the results, various parameters such as kind and amount of nanocarbons, hydrophobicity of carbon nanostructures, homogeneity and chemically immobilization of nanocarbons on glass support, and finally basal/edge planes in nanocarbon’s matrix are considered as the most effective factors during evaluation of mode-locking process with laser at 1064 nm. This technique simply predicts the correlation between some surface properties of nanocarbons and physical properties such as mode-locked laser without need to adopt any sophisticated surface analytical techniques.



Frequency domain showing real and imaginary phases using DFT algorithm

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Discrimination of chronic kidney disease stages by chemometric classification of infrared spectroscopic data obtained from serum sample

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Infrared (IR) spectroscopic analysis of biofluids results in a fingerprint of their biochemical characteristics in the form of an spectrum; this has given rise to the new field of “Biospectroscopy”. Current work describes the application of Mid-IR spectroscopy tools and chemometric data processing consisting of an experimental considerations include optimization of specimen preparation, objective acquisition of a sufficient number of spectra, linking of the derived spectra with serum architecture, and finally quadratic discriminant analysis (QDA) for multistage analysis of chronic kidney disease (CKD). Spectroscopic studies were performed in 600-4000 cm^{-1} spectral region by ATR sampling techniques. IR spectroscopy generates complex data sets and analyses are best when initially based on a multivariate approach QDA modified by variable selection strategy. Different prediction models were developed using QDA combined with successive projection algorithm (SPA) method and genetic algorithm (GA) as wavelength selection strategies. Results showed 96.7% and 93.3% of accuracy for SPA-QDA and GA-QDA models respectively. It was concluded that analysis of blood serum by ATR-FTIR spectroscopy and QDA-GA chemometric technique would be a reliable approach for detection of CKD.

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Dispersive and ultrasound assisted determination of trace amounts of gold with ionic liquid

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Abstract

Gold is a dense, soft, shiny, malleable and ductile metal. It is one of the most ancient metals used in the arts and jewelry and it is of growing importance in technology because of its resistance to corrosion [1]. It commands a premium price because of its low abundance in nature. Gold is extensively used in various areas such as the electronic apparatus [2]. As a result of the low levels of gold in most environmental samples, a variety of preconcentration methods for gold have been developed such as solid phase extraction, cloud point extraction, coprecipitation and liquid–liquid extraction in order to meet the sensitivity requirements of different detection techniques. A new methodology was developed for the determination of ultra-trace levels of gold in water, human hair and certified reference material. Ultrasound assisted-dispersive liquid-liquid microextraction with 1-hexyl-3-methylimidazolium hexafluorophosphate as extracting solvent was used to preconcentrate the ion pair formed between $AuCl_4^-$ and $[C_{23}H_{42}]N^+$ in a microliter-range volume of 1-undecanol. After optimization, the enrichment factor obtained was 225. Moreover, the extraction efficiency was around >96%. The repeatability, expressed as relative standard deviation ranged from ±4.6% to ±5.3%. The instrumental detection limit and calibration range were obtained 1.5 and 7.5-90 ng L^{-1}.

Keyword: Gold, Ionic liquid, Ion pair, ultrasound assisted

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Dispersive liquid - liquid microextraction for determination of cadmium (II) in water samples by Flame atomic absorption spectrometry.

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Cadmium is widely used in industry, especially in electroplating pigments and the production of Ni–Cd batteries [1]. Flame atomic absorption spectrometry (FAAS) has been widely used for determination of trace quantities of cadmium. However, conventional FAAS has a detection limit, which is not low enough to determine cadmium at trace levels. In order to achieve accurate, sensitive and reliable results at trace levels; preconcentration and separation steps are needed prior to analyte determination by FAAS [2]. A novel approach for preconcentration and analysis of trace amount of cadmium from water samples was proposed by dispersive liquid–liquid microextraction (DLLME). Cadmium(II) was complexed with 2-(2-(2-aminophenyl) disulfonyl) benzenamine to form hydrophobic chelates and then extracted into the fine drops of extraction solvent dispersed in the aqueous sample by dispersive solvent. After extraction, the sedimented phase was analyzed by FAAS. Some important parameters affecting the DLLME were investigated. Under the optimized conditions: (extraction solvent: 50 µL of CCl_4; dispersive solvent: 0.6 mL of acetone ; pH=7 and without salt addition), the limits of detection for Cd(II) was 0.5 µg L^{-1} and the relative standard deviation (RSD) was 1.11 % (n = 5) with a preconcentration factor of 168.6 times. Two real water samples (Isfahan industrial park Wastewater -Toos industrial park Wastewater) spiked with cadmium was detected by the developed method, and the relative recoveries obtained for Cd(II) was 96 % and 94 % respectively.

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Dispersive liquid–liquid microextraction based on ionic liquid against high concentration of salt for preconcentration of Cr (VI) in various samples

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Abstract

In the presence of salt, ILs dissolves completely and the cloudy solution is not formed. Consequently, IL-based DLLME cannot be applied for extraction and preconcentration of analyte from samples containing high salt concentration [1]. The main aim of this paper is to study the applicability of proposed IL-based DLLME followed by FAAS equipped with a homemade microsample introduction system. This method can solve the problems associated with the limited application of the conventional IL-DLLME in these samples. In this procedure, the hydrophobic chelate of chromium with ammonium pyrrolidinedithiocarbamate (APDC) was extracted into the fine droplets of 1-hexyl- 3-methylimidazolium hexafluorophosphate ([Hmim][PF_6]), which was dispersed into the aqueous sample solution [2,3]. To decrease the solubility of IL-phase in brines, $NaPF_6$ was added to the sample solutions as an ion-pairing agent. Under the optimum conditions, the limit of detection (LOD) was 0.16 μg L^{-1} and RSD for 20 μg L^{-1} of Cr was 2.68 % (n=10), The proposed method was successfully applied for the determination of Cr(VI) in water samples and food additives. The accuracy of the method was evaluated by analysis in electrothermal atomic absorption spectrometry (ET-AAS). The results showed that the proposed work is a powerful sample preparation technique for saline samples and can be used instead of the traditional liquid–liquid extraction method. In addition, it opened a new perspective for the widening application of DLLME.

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Dispersive liquid–liquid microextraction based on ionic liquid against high concentration of salt for preconcentration of Cr (VI) in various samples

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Abstract

In the presence of salt, ILs dissolves completely and the cloudy solution is not formed. Consequently, IL-based DLLME cannot be applied for extraction and preconcentration of analyte from samples containing high salt concentration [1]. The main aim of this paper is to study the applicability of proposed IL-based DLLME followed by FAAS equipped with a homemade microsample introduction system. This method can solve the problems associated with the limited application of the conventional IL-DLLME in these samples. In this procedure, the hydrophobic chelate of chromium with ammonium pyrrolidinedithiocarbamate (APDC) was extracted into the fine droplets of 1-hexyl- 3-methylimidazolium hexafluorophosphate ([Hmim][PF_6]), which was dispersed into the aqueous sample solution [2,3]. To decrease the solubility of IL-phase in brines, $NaPF_6$ was added to the sample solutions as an ion-pairing agent. Under the optimum conditions, the limit of detection (LOD) was 0.16 μg L^{-1} and RSD for 20 μg L^{-1} of Cr was 2.68 % (n=10), The proposed method was successfully applied for the determination of Cr(VI) in water samples and food additives. The accuracy of the method was evaluated by analysis in electrothermal atomic absorption spectrometry (ET-AAS). The results showed that the proposed work is a powerful sample preparation technique for saline samples and can be used instead of the traditional liquid–liquid extraction method. In addition, it opened a new perspective for the widening application of DLLME.

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Dispersive liquid–liquid microextraction combined with microvolume UV–Vis spectrophotometry for Sensitive determination of methyl orange

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Dyes are widely used in industries such as the textile, pulp mill, paper production, food technology, printing, leather tanning, and plastics industries. Many dyes are toxic to some organisms, carcinogenic and mutagenic to humans. Methyl orange (MO) is an intensely colored azo dye compound which is widely used for dyeing and printing of textiles, pharmaceutical, food industries and as indicator. The presence of azo group (N=N) on MO and its low biodegradability makes it an issue of concern for environmental science. MO is regarded as allergy materials after contacting by skin, which causes shin eczema and even be poisonous if swallowed [1]. Therefore, the development of simple and sensitive analytical methods for accurate determination of trace amount of methyl orange is important for its environmental monitoring. Dispersive liquid-liquid microextraction (DLLME) is an attractive mode of liquid microextraction which has received much attention for sample pretreatment. This method has been applied for the separation and preconcentration of trace metal ions and organic pollutants in the different matrices [2,3]. In the present study a dispersive liquid-liquid microextraction (DLLME) method coupled to UV-Vis spectrophotometry is developed for the sensitive determination of methyl orange**.** The method is based on extraction of methyl orange with DLLME procedure using carbon tetrachloride (extraction solvent) containing methyltrioctyl ammonium chloride which provides counter ion and also acts as an disperser. After extraction, the phase separation is performed with a rapid centrifugation, and methyl orange is determined in the enriched phase by UV-Vis spectrophotometry. Under the optimum extraction condition, the method yields a linear calibration curve in the concentration range of 5 to 400 ng mL^{-1}, and the limits of detection was 1.53 ng mL^{-1} for target analytes. The relative standard deviation for ten replicate measurements of 50 and 300 ng mL^{-1} of methyl orange was 3.61 and 1.43%, respectively. The proposed method was successfully applied to determination of trace amounts of methyl orange in real samples with satisfactory results.

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Dispersive liquid–liquid microextraction coupled with flow injection atomic absorption for trace determination of lead in the presence of 2- [(2-mercaptophenylimino) methyl] phenol

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Abstract: Dispersive liquid-liquid microextraction (DLLME) [1] coupled with flow injection flame atomic absorption spectroscopy (FI-FAAS) have been developed for preconcentration and determination of trace amount Pb(II). The proposed approach used 2- [(2-mercaptophenylimino) methyl] phenol (MPMP) as chelating agent and chloroform and ethanol were selected as extraction and dispersive solvent, respectively. The Pb(II) ions were extracted in the form of complex. After injection of the extracting mixture to a solution of Pb(II), a cloudy mixture was observed. A quick centrifugation induces phase separation and thus the settling of rich phase. The Pb(II) content in the rich phase is measured by FI-FAAS. The main factors affecting the DLLME such as pH, MPMP concentration, extraction and disperser solvent type and volume, extraction time, were investigated. Under the optimum condition (pH=8, [MPMP] $=6\times10^{-3}$ mol L^{-1} and extraction time of 10 min), a preconcentration factor of 50 was reached. The detection limit was 0.5 μg L^{-1} and the relative standard deviation for 10 replicate determination of 20 μg L^{-1} was 2.6%. The calibration graph was linear from 5 to 180 μg L^{-1} with the correlation coefficient of 0.99. The proposed method was fast, inexpensive, easy to operate with a high enrichment factor, consumes low volume of organic solvent and successfully applied to the extraction and determination of Pb(II) in water samples.

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Dispersive liquid–liquid microextraction method as an efficient microextraction technique for bioanalysis; Application in extraction and preconcentration of four antiarrhythic drugs in human plasma followed by high performance liquid chromatography-ultraviolet detection

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Abstract

Generally, antiarrhythic drugs therapy is preferred for patients with cardiac arrhythmia. Optimal therapy with these drugs requires therapeutic drug monitoring (TDM) in order to avoid adverse effects and obtain the desired clinical benefit [1]. Drugs and metabolites in complex matrices such as plasma cannot be injected directly into the analytical instruments without sample clean up. Therefore, there is a need for robust and sensitive high-throughput sample preparation with short turnaround time [2]. In this work, a fast and sensitive method was developed and validated for the simultaneous quantitation of four antiarrhythic drugs (propranolol, carvedilol, diltiazem, and verapamil) in human plasma samples. It involves dispersive liquid-liquid microextraction (DLLME) of desired drugs from 660 µL of plasma and separation using isocratic elution with ultraviolet detection at 200 nm. The complete separation of all analytes was achieved within 7 min. Different factors affecting the DLLME procedure such as type and volume of extraction and dispersive solvents, ionic strength, and sample pH were investigated and optimized. Under the optimal conditions the enrichment factors and extraction recovery ranged between 7.8-10.8 and 71.3-98.2%, respectively. The suggested method was linear ($r^2 \geq 0.998$) over the dynamic range of 0.02-0.80 µg/mL in plasma. The intra- and inter-day coefficient of variation (CV%) and relative error (RE%) values of the assay method were all below 20%, which showed good precision and accuracy. Finally this method was applied to the analysis of plasma samples obtained from the patients treated with these drugs.

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Dispersive micro-solid-phase extraction of benzodiazepines from biological fluids based on polyaniline/magnetic nanoparticles composite

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In this study, Fe_3O_4 nanoparticles modified by polyaniline, polypyrrole, and aniline-pyrrole copolymer were synthesized by chemical oxidative polymerization process in the presence of different dopants [1]. The results showed that modified nanocomposites with polyaniline and p-toluene sulfonic acid as dopant have higher extraction efficiency for extracting lorazepam and nitrazepame. The resultant magnetic sorbent was characterized by scanning electron microscopy (SEM), energy dispersive X-ray spectrometry (EDXS) and Fourier transform-infrared (FT-IR) spectroscopy. This nanocomposite in combination with high performance liquid chromatography-UV detection (HPLC-UV) was applied for extraction and determination of lorazepam and nitrazepame in urine and plasma samples. Effect of different parameters influencing the extraction efficiency including: sample pH, amount of sorbent, sorption time, eluent solvent and its volume, salt content, stirring rate, vortex rate and elution time were optimized. The obtained optimal conditions were: sample pH , 6; amount of sorbent, 5 mg; sorption time , 5 min; eluent solvent and its volume, 0.5 mM CTAB in acetonitrile, 150 µL; stirring rate, 1250 rpm; vortex rate and time, 2000 rpm, 2 min and without addition of salt. The calibration curves were linear in the concentration range of 1-2000 µg L^{-1}. The limit of detections (LODs) and relative standard deviations (RSDs) of lorazepam and nitrazepam were 0.2 and 0.5 µg L^{-1}; 4.1 and 6.1, respectively. Ultimately, the applicability of the method was successfully confirmed by extraction and determination of the target drugs in human urine and plasma samples in the range of microgram per liter and satisfactory results were obtained.

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Dispersive nano solid material-ultrasound assisted microextraction method for extraction and determination of bendiocarb and promecarb: Response surface methodology

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Carbamates are one of the important classes of the pesticides used as insecticides and fungicides in the early 1950s, and nowadays are widely used in agriculture due to their broad biological activity, low bioaccumulation potentials, and relatively low mammalian toxicities. However, the carbamates affect the nervous system by disrupting an enzyme that regulates acetylcholine, a neurotransmitter [1]. A new extraction method, based on dispersive nano solid material-ultrasound assisted micro-extraction (DNSUAME), was used for the preconcentration of the bendiocarb and promecarb pesticides in the water samples prior to high performance liquid chromatography (HPLC). The properties of NiZnS nanomaterial loaded on activated carbon (NiZnS-AC) are characterized by FT-IR, TEM, and BET. This novel nanomaterial showed great adsorptive ability towards the bendiocarb and promecarb pesticides. The effective variables such as the amount of adsorbent (mg: NiZnS-AC), the pH and ionic strength of sample solution, the vortex and ultrasonic time (min), the ultrasonic temperature (ºC), and desorption volume (mL) are investigated by screening 2^{7-4} experiments of Plackett–Burman (PB) design [2]. The important variables optimized by using a central composite design (CCD) were combined by a desirability function (DF) [3]. At optimum conditions, the method has linear response over 0.0033-10 μg mL^{-1} with detection limit between 0.0010 and 0.0015 μg mL^{-1} with relative standard deviations (RSDs) less than 5.5% (n = 3). The method has been successfully applied for the determination of the bendiocarb and promecarb pesticides in the water samples.

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DVD Substrate as Bipolar Electrode for Screening and Sensing Applications

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In this work we introduced an extremely low cost and easy method for fabrication of bipolar electrodes (BPE) for simultaneous and rapid screening of candidates for hydrogen evolution reaction (HER) and also for sensing applications. Our method takes the advantage of the thin silver reflective layer deposited on already available DVD-R polycarbonate substrate which in our design acts as BPE. Based on oxidation of the reflective layer of the DVD-R in anodic pole of the BPE which lefts behind a visually measurable dissolute length we correlated this dissolution length to the electrocatalytic activity of catalyst at the cathodic pole of the BPE as well as to the concentration of analyte in the solution. As a proof of concept p-benzoquinone (BQ) was tested as model target for sensing application and for screening application the electrocatalytic activity of Pt, Pd, Au, and pristine Ag DVD toward HER in 0.1M H_2SO_4 were compared. We measured the dissolution length by a ruler and our results indicate that for sensing application there is a nearly linear relationship ($l_{ox} = 3.272C_{BQ} + 8.3$, $R^2=0.997$, n=6) between the concentration of BQ and dissolution length of the BPE in the range of 0.0-2.5mM. Moreover, for screening of electrocatalysts, there is a linear relationship ($l_{ox} = -39.81\Delta E_{\text{onset}} + 43.4$, $R^2=0.998$, n=4) between the onset potential for HER (E_{onset}^{HER}) of electrocatalysts and the dissolution length of the corresponding electrode in an array of four BPEs each modified with electrocatalyst. An array of four BPEs is shown below in which at the right each electrode is modified with a catalyst and at the left the dissolution lengths corresponding with each of catalysts are visible. Using array makes it possible to compare the electrocatalytic activity of catalysts simultaneously in a single experiment run. The equation that governs the relationship between dissolution lengths and E_{onset}^{HER} is shown below in which l_0 is the initial length of the BPE prior to the beginning of the experiment and l_{ox} is dissolution length of BPE at the end of the experiment. E_{tot} is the driving voltage and $l_{channel}$ is the distance between two driving electrodes. E_{onset}^{ox} is onset potential for oxidation of the Ag layer.

$$l_{ox} = \left(-\frac{l_{channel}}{E_{tot}}\right)\Delta E_{\text{onset}} + l_0$$

$$\Delta E_{onset} = E_{onset}^{ox} - E_{onset}^{HER}$$

Dye removal activation by prepared FeOOH–MnO_2 nanocomposite with reactive Blue 19

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Industrial wastewater is usually one of the most polluting industries in the environment.Various industries such as textile, pharmaceutical, food, cosmetics and health products, paper making, leather form industrial waste water which can be color. Textile industries are one of the most important basic industries in each country. In this industry waste water of dying process forms an important part of colored wastewater [1].

FeOOH–MnO_2nanocompositewas prepared by mixing solutions of $KMnO_4$ and NaOH in cethyltrimethyl ammonium bromide media and FeSO4·7H2Oin cethyltrimethyl ammonium bromide media room temperature. The obtained suspension was continuously stirred for 1 h, and aged at 25 °Cfor 4 h.The product wascentrifuged, washed with water and dried at 60 °C for 24 h [2]. The nanocomposite was characterized by infra-red spectroscopy, x-ray diffraction and scanning electron microscopy.

The FeOOH–MnO_2nanocomposite was used as a photo catalyst in order to be removed dye from simulated colored wastewater. The adsorption behavior of reactive Blue 19was investigated for nanocomposite. The important factors such as the amount of nanocomposites, pH, temperature and dye concentration were determined.The removal of dye has been done over 92% in the best condition, pH =7, adsorbent 0.02 g anddye concentration 50ppm.These conditions were selected for other experiments in order to be specified kinetic parameters.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ۶ الی ۸ اسفند ماه ۱۳۹۲

Effect of experimental factors on the behavior of a thiocyanate selective electrode based on modified nano zeolite clinoptilolite with a cationinc surfactant

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Abstract

Zeolites are, narrowly defined, porous crystalline aluminosilicates that have a uniform pore structure and exhibit ion-exchange behavior.The negative charge of zeolites is suitable for their surface modification using cationic surfactants such as tetraethylammonium hyoroxide ion. At surfactant concentrations greater than the critical micelle concentration (CMC) and in the presence of a sufficient surfactant, the adsorbed surfactant molecules primarily form a bilayer on the external surface of zeolites. This bilayer formation results in a charge reversal on the external zeolite surface, providing sites where anions will be retained and cations repelled, while neutral species can partition into the hydrophobic cores. In this study mide the clinoptilolite zeolite in nanoscale mechanical method was developed. The decrease in the crystal sizes results in high external surface areas reduced diffusion path lengths,and more exposed active sites, the improved properties of nanocrystalline zeolites for adsorption and intracrystalline diffusion afford many opportunity for their application in environmental catalysis, membranes,… [1-3]. The thiocyanate selective electrode was produced by tetraethylammonium hyoroxide as a modifier for modifying the nano clinoptilolite particles (SMZ). This SMZ used in membrane texture. The sensor comprises a SMZ as a modifier, dioctyl phthalate (DOP) as a softner and polyvinyl chloride (PVC) as a plasticizer**.** The best performance was exhibited by the membrane having a composition of SMZ: PVC: DOPin the ratio 4:32: 64. The EMF response of the electrode to varying conceratrations of thiocyanate species indicated atinear range from 1.0×10^{-7} to 1.0×10^{-1} M. The average slope of calibration carves was -59.3±0.3 mV per decade of SCN^- concentration with detection limits of 3.0×10^{-8} M. The response time of this sensor is about 4- 6s. The SMZ – PVC has applicability in a pH range 3.0-8.5. The electrode exhibits good Nernstian behavior in the range of 25- 35 °C. The long term stablility of the electrode was investigated by recording the response of the optimized electrode over a period of 155 days. The proposed thiocyanate selective electrode was also applied to the determination of thiocyonate ions in the river and tap water samples.

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Effect of experimental factors on the behavior of a thiocyanate selective electrode based on modified nano-clinoptilolite with a cationinc surfactant

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Abstract

Thiocyanate finds many industrial applications and though not as toxic as cyanide, it is harmful to aquatic life. If the contenr of thiocyanate ion is a little higher in the body than normal, the protein dialsis will be affected and it may even result in coma. Because of their unique advantages of ISEs such as simplicity, speed of analysis, fast response time, low cost, wide linear rang, reaonable selectivity etc, they led to the development of potentiometric sensors for many inorgonic and organic species. In this study the thiocyanate selective electrode was produced by tetraethylammonium hyoroxide as a modifier for modifying the nano clinoptilolite particles (SMZ). The best performance was exhibited by the membrane having a composition of SMZ: PVC: DOPin the ratio 4:32: 64. The EMF response of the electrode to varying conceratrations of thiocyanate species indicated atinear range from 1.0×10^{-7} to 1.0×10^{-1} M. The average slope of calibration carves was -59.3±0.3 mV per decade of SCN^- concentration with detection limits of 3.0×10^{-8} M. The response time of this sensor is about 4- 6s. The SMZ –PVC has applicability in a pH range 3.0-8.5. The electrode exhibits good Nernstian behavior in the range of 25- 35 °C. The long term stablility of the electrode was investigated by recording the response of the optimized electrode over a period of 155 days. The proposed thiocyanate selective electrode was also applied to the determination of thiocyonate ions in the river and tap water samples.

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دانشگاه صنعتی اصفهان ، ۶ الی ۸ اسفند ماه ۱۳۹۲

Effect of ionic liquids on extraction efficiency of solid-phase microextraction based sol−gel technique

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The coating type is one of the most critical aspects in the solid-phase microextraction (SPME) techniques [1]. The porous structure of the sol–gel coating offers a high surface area; allowing high extraction efficiency and the coating composition can be altered with a relative ease to give different selectivity characteristics. Strong adhesion of the coating onto the support due to chemical bonding is a very important characteristic which increases the coating stability towards the organic solvents and high desorption temperatures [2]. The current strong interest in ionic liquids is motivated by their unique combination of properties such as negligible vapour pressure, thermal stability, miscibility with water and variety of organic solvents and also variable viscosity .In several cases, ILs had significant effects on the porous structure of sol−gel materials [3], reduction in cracking and shrinking during solvent evaporation from the sol−gel pores, and sol−gel reaction kinetics. Ionic liquid mediated sol–gel sorbents were developed for headspace solid-phase microextraction (HS-SPME) for the extraction of naphthalene, fluorene and anthracene (PAHs) compounds by GC−FID. Three different coating fibers were prepared including: poly(dimethylsiloxane) (PDMS), coatings prepared from poly(dimethylsiloxane) in the presence of two different ionic liquids and conditioned at a higher temperature than decomposition temperature of ionic liquids (PDMS-IL). Effective parameters were optimized for extraction of model analytes and figure of merit of the method were investigated in water and juice fruits samples. Under optimized conditions, the dynamic linear range with PDMS-IL$_1$ and PDMS-IL$_2$ fibers were 0.003−400 and 0.005−400 ng/mL, limit of quantifications (S/N = 10) were 0.003−0.005 and0.005−0.008 ng/ml and the detection limits (S/N = 3) were 0.0009−0.003, 0.003−0.005 ng/mL, respectively. The relative standard deviations (RSD) for one fiber (repeatability) (*n* = 5), were obtained from 4.2 up to 7.5 % and between fibers or batch to batch (reproducibility) (*n* = 3) in the range of 6.8 –8.9% for three fibers. The developed method was successfully applied to the real water samples while the relative recovery percentages obtained for the spiked water and juice fruits sample with 0.1 ng/ml of the analytes, were from 80 to 101.3%.

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Effect of non-aqueous solvents on stoichiometry and thermodynamics of complexation process between 1,13-bis(8-quinolyl)-1,4,7,10,13-pentaoxatridecane (Kryptofix5) and ZrO^{2+} cation using conductometry method

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Abstract:

Conductance measurements of an electrolyte solution in the presence of a crown compound provide two valuable pieces of information. The first, is detection of complexation between the crown compound and the cation. Furthermore, the stability constant of the crown compound cation complex can be determined from the conductance data [1]. Macrocycle design parameters, such as cavity size, the type and the number of donor atoms, the type and the number of proton ionizable groups within and without the macrocycle cavity, chirality's, the substituent groups,and steric hindrance and also the solvent parameters can all beused to obtain the desired selectivities [2, 3].In the present work, the complexation process between ZrO^{2+} cation and the macromolecule ligand, 1,13-bis(8-quinolyl)-,4,7,10,13-pentaoxatridecane (Kryptofix5) was studied in methanol-acetonitrile (MeOH-AN), methanol -ethylactate (MeOH-EtOAc), methanol-dimethylformamide (MeOH-DMF) and methanol -1,2-dichloroethane (MeOH-DCE) binary solutions at different temperatures using the conductometric method. The conductance data show that in most cases, the stoichiometry of the complex formed between kryptofix5 and ZrO^{2+} cation is 1:1 [M:L], but in some solvent systems also, a1:2 [$M:L_2$] complex is formed in solutions. The values of stability constant of $(Kryptofix5.ZrO)^{2+}$ complex which were obtained from conductometric data, show that the stability of the complex is affected by the nature and also the composition of the solvent system and in all cases, a non-linear behavior is observed for the variation of ($\log K_f$) of the $(Kryptofix5.ZrO)^{2+}$ complex versus the composition of the binary mixed solvents. The values of thermodynamic quantities ΔH_c° and ΔS_c° for formation of $(Kryptofix5.ZrO)^{2+}$ complex were obtained from temperature dependence of the stability constant using the van't Hoff plots. The experimental results show that depending on the nature and composition of the solvent systems, the complex is enthalpy stabilized or destabilized, but in most cases, it is stabilized from entropy view point and both thermodynamic parameters are affected by the nature and composition of the binary mixed solutions.

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Effective Removal of Methylene blue over Silica-Coated Fe_3O_4 Magnetic Nanoparticles Modified with Castor Oil

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Dye effluents, which are discharged from the dyestuff manufacturing, dyeing, printing and textile industries, are mostly toxic and/or carcinogenic to organism. Majority of dyes are synthetic and are usually composed of aromatic rings in their structure, inert and non-biodegradable when discharged into waste streams. Among of these dyes, Methylene blue is a cationic dye, which is most commonly used for coloring paper, dying cottons, wools, *etc*. Acute exposure to Methylene blue will cause increased heart rate, vomiting, shock, Heinz body formation, cyanosis, jaundice, quadriplegia, and tissue necrosis in humans [1]. In order to remove the Methylene blue from wastewater, conventional methods such as flocculation, biodegradation, and reverse osmosis have been developed [2]. In recent years, adsorption technology has attracted particular attention due to its simplicity in operation and availability for a wide range of adsorbents. Recently, magnetic nanoparticles due to their unique properties such as excellent magnetic responsivity, high dispersibility, relatively large surface area and easiness of surface modification have been used as a sorbent for removal of dyes [3].

In the present study, a novel magnetic sorbent material prepared by modifying silica-coated magnetic nanoparticles (Fe_3O_4/SiO_2) with castor oil has been reported in the adsorption of methylene blue from aqueous solution. The chemical structures of the sorbent were characterized by X-ray diffraction (XRD), thermo gravimetric analysis (TGA) and Fourier transform infrared spectrophotometer (FT-IR). The effects of contact time, pH, temperature of dye solution and adsorbent dose on the adsorption percentage have been investigated to optimize the conditions leading to maximum removal efficiency. The best kinetic and isotherm models was found from experimental data.

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Effective removal of Ni (II) ions from aqueous solutions using nanocrystalline Zeolite Na-P1

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In this study, we have synthesized nanocrystalline zeolite Na-P1 with crystal sizes around 70 nm and investigated its sorption activity for Ni (II) ions removal from aqueous solutions. Modification of nano-zeolite and its Ni-exchanged forms were done by dimethylglyoxime (DMG). Removal of Ni (II) by synthesized nanocrystalline Na-P1 was investigated in batch procedure. The sorption involves a film diffusion, an intra-particle diffusion, and a chemical cation-exchange between the Na^+ ions of nanocrystalline Na-P1 and the Ni^{2+} ions. The effects of analytical parameters such as pH, dose of DMG, concentration of nickel solution and contact time were studied [1, 2]. The most effective pH range for the removal of Ni (II) ions was found to be 6.0–8.0. The pseudo-first order and pseudo-second order equations were used to test the experimental rate data and to examine the controlling mechanism of the sorption process. Langmiur and Freundlich isotherm models were adopted to describe the adsorption isotherms and Freundlich isotherm expression were found to give a better fit to the experimental equilibrium data compared to Langmuir model [3]. The results indicated that synthetic nanocrystalline Na-P1 can be used as an efficient ion exchange material for the removal of nickel ions from aqueous solutions.

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Effects of carbon nanotubes (CNTs) on the performance of cross-linked EPDM membrane for CO_2/N_2 separation

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Abstract

A new type of mixed matrix membrane, consisting of multi-walled carbon nanotubes and ethylene propylene diene monomer (MWCNTs/EPDM) is prepared by the solution casting and solvent evaporation method. Also we use peroxide vulcanization to increase efficiency of nanocomposite membrane. Therefore 3 phr of dicumyl peroxide were added to the solution samples and dissolved by Stirring. The performance of the membrane was investigated for separation of CO_2/N_2 gases. The structural properties, surface and cross-sectional morphologies of these prepared membranes have been characterized by scanning electron microscopy (SEM). Fourier transform infrared spectroscopy (FTIR) was carried out to determine the effect of MWCNT incorporation on intermolecular interactions. The results showed a good distribution of carbon nanotubes into rubbery polymer matrix. The effects of adding different amounts of multi-walled carbon nanotubes between 0 and 5% of polymer weight with 40 minutes sonication to ensure uniformity have been studied. Gas permeation measurements showed increased permeability for both CO_2 and N_2 and increase in CO_2/N_2 selectivity, by the increase in CNT contents of the membrane samples. Also effect of various feed pressures (1- 6 bar) on the membrane performance in separation of CO_2/N_2 at ambient temperature have been investigated. With the increase in feed pressure, a slight increase was observed in the permeability of gases.

Keywords: Carbon nanotubes, CO_2/N_2 separation, Membrane, EPDM.

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Effects of Coating of Ammonium Perchlorate with Organosilicon Polymer on Its Thermal Decomposition and Agglomeration

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Abstract:

Ammonium perchlorate (AP) is common oxidizer which is used in solid propellant formulations. Coating is a method to prevent agglomeration or to improve thermal decomposition properties of AP. Organosilicon compounds are one of coating agent group that is used for coating of AP[1-3]. In this research AP particles are coated with poly(methyl hydrogen siloxan) as organosilicon polymer by solvent method and compared with industrial AP (coated with sodium lauryl sulfate) and uncoated AP used as reference sample . This coating agent can create steady and very thin film on surface of AP particles (less than 100 nanometer) so purity of AP that is coated in this research is more than industrial AP. Differential scanning calorimetry (DSC), thermogravimetry (TG), and scanning electron microscopy (SEM) have been exploited to investigate the thermal decomposition properties, heat of decomposition, and coating morphology of pure and coated samples. The results showed that coated AP with poly(methyl hydrogen siloxan) has eliminated AP agglomeration completely. The thermal analysis of coated AP, prepared at optimized coating conditions, showed that its thermal decomposition temperature increases about 15°C with compare to uncoated sample whereas phase transition temperature is constant approximately. Also heat of decomposition of poly(methyl hydrogen siloxan) coated AP is more than industrial AP about 30 j/g. In addition, TG curves show that start and end of weight loss temperature of coated AP are shift to higher temperature. In the other word, with this coating, thermal stability and heat of decomposition of AP are increased and problems of storing and moisture absorption of AP are solved.

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Effects of Organosilicon Polymer Nano Coating on Agglomaration and Thermal Decomposition of Ammonium Perchlorate

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(Moosem Research Center)

Abstract:

Ammonium perchlorate (AP) is an inorganic salt that is used widely in laboratories and industrials. Agglomeration and no control on particle size is important problem about AP. Coating is a method to prevent agglomeration or to improve thermal decomposition properties of AP. Organosilicon compounds are one of coating agent group that is used for coating of AP[1-3]. In this research AP particles are coated with poly(methyl hydrogen siloxan) as organosilicon polymer by solvent method and compared with industrial AP (coated with sodium lauryl sulfate) and uncoated AP used as reference sample . This coating agent can create steady and very thin film on surface of AP particles (less than 100 nanometer) so purity of AP that is coated in this research is more than industrial AP. Differential scanning calorimetry (DSC), thermogravimetry (TG), and scanning electron microscopy (SEM) have been exploited to investigate the thermal decomposition properties, heat of decomposition, and coating morphology of pure and coated samples. The results showed that coated AP with poly(methyl hydrogen siloxan) has eliminated AP agglomeration completely. The thermal analysis of coated AP, prepared at optimized coating conditions, showed that its thermal decomposition temperature increases about 15°C with compare to uncoated sample whereas phase transition temperature is constant approximately. Also heat of decomposition of poly(methyl hydrogen siloxan) coated AP is more than industrial AP about 30 j/g. In addition, TG curves show that start and end of weight loss temperature of coated AP are shift to higher temperature. In the other word, with this coating, thermal stability and heat of decomposition of AP are increased and problems of storing and moisture absorption of AP are solved.

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Effects of particle size on the activity and selectivity of MoO_3/CNTs catalyst in ethanol and higher alcohols synthesis from syngas

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Abstract

The influence of Mo particle size on catalytic activity and products selectivity in alkalized MoO_3 nano catalysts has been investigated. Nano catalysts are prepared using microemulsion technique with water-to-surfactant ratios of 1-12. Three different techniques, including XRD, TEM and hydrogen chemisorption were used to determine the molybdenum average particle sizes and their activity and selectivity in higher alcohols synthesis (HAS) been assessed in a fixed bed micro-reactor at 330^{o}C, 70bar and 3.6 nLit/h/g catalyst. The average MoO_3 particle sizes are changed from 4.5 to 11.9 nm. Experimental results showed that changing particle sizes from 11.9 to 4.5 nm decreased the methanol formation rate from 0.00534(mol/hr.gr Cat) to 0.00634 and increased ethanol formation rate from 0.00581 to 0.00787 and higher alcohols formation rate from 0.00473 to 0.00657. A model was derived to calculate the alcohol formation rates versus catalyst average particle sizes. Analysis of adsorption on catalyst using kinetic models that used widely for higher alcohol synthesis and also taking into account changing in catalyst properties that occurs because of different particle size were created an acceptable quantitative model for alcohol synthesis. The model not only matches experimental and theoretical results but also showed that MoO_3 catalyst has size dependent structure and for prediction of product’s selectivity it is easier to use this mathematical model.

Keywords: *Higher alcohol, Rate, Particle size, Size dependent kinetic, Model*

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Abstract

The influence of Mo particle size on catalytic activity and products selectivity in alkalized MoO_3 nano catalysts has been investigated. Nano catalysts are prepared using microemulsion technique with water-to-surfactant ratios of 1-12[1]. Three different techniques, including XRD, TEM and hydrogen chemisorption were used to determine the molybdenum average particle sizes and their activity and selectivity in higher alcohols synthesis (HAS) been assessed in a fixed bed micro-reactor at 330^{o}C, 70bar and 3.6 nLit/h/g catalyst . The average MoO_3 particle sizes are changed from 4.5 to 11.9 nm. Experimental results showed that changing particle sizes from 11.9 to 4.5 nm decreased the methanol formation rate from 0.00634 (mol/hr.gr Cat) to 0.00534 and increased ethanol formation rate from 0.00581 to 0.00787 and higher alcohols formation rate from 0.00473 to 0.00657. A model was derived to calculate the alcohol formation rates versus catalyst average particle sizes[2]. Analysis of adsorption on catalyst using kinetic models that used widely for higher alcohol synthesis and also taking into account changing in catalyst properties that occurs because of different particle size were created an acceptable quantitative model for alcohol synthesis[3]. The model not only matches experimental and theoretical results but also showed that MoO_3 catalyst has size dependent structure and for prediction of product's selectivity it is easier to use this mathematical model.

Keywords: *Higher alcohol, Rate, Particle size, Size dependent kinetic, Model*

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Electrocatalytic determination of glutathione in the presence of amoxicillin at a modified nanocomposite carbon paste electrode

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Abstract: Biological thiols, such as glutathione (GSH), occur widely in living tissues. GSH is synthesized in two steps catalyzed by ɣ-glutamyl-cysteine (Glu-Cys) synthetase and GSH synthetase, and then degraded into cysteinyl-glycine (Cys-Gly) by ɣ-glutamyltranspeptidase [1]. Amoxicillin, D-α-amino-p-hydroxybenzylpenicillin trihydrate, is one of the most frequently used β-lactam antibiotics in the world and it is employed to treat humans and animals [2]. Amoxicillin or clavulanic acid and their combined administration effective on the biochemical liver parameters, reduced glutathione (GSH), lipid peroxidation measured as hepatic malondialdehyde (MDA) levels. Therefore, simultaneous determination of these compounds is very important. The electrochemical behaviors of the compounds at modified nanocomposite carbone paste electrode were studied using cyclic voltammetry, square wave voltammetry, and electrochemical impedance spectroscopy. The results indicated that the chemically modified electrode exhibited an efficient electrocatalytic activity towards the oxidation of these compounds at pH 7.0. The separation of the anodic peak potentials of GSH and amoxicillin reached to about 0.3 V, using square wave voltammetry. The modified electrode was successfully used for the determination of the analytes in real samples with satisfactory results.

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Electrocatalytic Determination of Homocysteine Using a Platinum Coated Nanoporous Gold Film Electrode

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Abstract

Determination of Homocysteine using a nanoporous film-based gold catalyst design using the underpotential deposition. The procedure consisted of the underpotential deposition (UPD) of copper on the gold nanoporous film [1]. The deposition of platinum occurred as a spontaneous redox process in which a copper layer, obtained by underpotential deposition, was oxidized by platinum ions, which were reduced and simultaneously deposited [2]. Homocysteine at the surface of the modified electrode was studied using cyclic voltammetry, chronoamperometry, and electrochemical impedance spectroscopy. Under the optimized conditions, the amperometry peak current of Homocysteine increased linearly with Homocysteine concentration in the ranges of 4.84×10^{-5} to 4.74×10^{-9}. The detection limit was 3.6×10^{-9} mol L^{-1} homocysteine. The diffusion coefficient and kinetic parameters (such as electron transfer coefficient and the heterogeneous rate constant) for Homocysteine oxidation were also determined. The RSD% for Homocysteine were 2.8%. The proposed sensor was successfully applied for the determination of Homocysteine in human patient urine and tablet samples. Homocysteine is an amino acid, (2-amino-4-mercaptobutyric acid) which was first synthesized in 1932 by du Vigneaud in the course of his research into sulfur chemistry and metabolism. It is an important amino acid, which contains a free thiol moiety and as such has several important roles within physiological matrices. Although it is not found directly in the diet, it is an intermediate formed during the metabolism of methionine to cysteine [3].

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Electrocatalytic Oxidation of Hydrazine on a Nanoporous Co/Co-Ni-Ag Electrode in Alkaline Media

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Abstract

An electrochemical approach to nanostructured Co/Co-Ni-Ag catalyst with high activity design using the electrode position process and galvanic replacement technique is presented [1]. The procedure consisted of the electrode position of Co-Ni-Zn on the Co coating, with subsequent replacement of the Zinc by Silver at open circuit in a Silver containing alkaline solution. The surface morphologies and compositions of coatings were determined by energy dispersive X-ray (EDX) and scanning electron (SEM) microscopy techniques. The characterization showed that the leached Co/Co-Ni-Ag coating has a nanoporous structure [2]. The electrocataiytical oxidation of Hydrazine on Co-Ni-Ag electrode was studied using cyclic voltammetry (CV), choronoamperometry and electrochemical impedance spectroscopy (EIS). Electrocatalytic activity towards hydrazine oxidation in alkaline solutions was evaluated by cyclic voltammetry (CV). Moreover, the effects of various parameters such potential scan rate (10- 100 mVs^{-1}), Hydrazine concentration (0.1-1 mM) and solution temperature (25-65 °C) on the electro-oxidation of Hydrazine have also been investing a ted. The calibration curve was linear in the concentration range 0.1 mM to 1 mM with the detection limit of 3 µM. The anodic peak current density was found to be up to ten times higher on Co/Co-Ni-Ag electrode for Hydrazine compared to that flat Ag and confirmed the better electro-catalytic activity and stability of these new electrodes. These results indicate that the system studied in the present work is the most promising system for use direct Hydrazine fuel cells (DHFC) [3].

Keywords: Hydrazine; Nanoporous; Co/Co-Ni-Ag; Alkaline Media

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Abstract

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Electrochemical Analyses of Ag-Loaded on Silica-Supported Carboxylic Acid Dendrimers

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This work explains the ability of silica-supported dendrimers as substrates for loading of metal nanoparticles like Ag nanoparticles (AgNPs). Silver nanoparticles were formed by reducing $AgNO_3$ to AgNPs on dendrimers using $NaBH_4$ as a reductant [1]. This synthesized Ag loaded dendrimers were characterized using scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FT-IR), and transmission electron microscopy (TEM). The FT-IR spectra represented the synthesis of dendrimers. The SEM and TEM images showed the average size of Ag nanoparticles around 15 nm. The optimized amounts of the dendrimers were blend with graphite and paraffin oil. The carbon paste electrodes were studied using electrochemical methods including cyclic voltammetry, linear square wave voltammetry, and electrochemical impedance spectroscopy. The stability of electrodes was studied in different pH media of phosphoric buffer solutions. Cyclic voltammograms represented different effects of media on the peak potential locations, peak current intensities, and baseline currents of electrodes. The studies of square wave voltammetry showed the effects of scan rates on the voltammograms. The EIS was used to compare the conductivity of electrodes. These studies represented the differences of the dendrimers by changing their lengths and compositions. In further studies, the dendrimers can be investigated to analyze of different species.

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Electrochemical Analysis of a PEMFC under Different Operation Condition

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In this study, the steady state behavior of a PEM fuel cell is evaluated experimentally for different operation conditions by i-V curves. The applied conditions were: low and high cathode relative humidity (RH_C); 50% and 100% of RH_C and low and high oxidant concentration; air versus oxygen. The experimental data, obtained under different operation condition, are used in estimating the parameters of a completely analytical model that describes i-V curves. Polarization curves were fitted with analytical modeling equation by an optimization algorithm [1]. The analytical model consists of the three fundamental losses experienced by a PEM fuel cell, namely: activation, ohmic, and concentration losses. While the Tafel constants, limiting current density and the concentration loss constant are estimated through regression, the ohmic resistance is obtained through measurements. The estimated parameters derived in the literature are found to reasonably fit the obtained experimental data. Results indicate that decreasing in RH_C and oxidant concentration (using air instead of oxygen) is caused to reduce oxygen partial pressure on the catalyst surface, resulting in decreasing exchange current density, Tafel slope and limiting current density. Therefore, decreasing the oxidant partial pressure on the catalyst surface leads the high activation and concentration overpotential at all current density region [2].

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Electrochemical Analysis of Low-cost Nanocatalysts in Cathodic Reactions of Direct Borohydride Fuel Cell

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This paper will focus on benchmark of low-cost and selective cathode catalyst for ORR in alkaline direct borohydride fuel cell (ADBFC) by investigating of activity of the commercial 10 wt% Pt/C and nonplatinium electrocatalysts. The electro-catalytic properties of catalysts in oxygen reduction reaction (ORR) and borohydride crossover were studied using a three-electrode system and single direct borohydride fuel cell. The performance of the ADBFC and polarization behaviors of the both cathode and anode electrodes under different conditions were evaluated and discussed in this study. The anodes polarization curves of both cells are the same at low current density, which let us, make cathode kinetic comparative study. Due to the diffusivity of borohydride through the anion-exchange membrane from the anode side to the cathode side of direct borohydride fuel cell, borohydride could be oxidized electrochemically on the cathode side depending on the typical cathode catalyst [1]. It reduce the OCV and power density of the DBFC. The difference in performance of these catalysts can be attributed not only to more oxygen reduction reaction (ORR) activity but also to less sensitivity of nonplatinum cathode to oxidation of borohydride which the second one was obtained by results of cyclic voltammetry (CV) (borohydride tolerance among the cathodes). The performance was explained based on borohydride tolerance. The cyclic voltammetry curves shift to the direction of oxidation current by using the platinum catalyst, which is caused by the oxidation of crossover BH_4^- from anode electrode to the cathode. In addition, effect of concentration on borohydride crossover was investigated by this technique. Hence, the cells with the non-Pt cathode showed the higher activity to that with the Pt cathode.

Keyword: Direct Borohydride Fuel Cell, Cyclic Voltammetry, Polarization, Crossover, Cathodic Reactions.

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Electrochemical and Theoretical Evaluation of*PistaciaTerebinthus*Leaves Extract for Carbon Steel in H_2SO_4 solution

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Abstract

Use of inhibitors is one of the most practical methods for protection against corrosion especially in acid solutions to prevent metal dissolution and acid consumption.Organic chemicals operating as corrosion inhibitors were developed in the petroleum industry in the 1950s and were introduced to the concrete industry in the early 1990s [1].Recently, usage of toxic materials as inhibitors has been limited because of their environmental threat [2].Plant extracts are incredibly rich sources of naturally synthesized chemical compounds (e.g. amino and organic acids, alkaloids, polyphenols, tannins) and most are known tohave inhibitive action [3].Presently, to the best of our knowledge, there is no reported work on inhibitive effects of *PistaciaTerebinthus*extract on acid corrosionof carbon steel in sulphuricacid solution.In the present work, the use of *PistaciaTerebinthus*leaves extract as carbon steel corrosion inhibitor in H_2SO_4 solution was investigated by weight loss measurements, potentiodynamic polarization and electrochmical impedance spectroscopy (EIS) methods. The effects of extract concentration, solution pH, immersion time and temperature on the performance of extract were studied. Tafel polarization study revealed that *PistaciaTerebinthus*leaves extract acts as a mixed type inhibitor. The inhibition action of the extract was discussed in view of Langmuir adsorption isotherm. Thermodynamic parameters for the corrosion process were obtained and interpreted. The inhibition efficiency increases with increasing extract concentration. The results show that at the highest extract concentration of 2.0 g L^{-1}, the inhibition efficiency is increased markedly and reached 94%. Quantum chemical calculations using the density functional theory (DFT) and semi-empirical method (AM1) were performed to illustrate the process of adsorption of some specific components of the extract.

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Abstract

Use of inhibitors is one of the most practical methods for protection against corrosion especially in acid solutions to prevent metal dissolution and acid consumption.Organic chemicals operating as corrosion inhibitors were developed in the petroleum industry in the 1950s and were introduced to the concrete industry in the early 1990s [1].Recently, usage of toxic materials as inhibitors has been limited because of their environmental threat [2].Plant extracts are incredibly rich sources of naturally synthesized chemical compounds (e.g. amino and organic acids, alkaloids, polyphenols, tannins) and most are known tohave inhibitive action [3].Presently, to the best of our knowledge, there is no reported work on inhibitive effects of *PistaciaTerebinthus*extract on acid corrosionof carbon steel in sulphuricacid solution.In the present work, the use of *PistaciaTerebinthus*leaves extract as carbon steel corrosion inhibitor in H_2SO_4 solution was investigated by weight loss measurements, potentiodynamic polarization and electrochmical impedance spectroscopy (EIS) methods. The effects of extract concentration, solution pH, immersion time and temperature on the performance of extract were studied. Tafel polarization study revealed that *PistaciaTerebinthus*leaves extract acts as a mixed type inhibitor. The inhibition action of the extract was discussed in view of Langmuir adsorption isotherm. Thermodynamic parameters for the corrosion process were obtained and interpreted. The inhibition efficiency increases with increasing extract concentration. The results show that at the highest extract concentration of 2.0 g L^{-1}, the inhibition efficiency is increased markedly and reached 94%. Quantum chemical calculations using the density functional theory (DFT) and semi-empirical method (AM1) were performed to illustrate the process of adsorption of some specific components of the extract.

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Electrochemical and theoretical investigation of N-benzylidene derivatives on corrosion inhibition behavior of copper in hydrochloric acid

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Copper is widely used for industrial applications due to its excellent electrical and thermal conductivities and good mechanical workability [1]. Copper is resistant to the influence of atmosphere and many chemicals.

To improve the corrosion resistance properties of copper, great efforts have been gives to the investigations [2]. One suitable approach to minimizing corrosion damage is to use corrosion inhibitors which are substance used in very low amounts that efficiently slow the rate of corrosion.

Heterocyclic organic compounds containing sulphur and/or nitrogen heteroatoms which are available for bonding with metal surface are commonly used as inhibitors. N-benzylidene derivatives are illustrious due to their good inhibitor efficiency in various media.

The inhibition effect of two N- Benzylidene derivatives namely N-benzylidene Pyridin – 2 amine and N-(4-cholorobenzylidene) Pyridin – 2 amine have been investigated against the corrosion of copper in 6.0 M HCl solution. Corrosion inhibition behavior were investigated by electrochemical impedance spectroscopy (EIS), electrochemical polarization measurement and quantum chemical studies. Among the studied compounds, N-(4-cholorobenzylidene) Pyridin – 2 amine exhibited the best inhibition efficiency 99% at 0.01 M.

The EIS results show that equivalent circuits of blank and inhibitors are fitted with the RQRW and RRQ modes, respectively. The results demonstrated that the investigated compounds behave as good inhibitors for copper corrosion in acidic solution. Their efficiency increases with increase in concentration from 0.0005 M to 0.01 M. The results of potentiodynamic polarization show that N-benzylidene are cathodic type inhibitor. Adsorption of these compounds obeyed the Langmuir adsorption isotherm. The electronic properties obtained using quantum chemical approach, were correlated with the experimental results.

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Electrochemical behavior of dopamine at a ruthenium oxide nanoparticles modified glassy carbon electrode

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Dopamine (DA) as a catecholamine is an important neurotransmitter in the mammalian central nervous system and plays a pivotal role in the regulation and control of movement, motivation and cognition [1]. Since DA is an oxidizable compound, it can be easily detectable by electrochemical methods based on anodic oxidation. In addition, a lot of research has been conducted in recent years on the attractive properties of nanoparticles and their electrochemical and electrocatalytic behavior [2]. Due to different oxidation states of ruthenium, its compounds have been used as excellent electron transfer mediators for modification of different electrode surfaces [3]. In present work, the voltammetry responses of DA at an activated glassy carbon electrode (AGCE) and activated ruthenium oxide nanoparticles modified glassy carbon electrode (ARuON-GCE) show an anodic prepeak and a cathodic postpeak other than the diffusion main couple in a wide range of pH. Also, the results of this study show the reversibility and peak current of the redox process of DA is significantly improved at ruthenium oxide nanoparticles modified glassy carbon electrode (RuON-GCE) when compared to the bare glassy carbon electrode (BGCE). This encourages us to study based on our previous experiences, electrochemical behavior of DA at RuON-GCE in different pHs and conditions.

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Electrochemical behavior of salicylic acid at the surface of carbon paste electrode modified with multiwall carbon nanotubes: application to determination of salicylic acid in biological samples

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Abstract

Application of chemically modified electrodes (CMEs) in the electrochemical determinations has been widely considered as a selective and sensitive analytical method for the detection of trace amounts of biologically important compounds [1]. One of the most important properties of CMEs, in comparison to unmodified electrodes, has been their ability to catalyze the electrode process by significant decrease in the needed overpotential. With respect to relatively selective interaction of the electron mediator with the target analyte in a coordination fashion, these electrodes are capable to considerably enhance the selectivity in the electroanalytical methods. Carbon nanotubes (CNTs) are a new form of elementary carbon composed of graphitic sheets rolled into closed concentric cylinders with nanometer diameters and micrometer lengths. Since their discovery in 1991, CNTs have received considerable attention from chemists, physicists and materials scientists due to their unique electrical conductivity, chemical stability and high mechanical strength. [2]. Salicylic acid (SA) is the main metabolite of acetylsalicylic acid, that is, of one of the drugs most widely used in the world as a painkiller and anti-inflammatory, but banned from veterinary therapeutic treatment. Salicylic acid is therefore monitored in the urine and blood of animals to be slaughtered [3]. In this paper, we prepared a multiwall carbon nanotubes modified carbon paste electrode (MWCNT/CPE) for sensitive determination of SA. Some electrochemical techniques such as electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) was used for characterization of the modified electrode. Differential pulse voltammograms of SA showed enhanced redox responses for the MWCNT/GCE with respect to the bare GCE. Electrochemical behavior of SA was investigated by CV, chronoamperometry, chronocoulometry and linear sweep voltammetry (LSV) techniques. Experimental parameters, such as amount of MWCNT, pH, scan rate, step potential and modulation amplitude were optimized for SA measurement. Under the optimum pH of 2.0 in 0.2 M phosphate buffer solution, the anodic peak current showed a linear relation versus SA concentration in the range of 0.4-14.4 μM with a detection limit of 41.0 nM (signal to noise = 3) using differential pulse voltammetry. Moreover, this method demonstrated good reproducibility and long-term stability. Then, the proposed methode have been applied to the determination of SA in urine and plasma samples with good recovery.

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Electrochemical behaviour and voltammetric determination of sulfamethazine using a multi-walled carbon nanotube composite film-glassy carbon electrode

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Sulphonamides are synthetic antibacterial drugs with low potency used against a wide range of parasitic organisms including Plasmodia,Toxoplasma gondii and Pneumocystis carinii. Their mechanism of action is based on the inhibition of dihydropteroate synthetase. Sulphonamides are used for therapeutic, prophylactic and growth-promoting purposes in animals and in the treatment of trachoma and ocular toxoplasmosis in ophthalmology [1]. In this work, a simple and rapid electrochemical method was developed for the determination of sulfamethazine (SMT) at a glassy carbon electrode modified with multi walled carbon nanotube (MWCNT-GCE), using cyclic voltammetry. SMT showed an irreversible oxidation peak at bare electrode at potential of 1.08V. With this modified electrode the oxidation peak potential of SMT was shifted about 0.12 V toward a less positive value, presenting a peak current much higher than those measured on an unmodified GCE. Under the optimal experimental conditions the peak current of SMT was found to be proportional to its concetration over two linear ranges of 10-200 µM and 300-3000 µM [2]. The kinetic parameters such as the electron transfer coefficient between SMT and modified electrode, α, and the charge transfer rate constant, k_s, for oxidation of SMT at the MWCNT–GCE surface were determined according to the Laviron procedure [1]. The limit of detection (LOD) was obtained as 6.15 µM. The influence of potential interfering substances on the determination of SMT was examined. The method was successfully applied to determination of SMT in serum sample [2].

Keywords: sulfamethazine; multi-walled carbon nanotube; cyclic voltammetry.

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Electrochemical behaviour of thin film of Iron-cobalt layered double hydroxide (Fe/Co–LDH) prepared by electrodeposition

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Layered double hydroxides (LDHs) are a family of anionicclays consisting of positively charged brucite-like layersamong which are located anions and water molecules. Theirstructure is closely related to that of brucite, $\mathrm{Mg(OH)_2}$. The composition of LDHs can be described by the generalformula $[\mathrm{M^{II}}_{1-x}\mathrm{M^{III}}_x(\mathrm{OH})_2]^{x+}[\mathrm{A^{n-}}_{x/n}.y\mathrm{H_2O}]^{x-}$, where $M^{II}$is a bivalent cation ,$M^{III}$is a trivalent cation, A is an interlayer anion.

Many methods, such as sputtering, pulsed laser deposition, sol–gel and chemical vapor deposition, have been developed to deposit thin films of metal hydroxides or oxides on different materials. The electrochemical deposition has several advantages over other methods such as: (i) the film thicknesscan be modified from about 100 nm to a few microns; (ii)it is suitable for the coating of substrates with complex geometry; (iii) milder working conditions are required than with other techniques of thin film preparation; (iv) lastly, expensive equipment is not requiredand it is cost-effective. In general, the thickness, uniformity and morphology of the coating could be simply modulated either with the modification of electrochemical parameters, suchas applied potential and deposition time and concentration of metals in the working solution.

In this work electrochemical deposition of Fe/Co-LDH based on electrogeneration is considered as a novel method for preparation of LDHs.The film of $Co/Fe-NO_3$ LDH was deposited on the electrode surfaceby cathodic reduction of a 0.16 mMsolution containing $Co(NO_3)_2$and $Fe(NO_3)_3$ at different Co/Fe molar ratios (2/1, 3/1, 4/1, 5/1) and0.15 mMKNO_3 as supporting electrolyte. The electrochemical reaction was carried out in a single compartment,three-electrode cell, by applying a potential of −0.9V vs.SCE. Different deposition times (30, 60, 80, 120 s) were considered.The electrochemical behaviour of the film deposited on glassy carbon electrodehas been deeply studied in 0.1M NaOH solution.

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Electrochemical detection of trace insulin at poly-Co(Phen)$_3$/Carbon - Nanotube-modified Electrodes

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Abstract

The present study demonstrateselectro-catalytic oxidation and detection of insulin at a glassy carbon electrode chemically modified with MWCNTs and tris(1,10-phenanthroline)cobalt complex, (Co(Phen)$_3$), was thoroughly investigated. The modified electrode was prepared with electropolymerization of Co(Phen)$_3$ by cyclic voltammetry on the surface of the MWCNTs/GC electrode. The effect of various factors such as electropolimerization pH, amount of Co(Phen)$_3$, deposition time, number of cycle electropolimerization and pH of insulin determination were studied and the optimum value for each factor is suggested. The apparent electron transfer rate constant (k_s) and transfer coefficient (α) were determined by cyclic voltammetry and they were about 2.5 s^{-1} and 0.6, respectively. The modified electrode showed excellent electro-catalytic activity toward insulin electro-oxidation at physiological pH value (pH = 7.4). Anodic oxidation of insulin at the poly-Co(Phen)$_3$/MWCNTs-modified glassy carbon electrode occurred at low overpotential (0.6 V versus Ag/AgCl), treatment of the voltammetric data showed that it was a purely diffusion-controlled reaction involving one-electron in the rate-determining step. Under optimized condition in cyclic voltammetry method, the concentration range, detection limit and sensitivity were 0.05 – 7 µM, 0.01 µM and 8.05 µA/µM, respectively. This modified electrode provides aneasy sensitive method for the detection of insulin.

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Electrochemical determination of acetaminophen using a carbon ceramic electrode modified by Orthoferrite Strontium nanocrystal

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Perovskites prepared in the nano-scale have recently received extensive attention due to their catalytic nature when used as electrode modifiers. The high electronic conductivity, magnetic properties, electrically active structure, thermoelectric properties, dielectric property, thermal and chemical stability have made the perovskite type oxides attractive materials for their applications in electrochemical sensors[1]. Recently, the sol–gel derived ceramic carbon composite electrodes (CCEs) have become increasingly used for the fabrication of effective electrochemical sensors and biosensors owing to its simplicity, versatility, physical rigidity, transparency, porosity, efficient encapsulation, and flexibility in the fabrication procedure [2]. Acetaminophen (ACE) is the most extensively employed drug as pain reliever and fever reducer. However, overdoses of ACE cause liver and kidney damage and may lead to death. So determination of ACE is very important [3].

Herein, this paper includes the preparation of a new modified CCE based on Orthoferrite Strontium nanocrystal (SrFeO3 nanoparticles) for the electrochemical determination of ACE. The electrochemical behavior of proposed modified electrode has been studied in phosphate buffer solution of pH 5.0 with cyclic voltammetry. The catalytic peak current obtained, was linearly dependent on the ACE concentrations up to 1360.0 µM with sensitivity of 0.013 µA μM^{-1}. The detection limit for ACE was 0.3 µM. The result demonstrated that the proposed modified electrode exhibited enhanced electrocatalytic activity and good stability for the detection of ACE .Finally, this sensor has been used for the determination of ACE in ACE tablet sample successfully.

Keywords: Orthoferrite Strontium nanocrystals(SrFeO3), carbon ceramic electrode, Acetaminophen (ACE) , electrochemical determination.

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Electrochemical determination of hydrogen peroxide using copper/porous siliconnanocomposite based non-enzymatic sensor

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Copper/porous silicon (Cu/PSi)nanocomposite powder as a new electrode material, have been synthesizedby electrodlessdeposition of coppernanoparticles on the etched PSi powder in a solution containing Hydrofluoric acid and $CuSO_4$.Thenanocomposite wascharacterizedwith different techniques such asscanning electron microscopy (SEM),X–ray diffraction (XRD), FT–IR spectroscopy and cyclic voltammetry (CV). The modified electrode with this nanocomposite showed sensitive and selective electrochemical response to Hydrogen peroxide (H_2O_2) reduction. The effects of different parameters such as pH, electrode composition and applied potential on H_2O_2 reduction were investigated. The new developed non-enzymatic H_2O_2sensor was used for determination of H_2O_2 in neutral media (pH=7) using chronoamperometry. Wide linear range (0.50 µM to 3.78 mM), low detection limit (0.27 µM), fast response (less than 5 second), good signal reproducibility (R.S.D=1.5%.),long term stability (more than one month), good selectivity (no interference from common species such as ascorbic acid, dopamine, uric acid and nitrite), low cost and easy preparation method made this new electrode material a goodcandidate for routine analysis of H_2O_2 in comparison with other non-enzymatic H_2O_2sensors[1,2].

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Electrochemical determination of methyldopa on a Mg-Al layered double hydroxide and CNT modified carbon paste electrode.

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Abstract

Methyldopa is used as a sympatholytic or antihypertensive. Methyldopa is capable of inducing a number of adverse side effects. Side effects may include: Depression and suicidal ideation, as well as nightmares, Decreased alertness, awareness, and wakefulness, Impaired attention, focus, and concentration. So it is important to determine this drug.

Layer double hydroxides (LDHs) also called anionic clays have a layered structure of positively charged metal separated by compensating intercalated anions and water molecules. This material exhibit attractive physical and chemical property such as biocompability, exceptional absorptivity, anion exchange capacity, good catalytic activity, high chemical and physical stability and low production cost. [1]

In addition, CNT can provide the good electrical conductivity and LDH can provide good chemical activity. so mixture of LDH and CNT can improve sensitivity and selectivity.

In this work a biocompatible CNT and Mg-Al-graphene LDH modified carbon paste electrode for the selective determination of methyldopa was constructed.

The electrochemical behavior of the modified electrode was investigated using the cyclic voltammetry method. Chemical parameters such as pH, composition of electrode, potential and time of applied potential were investigated. Optimum values were 2,5%Mg/Al/Graphen-3%CNT,0.4V,180s respectively.

The modified electrode enhances the sensitivity for the detection of methyldopa in comparison to the electrode without LDH. In addition dependence of peak current to scan rate was investigated which was showed the mechanism of the reaction in forward is diffusion based and for backward is adsorptive.

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Electrochemical determination of trace amounts of zearalenone using carbon paste electrode modified with carbon nanotubes

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Abstract

The toxicity of mycotoxins in food and animal feed has been widelyrecognized.Zearalenone(ZEN) is a nosteroidal estrogenic mycotoxinand itcan bind to mammalian oestrogenreceptors, though it is quite similar to estradiol. ZEN inducesoestrogenic effects in mammals and interferes with conception,ovulation, implantation, foetal development and viability of newbornanimals.ZEN can enter into the human food chain directly through foods of plantorigin and indirectly through foods of animal origin[1-2]. Therefore, its determination in real samples is essential.

Inthe present study, for the first time, a novel, simple, rapid and efficient method for the preconcentration and determination of zearalenone was presented by using differential pulse anodic stripping voltammetric method. A carbon paste electrode modified with multi-walled carbon nanotubes was purposed for selective accumulation and determination of trace amounts of zearalenone. The analytical procedure was consisted of a closed-circuit accumulation step onto the modified carbon paste electrode. An anodic peak was observed at 0.40 V, which is related to the oxidation of accumulated and reduced zearalenone on the electrode surface. The calibration graph was linear in the range of 2.00-50.00 ng mL^{-1}. The limit of detection was found to be 0.85ng mL^{-1} and the relative standard deviations for five replicated determinations at 5.00 and 30.00 ng mL^{-1} of ZEN were obtained to be 2.71 and 1.44%, respectively. The modified electrode was applied successfully for the analysis of zearalenone in different beverages samples.

Keywords
Electrochemical determination of zearalenone,Modified carbon paste electrode, Multiwalled carbon nanotubes

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Electrochemical determination of uric acid using a carbon ceramic electrode modified by nanozeolite

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In 1994, Lev and co-workers [1] introduced the sol-gel derived carbon-ceramic electrodes (CCE) kind of electrode had been largely utilized for the design of electrochemical devices whose surfaces could be renewed by a simple polishing step. Now, the sol-gel process conducted in the presence of graphite powder was offered for the fabrication of CCE as a new kind of carbon substrates in electrochemical systems. Considering the stability, permeability, simplicity, easy production, good porosity and especially low cost of carbon-ceramic substrate, it is one of the best materials that can be used as electrocatalyst supporter in modified electrodes. During the last 2 decades, zeolite modified electrodes (ZMEs) have been widely investigated, because of their chemical, physical and structural characteristics which make them of high interest in the design of electroanalytical systems [2]. Uric acid (UA) is the primary end product of purine metabolism. Its abnormal concentration levels will lead to several diseases such as hyperuricemia and gout. Other diseases such as leukemia and pneumonia are also associated with enhanced urate levels [3]. So determination of UA is very important in medicine.

In the present study, a modified carbon ceramic electrode (CCE) was developed with natrolite nanozeolite for the electrochemical determination of UA. Electrochemical behavior of modified electrode was investigated by cyclic voltammetry and chronoamperometry techniques in buffer solution with pH 1. Under the optimized conditions the calibration curve was linear in the concentration range 5–120 μM. The detection limit of UA was 6.2×10^{-7} μM and for seven successive determinations of 50 and 200μM of the UA, the relative standard deviations were 2.1 and 1.3%, respectively. The modified electrode was successfully applied for the electrochemical determination of UA in human urine and serum blood samples.

Keywords: Nano zeolite, Carbon ceramic electrode, Uric acid. Electrochemical determination.

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Electrochemical hydride generation coupled to funnel assisted headspace liquid phase microextraction and UV-Vis spectrophotometry for determination of arsenic

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Hydride generation (CHG) is a well-established technique for determination of toxic arsenic (As) [1]. Electrochemical hydride generation (ECHG) has been introduced as a suitable alternative to common hydride generation by $NaBH_4$, and has been shown, to be capable of circumventing some of the disadvantages associated with the $NaBH_4$-acid reducing system [2]. This paper describes a novel method for the quantitative determination of arsenic in food and environmental samples. The method is based on electrochemical reduction of As to Arsine (AsH_3) in an electrochemical cell and subsequent reaction of AsH_3 with silver diethyldithiocarbamate to give an absorbing complex at 525 nm. The electrochemical generator consisted of a cathode cell separated from the anode cell by a nafion ion exchange membrane. Arsine is generated at the cathode and reacts with silver diethyldithiocarbamate in a headspace microfunnel. The affecting parameters on the efficiency of the electrochemical hydride generation and the microextraction steps have been examined and optimized. The analytical performance of the method has been evaluated under optimized operating condition (graphite cathode, 150 mA current, 0.3 M H_2SO_4 as catholyte, 10 min for reaction duration) and the analytical figures of merits has been reported. As advantage, the system is very fast, efficient and environmentally friendly for monitoring total arsenic food and waters.

Keywords: Electrochemical hydride generation, Arsenic, Funnel assisted liquid phase microextraction, UV-Vis spectrophotometry.

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Electrochemical Hydrogen Storage using Modified Pt Electrode with Graphene Quantum Dots

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As hydrogen is a renewable source of energy, this gas has been categorized as green source [1, 2]. In future, human would have much more environmental problems due to utilizing the fossil fuels. Also, using green source of energies, such as wind, solar and geothermal energies, waste and pollution would be declined [3]. Hydrogen, unlike fossil fuels, is clean energy sources, which does not release harmful gases into the environment. In this study, a novel electrochemical cell was designed using glass and Teflon. A three-electrode system was used to study the electrochemical hydrogen storage behavior of the composite using KOH (~1.0 M) as electrolyte. These electrodes include graphene-modified Pt disk, glassy carbon and Ag/AgCl (3.0 M) as working, counter and reference electrodes, respectively. The experiments were achieved in ambient pressure and temperature. Cyclic voltammetric (CV) measurements were also investigated using Potentiostat–Galvanostat, µAutolab type Ш set in the potential range of -1.0 to +1.0 (vs. Ag/AgCl) during evaluation of hydrogen evolution reaction of nanocarbons. Fig. 1 shows the voltammogram at optimum condition. According to the voltammograms peak shape, well-defined and sensitive peak was observed at potential of ~ 0.85 V (vs. Ag/AgCl). As shown, significant sensitivity of the synthesized graphene is clearly evaluated via following the measured current (between 2-3 mA). Compared to the previously reported articles, graphene is considered as excellent candidate for hydrogen storage and evolution.

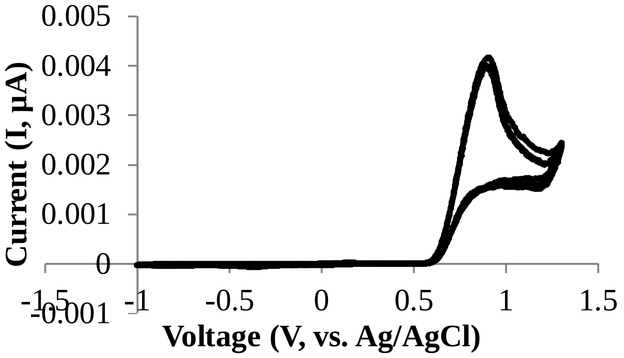

Fig. 1. Voltammogram showing hydrogen storage behavior of graphene.

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بیستمین کنفرانس شیمی تجزیه ایران
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Electrochemical impedance spectroscopy characterization of Graphene/PolyanilineNanocompositesModifiedand its electrocatalytic activity on hydroquinone

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This work explored the effect of graphene surface modification on the electrochemical performance of polyaniline-based nanocomposite[1,2].Electrochemical experiments such as cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) were carried out by using three electrode system where platinum and saturated calomel electrode (SCE) were used as counter and reference electrodes ,respectively.For Cyclic voltammetry (Ivium soft Instrument)was used, For the electrochemical characterizations potential window was chosen from (- 0/2V to 0/2V) ,measurements were performed in 1 M KCl solution at different scan rates from 10 to 200 mV/s. Impedance measurements were also carried out in 1 M KCl solution including hydroquinone. The specific capacitance of the graphene/polyanilinecomposite was 235 F/g, but it decreased to 185 F/g after graphenemodification[3]. The thermal stability of the composite also increased after the graphene modification. The graphene/polyanilinenanocomposite shows much higher impedance in the lower frequency region compared to that of other graphenenanocomposites. In conclusion, the nanocompositebased on modified graphene shows better application potential in supercapacitors or other energy storage devices.

Keywords: impedance spectroscopy, Nanocomposites, Graphene/Polyaniline, hydroquinone

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Electrochemical oxidation of 4-(Piperazin-1-yl)phenols in aqueous and organic solvents

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Abstract

Electrochemistry provides very versatile means for the electrosynthesis, mechanistic and kinetic studies. [1]. On the other hand solvents can have a significant effect on solubility, stability and reaction rates and choosing the appropriate solvent allows for thermodynamic and kinetic control over a chemical reaction. Different solvents can affect the equilibrium constant of a reaction by differential stabilization of the reactant or product. Recently, we have dealt with the electrochemical oxidation of 4-(Piperazin-1-yl)phenols (**1a,b**) in aqueous solution [2]. The oxidation of these compounds is followed by many chemical reactions such as hydrolysis, hydroxylation, and/or dimerization reaction [3]. In this work the electrochemical oxidation of 4-(Piperazin-1-yl)phenols was studied in the water, acetonitrile and nitromethan by means of cyclic voltammetry and controlled-potential coulometry. The results revealed that *p*-quinone-imine generated of oxidation 4-(Piperazin-1-yl)phenols after the hydrolysis reaction participate in Michael-addition reactions with released piperazine of hydrolysis reaction of *p*-quinone imines. The present work has led to the development of a facile and environmentally friendly electrochemical method for the synthesis of some new derivatives of *p*-benzoquinone under green conditions. In addition, the effect of water as a solute on the electrochemical response of 4-(Piperazin-1-yl)phenols (**1a,b**) was examined in the acetonitrile (AN) and nitromethane (NM) solvent.

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Electrochemical Oxidation of N,N,N',N'-tetrmethyl-1,4-phenylendiamine in Non-aqueous Solvents; Evaluation and Interpretation of Redox Potentials

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Electrochemical behavior of N,N,N',N'-tetrmethyl-1,4-phenylendiamine (TMPD) was studied in eight non-aqueous solvents by means of cyclic voltammetry technique. Voltammograms exhibit two consecutive quasi-reversible one electron transfers, which half-wave potential separation ($\Delta E_{1/2}$) is consistent with donor number of solvents. Oxidation of radical cation ($TMPD^{+\bullet}$) to dication ($TMPD^{2+}$) is coupled with chemical follow-up reactions in basic solvents such as DMSO, DMF and MeOH. Values of heterogeneous electron transfer rate constants (k_s) were determined by digital simulation method and solvent dynamical effects on the kinetic of electron transfer processes have been studied. A semi linear relation has been found between $\ln k_s$ and $\ln \tau_L$ of given solvents.

Keyword: N,N,N',N'-tetrmethyl-1,4-phenylendiamine, Solvent effect, Cyclic voltammetry.

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Electrochemical Oxidation of *N,N´*-Diphenyl-1,4-Phenylenediamine in the presence of some thiols as the nucleophiles

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Electrochemical oxidation of *N,N'*-dipheny-l,4-phenylenediamine has been studied in the absence and presence of some thiols derivatives as a nucleophile in water/ethanol (25:75) solutions using cyclic voltammetry and controlled-potential coulometry. The results indicate that, in acidic medium, *N,N'*-dipheny-l,4-phenylenediamine is oxidized in two steps through an *EE* mechanism. The first step is formation of radical cation *N,N'*-diphenylcyclohexa-2,5-diene-1,4-diamine and second step is formation of *p*-quinineimine of *N,N'*-diphenyl-4-phenylenediamine (*N*-(4-(phenylimino)cyclohexa-2,5-dienylidene)benzenamine). The radical cation can participate in disproportionation reaction and produce preliminary *N,N'*-dipheny-l,4-phenylenediamine and *N*-(4-(phenylimino)cyclohexa-2,5-dienylidene. In the presence of thiol derivatives, *p*-quinineimine participates in a Michael addition reaction via an $EC_{disp}C$ mechanism and converts to the organosulfone derivatives. In this work, we prepared thiol derivatives of in good yields using controlled-potential electrochemical oxidation of at a carbon electrode in a divided cell. The obtained products were purified and characterized by spectroscopic IR and MS techniques.

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Electrochemical oxidation of *N*,*N*-dialkyl-*p*-phenylenediamines in the Presence of Sulfite

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Abstract

p-phenylenediamine and related compounds are of particular interest due to their wide range of chemical applications and biological issues. Despite the wide applications of these compounds in processes such as rubber products (as antioxidants), hair dye formulations (as precursor) and in the manufacture of azo dyes (as intermediate), however, they induce oxidative stress and are extreme sensitizer that may lead to contact allergy and dermatitis as well as severe facial swellings [1].

In this work, the electrochemical oxidation of *N*,*N*-dialkyl-*p*-phenylenediamines have been studied in the presence of sulfite ion as a nucleophile in aqueous solution using cyclic voltammetry and controlled-potential coulometry. A plausible mechanism for the oxidation of *N*,*N*-dialkyl-*p*-phenylenediamine and their reactions with sulfite is presented. The results indicate that the quinone-diimine obtained from electooxidation of *N*,*N*-diethyl-p-phenylenediamine [2,3] participats in a Michael type reaction with sulfite ion to form the corresponding sulphonated derivatives. The electrochemical synthesis has been performed at carbon rod electrodes in a divided cell using a constant potential. High product yield and mild reaction conditions are important features of this method.

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Electrochemical Oxidation of Urazole Derivatives in the Presence of 1,2-Dimethyl-1*H*-indol

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Abstract

Indole derivatives are important therapeutic agents in medicinal chemistry including anticancer, antioxidant, antirheumatoidal and anti-HIV, and also play a vital role in the immune system [1]. Some indole derivatives were found to be potent scavenger of free radicals [2]. On the other hand, urazole derivatives were found to be potent cytotoxic agents in murrain and human cancer cell lines and also reduce DNA synthesis considerably with moderate reduction in RNA synthesis [3]. Following our experience in the electrochemical synthesis of organic compounds based on the *in situ* generation of Michael acceptors, we thought that synthesis of new derivatives of indoyl-urazole might be useful from the point of view of pharmaceutical research. In this work, the electrochemical oxidation of 4-substituted urazoles has been studied in the presence of 1,2-dimethyl-1*H*-indol as nucleophile in water/ethanol (60/40) using cyclic voltammetry and controlled-potential coulometry. The results indicate that the electrochemically generated 4-alkyl-4*H*-1,2,4-triazole-3,5-diones participate in Michael type addition reaction with 1,2-dimethyl-1*H*-indol and convert to the corresponding new indoyl-urazole derivatives. The electrochemical synthesis of these compounds has been successfully performed in one-pot process with high yields, without toxic reagents and solvents at a carbon electrode using an environmentally friendly method. The products are identified by ^{1}HNMR, ^{13}CNMR, IR and MS.

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Electrochemical Sensing of Pb^{2+} by Using Fe_3O_4-Graphene Magnetic Nanocomposite

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Abstract

New nanomaterials such as graphene, due to showing specificcharacteristics, are promising candidate for nanoelectronics,catalysis andsensorsapplications[1-3]. Combination of its unique electronic, mechanical, and thermalproperties with other properties such as magnetic property leads to increases application of graphene in new fields. Herein, magnetic graphene and graphene oxide nanosheets were synthesized and characterized by physical, chemical and electrochemical techniques. Interaction of graphene, graphene oxide, and magnetic graphene and graphene oxide nanosheets with two valance metal ions was investigated by tracing square wave voltammetry responses. Both modified carbon paste electrode by magnetic graphene and graphene oxide nanosheets had better response between them, to Pb^{2+} ion. In optimized experimental and instrumental conditions, a linear calibration curve from 1×10^{-9} to 1×10^{-3} M Pb^{2+} with slope as 382.52 **μ**A, r=0.996, and detection limit as 3.07×10^{-10} M Pb^{2+} was observed for modified carbon paste electrode by magnetic graphene nanosheets, and two linear calibration curve from 1×10^{-9} to 5×10^{-6} and from 1×10^{-9} to 5×10^{-6} M Pb^{2+} with slope as 42.51 **μ**A, r=0.994, and slope as 409.63, r=0.998, respectively, and detection limit as 8.13×10^{-10} M Pb^{2+} were observed for modified carbon paste electrode by magnetic graphene oxide nanosheets. Prepared sensors had good sensitivity, reputability, and reproducibility in this work. The data will be presented and discussed.

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Electrochemical sensor based on polypyrrole, sol-gel, and gold nanoparticles nanocomposite molecularly imprinted film for the determination of lorazepam

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A new imprinted electrochemical sensor based on a pencil graphite electrode modified with one-step electropolymerization of the molecularly imprinted polymer (MIP) composed from polypyrrole, sol-gel, gold nanoparticles (AuNPs), for determination of lorazepam was developed. The integration between AuNPs, conductive polymers, and sol-gel technology is a way to take advantage of the properties of all materials [1-3]. In addition, it is possible to design the MIP film properties and polarity through proper selection of the sol-gel precursor and conductive polymer derivatives. This MIP sensor has the advantages of low cost, ease of preparation, higher functional groups, better conductivity, good selectivity and sensitivity. The fabrication process of the sensor was characterized by cyclic voltammetry and electrochemical impedance spectroscopy. The experimental parameters that affect the performance of the MIP sensor were studied and optimized. The MIP sensor was evaluated to verify its electrochemical properties such as conductivity, linearity, selectivity, stability, repeatability and reproducibility. Under the optimized condition the linear range of the imprinted sensor were in the concentration range from 0.2 to 2.0 and 2.0 to 20.0 nM, with the limit of detection of 0.09 nM. Furthermore, the proposed method was successfully intended for the determination of lorazepam in real samples (tablet, plasma, and urine).

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Electrochemical sensor for Amantadine hydrochloride based on a competitive host–guest complexation

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Abstract:

A sensitive and simple electrochemical sensor for the determination of amantadine (AMD), an electrochemically inactive pharmaceutical compound, was constructed. β–cyclodextrin (β-CD) was immobilized on the poly (p-aminobenzene sulfonic acid) (p-ABSA) film modified electrode as a molecular host, ferrocene (FC) was chosen as the electrochemical probe. In the presence of AMD, the captured FC molecules were replaced by AMD via an equilibrium established between the two species, lowering the oxidation current of FC. The extent of current reduction (ΔI) was linearly proportional to AMD concentration in the range of 3.6×10^{-8} M to 1.4×10^{-5} M with a detection limit of 1.0×10^{-8} M. The formation constant of FC and AMD with the immobilized β-CD was estimated. In order to evaluate the applicability of the proposed modified electrode in real sample analysis, it was used in the determination of AMD in blood serum and tablet samples. Standard addition method was used for calculating AMD concentration. Data show that difference between the peak currents of the redox reaction (FC) in the presence and absence of AMD (ΔI) increased with increasing pH from 2 to 7.5, but decreased with the increasing pH more than 7.5. AMD added to solution in low scan rate. because in the low scan rate, time for competitive reaction increase and AM can replace with the FC.

Electrochemical sensor for sensitive determination of acetaminophen using a carbon paste electrode modified by silver nanoparticles

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Silver nanoparticles (AgNPs) due to its excellent surface-enhanced Raman scattering (SERS) activity has attracted much interest in biosensing applications [1]. Silver nanoparticles facilitate acetaminophen oxidation, showing excellent catalytic activity. However, there are no reports for the direct electroanalytical detection of acetaminophen (ACE) with modified carbon past electrodes by AgNPs. Chemical reduction of silver cation in the presence of silica was used for the preparation of silver nanoparticles. Then, AgNPs were characterized by transmission electron microscopy**.**

ACE is an antipyretic and analgesic drug widely used, it is an effective and safe analgesic agent used worldwide for the relief of mild to moderate pain associated with headache, backache, arthritis and postoperative pain. It is also used for reduction of fevers of bacterial or viral origin [2, 3]. So it is very important to establish a simple, fast, sensitive and accurate detection method for ACE.

In the present study, a modified carbon past electrode (CPE) was developed with silver nanoparticle for the electrochemical determination of ACE. Electrochemical behavior of modified electrode was investigated by cyclic voltammetry technique. pH 1.0 was chosen as optimal pH. Under optimal conditions, the differential pulse voltammetry current of the sensor was linear with acetaminophen concentration across a 0.57–410.4 µM range and associated with a detection limit of 0.081µM. significantly, the detection limit is comparable or better than many current electrochemical acetaminophen assays, and the sensor is potentially much cheaper and easier for fabrication. The result demonstrated that the proposed modified electrode exhibited enhanced electrocatalytic activity and good stability for the determination of ACE . The modified electrode was successfully applied for the electrochemical determination of ACE in ACE tablets.

Keywords: Electrochemical sensor, Carbon paste electrode, Silver nanoparticles, Acetaminophen.

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Electrochemical Study of catechols in the presence of 4,6-Dimethyl-2-mercaptopyrimidine

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Electrochemical methods are widely used for the study of electroactive compounds in pharmaceutical forms and physiological fluids due to their simple, rapid, and economical properties [1]. Unique selectivity because of in situ formation of an active species at the interface, inversion in polarity by transfer of electron and variability in product formation by control of potential are some of advantage of electrosynthesis. Michael addition reaction, as a one of the effective organic reaction, has been shown its importance during last years. Electrochemical oxidation of catechols has been studied in the presence of 4,6-dimethyl-2-mercaptopyrimidine as nucleophile in aqueous solution using cyclic voltammetry and controlled-potential coulometry methods. Among the numerous methods to synthesize substituted catechols, Michael-addition reactions play an important role. We derived catechol derivatives in good yields based on controlled potential electrochemical oxidation at carbon electrode in a divided cell.

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Electrochemical study of Haemalum in presence of Phosphate and EDTA

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Abstract

Hematoxylin is extracted from the Central American logwood tree; the dye is classified as a flavone or pigment. A comprehensive review on the chemistry, application and extraction of hematoxylin has been published by Puchtler et al. [1]. Hematoxylin is one of the oldest dyes used in histochemistry. In this work, electrochemical behavior of Hamatoxylin [2] in the presence of $AlCl_3$ and H_3BO_3 has been studied then synthesis of haemalum in the presence of hematoxylin, potassium iodate (oxidizing), distilled water and etc [3]. Upon oxidation, hematoxylin changes color from yellow to dark blue. The electrochemical behaviour of haemalum in the presence of Phosphate and EDTA has been studied using cyclic voltametry as a diagnostic technique. The results indicate that this system is involved in the CE process. CE is an infrequently observed mechanism with individual I - E curves, and it contains a preceding chemical reaction for the production of electroactive species.

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Electrochemical study of the adsorption of 4, 4' biphenol onto a glassy carbon electrode

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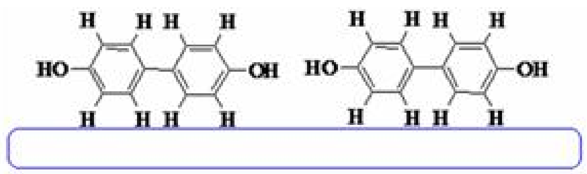
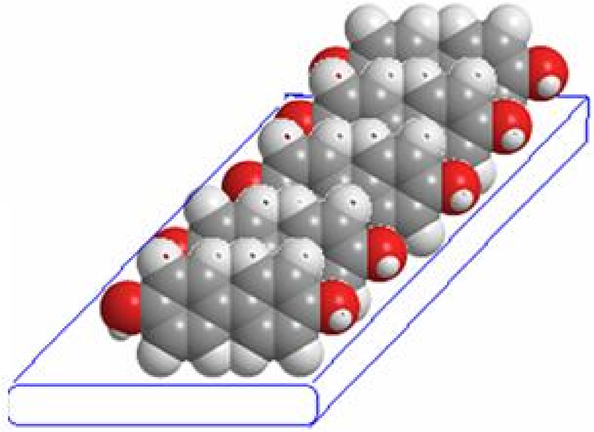
Semiintegral analysis is used as an effective method for determining surface coverage (i.e., surface excess concentration, Γ^*) from cyclic voltammetry in the presence of weak adsorption. Bot surface excess concentration (Γ^*) and bulk concentration (C^*) can be determined from the semiintegration of a single voltammogram. The approach is illustrated using 4, 4' biphenol and its oxidation form (4,4'-diphenoquinone) where the adsorption and diffusion response are indistinguishable, characteristic of weak adsorption. A surface excess concentration (Γ^*) of 0.43±0.06 (for 4, 4'-biphenol) and 0.6±0.047 (for 4,4'-diphenoquinone) nmol/cm^2 was determined via semiintegration and confirmed using chronocoulometry with good agreement [1]. Therefore Semiintegral analysis and chronocoulometry are used as effective methods for determining surface coverage (i.e., surface excess concentration, Γ^{*}) from voltammogram and chronocoulogram in the presence of weak adsorption. Also, advantages and disadvantages of these methods are investigated.

Eventually, a method to determine the orientation of molecules adsorbed at solid-liquid interfaces is described. This method has been applied to determine orientational status of molecules adsorbed from solution onto glassy carbon electrode. Comparison of surface excess concentration measurements with values calculated for various possible molecular orientations indicates the predominant orientations of the adsorbed molecules (4, 4'-biphenol and 4,4'-diphenoquinone) are edgewise. Calculations were based upon covalent and Van Der Waals radii as tabulated by Pauling and were tested against the results of classical adsorption experiments. [3]





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Electrochemical Synthesis of a catechol derivative and determination of captopril and acetaminophen at a nanostructure modified electrode

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Captopril (CA), (S)-1-(3-mercapto-2-methyl-1-oxopropyl)-l-proline, is the first discovered orally active angiotensin- converting enzyme (ACE) inhibitor. The CA is used for the treatment of hypertension, congestive heart failure, and left ventricular dysfunction after myocardial infarction [1]. In this work, we synthesized a catechol derivative using the electrosynthesis method by electrochemical oxidation of catechol derivative in the presence of dimedone as nucleophiles in aqueous solution [2]. The electrochemical behavior of the synthesized compound was studied at carbon paste electrode modified with TiO_2 nanoparticles. The apparent charge transfer rate constant, k_s, and transfer coefficient, α, were calculated. The experimental results indicate that modified electrode offers several advantages such as high repeatability, good stability and high apparent charge transfer rate constant. In the second part of the work, the mediated oxidation of CA at the modified electrode was described. The oxidation peak potentials of CA at modified electrode in cyclic voltammogram was occurred around 120 mV (at pH 7.0) while this peak potential at unmodified carbon paste electrode was appeared around 700 mV at the same scan rate. The values of electron transfer coefficients (α), catalytic rate constant (k), diffusion coefficient (D) and detection limit were calculated for CA, using electrochemical approaches [3]. Finally, this method was used for the determination of CA in real sample.

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Electrochemical synthesis of Fe-Al layered double hydroxide, on glassy carbon electrode: Application for electrocatalytic reduction of isoniazid

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Layered double hydroxides (LDHs) also known as anionic clays, consist of positively charged brucite-like host layers and can be described by the general formula as $[M^{II}{}_{1-x}M^{III}{}_{x}(OH)_2]^{X+}[A^{n-}{}_{x/n}\cdot yH_2O]^{X-}$,where M^{II} and M^{III} represent divalent and trivalent metal ions, A^{n-} is an n-valent anion [1,2].Increasing attention has been paid to layered double hydroxide (LDH) film modifiedelectrode attributing to itsdesirable properties for fabrication of electrochemical sensor [3].

In this research, the Fe-Al LDH film modified glassycarbon electrode was characterized by electrochemical methods.Themodified electrodes with film of the Fe/Al-NO3 double layered hydroxide have been obtained by one-step electrosynthesis. The film of Fe/Al-NO_3LDH was deposited on the electrode surface by cathodic reduction of a solution containing $Fe(NO_3)_3$ and $Al(NO_3)_3$ at Fe/Al molar ratio 3/1 and KNO_3 as supporting electrolyte. The electrochemical reaction was carried out in a single compartment, three-electrode cell, by applying a potential of −0.9V vs. SCE for 90s.

Issonicotinic acid hydrazide or isoniazid (INZ) is a tuberculostatic agent effective against mycobacterium strains widely used for clinical purposes.The electrochemical reduction behavior of isoniazid at the bare electrode and the differential modified electrodes was investigated by cyclic voltammetry.At all the electrodes, isoniazid showed a quasi-reversible electrochemical behavior.The cathodic peak currents of isoniazid obtained at LDH/GCE were a significant higher than at thebare GCE, and the reduction peak potential shiftedpositively to higher potential.The results showed that the modified electrode Fe-Al-LDH/GC can be able to catalyze the electroreductionof the isoniazid.

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Electrochemically Deposition of Sol-Gel/Cu Nanocomposite as a Single Step Technique for Preparation of Solid Phase Microextraction Fibers

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In this study, electrochemically co-deposited sol-gel/Cu nanocomposite is introduced as a novel and single-step technique for preparation of efficient and unbreakable solid phase microextraction (SPME) fibers; having strong interaction between the substrate and the coating. The fibers are prepared without modification of substrate surface step. Details of electrochemical co-deposition procedure is reported elsewhere [1] with some modifications; briefly, the solution for electrodeposition of silica-based sol-gel films consisted of 400 µL of sol-gel precursor, 5 mL of phthalate buffer (1 mM, pH =3.5) containing 0.2 M KNO_3. Hydrolysis was carried out by stirring the above described solution for 2 hr at the room temperature. Cupric ions (0.5-20 mM) were added to the solution at this stage. Then, the electrodes were placed in the electrochemical cell and a constant negative potential (between -0.5 and -1.3 V vs Ag/AgCl) was applied to the electrode for 5-20 min under continuous stirring. The applicability of prepared nanocomposite films was evaluated through extraction of some environmental pollutants as model compounds from the headspace of aqueous samples in combination with gas chromatography–mass spectrometry (GC–MS). Different parameters affecting the structure and composition of the deposited films including applied potential, electrodeposition time, and precursor concentration; and the parameters affecting extraction efficiency such as extraction temperature, extraction time, and salt content were investigated. The results showed that morphology and grain size of the films are strongly affected by the ratio between the sol–gel precursor and Cu^{2+} ions. Furthermore, potential of deposition influences the composition of films as it controls the kinetics of sol–gel/Cu co-deposition.

Finally, characterization of the deposited films was accomplished by scanning electron microscopy (SEM) and thermogravimetric analysis (TGA).

The LOD ranged from 0.005 to 0.010 ng/mL and repeatability at the 1 ng/mL was below 12%. Electrodeposited films were characterized for their surface morphology and thermal stability using SEM and thermogravimetric analysis, respectively. SEM analysis revealed formation of porous substrate and subsequently porous coating on the wire surface and thermogravimetric analysis showed high thermal stability of the prepared fiber.

[1] R. Toledano, R. Shacham, D. Avnir, D. Mandler, Chem. Mater. 20 (2008) 4276.

Electrochemically Deposition of Sol-Gel/Cu Nanocomposite as a Single Step Technique for Preparation of Solid Phase Microextraction Fibers

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Electrochemically oxidation of Captopril Using 4,4'-Biphenol as a Homogeneous Mediator

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Abstract

CSH is a well known drug applied to block an enzyme system, lowering high blood pressure and reducing peripheral resistance. It is also take up free radicals in living systems and exhibit antioxidant properties [1-3].In this work describes the electrochemical behaviour of captopril (CSH), on glassy carbon in the presence 4,4'-biphenol, using cyclic voltammetry as a diagnostic technique. The results indicate a complicated catalytic system. The electrochemical behavior of 4,4'-biphenol as a suitable mediator for the electrooxidation of captopril in aqueous media. The diffusion coefficient of captopril (CSH) and the catalytic rate constant for the catalytic reaction of 4, 4'-biphenol with captopril (CSH) was also estimated using digital simulation method. The other kinetics parameters of this process were calculated in various pHs. The dependence of peak currents on pH, concentration and the potential scan rate was investigated.

Keywords: Electrocatalytic, 4,4'-biphenol, Captopril, Cyclic voltammetry

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Electrochemically pretreated glassy carbon electrode for determination of methimazole in pharmaceutical preparations

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Methimazole (2-mercapto-1-methyl imidazole, MMI), the active metabolite of carbimazole, is one of the most widely used anti thyroid drugs in the treatment of human hyperthyroidism, which inhibits thyroid hormone biosynthesis by preventing the organification of iodide in the thyroid [1]. A simple, inexpensive and rapid electrochemical method for the determination of methimazole, was developed using an electrochemically pretreated glassy carbon electrode (PGCE). It is well known that the electrochemical pretreatment of glassy carbon electrode introduces oxygen-containing functional groups on the electrode surface [2]. The experimental results showed that methimazole exhibited a oxidation peak at 0.4 V. Compared to untreated electrode, the modified electrode showed a large negative shift in the oxidation peak of methimazole (~300 mV) which indicate the electrocatalytic effect of the modified electrode. Cyclic voltammetry was used to study The effects of pH and scan rate on peak current and peak potential for the irreversible oxidation peak of MMI. Square-wave voltammetry on PGCE showed a linear dependence on the concentration of methimazole in the range of 7.44×10^{-5}-1.30×10^{-3}M (pH 7) with a detection limit of 3.77×10^{-5} M (S/N = 3). The voltammetric response was quite reproducible (RSD < 0.41%). The proposed method was successfully applied to the determination of methimazole in thyramozol tablets.

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Electrochemistry and Electrocalalysis of Hemoglobin at Glassy Carbon Electrodes Modified with Dendrimer-Encapsulated Fe-Ni Nanoparticles

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Abstract

Direct electron transfer between an electrode material and a redox-active protein or protein clusters has become an intriguing phenomenon for last three decades [1]. In general, the functions of most proteins depend upon precise three dimensional structures [2], which are affected by a number of noncovalent interactions such as hydrogen bonds, van der Waals interactions, electrostatic interactions, hydrophilic/hydrophobic effects, and soon [3]. Hemoglobin (Hb), is considered to be an ideal model protein for the study of the electron transfer of heme molecules because of its commercial availability, moderate cost as well as well-known and documented structure. However, it is difficult for Hb to exchangeelectron directly with electrode surface due to the inaccessibility ofits electroactive center and loss of electrochemical activities maybeoccurred when Hb adsorbed directly on the electrode surface. In this study, we investigate the direct electron-transfer reactivity of immobilized hemoglobin (Hb) in a Fe-Ni dendrimer-encapsulated nanoparticles (DENs) were prepared within fourth-generation, hydroxyl-terminated, poly(amidoamine) dendrimers and immobilized on glassy carbon electrode. Electrochemical measurements show that the presences of Fe-Ni nanoparticles facilitate the direct electron transfer between the immobilized Hb and the surface conductivity. The biosensor was used to catalyze the reduction of hydrogen peroxide. The proposed method opens a way to develop biosensors by using nanostructured materials mixed with low electrical conductivity matrixes.

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ELECTROCHEMISTRY FOR GREEN ORGANIC SYNTHESIS.

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The twelve principles of green chemistry, include, prevention of the waste, high atom economy, less hazardous chemical synthesis, designing safer chemicals, safer solvents and auxiliaries, using renewable feedstocks, reduce derivatives, catalysis, design for degradation, real-time analysis for pollution prevention, inherently safer chemistry for prevention of accidents. On the other hand, there are several features of electrosynthesis that are often cited as being environmentally favorable. Electrons are considered to be clean reagents to effect on oxidation and reduction reactions; that, under usual circumstances, require potentially polluting metal-based reagents. Such procedures have considerable waste streams which require downstream remediation. In addition, electrosynthesis is characterized by high selectivity, good atom economy, readily available starting materials, low- temperature and energy consumption, low costs for reagents and material failure. Furthermore, the electrodes may be regarded as heterogeneous catalysts that are easily separated from the products. So, it can be concluded that, electrosynthesis is a green tool for organic synthesis. This special issue focuses on using green electrochemical methods for organic synthesis.

KEYWORDS:

Electrosynthesis, Green chemistry, Atom economy, Clean reagents, Heterogeneous catalyst.

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KEYWORDS:

Electrosynthesis, Green chemistry, Atom economy, Clean reagents, Heterogeneous catalyst.

Electrochmical oxidation of *N*,*N*-dialkyl-*p*-phenylenediamines in the presence of the coumarins

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A lot of coumarins have been identified from natural sources. Coumarins have attracted intense interest in recent years because of their diverse pharmacological properties. Electrochemical has emerged as a powerful tool for the synthesis of complex organic molecules.Michael addition reaction of nucleophiles to unsaturated carbonyl compounds is a very useful synthetic method in organic chemistry providing efficient access to a variety of natural products or compounds with pronounced biological activity [1]. Electrochemical synthesis of *N*,*N*-dialkyl-*p*-phenylenediamines [2] has been studied in the presence of 4-hydroxycoumarin and 4-hydroxy-6-methylcoumarin and 4-hydroxy-6,7-dimethylcoumarin [3]. Among the numerous methods to synthesize substituted *N*,*N*-dialkyl-*p*-phenylene-diamines, Michael addition reactions play an important role. In this context, as a part of a program to electrochemical synthesis of novel compounds based on the in situ generation of Michael acceptor. We derived new *N*,*N*-dialkyl-*p*-phenylenediamines derivatives in good yields based on controlled potential electrochemical oxidation at carbon electrode in a divided cell. In this work, also, we compared the obtained products with the products of electrochemical oxidation of *N*,*N*-dialkyl-*p*-phenylenediamines and in the presence of 4-hydroxy-6,7-dimethylcoumarin.

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Electrodeposition of polyaniline/grapheme nanocomposite on composite graphite and investigation of its electrocatalytic activity on resorcinol

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Abstract

The polyaniline/graphene (PANI/GR) nanocomposites modified composite graphite electrode was prepared by electrochemical synthesis of aniline monomer and graphene oxide in phosphoric acid. The modified electrode based on polyaniline/graphene (PANI/GR) nanocomposite for investigation of its electrocatalytic activity on resorcinol is presented. The influence of PH and resorcinol concentration on the electrocatalytic ability of the PANI/GR composite modified electrode was also studied. Fourier transform infrared spectroscopy (FTIR), SEM and UV/Vis absorption spectra are used to demonstrate non-covalent interactions between ANI and GR in the PANI/GR particle.The high electrocatalytic ability of PANI/GR is established through cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) and liner sweep voltammetry.This electrochemical sensor shows a favorable analytical performance for resorcinol with detection limit of 10^{-6}mM with correlation coefficient (R) of 0.9897 and short response time(11s) were observed by PANI/GR composites modified thin film electrode and very high sensitivityof 10^{-3}mM.

Keywords: Resorcinol; Graphene; Polyaniline; Modified electrode; Electrocatalytic

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دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

Electrodeposition of polyaniline/graphenenanocomposite on composite graphite and investigation of its electrocatalytic activity on resorcinol

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Abstract

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Keywords: Resorcinol; Graphene; Polyaniline; Modified electrode; Electrocatalytic

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Electrodeposition of quercetin on the electrospun zinc oxide modified carbon past electrode for enhanced electrochemical determination of uric acid

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A distinctive voltammetric method has been belt up for determination of uric acid (UA) at a carbon paste electrode modified with zinc oxide nanofibers and quercetin. Zinc oxide nanofiber (ZNF) was fabricated via electrospinning and subsequent calcination process at 510 ^{o}C [1]. The synthesized ZNFs were characterized by X–ray powder diffraction (XRD), transmission electronic microscope (TEM) and scanning electronic microscope (SEM). The electrode was made up through the electrodeposition of quercetin on a Zinc oxide nanofibers carbon paste electrode (Q/ZNF/CPE). The Q/ZNF/CPE offered substantially lower over potential for electro-oxidation of UA in acetate buffer solution (ABS) (pH 4.45) compared with Q/CPE, ZNF/CPE and bare CPE. Furthermore the effects of pH, scan rate, accumulation time and potential, interferences effect and effect of quercetin electrodeposition on the surface of ZNF/CPE were studied in details [2]. The studies suggested that electrode reaction process is totally irreversible and adsorption controlled. Differential pulse voltammogram peak current of UA increased linearly with its concentration in two dynamic ranges of 0.2–1 μM and 5–15 μM with a sensitivity of 6.0185 and 0.2012 μA/μM and detection limits for UA was obtained to be 0.05μM with RSD = 0.2 %. The prepared electrode was applied for determination of UA levels in serum and plasma of a healthy person and who has leukemia. The results indicate that the proposed method is sensitive, selective, fast and simple for determination of UA.

Key words: Electrospining, Electrodeposition, ZnO nanofiber, uric acid, quercetin

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Electromambrane surrounded solid phase microextraction using electrochemically synthesized poly pyrrole fiber

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Solid phase microextraction (SPME) is known as a popular method for extraction of analytes from aqueous or gaseous samples [1]. However, this method faces some problems in analysis of nonvolatile or ionizable species in complicated matrices due to fiber saturation with interferences. Therefore, combination of electromembrane extraction (EME) and SPME was introduced for the first time in 2013 by Rezazadeh et al. termed electromembrane surrounded solid phase microextraction (EM-SPME) [2]. In the present work, EM-SPME using conductive polymers as the sorbent was carried out for the first time for analysis of some antidepressant drugs as the model analytes. Polypyrrole is one of the most studied conducting polymers as a result of simple preparation procedure, high conductivity and relative stability.The organic liquid membrane was immobilized in the pores of a hollow fiber (HF) and the basic analytes were migrated in an electrical field from aqueous sample solution through the liquid membrane and into aqueous acceptor phase and then they were adsorbed on the solid sorbent, which acts as the cathode. Effective parameters such as composition of organic liquid membrane, pH of donor and acceptor phases, applied voltage and extraction time were optimized for extraction of amitriptyline (AMI) and doxepin (DOX) and figures of merit of the method were investigated in human plasma, and urine samples. Regardless of high sample cleanup, which make the proposed method suitable for analysis of drugs from complicated matrices, clean chromatograms were obtained in comparison with previous EM-SPME work. Also, admissible repeatability and reproducibility (intra- and inter-assay precision ranged between 3.2-7.8% and 7.0-12.8%, respectively) were obtained in different extraction media. Linearity of the method was studied up to 500.0ng mL^{-1}for both of the analytesand correlation coefficients higher than 0.9935 were achieved. Finally, the proposed method was applied for analysis of AMI and DOX in different real samples and reasonable data were obtained.

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Electromambrane surrounded solid phase microextraction using electrochemically synthesized poly pyrrole fiber

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Electromembrane extraction using cylindrical electrode: A new view for augmentation of extraction efficiency

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Electromembrane extraction (EME) as one of liquid phase microextraction (LPME) methods was introduced by Pedersen-Bjergaard et al. in 2006 [1]. EME has many advantages in comparison with hollow fiber based liquid phase microextraction (HF-LPME) and provides more efficient extractions and sample clean up [2]. However, EME suffered from some drawback such as low repeatability and extraction efficiency, high voltage usage and it is dangerous, SLM instability and sparking. Driving force for separation of ionizable components in EME is the applied voltage which causes formation of electrical field between electrodes. Potency of electrical field has a close relationship with the extraction efficiency. However, to the best of our knowledge there is no currently report of employing electrodes with different geometric shape for enhancement of extraction efficiency. In this work, the two shapes of electrodes (wire electrode versus cylindrical electrode) as outer electrode (the electrode in donor phase) were used and the effect of electrodes shape on the performance of EME extraction was examined. Mefenamic acid (MEF) and diclofenac (DIC) have been used as model analytes.

Effective parameters such as composition of organic liquid membrane, pH of donor and acceptor phases, applied voltage and extraction time were optimized for extraction of MEF and DIC and figures of merit of the method were investigated in human plasma and urine samples. Regardless of high sample cleanup, high SLM stability and low voltage usage without any sparking were obtained. Effective parameters on EME such as extraction time, applied voltage and acceptor/donor phases composition are optimized using the experimental design. Under optimized conditions preconcentration factors in the range of 47–355 and recoveries in the range of 94–105 were obtained in various biological matrices. The linear dynamic ranges of 2.5–500 ng mL^{-1} (with correlation coefficient higher than 0.9986) and limits of detection of 1.0 ng mL $^{-1}$ were obtained for both of the model analytes.

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Electropolymerization of 2,4-dinitrophenol on MWCNT/Glassy carbon modified electrode and using it in adsorption stripping voltammetry for monitoring of Hg^{2+} in some lagoons South of Caspian Sea

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The goal of electro organic synthesis is efficient production of pure compounds with unique structures and properties. In this direction, it is possible to electro synthesis some functional polymers which can act as ligand [1,2].

In this work 2,4-dinitrophenol was electropolymerized on lead cathode in aprotic medium, in a separated cell under constant current. After separation and purification of electrode-product, it was characterized by C.V, FT-IR, DSC. In another experiment 2,4-dinitrophenol was polymerized on MWCNT/Glassy carbon modified electrode by cyclic voltammetry. This new modified electrode showed high tendency to adsorb Hg^{2+}.

Therefore it was used as a new electrode in adsorption stripping anodic voltammetry (AdSAV). All parameters effecting the performance of AdSAV like pH, time of adsorption, effect of various cation and anion, buffer system and ionic strength were studied and optimized. This method was applied for monitoring of Hg^{2+} in water of some lagoons south of Caspian Sea. The limit of detection and qantitation were 0.3 and 1.1 ng.ml^{-1}.The limit of detection (LOD) of the proposed method was lower than ICP-OEC.

Keyword: Electropolymerization. 2,4-dinitrophenol. absorption stripping voltammetry. mercury ion in lagoons.

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Electropolymerized polypyrrole/multiwalled carbon nanotubes/TiO_2 composite on stainless steel wire for solid-phase microextraction of BTEX coupled to GC-FID

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Abstract

A novel solid-phase microextraction (SPME) fiber based on polypyrrole-carbon nanotubes-titanium oxide (PPy-CNT-TiO_2) composite is coated onto stainless steel wire, was prepared via electrochemical techniques. The fiber was used for the headspace solid-phase microextraction (HS-SPME) of benzene, toluene, ethylbenzene and *o*-xylene (BTEX), followed by gas chromatography-flame ionization detector (GC-FID). The morphology of the fiber surfaces was investigated by scanning electron microscopy (SEM) and Energy Dispersive X-ray Spectrometer (EDS). Due to the unique properties of TiO_2 [1], CNTs and the inherent advantageous features and also the performance of the conductive polymer coating [2], this innovative fiber exhibited porous surface structure, reproducible preparation, high sensitivity, long lifetime, high thermal stability (over 400 ^{o}C) and strong adhesion of the coating to the steel wire. The new PPy-CNT-TiO_2 fiber is fairly simple to prepare, robust, with good extraction efficiency. Important parameters influencing the extraction efficiency such as desorption temperature and time, extraction temperature, extraction time, stirring speed and salt effect were investigated and optimized [3]. Under the optimal conditions, the method detection limits (S/N=3) were in the range of 0.01–0.04 ng mL^{-1} and the limits of quantification (S/N=10) between 0.03–0.09 ng mL^{-1}. The relative standard deviations (RSDs) for one fiber (repeatability) (n=5) were obtained from 3.5 up to 5.6% and between fibers or batch to batch (n=3) (reproducibility) in the range of 5.9–8.1%. The developed method was successfully applied to real water samples while the relative recovery percentages obtained for the spiked water samples were from 93.7 to 106.2%.

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Electrospun acrylonitrile-maleic acid copolymer nanofibers as a solid-phase extraction sorbent for the determination of trace Pb and Ni in environmental water

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Present analytical and separation methods can resolve practically all kinds of the analytes with detection limits down to the femtogram range. However, their sensitivity and selectivity are usually insufficient for direct determination of the target compound at a very low concentration level in complex matrix samples. Therefore, a sample pretreatment step prior to analysis is usually necessary. solid-phase extraction (SPE) has offered several advantages in terms of selectivity, extracts, reproducibility, and avoidance of emulsion formation [1, 2]
electrospun nanofibers, which possess high surface area than have great analytical potential as an effective SPE sorbent. Electrospinning is a process that easily produces a polymer with nanoscale fibrous structures. As a result, these fibers possess a high aspect ratio that leads to a larger specific surface [3, 4].

This paper describes the novel synthesis and preparation of acrylonitrile-maleic acid copolymer nanofibers through the use as a solid-phase extraction (SPE) sorbent to directly extract Pb and Ni from environmental water. The target compound was then monitored by a atomic absorption system. Parameters of influencing the extraction efficiency, such as fiber packing amount, eluted solvent, pH and ionic strength were investigated. Under the optimized conditions, a linear response for Pb and Ni over the range of 20–2000 ngmL^{-1} were achieved with a γ2 value of 0.9996, 0.9932. The precision of the method was examined with relative standard deviations of 2.7, 2.2% corresponding to 50 ngmL^{-1} for spiked Pb and Ni, respectively. The relative recoveries of 93.7–104.2% were obtained. The limit of detection was determined to be 8 ngmL^{-1} and 2 ngmL^{-1}. A comparison of the SPE using nanofibers as sorbents and the most commonly used octadecylsilica SPE cartridges was carried out in terms of absolute recovery, sensitivity, and reproducibility for the compounds investigated. Finally, the method was applied to four real water samples.

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Electrospun based nanofibers for on-line micro solid phase extraction of clodinafop propargyl from water, soil and wheat samples

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Sample preparation is an important step towards isolating the desired compounds from complex matrices which eventually influences on the reliability and accuracy of the analysis.

Preparation of new sorbents with improved entrapment properties for trace analysis is a demand and is highly focused by many researchers [1]. To apply a high electrostatic field to polymer solution to produce non-woven webs of ultrafine fibers with the diameters ranging from few nanometers to sub-micrometers is regarded as an interesting approach. This approach is the base of the electrospinning technique that has been widely investigated in the past decade as a low-cost and convenient method to produce microfibers and nanofibers for various applications [2]. Also, electrospun nanofibers have been employed for the extraction purposes due to their inherent high aspect ratios [3].

In the present work, an on-line extraction-determination device was set up for micro solid phase extraction (μ-SPE) of clodinafop propargyl from water, soil and wheat samples using an electrospun polyamide nanofibers-base sorbent. The prepared nanofibers were packed in a stainless steel tube which conveniently acted as a high performance liquid chromatography (HPLC) injection loop. The obtained scanning electron microscopy (SEM) data revealed that the synthesized nanofibers have sufficient homogeneity and porosity with diameters between 500 to 550 nm. Influential parameters affecting the extraction efficiency were optimized using a distilled water sample fortified with 25 μg L^{-1} of clodinafop propargyl. An enrichment factor of 440 achieved for clodinafop propargyl indicating the ability of the developed technique. Under the optimum condition, the linearity for the analyte was in the range of 6-700 μg L^{-1} while the limit of detection (LOD) and limit of quantification (LOQ) of 2 and 6 μg L^{-1} could be conveniently achieved, respectively. The relative standard deviation (RSD%) of the whole procedure was 5.8 when water samples were spiked at 25 μg L^{-1} from the selected analyte. To investigate the matrix effect, the developed method was applied to real water samples including paddy and river waters as well as wheat and soil samples. The relative recovery percentages for the spiked samples were in the range of 63 to 95 %.

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Electrospun carbon nanofiber solid-phase microextraction coatings for preconcentration of Chlorobenzenes prior to gas chromatography-electron capture detector

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Chlorobenzenes are used as industrial solvents, pesticides, dielectric fluids, deodorant, and chemical intermediates. They can enter the aquatic environment through solid and liquid effluents and atmospheric discharges. Because they are relatively resistant to abiotic and biotic degradation they tend to persist in the environment. Chlorobenzenes are essentially ubiquitous in the aquatic environment; they can be detected in water, sediment and aquatic biota [1].

Electrospinning is a simple, inexpensive technique that createsnanofibers through an electrically charged jet of polymer solutionor melt. This method should provide anapproach to generate high surface area fibers and the versatilityto alter the SPME fiber composition by changing the polymersolution for electrospinning [2,3].

In this study, SPME fibers were produced by electrospinning of polyacrylonitrile on aliminium wires and then the nanofibers were pyrolyzed to generate carbon nanofiber SPME coatings. The fiber was then applied for preconcentrationand determination of 1,4-dichlorobenzene (1,4-DCB), 1,2,3-trichlorobenzene (1,2,3-TCB) and 1,2,4-trichlorobenzene (1,2,4-TCB) in water samples. Various factors which influenced the recovery of the analytes were investigated such as: fiber thickness, salt addition, extraction time, extraction temperature, desorptiontemperature and time. Detection limits were 10, 40, and 400 ng/Lfor 1,2,4-TCB, 1,2,3-TCB, and 1,4-DCB, respectively. All RSDs were <9% (forn = 5). The proposed method was applied for the analysis of chlorobenzes in surface waters.

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Electrospun modified polyamide nanofibers as a novel fiber coating for rapid determination of chlorophenols

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In solid phase microextraction, sampling, extraction and preconcentration are performed in a single step. The developments of new fiber coatings are mostly focused on improving the thermal, mechanical and chemical stability, imparting diverse functionalities and polarities, and enhancing the fiber capacity. Electrospinning is a simple and convenient method for producing nanofibers with adjustable diameters, polarities and porosities. They have malleability to conform to a wide variety of shapes and sizes [1]. To follow our research interests [2], a mixture of PA and polyethylene glycol (PEG) was synthesized via electrospining and the resultant nanfibers coating was employed for headspace solid phase microextraction (HS-SPME) and rapid determination of chlorophenols. The major polymer solution contained PA along with PEG, acting as a side low molecular weight polymer. After synthesizing the PA−PEG fiber coating, PEG was subsequently removed by water (modified PA), confirmed by Fourier transform infra-red spectrometry. The scanning electron microscopy images showed an average diameter of 640 and 148 nm for PA and PA−PEG coatings, respectively while the latter coating structure was more homogeneous and porous. The extraction efficiencies of PA, PA-PEG and modified PA fiber coatings were assayed by isolation of a number of chlorophenols from real water samples. Eventually the extractants were analyzed by gas chromatography-flame ionization detector and gas chromatography-mass spectrometry. The effect of added amounts of PEG to the main PA constituent and the flow rate of electrospinnig solution were also studied. Various influential parameters associated with the extraction efficiency such as the salt content, desorption condition, extraction temperature and time were optimized. The detection limits of the method were in the range of 0.8−25 ng L^{-1}, while the relative standard deviations (RSD %) at two concentration levels of 200 ng L^{-1} and 80 ng L^{-1} were between 2.1 and 12.2%. The calibration curves for the selected phenols were linear in the range of 8−1500 ng L^{-1}. The analysis of real water samples led to relative recoveries values between 85 and 98% for all the selected phenols.

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Electrospun titania sol-gel based ceramic composite nanofibers for on-line μ-SPE-HPLC of naproxen and clobetasol from biological samples

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Titanium (IV) tetraisopropoxide (TTIP) was employed as the metal oxide sol-gel precursor to prepare ceramic composite nanofibers by electrospinning system. The sorption characteristic was investigated through a micro solid phase extraction (μ-SPE) set up in on-line combination with high performance liquid chromatography (HPLC). To facilitate the electrospinning process, poly(vinylpyrrolidone) (PVP) was also added to the sol of titania. Electrospinning system was employed to assist the sol-gel process to obtain higher aspect ratio nanofibers with the enhanced surface area. Four ceramic composites nanofibers based on titania were prepared. Sorbent 1 was prepared by addition of Fe and Co to TTIP; sorbent 2 included composite of Fe and Mn added to TTIP; sorbent 3 was a composite of Fe and Co added to TTIP and finally, sorbent 4 was prepared by addition of mixture of Co, Mn and Ni and Fe to TTIP precursor. The images obtained from a scanning electron microscope (SEM) revealed the good homogeneity of all sorbents with the high aspect ratio which originated from the essence of the electrospinning process. Naproxen and clobetasol were selected to evaluate the extraction efficiencies of the prepared sorbents. The obtained data showed that all sorbents have good efficiencies to preconcentrate the analytes and the type of metal incorporated in the titania-based composite structure has no significant effect on the extraction efficiency. One of the sorbents, Sorbent 2, was chosen as a representative for further analysis and validation of the developed method. The linearity of the analytes was in the range of 1-1000 μg L^{-1}. LOD was found to be 0.3 and 2 μg L^{-1} for naproxen and clobetasol, respectively.

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Electrosynthesis of a catechol derivative and its application as an electrocatalyst for simultaneous determination of levodopa and carbidopa at a multi wall carbon nanotubes modified electrode

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The modification of electrodes using redox modifiers is an interesting field in analytical chemistry. Redox modifiers are electroactive compounds that effectively shuttle electrons between the analyte and the electrode [1]. In this research, a catechol derivative multi−wall carbon nanotube modified carbon paste electrode was constructed and used as an excellent electrocatalyst to oxidation levodopa (LD) and carbidopa (CD). In the first part of the work, we synthesized a catechol derivative using the electrosynthesis method by electrochemical oxidation of catechol derivative in the presence of dimedone as nucleophiles in aqueous solution [2]. A modified carbon paste electrode based on an electro synthesized compound of catechol derivative and carbon nanotubes (CNTs) was prepared. Cyclic voltammetry was used to investigate the redox properties of this modified electrode at various scan rates. Next, the mediated oxidation of LD at the modified electrode was described. At the optimum pH of 7.0, the oxidation of LD occurs at a potential about 330 mV less positive than that of an unmodified carbon paste electrode. Based on differential pulse voltammetry (DPV), the oxidation of LD calculates a dynamic range and a detection limit. DPV was used for simultaneous determination of LD and CD at the modified electrode, and quantitation of LD and CD in some real samples by the standard addition method.

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Electrosynthesis of heparin doped polypyrrole and electrochromic characterization

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Abstract

An electrochromic material is the one that changes color in a persistent but reversible manner by an electrochemical reaction and the phenomenon is called electrochromism. Conducting polymers (CPs) are finding increasing use in electrochromic applications because of their cathodic, anodic and multicolor control, ease in processability, flexibility, rapid redox switching, high optical contrast, and long-term stability [1]. The physical material properties such as color, conductivity, and surface energy can be controlled by electrochemical doping in a CP. By doping a CP, new energy levels are created within the band gap due to generation of polarons and bipolarons in the polymer bulk [2]. The electrochromic properties of CPs have became of great importance in modern reversible electro-switching optical design of polymer based optical sensors and devices. In this paper, we report on the electrochemical preparation of the conducting polypyrrole (PPy) synthesized with entrapped heparin (Hep) anions. Heparin, a widely used anticoagulant, is a highly sulfated linear natural polysaccharide [3]. The electrochromic properties of polypyrrole film are difficult to exploit due to the dissatisfactory stability. We have chosen Hep as the doping anion since its polyanionic nature predicts that it would increase the stability of PPy film. The Hep-doped PPy was electrochemically synthesized on a fluorine doped tin oxide FTO/glass electrode in aqueous solution. The resultant polymer was characterized via electrochemical and spectroelectrochemical methods. The spectroelectrochemical analysis revealed the PPy-Hep has distinct electrochromic properties with respect to the PPy. Polyanion-doped polymer exhibited a switching time of less than 1.5 s and the maximum transmittance contrast (ΔT%) is 48% at 800 nm. In addition presence of Hep causes drastic enhancement of electro-optical stability of PPy. The introduction of Hep polyanion can lead to enhance the formation of an ordered PPy-based film which can be reason for improvement of stability.

Keywords: Polypyrrole; Heparin; Electrochromic; Optical sensors; Spectroelectrochemistry.

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Enhanced electrochemiluminescence [Ru(bpy)$_3^{2+}$] with Sm$_2$O$_3$ nanoparticle and its sensing of L-Proline

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Abstract:

The analysis of l-proline has attracted considerable attention because of its important physical functions, broad distribution in nature and wide application in human life. For monitoring and warning on food stuff security and health, multifarious methods based on spectrophotometric, colorimetric, chromatographic and electrochemical techniques have been developed for determination of amino acids[1], some of them are complex, time consuming and require tedious sample pretreatment, and the process limits the application of these[2]. A novel, sensitive, rapid and inexpensive [3] electrochemiluminescence (ECL) detection of l-proline at Tris(2,2’-bipyridyl)ruthenium(II) [Ru(bpy)$_3^{2+}$]-Sm$_2$O$_3$ nanoparticle-modified carbon paste electrode was developed. The immobilized [Ru(bpy)$_3^{2+}$]-Sm$_2$O$_3$ nanoparticle-modified CPE shows good electrochemical and photochemical activities. The modified electrode showed an electro-catalytic respond to the oxidation of l-proline. The optimized condition for the highest intensity is pH= 8.5 (0.1M PBS). The electrochemical and electrochemiluminescence characterizations will be made by cyclic voltammetry and electrochemical impendence spectroscopy. The sensor showed good selectivity and long term stability.

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Enhancement of bimetallic Fe-Mn /CNTs nano catalyst activity and product selectivity using microemulsion technique

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Abstract

Bimetallic Fe-Mn nano catalysts supported on carbon nanotubes (CNTs) are prepared using microemulsion technique with different water-to-surfactant ratios of 0.4-1.6. The nano catalysts were extensively characterized by different methods (such as XRD, TPR, TEM, and BET) and their activity and selectivity in Fischer–Tropsch synthesis (FTS) have been assessed in a fixed bed micro-reactor. The physico-chemical properties and performance of the nanocatalysts were compared with the catalyst prepared by impregnation method. Very narrow particle size distribution has been produced by the microemulsion technique at the relatively high loading of active metal. The TEM images showed that small metal nano particles are confined inside the CNTs and located on the outer surface of the CNTs (3–7 nm). Using microemultion technique with water to surfactant ratio of 0.4 decreased the average iron particle sizes to 5.1 nm. The percentage reduction and percentage dispersion are almost doubled. Activity and selectivity were found to be dependent on the catalyst preparation method and iron average particle sizes. The CO% conversion and FTS rate increased from 49.09 to 70.98% and 0.144 to 0.289 (gr. HC/gr. cat./hr), respectively.While the WGS rate decreased from 0.097 to 0.056 (gr. CO_2/gr. cat. /hr). C_5^+ liquid hydrocarbons selectivity decreased slightly and olefins selectivity almost doubled.

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Enhancement of carbon nanotubes supported Cobalt Fischer-Tropsch synthesis nano catalyst performance using microemulsion technique

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Abstract

Cobalt nano catalysts supported on carbon nanotubes (CNTs) are prepared using microemultion technique with water-to-surfactant ratios of 0.5-1.5. The nano catalysts were extensively characterized by different methods and their activity and selectivity in Fischer–Tropsch synthesis (FTS) have been assessed in a fixed bed micro-reactor[1]. The physico-chemical properties and performance of the nanocatalysts were compared with the catalyst prepared by impregnation method[2]. Very narrow particle size distribution has been produced by the microemulsion technique at the relatively high loadings of active metal (15 wt.% Co). According to the TEM pictures small Co particles (2–7 nm) are mostly confined inside the CNTs. Comparing with the catalyst prepared by impregnation; using microemultion technique with water to surfactant ratio of 0.5 decreased the average cobalt oxide particle sizes to 4.9 nm, the %dispersion is almost doubled and the %reduction increased by 27.1%. Activity and selectivity were found to be dependent on the catalyst preparation method and water to surfactant ratio (as well as on cobalt particle sizes)[3]. The %CO conversion and FTS rate increased from 56.12 to 73.08 and 0.280 to 0.362 (gr. HC/gr. cat./hr), respectively. While the CH_4 and light gaseous hydrocarbons selectivities increased and the C_5^+ liquid hydrocarbons selectivity decreased slightly.

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بیستمین کنفرانس شیمی تجزیه ایران
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Evaluation of binding selectivities of some calix[4]arene derivatives towards alkali metal cations using electrospray ionization mass spectrometry

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Calix[4]arenes are complex macromolecules with specific structural features that have been synthesized to enhance metal recognition properties. Electrospray ionization mass spectrometry (ESI/MS) can be used as an efficient analytical characterization technique to evaluate structure/selectivity correlations for host-guest complex between these macromolecules and target metal cations [1]. In this work, binding selectivities of some calix[4]arene derivatives toward alkali metal cations have been studied using ESI/MS. In the mixtures of different guest ions of K^+, Rb^+ and Cs^+ and a specific calix[4]arene derivative, the intensities of the host-guest complexes at M/Z values in the mass spectra are correlated with the relative binding selectivities. The concentration of each component in the mixture was 2×10^{-4} M. All complexation experiments were performed in LC/MS grade methanol in order to remove source of trace metal interferences. A syringe pump was used to deliver the solutions to electrospray needle in direct infusion mode at flow rate 0.45 mL/h and needle voltage was held at 3.5 kV. Validity of the ESI/MS results was confirmed by comparison of the experimental and calculated selectivities of dibenzo-18-crown-6 (DB18C6) as reference crown. The calculated selectivities of reference crown were obtained by solving a set of 3 nonlinear chemical equilibrium equations. It was observed that selectivity trends of calix[4]arenes are significantly influenced by chemical identity and steric hindrance of functional groups in their upper and lower rims.

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Experimental and Computational Study of the Optical Electron Transfer in D5 and D35 Dye-Sensitized Solar Cells Based on Cobalt Redox System

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A combined experimental and computational investigation to understand the nature of the interactions between cobalt II/III redox mediators and their impact on the performance of the corresponding dye-sensitized solar cells (DSCs) are reported. In this project we have used the density functional theory (B3LYP/6311++G**) computational method to optimize molecular structure and electronic interactions of a series of organic dye sensitizers and cobalt complex redox mediator, and to calculate the infrared (IR), raman and vibrational circular dichroism (VCD) spectra. In the experimental section of the project, dye sensitized solar cells are fabricated by D35 organic dye as a sensitizer and cobalt tris bipyridine as a redox mediator system. Parameters of the prepared DSCs are investigated by the current density-voltage (J-V) and incident photon to current efficiency (IPCE) measurements. The J-V curves results show enhancement of the short circuit photocurrent density (J_{sc}), fill factor (FF) and overall efficiency of the DSCs with titania thin layer film as a blocking layer (prepared by sol-gel method). The experimental results in this project are in good agreement with the computational results.

Keywords: Dye-sensitized Solar Cell; TD-DFT; Cobalt Complex; Redox Mediator; Marcus Diagram.

Experimental design for the optimization of dispersive liquid-liquid microextraction followed by HPLC-DAD determination of Cr (III) and Co (II) based on the complexation reaction with morpholin -4-carodithioate

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Abstract

In this study, a dispersive liquid-liquid microextraction (DLLME) method was used for preconcentration of Cr (III) and Co (II)cations from water samples. Morpholin-4-carodithioate (MDTC) was used as a chelating agent prior to extraction. High-performance liquid chromatography-diodearray detection (HPLC-DAD) was used for quantitation of the analytes after preconcentration.The effect of extraction, nature and volume ofdisperser solvent, pH value of sample solution, extraction timeand extraction temperature were investigated[1,2]. Under of optimum conditions the calibration graphs were linear in the range of (10-4000µg L^{-1}) with detection limits of 3µg L^{-1} for Co(II) and (5-4000 µgL^{-1}) and 3µg L $^{-1}$ for both Cr(III). Recoveries and enrichment facture for Cobalt and Chromium were obtained (90%, 63.69) and (84%, 58.42), respectively.The DLLMEHPLC-DAD method was successfully applied to the analysisof Cr (III) and Co (II) in aqueous samples.

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Abstract

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Extraction and concentration of trace insecticides Diazinon and Chlorpyrifos of water by dispersive liquid-liquid micro-extractin(DLLME) and measured by gas chromatography

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Abstract : A rapid and sensitive method has been established for the determination of Diazinon and Chlorpyrifos in water samples by using dispersive liquid–liquid microextraction coupled with Gas Chromatography[1]. In this method, a mixture of 700 µL methanol (disperser solvent) and 70µL n-heptan (extraction solvent) was rapidly insected by a syringing to 8 water sample that containing 100 ng/lit Two pesticides were (analyte). After, 10 min (extraction time), samples were centrifugation for 5 minutes, then extract phase (organic) designed on top of the test tube, was collected and the aqueous phase was separated. After this step, the extracted material with a syringe 10 µL was injected the gas chromatograph. The effects of some parameters affecting extraction, including : volume of extraction solvent, volume of disperser solvent, carrier gas flow rate, extraction time, salt increasing the rotation speed of the centrifuge were optimized. Concentration factor of 114 was achieved under optimal conditions. The calibration curve in the range of 10-100 ng/ml for diazinon and range of 1-100 ng/ml for chlorpyrifos was linear. Detection limit of this method for diazinon 1.24 ng/ml and for chlorpyrifos 0.155 ng/ml relative standard deviation (RSD %), for 5 repeated measurements for diazinon 2.25% for chlorpyrifos 2.36%, respectively.

The proposed method successfully to measure trace amounts of two pesticides (diazinon and chlorpyrifos), in water samples were used. The main advantage of using dispersive liquid-liquid microextraction: simplicity, speed, low cost, high concentration factor, good repeatability and low consumption of solvent extraction.

Key word: Dispersive liquid-liquid microextraction , Gas chromatography, Diazinon, Chlorpyrifos.

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دانشگاه صنعتی اصفهان ، ۶ الی ۸ اسفند ماه ۱۳۹۲

Extraction and determination of iodide by hollow fiber based liquid-liquid-liquid microextraction followed by flow injection, photochemical reaction and UV-Vis detection

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Abstract

Iodide and iodate is used by the thyroid gland to make iodo-hormones essential for brain and neural development in fetuses and infants. A diet deficient in iodine can result in a variety of growth and developmental problems and when severe may cause cretinism. Several analytical methods have been applied for the determination of iodate and iodide, such as: electrochemistry, ion chromatography, ICP-MS and ICP-AES. In the present work, a very simple, rapid and sensitive flow injection spectrophotometric method has been described for the determination of iodate and iodide.

Over the last years, some new miniaturized extraction procedures have been introduced with success, which can be grouped in the so-called liquid-phase microextraction (LPME) techniques [1]. LPME emerged from liquid–liquid extraction (LLE) which is probably the most widely used sample extraction procedure despite its clear disadvantages such as high consumption of time and solvents. In general, the LPME technique combines extraction, concentration and sample introduction in one step.

In this study, hollow fiber based liquid-liquid-liquid microextraction (HF-LLLME) followed by flow injection and spectrophotometric detection was applied as a simple method for the extraction and determination of iodide in milk and river water. For the determination of iodate in edible salt, the iodate was converted to iodide. The extraction of iodide was carried out using a cationic surfactant (Aliquat 336) which was dissolved in hexyl acetate as an organic phase for filling the porous of the hollow fiber wall and 0.1 M ClO4- solution as acceptor phase in the lumen of the fiber. Iodide was oxidized to iodine in a photochemical reaction coil. A mercury lamp was used as a UV source for the reaction. The iodine showed a maximum absorption at 445 nm. The factors affecting the HF-LLLME including type of organic solvent, concentration of surfactant, type of acceptor phase, concentration of acceptor phase, ionic strength, volume of donor, sample stirring rate and extraction time were optimized. Effect of length of the reaction coil and carrier flow rate were also investigated. Analytical parameters including: intra-and inter-day relative standard deviation (4.5% and 11%, respectively), linear dynamic range (5-1000 µg L-1) and enrichment factor (350) were calculated.

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Extraction and determination of nitrite by hollow fiber based liquid-liquid-liquid microextraction followed by flow injection analysis and spectrophotometric detection

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Abstract

High concentration of nitrite in drinking water, rivers and lakes is a critical hazard to human health and environment. Nitrite mostly uses as preservatives. Irreversible reaction of nitrite with hemoglobin, may causes methemoglobin disease, which leads to reduce the blood capacity of oxygen transporting. Nitrite also can harm the nervous system. So determination of nitrite is very important due to it's harmful effects on human health. In this case a fast, sensitive, selective, and inexpensive method is required for determination of nitrite[1-2].

Hollow fiber based liquid-liquid-liquid microextraction (HF-LLLME) followed by flow injection analysis and spectrophotometric detection was used as a simple and sensitive method for determination of nitrite in water and food samples. The extraction of nitrite was carried out by using isoamyl benzoate containing 4% surfactant as the organic phase for filling the pores of the hollow fiber, and 0.25 M NaI solution as acceptor phase in the lumen of the fiber. After that, extracted nitrite was reacted with sulfanilic acid and phenol to form an azo dye, showing a maximum absorption at 440 nm. The factors affecting the HF-LLLME, including type of organic solvent, concentration of surfactant, type and concentration of acceptor, extraction time and stirring rate were investigated. Moreover, the interference of some anions and cations in determination of nitrite were also studied. The enrichment factor, linear rang and the limit of detection were obtained 75, 2-500 and 0.6 µg/L, respectively for 4 mL of sample solution. The method was applied for the determination of nitrite in various water and food samples such as potato and sausage.

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Extraction and determination of pyridine derivatives in human urine using electromembrane extraction coupled to dispersive liquid–liquid microextraction followed by gas chromatographic analysis

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There are most of pyridine derivatives in cigarette smoke which have relatively high toxicity. So it is important to diagnosis smokers for many agencies or clinical centers. The presence of these toxic compounds in cigarette smoke has been recognized as an important issue for development of different methods for the analysis of these compounds, but most of these methods were performed in water and not complicated matrices such as biological fluids. Sample preconcentration and cleanup must be carried out in complicated matrices. A large number of modern sample preparation techniques such dispersive liquid-liquid microextraction (DLLME) [1] and electromembrane extraction (EME) [2] methods have been introduced. Recently, Yamini et al. have coupled EME to DLLME so that they benefited from high cleanup ability of EME method as well as DLLME compatibility with GC instrument [3]. In this work, EME-DLLME is used for extraction and determination of some of pyridine derivatives (3-mthylpyridine, 2, 4-lutidine, quinoline and 4-dimethylaminopyridine) in urine samples. To this end, ionized forms of the analytes were first extracted into an aqueous acceptor phase using EME method. Then, DLLME was applied to transfer the target analytes into final organic phase which is GC compatible. Both of EME and DLLME variables were optimized. The SLM was a mixture of 90% NPOE and 10% di-(2-ethylhexyl) phosphate (DEHP). An electric field of 100 V was applied to transfer the analytes into acidic acceptor solution with pH 1.0. Acceptor solution were transferred to 1 mL of the alkaline solution (pH = 13) and then DLLME procedure was performed. Chloroform and methanol were used as extraction and disperser solvent, respectively. Preconcentration factors in the range of 40-263 and satisfactory repeatabilities (2.3 < RSD% < 5.3) were obtained in different matrices. The method was applied for determination and quantification of pyridine compounds in smokers' real samples. The proposed technique can be introduced as a simple, fast and inexpensive method for diagnosis smokers.

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Extraction and determination of sunset yellow acidic artificial dye in fruit drink by polyamide based magnetic nanosorbent and UV-Vis spectrophotometry

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Abstract:

Soft drinks and orange juices are very popular all over the world. Sunset yellow (yellow 6; Disodium 6-hydroxy-5-(4-sulfophenyl) azo-2-naphthalenesulfonate , E110) was used in food and drinks as color additives earlier but due to its adverse effects on human health their determination in different food matrices, during the recent years has received increasing attention all over the world. However, this harmful color additive is being used in commercial orange drinks. Yellow 6 is acidic and soluble in water and methanol. Sunset Yellow has been previously evaluated by the Joint FAO-WHO Expert Committee Report on Food Additives (JECFA) and the Scientific Committee on Food (SCF). Both committees established an Acceptable Daily Intake (ADI) of 0-2.5 mg/kg bw/day. E 110 side effects are gastric upset, diarrhea, vomiting, nettle rash, swelling of the skin and migraines, linked to hyperactivity in young children. Thus its determination in foods is crucial. In this work, the polyamide based magnetic nanosorbent was used for Sunset Yellow assay in fruit drink .Sunset yellow color adsorbed by magnetic polyamide nano adsorbent and then the analyte was desorbed from nanosorbent with solvent methanol and ammonia mixed and determined quantitatively with UV-Vis spectroscopy . The UV-visible absorption maxima of yellow 6 are 482 nm. The effective parameters such as pH of feed and desorbed solvent, donor phase volume (7.0 mL), desorption solvent volume (3.0 mL) and extraction time (5.0 minutes) were investigated. Method was evaluated statistically and real samples were tested according the optimum conditions.

Keywords: Polyamide based magnetic nanosorbent, Sunset Yellow, UV-Vis spectroscopy.

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Extraction and high preconcentration of triazole pesticides residues in honey using a new microextraction method based on elevated temperature dispersive liquid-liquid microextraction prior to gas chromatography-nitrogen phosphorous detection

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Sample preparation is a tedious and yet unavoidable procedure in analytical chemistry [1]. The objective of this challenging and critical step is to transfer the analyte into a form that is pre-purified, concentrated and compatible with the analytical system [2, 3]. Extraction can vary in degree of selectivity, speed and convenience and depends not only on the approach and conditions used but also on the geometric configurations of the extraction phase [4]. In the present study, a rapid, highly efficient, and reliable sample preparation method named elevated temperature dispersive liquid-liquid microextraction followed by gas chromatography-nitrogen/phosphorus detection was developed for the extraction, preconcentration and determination of five triazole pesticides (penconazole, hexaconazole, diniconazole, tebuconazole, and difenconazole) from honey samples. This method consists of a dispersive liquid-liquid microextraction technique which is assisted by temperature. In this method the temperature of high volume aqueous phase is adjusted at an elevated temperature and then a disperser solvent containing an extraction solvent is rapidly injected into it. After cooling to room temperature the phase separation is accelerated by centrifuging. Various parameters affecting the extraction efficiency such as type and volume of the extraction and disperser solvents, temperature, salt addition, and pH were evaluated. Under the optimum extraction conditions, the method showed low limits of detection and quantification between 0.05 – 0.21 ng g^{-1} in honey (15 - 70 ng L^{-1} in solution) and 0.15 –1.1 ng g^{-1} in honey (45 - 210 ng L^{-1} in solution), respectively. Enrichment factors and extraction recoveries were in the ranges of 1943 – 1994 and 97 – 100 %, respectively. The relative standard deviations for the extraction of 1.5 ng g^{-1} of each pesticide were less than 6 % for intra-day (n = 6) and inter-days (n = 4) precisions. Finally the method was successfully applied to determination of analytes in honey samples and difenconazole was determined at ng g^{-1} level.

Key words: Elevated temperature dispersive liquid-liquid microextraction, Gas chromatography

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Extraction of Carvacrol from *Satureja khuzestanica* using MSPD method and measured with GC-MS chromatography

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Carvacrol as a phenolic compound is the major component in *Satureja khozestanica* essential oil. Matrix-solid phase dispersion (MSPD) method coupled with gas chromatography flame ionization detector (GC-FID) as a quick and easy extraction technique has been developed to extract carvacrol from plants and herbal medicines [1-2]. Influence of important parameters on MSPD method efficiency, such as the sorbent material, the ratio of sample to sorbent material, elution solvent, and volume of the elution solvent have been evaluated and optimized. Carvacrol was successfully extracted by diatomaceous earth as sorbent with 350 μL of dichloromethane as elution solvent. The calibration curve showed good linearity (r^2=0.9965) and precision (RSD<8.16%) in the concentration range of 0.5–100 μg/mL for carvacrol. The limit of detection (LOD) and limit of quantification (LOQ) were 3.0 and 10 μg mL^{-1}, respectively. The recoveries were in the range of 74.4–80.5% with RSD values ranging from 8.4 to 9.8%. The reported MSPD extraction method revealed to be simpler and faster than conventional methods used to quantify carvacrol from plants and herbal medicines.

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Extraction of methocarbamol from biological fluids with magnetic nanoparticles decorated polypyrrole/multiwalled carbon nanotubes as adsorbent followed by electrospray ion mobility spectrometry

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Abstract: A new type of solid phase extraction that is a topic of great interest to researchers is called magnetic solid-phase extraction (MSPE). By using magnetic modified adsorbents, the extraction capacity and efficiency increases, because of the significantly higher surface area-to-volume ratio and the magnetic adsorbents can be easily removed from sample solution with the assistance of an external magnetic field [1]. In this work, magnetic nanoparticles decorated polypyrrole/multiwalled carbon nanotubes (Fe_3O_4-PPy/MWCNTs) was chemically synthesized [2] and applied as a novel adsorbent for the preconcentration of methocarbamol in biological fluids followed by electrospray ionization ion mobility spectrometry (ESI-IMS). The properties of Fe_3O_4-PPy/MWCNTs adsorbent were characterized by scanning electron microscopy, fourier transform infrared spectroscopy and x-ray diffraction. In this study, in addition to the above mentioned adsorbent, two different magnetic nanoparticles adsorbents, i.e. Fe_3O_4-MWCNTs and Fe_3O_4-PPy were also evaluated to extract the various drugs with different log K_{ow} such as methocarbamol, metoprolol, chloramphenicol, dextromethorphan and cyproheptadine. The results showed that the extraction efficiency of Fe_3O_4-MWCNTs and Fe_3O_4-PPy for extraction of compounds with lower log K_{ow} such as methocarbamol, methoprolol and chloramphenicol were lower than Fe_3O_4-PPy/MWCNTs. The hydrophilic (PPy) and hydrophobic (MWCNTs) properties of the Fe_3O_4-PPy/MWCNTs adsorbent improved extraction efficiency of the polar analytes. The effect of parameters on the extraction efficiency for the preconcentration of methocarbamol by Fe_3O_4-PPy/MWCNTs adsorbent were investigated. Under optimized conditions, the linear dynamic range was found to be 1-100 ng/mL with detection limit of 0.3 ng/mL in water samples. The relative standard deviation was 5.9% for three replicate measurements of methocarbamol at 50 ng/mL in water samples. This method was applied for determination of methocarbamol in biological samples.

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Extraction of Oleuropein from *Olea uropea* using MSPD method and measured with HPLC

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Matrix solid-phase dispersion (MSPD) methodology as a quick and easy extraction method has been developed to extract oleuropein from *Olea europaea* leaves. This method has been compared with conventional maceration and Soxhlet extraction (SE) methods [1]. Variables affecting MSPD, such as the sorbent material, the ratio of sample to sorbent material, elution solvent, and volume of the elution solvent have been fully evaluated and optimized. Oleuropein was successfully extracted by silica as sorbent with 350 μL of dichloromethane- methanol (70:30, v/v) as elution solvent. The calibration curve showed good linearity (r^2=0.9985) and precision (RSD<4.5%) in the concentration range of 0.5–100 μg/mL for oleuropein. The limit of detection (LOD) and limit of quantification (LOQ) were 0.14 and 0.5 μg/mL, respectively. The recoveries were in the range of 88.0–94.0% with RSD values ranging from 6.0 to 9.8%. The extraction yields obtained by the MSPD method were comparable with those obtained by the maceration and SE extraction methods. The reported MSPD extraction method revealed to be eco-friendly, simpler and faster than conventional methods used to quantify oleuropein in *Olea europaea* leaves.

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Extraction of trace amounts of Cr(VI) from tannery waste water by magnetic nano particle supported by ionic liquid and followed by ICP-AES determination.

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Chromium in the form of hexavalent ion, Cr(VI), is a highly toxic species for human and micro organisms. In addition, Cr(VI) is also known by the US-EPA as well as other agencies as a carcinogenic agent [1]. On the other hand, the most naturally occurring species of chromium is trivalent chromium, Cr(III), which known as an essential trace element for some systems. Therefore, such variation and vibration between toxicity and nontoxicity of chromium species must direct the attention of researchers toward finding possible methodology to identify and determine the accurate concentration of each species of chromium with high precision and accuracy.

Direct determination of Cr(VI) ions at trace level is limited due to low concentration and matrix interferences. Therefore, in trace analysis, to improve the detection limits, preconcentration of trace elements is frequently necessary. Room temperature ionic liquids (RTILs) are generally composed of organic cations and inorganic anions and are liquids at room temperature. They have been shown to be suitable reaction media for synthesis, catalysis, electrochemistry, separation and extraction, chromatography, enzyme immobilization [2], etc. magnetic nanoparticles which have recently appeared as a new type of catalyst supports because of their good stability, easy synthesis and facile separation by magnetic fields have been used to immobilize ionic liquids [3].

In the present study preconcentration of hexavalent chromium by new SPE with mechanism of anion exchange has reported in the waste water of Charmshahr industrial town (Varamin) which has a lot of tannery. Determination of the extracted chromium was performed by ICP-AES. The effect of different parameters such as sample volume, extraction time, desorption time, desorption volume, desorption solvent, pH and amount of nanoparticle on the extraction efficiency of chromium was investigated and optimized. Under optimized conditions extraction recoveries in the range of 10-33% and satisfactory repeatability (RSD 3-5%) were obtained. LOD of 0.1 μg L^{-1} and linear dynamic range of 0.1 to 25 μg L^{-1} (with correlation coefficient better than 0.9979) were obtained.

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Extraction of trace amounts ofCr(VI) from tannery waste water by magnetic nano particle supported by ionic liquid and followed by ICP-AES determination.

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Chromium in the form of hexavalent ion, Cr(VI), is a highly toxic species for human and micro organisms. In addition, Cr(VI) is also known by the US-EPA as well as other agencies as a carcinogenic agent[1]. On the other hand, the most naturally occurring species of chromium is trivalent chromium, Cr(III), which known as an essential trace element for some systems. Therefore, such variation and vibration between toxicity and nontoxicity of chromium species must direct the attention of researchers toward finding possible methodology to identify and determine the accurate concentration of each species of chromium with high precision and accuracy.

Direct determination of Cr(VI) ions at trace level is limited due to low concentration and matrix interferences. Therefore, in trace analysis, to improve the detection limits, preconcentration of trace elements is frequently necessary. Room temperature ionic liquids (RTILs) are generally composed of organic cations and inorganic anions and are liquids at room temperature. They have been shown to be suitable reaction media for synthesis, catalysis, electrochemistry, separation and extraction, chromatography, enzyme immobilization[2], etc. magnetic nanoparticles which have recently appeared as a new type of catalyst supports because of their good stability, easy synthesis and facile separation by magnetic fields have been used to immobilize ionic liquids[3].

In the present study preconcentration of hexavalent chromium by new SPE with mechanism of anion exchange has reported in the waste water of Charmshahr industrial town (Varamin) which has a lot of tannery.Determination of the extracted chromium was performed by ICP-AES.The effect of different parameters such as sample volume, extraction time, desorption time, desorption volume, desorptionsolvent, pH and amount of nanoparticle on the extraction efficiency of chromiumwas investigated and optimized.Under optimized conditions extraction recoveries in the range of 10-33% and satisfactory repeatability(RSD 3-5%) were obtained.LOD of0.1 µg L^{-1} and linear dynamic range of 0.1 to 25µg L^{-1}(with correlation coefficient better than 0.9979) were obtained.

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Extraction of ultra-trace amounts of iron by ion pair formation-solid phase extraction method and determination by ETAAS

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Abstract

Iron is the fourth most abundant element in the earth’s crust. It is present in a variety of rock and soil minerals [1]. Iron is necessary for hemoglobin synthesis and oxidative processes of living tissues, as it exists at the active site of molecules responsible for oxygen transport and mitochondrial electron transfer and provides a fundamental structure of myoglobin, hemenzymes and many co-factors involved in enzyme activities [2]. Iron also plays a key role in some important infectious diseases such as malaria. Total iron concentration should not exceed 5 mg L^{-1} in order to meet EU legislation [3]. In present investigation, a new and green separation method based on ion pair formation-solid phase extraction (IP-SPE) combined with ETAAS was developed for the first time to the preconcentration and determination of iron in several real samples. It was used the ion pair formed between $FeCl_4^-$ with benzyldimethyltetradecyl ammonium chloride dehydrate as an ion pair forming agent that immobilized on the surface of multi-walled carbon nanotubes. Under the optimized conditions, the detection limit of 0.17 ng L^{-1}, calibration graph of 0.7-9.4 ng L^{-1} and the precision of 5.3% were obtained. The method was applied to the determination of iron in human hair and certified reference material: (MA-1b reference gold ore).

Keyword: Iron, Solid phase extraction, Ion pair

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Fabricating of humidity sensor by graphite/SnO_2 thin film Synthesized by spray pyrolysis technology

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Abstract

Highly sensitive G/SnO_2 semiconductor for humidity sensor was fabricated on glass substrates by spray pyrolysis methods [1]. The characterization of these composite materials with Furrier Transform Infra Red (FT-IR) scanning electron microscopy (SEM), X-ray diffraction (XRD) and energy dispersive X-ray spectroscopy (EDX) points to the formation of crystalline SnO_2 on graphite sheets demonstrated. Humidity sensors based on semiconductor thin film exhibit excellent sensitivities with resistance varying from 1500 Ω to 11 kΩ over a relative humidity (RH) range of 11%–95% at room temperature. The end result of measuring resistance and Response – Recovery behavior of the humidity sensor are also discussed in detail. It is also observed that pyrolysis temperature results in increasing of sensitivity and reduction of recovery time up to 70% in sensor with 50 nm, while the response time is 60 s for the sensor. Stability of the sensor demonstrated in long period of time.

Keywords: humidity sensor, graphite/SnO_2 thin film, spray pyrolysis technology

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Fabrication a platform of chitosan-carbon nanotube composite for DNA-Cu complexes interaction

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The interaction of two new water-soluble [Cu(4,7-dmp)(phen-dione)Cl]Cl (1) and [Cu(2,9-dmp)(phen-dione)Cl]Cl (2) which dmp is dimethyl-1,10-phenanthroline and phen-dion represents 1,10-phenanthroline-5,6-dion, with immobilized ss and dsDNA on a chitosan–carbon nanotubes composite modified glassy carbon electrode were investigated by cyclic voltammetry technique. In solution interactions, spectroscopic and electrochemical evidences indicated outside binding of these complexes. In detail, these two complexes (1) and (2) interact with DNA via groove binding and partially intercalative modes, respectively. The electrochemical characterization experiments showed that the nanocomposite film of chitosan–carbon nanotubes could effectively immobilize DNA and greatly improve the electron-transfer reactions of the electroactive molecules that latter finding is the result of strong interactions between captured DNA and Cu complexes. In addition to, the significant differences in the electrochemical signals obtained at the ssDNA- and dsDNA-modified electrodes suggested that these copper complexes could be electroactive probes to differentiate between ssDNA and dsDNA in biosensing analysis [1]. A precise understanding of DNA binding properties is essential for their potential applications as chemical probes of nucleic acid conformation and drug design [2,3].

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Fabrication of Platinum Nanoporous Gold Film Electrode for Elector Catalytic Determination of Glutathione

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Abstract

Metal nanopore structure particularly gold[1] platinum [2] and nickel [3] nano structure film are attracting much interested due to their applications in catalysts chemical and electrochemical sensors. This research involves three main parts. The first part focuses on fabrication of platinum coated nanoporous gold film electrode (PTNPGF) via a three step process. In part two of this research the effective parameters on fabrications of electrode were evaluated and optimum condition was reported. In part three of the electro catalytic activity of PTNPGF electrode determination was compared with a smooth gold electrode a nanoporous gold electrode and a pt-decorated smooth gold electrode in the same conditions. The observed result exhibit that PTNPGF electrode has the best electrode. At the optimized conditions, the linear range for the determination of glutathione was 4.62×10^{-5} to 4.84×10^{-9}. The detection limit was 3.1×10^{-9} mol L^{-1} Glutathione. The RSD% for Glutathione were 3.3%.

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Fabrication of a Novel Carbon Dioxide Sensor via Modification of a pH Glass Electrode with Inner-Shell Membrane of Bird’s Egg

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Abstract

To increase the efficiency as well as the reducing emissions of hazardous gases, instantaneous information about the composition of gases is essential [1, 2]. For this purpose, sensing methods are considered as appropriate approaches that are adopted to monitor gas compositions instantly. In this study, a novel potentiometric carbon dioxide (CO_2) sensor was fabricated via modification of a pH glass electrode with inner-shell membrane of a birds ‘egg. According to the figures of merits, the fabricated sensor gave a linear output from 180 to 1320 parts per million (ppm). Detection limit was also ~21 ppm. Relative standard deviation (RSD) for seven replicate analyses was 2.37%. Based on 90% of maximum response (t_{90}), the response time was ~1.5 min with recovery time equal to ~6.3 min (t_{90}). Calibration sensitivity was measured to -0.0016 pH/ppm (~-57.91 mV/decade). No interference was observed, during introducing at most 1000 folds of foreign gas species such as O_2, H_2, CO, O_3 and volatile organic compounds (VOCs) such as C_2H_2 and CH_4 to 500 ppm of CO_2 solution. Acceptable flexibility was evaluated for the introduced membrane, during its humidifying with water. The lifetime of the humidified membrane was at least 10 days. Reliability of the sensor was also evaluated via determination of CO_2 in different air samples.

Keywords: CO_2 gas sensor; Glass membrane electrode; Potentiometry; Inner-shell membrane; Bird’s egg.

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Fabrication of a Selective Electrochemical Sensor for Determination of Acetaminophen in Some Drug Formulations and Biological Fluids Based on Glassy Carbon Modified with Multi-Walled Carbon Nanotubes

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Abstract

Since the discovery of carbon nanotubes (CNT) in 1991, research on CNT has progressed rapidly, and has became one of the most attractive part of nanotechnology [1]. Carbon nanotubes are molecular-scale wires with high electrical conductivity, high chemical stability, and extremely high mechanical strength and modulus [2]. There have been many new applications due to their unique chemical, electronic and mechanical properties. Recently, electrochemical studies suggested the ability of CNT to promote certain kinds of electron-transfer reactions [3], to minimize surface fouling, and to enhance electrocatalytic activity.

In this work, a highly sensitive electrochemical sensor for the determination of acetaminophen was fabricated using Multi- walled carbon nanotubes (MWCNTs) modified glassy carbon electrode. We observed that the modified electrode has shown higher sensitivity than bare glassy carbon electrode and produced a pair of redox peaks at 0.213 to 0.625 V (Ag/AgCl). Some parameters as kind of supporting electrolyte, pH, volume of nanotube and scan rate optimized for achieving to higher sensitivity. It was shown that at pH 9 of Britton Robinson buffer, 20 μl of MWCNTs and 100 mV s^{-1}, the calibration curve was linear in the ranges of 0.1 to 30 μM and 30 to 100 μM. The fabricated electrochemical sensor was successfully applied to the determination of acetaminophen in some drug formulations, whole blood and plasma.

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Fabrication of an electrochemical sensor based on computationally designed molecularly imprinted polymers for determination of mesalamine in real samples

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Abstract

A novel electrochemical sensor based on mesalamine (MESAL) molecularly imprinted polymer (MIP) film on a glassy carbon electrode was fabricated. This film was cast on glassy carbon electrode by electrochemical polymerization in solution of o-phenylenediamine (OP), gallic acid (GA), p-aminobenzoic acid (ABA) and template mesalamine via cyclic voltammetry scans and further deposition of Ag nanoparticles (Ag NPs) [1]. Density functional theory (DFT) was developed to study the intermolecular interactions in the polymerization mixture and to find suitable functional monomers in MIP preparation. DFT results show that spin multiplicity of electronic ground state mesalamine before and after complex forming with functional monomers of OP, GA, ABA compounds remain singlet [2,3]. Theoretical result with DFT approach with polarizable continuum model and electrochemical experimental results are agreement in all cases. The surface feature of the electrode modified with Ag NPs molecularly imprinted polymer (Ag NPs-MIP/GCE) was characterized by cyclic voltammetry (CV), scanning electron microscopic (SEM) and electrochemical impedance spectroscopy (EIS). Under the optimal experimental conditions, the peak currents were proportional to the concentrations of mesalamine in the range, from 0.025 to 100 µM, with the detection limit of 0.015 µM. Due to the high affinity, selectivity and stability the Nano Ag imprinted sensor provides a simple detection platform for mesalamine real samples.

Keywords: Mesalamine; Molecular imprinted polymer; Electropolymerization; Computational design; Continuum and Onsager reaction field models; Silver nanoparticles

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Fabrication of Bismuth-chitosan modified glassy carbon electrode and its application in measurement of Tartrazine synthetic dye in food and drug samples

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Abstract:

Bismuth-film modified electrodes (BiFE) have been used as a suitable alternative for dropping mercury electrodes (DME) since 2000 [1,2]. These electrodes have high hydrogen over potential and limited oxygen interferences.

Synthetic dyes have wide applications in various industries such as food, drug and cosmetics. In comparison to natural colors, synthetic dyes have advantages such as:high stability when they are exposure to light, pH and oxygen, being monotonous, low biological contamination and low price[3].Tartrazine(Trisodium(4E)-5-oxo-1-(4-sulfonatophenyl)-4-[(4-sulfonatophenyl) hydrazono]-3-pyrazolecarboxylate) is a synthetic dye and is used in food and drug samples. Tartrazine is also known as E102 and acid yellow 5. Allowed uses amount of this color is receiving daily 7.5 mg per kilogram of body. But overuse of it in products can cause harmful effects on human health. So measuring this color that is used in many productsespecially food and drugs, is the matter of this research.

In this study, glassy carbon electrode is modified with bismuth by ex-situ method. Moreover, this electrode is modified by chitosan. Because chitosan is known for following properties: biological compatible with other elements and non-toxicity. Also it doesn't contaminate electrode's surface. Effects of some parameters such as applied potential, bismuth concentration, pH, electrolyte concentration and deposition time were studied. The optimum conditions are: acetate buffer solution 0.1M (pH=4.5) containing 0.18 mg/ml Bi^{+3} ions by electrolysis at -0.75 V vs.SCE for 200s,while stirring the solution.

Electrochemical behavior of Tartrazine was investigated on the modified electrode.Also, Tartrazine was determined in food and drug samples on bismuth-chitosan modified glassy carbon electrode by differential pulse voltammetry (DPV) technique.

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Fabrication of electrically-conducting polypyrrole /polyvinylpyrrolidone core–shell nanofibers via electrospinning

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Nanomaterials are nowadays considered as well established and interesting class of materials due to their small sizes. This property can improve and bring new features which are somehow impossible to achieve with bulk materials [1]. Some of these properties include unique electronic/optic properties such as plasmon resonances in nanoparticles, superparamagnetic behaviors for magnetic nanoparticles, extraordinary high surface area per unit mass, together with remarkable high porosity, excellent structural mechanical properties, high axial strength combined with extreme flexibility, low basis weight and cost effectiveness, appropriate platform for the efficient separation, extraction and enrichment [2, 3]. The electrospun nanofibers have found a great deal of applications in fabricating nanofibers. Core-shell nanofibers in particularly have gained many attentions, which is due to their great potential applications and platforms for nanochannel, nanocapsule and small encapsulating devices. Electrospinning of conductive polymers such as polypyrrole (PPy) is traditionally difficult which is due to its low solubility in most organic solvents. Core-shell electrospinning of PPy, with an appropriate polymer like polyvinylpyrrolidone (PVP) overcomes these problems, providing a simple method for the electrospinning of PPy. In this study firstly, a homemade coaxial electrospining setup was designed and constructed. The solutions for the core and shell materials were separately fed into the coaxial nozzle from which they were ejected simultaneously by utilizing one syringe pump. An amount of 0.1 g ferric chloride was added to dimethylformamide (DMF)/ethanol (1:1), After dissolving the salt, the pyrrole monomer was added to the solution, and the mixture was stirred for 24h to prepare the PPy solution. Also 20%w/v of PVP in DMF/ethanol (1:1) was prepared by dissolving PVP in the mentioned mixture solvent and stirred for an hour. Two polymeric solutions were drawn into two 1mL syringes separately and were connected to the core-shell setup and eventually placed in the syringe pump. A piece of aluminum foil was employed as a collector electrode. The coaxial nozzle of the constructed core-shell and the collector were connected to the terminals of the power supply. A distance of 12 cm was set between two electrodes. By applying a voltage of 15 kV between two electrodes, the core-shell nanofibers could be formed on the surface of the aluminum collector. A flow rate of 0.1 mL h^{-1} was used for the polymer solution pumping.

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Fabrication of electrochemicalcocaineaptasensorbased on immobilization of aptameronto glassy carbon electrode modified withgraphenenanocomposite

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The detection and quantification of trace amounts of illicit substances such as cocaine to be important for law enforcement and clinical medicine. Cocaine is a powerfully addictive stimulant drug that increases the level of dopamine, a brainchemical associated with pleasure and movement, in the brain'sreward circuit. Abusing cocaine has a variety of adverse effectson the body. Cocaine constricts blood vessels, dilatespupils, and increases body temperature, heart rate, and bloodpressure[1]. Thus, a simple, rapid, sensitive and selectivemethod for the determination of cocaine is of great interest.

Aptamers are specific single-stranded DNA or RNA molecules that have been selected in vitro from large randomized oligonucleotide libraries bySELEX (systematic evolution of ligands by exponential enrichment) [2].Very similar to antigens and antibodies, aptamers can bind specifically to abroad range of targets such as small molecules, proteins, amino acids, drugs and even viruses and whole cells.Owing to their numerous advantages such as easy labeling, good stability, reproducibility for synthesis and so on, aptamers are considered to be ideal candidates as molecular recognition elements in biosensors [3]. Among various aptamer-based biosensors, electrochemical aptasensors are proved to be powerful analytical tools in the detection of proteins and small molecules due to high sensitivity and selectivity as well as simple instrumentation.

The present study describes the fabrication of a novel electrochemical aptasensor for the determination of cocaine. The immobilization surface is prepared by the modification of a glassy carbon (GC) electrode with a robust nanocomposite containing nitrogen-doped graphene. The cocaine aptamer dually labeled with 3'-SH and 5'-NH_2 is covalentlyattached onto the nanocomposite using glutaraldehyde (GA) as the linking agent. The peak current of ferrocene increased linearly with increasing cocaine concentration.The aptasensor showed a low detection limit of 200 pM and high sensitivity of 0.67 μAnM^{-1} at a concentration range of up to 0.4 µM.The control experiments performed by using some other analgesic drugs to testify the selectivity of this sensing platform.The potential applicability of the aptasensor is successfully applied for measuring cocaine concentration in human blood serum and urine.The herein described methodology may hold great promise for fabrication of other aptasensors and immunosensors.

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Fabrication of electrochemicalcocaineaptasensorbased on immobilization ofaptameronto glassy carbon electrode modifiedwithgraphenenanocomposite

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The detection and quantification of trace amounts of illicit substances such as cocaine to be important for law enforcement and clinical medicine. Cocaine is a powerfully addictive stimulant drug that increases the level of dopamine, a brainchemical associated with pleasure and movement, in the brain'sreward circuit. Abusing cocaine has a variety of adverse effectson the body. Cocaine constricts blood vessels, dilatespupils, and increases body temperature, heart rate, and bloodpressure[1]. Thus, a simple, rapid, sensitive and selectivemethod for the determination of cocaine is of great interest.

Aptamers are specific single-stranded DNA or RNA molecules that have been selected in vitro from large randomized oligonucleotide libraries bySELEX (systematic evolution of ligands by exponential enrichment) [2].Very similar to antigens and antibodies, aptamers can bind specifically to abroad range of targets such as small molecules, proteins, amino acids, drugs and even viruses and whole cells.Owing to their numerous advantages such as easy labeling, good stability, reproducibility for synthesis and so on, aptamers are considered to be ideal candidates as molecular recognition elements in biosensors [3]. Among various aptamer-based biosensors, electrochemical aptasensors are proved to be powerful analytical tools in the detection of proteins and small molecules due to high sensitivity and selectivity as well as simple instrumentation.

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Fabrication of self-assembled monolayer of new 1,3-dithiolan derivative on nanoparticle of gold and its selective catalysis for the simultaneous determination of epinephrine, folic acid and acetaminophen

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Since epinephrine, folic acid and acetaminophen are electroactive components they can be determined electrochemically. However, it is very difficult to distinguish their response signal at bare electrodes because of their similar potentials and interference from each other. Therefore, it is very important to develop a modified electrode to resolve their voltammetric response from each other. In this paper, A monolayers of 1,3-dithiolan derivative was prepared on a gold nanoparticles deposited on gold electrode through self-assembly procedure to produce a gold 1,3-dithiolan self-assembled monolayers modified electrode. Characterization of the modified electrode was performed by using cyclic voltammetry.

A pair of well-defined quasi reversible redox peaks of 1,3-dithiolan derivative was obtained at the modified electrode by direct electron transfer between the 1,3-dithiolan derivative and the gold electrode. The apparent charge transfer rate constant, k_s, and transfer coefficient, α, were calculated. The modified electrode displayed strong resolving function for the overlapping voltammetric responses of epinephrine, folic acid and acetaminophen into three well-defined peaks. The peak potential of epinephrine oxidation at the surface of modified electrode shifts by about 200 mV toward negative values compared with that at the bare electrode. The values of electron transfer coefficients (α), catalytic rate constant (k), diffusion coefficient (D) and detection limit were calculated for epinephrine, using electrochemical approaches [2].

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Fast adsorption and removal of melanoidin from defense industries wastewater by using activated carbon

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Abstract

Alcohols are widely used as solvent in the military industries in the production of explosives and propellants. The ethanol is prepared from sugarcane molasses as raw material via fermentation biological process. One of biproducts in this process is melanoidin compound, that is caused a strong odor and dark brown in wastewater. Melanoidin are formed during a non-enzymatic browning reaction called the Maillard reaction, between amino compounds and carbohydrates. Consequently, discharge of melanoidin is caused serious problems such as prevent the penetration of sunlight and thus affect the photosynthetic activity of aquatic plants and dissolved oxygen concentration. Therefore, the alcohol industries wastewater is one of the pollution sources and its treatment from the environment is very important [1-2]. In this work, removal of melanoidin from wastewater was done by using activated carbon (AC) as an effective adsorbent. Effective Parameters in removal of melanoidin including pH, contact time, adsorbent amount and electrolyte were optimized. In an aqueous solution of melanoidin at 25 ^{0}C and pH 4.0, the adsorption capacity was evaluated using both the Langmuir and Freundlich adsorption isotherm models. Equilibrium data fitted very well with the Langmuir model and the maximum predicted adsorption capacities (q_{max}) was found to be 250 mg g^{-1} of the adsorbent. Finally, the removal efficiency was obtained to be ≥90%. The proposed method was successfully applied for removal of melanoidin in defense industries wastewater.

Keywords: Melanoidin, Removal, Activated Carbon, Adsorption isotherm.

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بیستمین کنفرانس شیمی تجزیه ایران
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Fast simultaneous determination of stabilizers by photochemically induced fluorescence and synchronous fluorimetry method

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Abstract

Diphenylamine (DPA) and its derivatives, as a group of emerging disinfection byproduct (DBP), have recently caused significant concerns among drinking water utilities because these compounds are generally much more potent rodent carcinogens than the currently regulated DBPs, and because most of nitrosamines are classified as probable human carcinogens by the International Agency for Research on Cancer [1]. In this work, a new simple and sensitive synchronous fluorescence method combined with PIF (photochemically induced fluorescence) for determination of NDPA (nitrosodiphenylamine) and DPA in gunpowder and well water samples has been developed. One of the components of the mixture is monitored by measuring its native fluorescence and the other after its UV irradiation and generation of a strongly fluorescent photoproduct. The performances of the PIF method are compared to literature data obtained with other techniques. The proposed method is not time consuming, do not involve any sample cleanups and derivatization. This method has been proved a very useful technique for simultaneous analyzing NDPA and DPA in mixture. The proposed method was found to be easier than the published chromatography and Mass methods, also less time consuming compared with other published LC methods for the simultaneous determination of NDPA and DPA, whereas there is no need for using internal standard, gradient elution, or time programming to adjust excitation and emission wavelengths. The technique offers good performance in terms of precision (R.S.D. < 3%), LOD (8×10^{-9} and 1×10^{-9} mol L^{-1} for NDPA and DPA, respectively) and is well suited for the quantification of NDPA and DPA in real samples without any previous treatment, make this method promising for routine analysis.

Keywords: Synchronous fluorimetry; Double scans; Photochemically induced fluorescence; Nitrosodiphenylamine; Diphenylamine

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Fatty acids profiling of Avocado seed extract using gas chromatography-mass spectrometry combined to multivariate chemometric techniques: A metabolomic study

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Fatty acids (FAs) play a significant role in growth, development and health conditions of plants, animals and humans. Avocado (*Persea Americana*) is being increasingly recognized as one of the greatest source of essential FAs, proteins, vitamins, antioxidants and minerals. Therefore, special efforts have been dedicated to profiling the metabolites and especially the FAs content of its pulp and seed [1]. Gas chromatography–mass spectrometry (GC–MS) is one of the most important and widely used techniques in analytical chemistry. With the development of metabolomics as an independent research field, GC–MS quickly turned into an essential tool of the new disciplines, being the most extensively exploited in plant metabolomics. In spite of recent technological advances, current GC-MS analyses are faced with some fundamental challenges that can directly affect qualitative and quantitative chromatographic results. Accordingly, multivariate chemometric resolution methods have been proposed to overcome different fundamental chromatographic challenges occurred during analysis of complex mixtures [2].

In the present study, the FAs content of Avocado seed were extracted by optimized dispersive liquid-liquid microextraction (DLLME) and then derivatized to FA methyl esters (FAMEs) and finally analyzed using GC-MS technique. The obtained GC-MS profiles of FAs in Avocado extract were segmented to desired number of chromatographic regions (i.e., fifteen). Then, the GC-MS data for standard mixture samples of target FAMEs and Avocado extract were column-wise augmented with elution times as rows and m/z values as columns of this data matrix. Multivariate curveresolution-alternating least square (MCR-ALS) was applied on this matrix under non-negativity, unimodality and component correspondence constraintsto exploit pure elution and mass spectral profiles along with calibration curves of seventeen FAMEs. In this way, pure information of target FAMEs were obtained in the presence of unknown interferences and chromatographic challenges, such as baseline/background contribution, elution time shifts, low S/N and peak overlap [2-3]. The linear dynamic ranges (LDRs) for FAMEs were in the range of 0.19-65.00 mgL^{-1}. The regression coefficients (R^2) were between 0.902 and 0.998. In addition, the relative errors (REs) in the calibration concentrations were below 8.0 % except for one case that was 20.0 %. All of these figures of merit supported the reliability of the proposed method for standard mixture samples. Finally, eight FAMEs out of seventeen were precisely determined in Avocado seed extract with concentration ranging from 0.38 to 49.63 mg L^{-1} and relative standard deviations (RSD, %) between 0.67-10.14. The results demonstrated that the DLLME-GC-MS method coupled with MCR-ALS algorithm is an efficient technique for fast, simple and cost effective quantification of FAs in highly complex natural samples.

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FeO loaded on synthesized zeolite P nanoparticles as a heterogeneous catalyst in photodegradation of 2,4-dichlorophenol

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Abstract

One of the important organic contaminants in industrial wastewaters are phenols and phenolic compounds and chlorophenols as phenolic types of pollutant in water and wastewater are widely recognized as one of the most important organic contaminants [1]. They pose severe problems to the environment, because they are carcinogens and mutagens [2]. Nowadays, heterogeneous photocatalysis processes are one of the most efficient methods for destroying organic pollutants and dyes in aqueous media and end products are CO_2 and H_2O in this method [3]. In this work, the photocatalytic degradation of 2,4-dichlorophenol as the deputy of organic pollutant in aqueous solutions with FeO/P zeolite as a photocatalyst under UV irradiation was studied. Photocatalyst was prepared by ion exchanging process of parent synthetic zeolite NaP with ammonium iron(II) sulfate hexahydrate aqueous solution (0.1 M) for 24 h followed by drying and calcination at 450 ◦ C for 8 h. The parent zeolite, ion exchanged media, and photocatalyst were characterized by XRD, SEM and FT-IR. The effect of key operating parameters such as catalyst dosage, substrate concentration and initial pH of the solutions were studied on the photodegradation of 2,4-dichlorophenol. The optimum values were 0.25 g L^{-1} of photocatalyst, pH 5 and concentration of 2,4-dichlorophenol solution; 200 mgL^{-1}. The process obeyed from the first order kinetics model.

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Fiber optics near infrared spectroscopy for characterization of polymeric nano fibers

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Evaluating the capability of electro spinning method as an efficient versatile approach for fabrication of nanofibers [1], a central composite design (CCD) model was used to design an experimental strategy for investigation of effective factors in production of poly (vinyl alcohol) (PVA) nanofibers fabricated from aqueous solutions of PVA. The studied variables were polymer solution concentration, applied voltage, distance between nozzle and collector plate and flow rate of solution injection [2]. Bead-free nanofibers, with fiber diameter ranging from 211-645 nm were produced with different process parameters. The influence of these parameters on diameter and morphology of obtained PVA nanofibers was studied by SEM analysis. Among all factors, concentration would strongly affect the nanofiber diameter. On-line control of the process in fabrication of polymeric nanofibers is of severe interest and infrared spectrometric fiber optics is known as a powerful process analyzer for the noninvasive, on-line and real-time monitoring. Thus, in the next step, nonlinear partial least squares (N-PLS) technique was utilized to estimate the average diameter of nanofibers based on fiber optic near-infrared spectra. Performing the Kennard-Stone (KS) algorithm, the model was trained and being validated respectively. It was observed that N-PLS using the KS algorithm is capable of generalization and could correctly predict the average diameter of nanofiber samples. In this study, 30 nanofiber samples were analyzed and reasonable results were obtained with high accuracies up to 99% and 92% in the calibration and validation sets respectively.

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First report on graphene quantum dots as novel and highly efficient nonmaterials for removal of new fuchsin dye under visible light

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Abstract: Environmental water pollution could lead to public health hazards (risk to human health), deterioration of water quality and damage to ecosystems (environmental risk) or may cause economic consequences (economic risk). Dyes are an abundant class of colored organic compounds that represent and increasing environmental danger. Generally, 30–40% of these dyes remain in the waste waters coming from these industries. These dyes are chemically and photolytically stable and the complex aromatic structures of these dyestuffs hinder in the natural bio-degradation process resulting in the turbidity as well as foul odors of these waste waters[1]. That is why; the color removal from waste waters has attracted the attention of the researchers worldwide, because not only these compounds themselves but their hydrolysis in the untreated waste waters produce other toxic compounds which pose threat to aquatic life[2].

In this paper, a novel and efficient method for decolorization of New Fuchsin (NF) dye based on the synthesized graphene quantum dot (GQD) was reported. Preparation of GQD was carried out by pyrolyzing method. Decolorization ability of the prepared GQD samples were investigated for removal of organic dye (i.e., NF) as model molecule. Effect of the experimental parameters such as pH of dye solution, contacting time, concentration of NF dye and amount of GQD on the decolorization efficiency of GQD were studied. Finally, the kinetic model for decolorization of NF in the presence of GQD was fitted and discussed.

Keywords: Graphene quantum dots, Decolorization, New Fuchsin.

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First-principles study on the electrochemical and optical properties of $Bi_{0.5}Na_{0.5}ZrO_3$ crystal and comparing to $Bi_{0.5}Na_{0.5}TiO_3$ crystal

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The $Bi_{0.5}Na_{0.5}ZrO_3$ (BNZ) ceramic which first introduced by K.Lily et al in 2007 is a lead-free compound which has perovskite structure [1]. In this paper, BNZ crystal has been investigated in tetragonal phase. The full potential and linearized augmented plane wave (FP-LAPW) method has been used for calculations in density functional theory (DFT) frame. The generalized gradient approximation (GGA) has been used for exchange and correlation effects. The density of states (DOS) of BNT and BNZ compounds is shown in fig. 1.

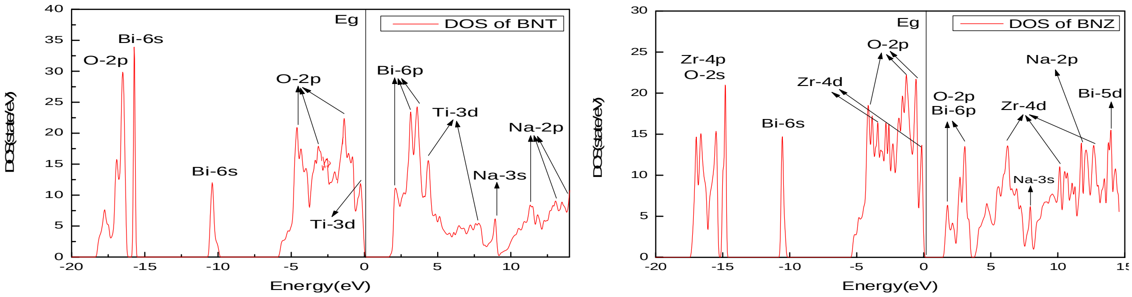

Fig. 1: Density of states of BNT and BNZ compounds.

The electronic energy gaps of BNZ and BNT are obtained 1.45 eV and 1.9 eV, respectively. This shows that replacing Zr atoms with Ti ones has reduced the energy gap. Besides, it is seen that BNZ has greater lattice parameters than BNT [2, 3]. These results can be mainly because of greater ionic radii of Zr with respect to Ti.

Also, the optical energy gaps of BNZ and BNT are 1.8 eV and 2.2 eV, respectively. The bond lengths of Zr-O and Ti-O are 2.04 Å and 1.97 Å, respectively. These values are less than ionic bond length so there must be covalent bonds between Zr and O as well as Ti and O. This also can be derived from DOS diagram where O-2p and Ti-3d as O-2p and Zr-4d can be seen in same energies.

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Flow injection simultaneous determination of nitrite and nitrate in water samples using kinetic catalytic spectrophotometric detection system

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Abstract

Nitrite and nitrate are two important wide spread contaminants chemical species of aqueous environment and serve as significant indicators of natural water quality [1]. The determination of nitrite and nitrate is essentially important because of their harmful effect on human body. The reaction between nitrite and secondary or tertiary amines leads to the formation of *N*-nitroso compounds, some of which are known to be carcinogenic, teratogenic and mutagenic [2]. The concept of flow injection analysis has been applied successfully to enhance simplicity, reproducibility and flexibility of chemical analysis especially in simultaneous determination of various spices [3].

An automated, inexpensive and rapid procedure for the simultaneous determination of nitrite and nitrate based on flow injection technique and spectrophotometric detection is presented. Nitrite signals are resulted by catalytic effect of nitrite ion on redox reaction between sulfonazoIII and bromate ion in acidic media which monitored spectrophotometrically at 570 nm. Nitrate ion content of the samples is converted to nitrite using an integrated copperized cadmium reduction column and resulted signals corresponds to sum of nitrate and nitrite concentrations. The sample plug after injection into water carrier split to two streams via a T-shaped connector, one of them merged directly to the monitoring reaction line and monitored as nitrite signal. Another stream is passed through the Cu/Cd column, mixed with the reagents through reaction coil and pumped toward the flow cell and monitored as sum of the nitrite and nitrate signals. A complete range of parameters including concentration of reagents and physical variables of FIA system were studied and optimized. Under the optimal conditions, calibration graph was linear in the ranges of 0.05-0.70 mgL^{-1} and 1.00-3.20 mgL^{-1} for nitrite while 0.06-0.75 mgL^{-1} for nitrate. Limit of determination values (S/N=3) were achieved 0.015 and 0.018 mgL^{-1} for nitrite and nitrate, respectively which can easily cover maximum admissible levels of these contaminants (3 and 50 mgL^{-1} NO_2 and NO_3) according to Iran standard no. 1053 for drinking water quality. Relative standard deviations for 0.3 mgL^{-1} nitrite and nitrate solutions were 0.33% and 1.6% (n=5), respectively. The proposed method was successfully applied for the determination of nitrite/nitrate ions in some mineral and bottled waters with sampling frequency of 12 h^{-1}.

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Fluorescence detection of formaldehyde by CdSe quantum dots

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A novel method for direct detection of formaldehyde based on the fluorescence quenching of L-cysteine (Cys) CdSe quantum dots (QDs) was developed. The CdSe QDs were synthesized according to the previous report with some modifications [1]. Typically, CdSe QDs were prepared using the reaction between Cd^{2+} and NaHSe solution. The concentration of Cys and the molar ratio of Cd^{2+}:Se:Cys were set as 3 mM and 1:0.25:2.4, respectively. The resulting products were precipitated by ethanol, and superfluous Cys and Cd^{2+} that did not participate in the reaction were removed with centrifugation. The resultant precipitate was redisposed in water. Concentration and average size of the QDs were obtained from absorbance measurements following the calibration method reported by Yu et al [2]. The developed method is on the base of the fluorescence quenching character of formaldehyde. In addition, effect of different parameters influencing the interaction efficiency of formaldehyde including, interaction time, concentration of QD and sample pH were investigated and optimized. Formaldehyde shuttles the electron from the conduction band to the valence band of the excited CdSe QDs and quench the fluorescence of QD [3]. Under the optimized condition, the fluorescence intensity of QDs decreased linearly with the increase of formaldehyde concentration in the range of 1.7–28 mg L^{-1} with R^2= 0.9933. The limits of detection ($3S_b$) and limits of quantification ($10S_b$) of the method were 0.71 and 1.7 mg L^{-1} (n=3), respectively. The relative standard deviation for water sample was 2.1% (n= 3).

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Fluorimetric determination of ascorbic acid based on enhencment effect on 2(2-hydroxy-4-methoxy benzylidene amino)-7, 6, 5, 4-tetrahydro benzo thiophene 3-carbonitrile fluorescence as new flourescer

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Abstract

Accurate quantification of the nutritional and antioxidant properties of raw materials is important in the diet and nutritional management. In this study, the 2(2-hydroxy-4-methoxy Benzylidene amino)-7,6,5,4-Tetrahydro Benzo thiophene 3-carbonitrile as a flourimetric probe method used for determination of ascorbic acid level in fruits..In the proposed method was tried an accurate, more sensitive and more suitable for quantitative measurements of vitamin C is applied. So fluorimetric method was used that was accurate and repeatable. A analytical method for extracting vitamin C in tropical fruits [banana, mango and pineapple] and 11 varieties other of fresh fruits were evaluated. These extracted vitamin C used spectrofluorimetrically for detecting ascorbic acid.

In this study a simple and sensitive fluorimetric method for the determination of ascorbic acid (AA) is described. This method for determining ascorbic acid based on its enhancement on the fluorescence thiophene dye. Ascorbic acid of the 14 varieties of fresh fruits were studied by spectrofluorimetric method proposed. The optimal conditions were found. The influence of different buffers and concentrations on the relative fluorescence intensity was tested. The method is based on the fluorescence variations of 2(2-hydroxy-4-methoxy Benzylidene amino)-7,6,5,4-Tetrahydro Benzo thiophene 3-carbonitrile (BTTC) by AA. The fluorescence intensity is measured in a 1 cm quartz cell with excitation and emission wavelengths of 260 and 273 nm, respectively. Under optimum condition, a linear relationship is obtained between the fluorescence intensity and the concentration of AA in the range of 0.43-74.12 µg ml^{-1}. The detection limit is 0.05 µg ml^{-1}, which is obviously lower than that of other methods reported. RSD = 1.35 % and has excellent detection limit was 0.05 when compared with other similar methods is quite comparable. Also interfering effect were investigated.

Keywords: Ascorbic acid, Spectrofluorimetric, BTTC.

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Forced vortex assisted liquid–liquid microextraction with central composite design optimization for preconcentration and spectrophotometric determination of omeprazole

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Abstract:

A new microextraction technique has been developed for separation and determination of omeprazole in biological samples using forced vortex assisted liquid–liquid microextraction (FVA-LLME) procedure coupled with UV-Vis spectrophotometry. FVA-LLME is based on the formation of a forced (rotational) vortex of organic phase in aqueous phase. Rotation of fine layer of organic phase as a solid body in an aqueous phase accelerates the mass transform and increase the extraction efficiency [1]. A response surface methodology (RSM) based on central composite chemometrics design (CCD) was used for multivariate optimization of the effects of five different parameters influencing the extraction efficiency of the method [2]. Under the optimal conditions, the calibration curve was linear in the range of 5–600 ng mL^{-1} of omeprazole with a R^2 of 0.996. The relative standard deviation (RSD) for the determination of 200 ng mL^{-1} omeprazole was 1.8% (n=5), and the detection limit (S/N=3) was 2.04 ng mL^{-1} in the original solution. The purposed method was successfully applied for determination of omeprazole in urine and plasma samples. The average recovery of spiked samples was 98.5%. A comparison of this method with previous methods demonstrated that the developed method is an accurate, inexpensive, rapid and reliable sample pretreatment method that gives very good enrichment factor and detection limit for extracting and determining omeprazole in biological samples.

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Formic Acid Electrooxidation on a Porous Nanostructured Co/Co-Ni-Au Electrode in Alkaline Media

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Abstract

The electrocatalytic oxidation of formic acid on Co/Co-Ni-Au electrode in alkaline solution has been investigated. A new porous Co/Co-Ni-Au nanocatalyst prepared by electrodeposition process and simple galvanic replacement technique in alkaline gold solution. The procedure consisted of the electrodeposition of Co-Ni-Zn on the Co coating, with subsequent replacement of the Zn by Au at open circuit in an Au containing alkaline solution.The morphology and surface characteristics of result electrode were investigated using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) respectively. The results showed that the Co/Co-Ni-Au coating were porous and composed of discrete Au nonoparticales with the crystallite size of about 60-80 nm. Electrocatalytic activity toward the formic acid electro-oxidation was assessed by cyclic voltammetry (CV), choronoamperometry and electrochemical impedance spectroscopy (EIS) methods. Moreover, the effects of various parameters such potential, scan rate (10- 100 mVs^{-1}), Formic acid concentration and solution temperature (25-65 °C) on the electro-oxidation of formic acid have also been investigated. The calibration curve was linear in the concentration range 1 mM to 10 mM with the detection limit of 0.1 mM Therefore the Co/Co-Ni-Au catalysts can be potential anode catalysts for the direct formic acid fuel cells (DFAFC) [3].

Keywords: Formic Acid; Nanostructured; Porous; Co/Co-Ni-Au; Alkaline Media

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Full automated hollow fiber liquid phase microextraction for determination of Megestrol Acetate and Levonorgestrel based on two immiscible organic solvent

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Megestrol acetate is a synthetic derivative of a naturally occurring progestational agent, which is chemically similar to progesterone. This drug is primarily used at high dosage for the treatment of metastatic breast cancer. Levonorgestrel is also a synthetic progestational hormone. Norgestrel was the first progestational hormone witch synthesized in the 1950s as a racemic product and Levonorgestrel is the biologically active levorotory enantiomer of norgestrel (D-norgestrel). It is used as a contraceptive agent at low doses either alone or in combination with estrogen as an oral pill [1]. Presence of these free drugs and their methabolites in biological sampels such as urine and plasma can show the percentage of drugs that would be effective as a pharmaceutic substance; however, this is necessary that we can detect the amounts of free drugs in biological samples. In this research an automated instrument for preconcentration and determination of two hormonic drugs (megestrol acetate and levonorgestrel) based on hollow fiber liquid-phase microextraction (HF-LPME) followed by high performance liquid chromatography (HPLC) with UV-Vis detection was applied [2]. The effect of different extraction parameters such as fiber length, extraction time, stirring rate and ionic strength on the extraction efficiency were investigated by using modified simplex and central composite design method as a screening method and optimization method, respectively. The effect of supported liquid membrane polarity and type of acceptor phase were optimized with one variable at a time. The calibration curves were linear in the range of 0.5 to 125 µg L^{-1}. LOD for both of the drugs was 0.5 ppb. The applicability of this technique was examined by analyzing drugs in urine, plasma and waste water samples. The relative recoveries of the drugs were in the range of 90 to 104 % that shows the capability of the method for extraction with suitable clean-up of these drugs from various matrices.

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Full automated hollow fiber liquid phase microextraction for determination of Megestrol Acetate and Levonorgestrel based on two immiscible organic solvent

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Megestrol acetate is a synthetic derivative of a naturally occurring progestational agent, which is chemically similar to progesterone. This drug is primarily used at high dosage for the treatment of metastatic breast cancer. Levonorgestrel is also a synthetic progestational hormone. Norgestrel was the first progestational hormone witch synthesized in the 1950s as a racemic product and Levonorgestrel is the biologically active levorotory enantiomer of norgestrel (D-norgestrel). It is used as a contraceptive agent at low doses either alone or in combination with estrogen as an oral pill [1]. Presence of these free drugs and their methabolites in biological sampels such as urine and plasma can show the percentage of drugs that would be effective as a pharmaceutic substance; however, this is necessary that we can detect the amounts of free drugs in biological samples. In this research an automated instrument for preconcentration and determination of two hormonic drugs (megestrol acetate and levonorgestrel) based on hollow fiber liquid-phase microextraction (HF-LPME) followed by high performance liquid chromatography (HPLC) with UV-Vis detection was applied [2]. The effect of different extraction parameters such as fiber length, extraction time, stirring rate and ionic strength on the extraction efficiency were investigated by using modified simplex and central composite design method as a screening method and optimization method, respectively. The effect of supported liquid membrane polarity and type of acceptor phase were optimized with one variable at a time. The calibration curves were linear in the range of 0.5 to 125 µg L^{-1}. LOD for both of the drugs was 0.5 ppb. The applicability of this technique was examined by analyzing drugs in urine, plasma and waste water samples. The relative recoveries of the drugs were in the range of 90 to 104 % that shows the capability of the method for extraction with suitable clean-up of these drugs from various matrices.

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Functionalization of iron magnetic nanoparticles for extraction of aflatoxins from foodstuffs and their determination by HPLC

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Research on aflatoxin is necessary because AFs are potentially hazardous to humans and animals. they display strong immunosuppressive, mutagenic, teratogenic and carcinogenic effects [1]. Some of the most famous include B1, B2, G1 and G2. The aim of this work was to prepare a suitable and simple method for the detection and measurement of aflatoxins in selected foodstuff. The method that be able for identifying and measuring aflatoxins by HPLC UV-based methods as general method. In this work magnetic nanoparticles were synthesized by precipitation method and then were coated with silica using sol–gel method. Surface treatment of silica-coated magnetic nanoparticles was carried out by amino-silane coupling agent, 3-aminopropyltriethoxysilane (APTES), finally albumin was covalently immobilized onto the amino-silane modified magnetic silica supports by the glutaraldehyde as spacer [2]. Extraction was done by these functionlized nanoparticles in aqueous solution. Results showed that the performance of the adsorbent is highly dependent upon the chemical environment and pH (pH=6). Other study that was performed evaluate the performance of this adsorbent with other absorbents synthesized such as magnetic nanoparticles coated with silica and dichlorodiphenylsilane-functionalized magnetic nanoparticles in order to adsorb aflatoxins. Results of this study also showed that interactions between aflatoxins and albumin-functionalized MNPs were much more than the others (>70%). Finally, in the optimized conditions (optimized parameters: amount of sorbent (50 mg), sample volume (1/5 mL), extraction time (30 min), type and volume of the solvent desorption (chloroform/acetone:2/1, 2 mL), (pH=6)) calibration curve was plotted for aflatoxins B1, B2, G1, G2 with $R^2>0.99$. The current method was applied successfully for the determination of this toxic materials at very low concentrations.

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Functionalized graphene nanosheets with 4-aminothiophenol as a stable supported for oxidation of formic acid base self-supported Pd–nanoclusters via galvanic replacement from three-dimensional Cu_2O nanocubes

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Abstract

An easy way to fabricate three-dimensional Cu_2O nanocubes decorated on functionalize reduced graphene oxide (rGO) is presented. The structure and composition of the Cu_2O nanocubes were verified using scanning electron microscopy and X–ray diffraction spectroscopy (XRD). The results from field emission scanning electron microscopy showed that decoration of Cu_2O nanoparticle on functionalized rGO leads to three-dimensional (3D) Cu_2O nanocubes. In addition, the XRD results showed the decoration of Cu_2O on the surface of functionalized rGO fabricated a face-centered cubic Cu_2O. Then Cu_2O was combined with Pd by galvanic replacement. Electrochemical studied showed that this bimetallic system enhances the electrocatalytic activity and stability of the nanoparticles for electro-oxidation of formic acid. The electrochemical study demonstrated that functionalized reduce graphene oxide supported Cu_2O nanocubes and combined with Pd by galvanic replacement (Pd/Cu_2O–NC@F–rGO) has superior catalytic activity towards the electro-oxidation of formic acid in acid media compared to the reduce graphene oxide supported Cu_2O nanoparticles and combined with Pd by galvanic replacement (Pd/Cu_2O-NP@rGO). While a potential cycling test was employed to confirm the stability of the electro-catalyst, the results showed high stability of Pd/Cu_2O–NC@F–rGO compared to the Pd/Cu_2O-NP@rGO catalyst with carbon monoxide poisoning of. In addition, the experimental results demonstrated an effective way for non-poisoned of the electrocatalysts using thiol functional group that can resolve key problems of anodic formic acid oxidation hampering fuel cell commercialization[1, 2].

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Gas chromatoghraphic determination of some phenolic compounds in fuels after simultaneous derivatization and microextraction

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Phenols are found among the oxygenated compounds present in petroleum. The interest in the determination of phenols in crude oils and fuels lies in the fact that, upon storage, phenols and other polar compounds promote deposit formation, with deleterious effects for fuel quality and stability [1]. Several sample preparation methods have been developed for the analysis of phenols such as liquid–liquid extraction, solid-phase microextraction, supercritical-fluid extraction, ultrasonic extraction, microwave-assisted extraction, and dispersive liquid–liquid microextraction.

The goal of this study was to develop a simple and rapid method for isolation, enrichment, and determination of phenols in fuels by combination of liquid-liquid extraction and air-assisted liquid-liquid microextraction (AALLME) followed by GC-FID determination [2]. This method consist of two steps: (i) extraction of phenols from fuels by back extraction into NaOH solution; and (ii) simultaneous derivatization and enrichment of phenols with during an AALLME procedure. In the second step, butylchloroformate (as derivatization agent) and tetrachloro ethane (as extraction solvent) were transferred into aqueous sample solution in a conical test tube. The mixture was repeatedly sucked into a glass syringe and injected into the tube. By this action the turbidity of solution increased more and more. After performing predetermined cycles, the mixture was centrifuged and 1 µl of the sedimented phase was analyzed by GC-FID. This technique was successfully applied for the extraction/preconcentration and derivatization of the selected phenols from different samples such as gasoline, kerosene, gas oil, and engine oil. Under the optimum conditions, enrichment factors, extraction recoveries, and limit of detection were in the ranges of 568 to 1128, 70-100%, and 0.81-2.00 µg L^{-1}, respectively.

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Gas chromatography-mass spectrometric detection Coupled with chemometrics methods for the determination of N-(2,6-dimethyl-4-(4-methyl piperazine-1-ylsulfonyl) acetamide in spiked human plasma samples

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Paracetamol (4-hydroxy acetanilide) is an analgesic and antipyretic agent. In this study, a simple, rapid, and sensitive gas chromatography-mass spectrometry (GC-MS) method was applied for the determination of N-(2,6-dimethyl-4-(4-methyl piperazine-1-ylsulfonyl) acetamide (2,6-D-4-MP-1-YA) as a new analgesic compound of paracetamol analogues [1,2] in human plasma. 2,6-D-4-MP-1-YA was extracted from plasma with high recovery using methyl tertbutyl ether and carbonate buffer (pH=9.0). Chemometric methods were applied to optimize the chromatographic conditions. Effects of chromatographic variables on retention of 2,6-D-4-MP-1-YA were fully investigated by using full factorial and central composite design. The main chromatographic variables, interactions between these variables, and quadratic terms were evaluated with fewer experimental runs. The proposed method was validated for parameters including specificity, limit of quantitation, linearity, accuracy, precision, recovery. The method was successfully applied to determine 2,6-D-4-MP-1-YA in the plasma of opium addict patients.

Key words: 2,6-D-4-MP-1-YA, GC-MS method, Human Plasma, Factorial Design.

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Gas phase Titrimetry of Acetylene bases on Image Processing of Glow Discharge Plasma during Ionization of Hydrogen as Titrant

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Abstract

Acetylene (C_2H_2) is considered as one of the most important and basic chemical raw materials that has extensive applications in chemical industry such as in the production of some organic chemicals, welding and flame cutting of metals [1, 2]. For this purpose, hereby in this study, a new gas phase titrimetry is titrations done in the gas phase, specifically as methods for determining reactive species by reaction with an excess of some other gas, acting as the titrant [3]. A novel gas phase titrimetry is introduced for acetylene (C_2H_2) determination at parts per million (ppm) levels based on the non-thermal glow discharge plasma-based ionization of hydrogen (H_2) standard solution. For this purpose, a constant flow of H_2 solution is introduced to an ionization chamber containing a fixed volume of C_2H_2 solution (several μL). The end point (EP) of the titration is indicated by the image processing of the plasma generated between a two-electrode system including Al rod as anode and multi-walled carbon nanotubes (MWCNTs) as cathode using a charged coupled device (CCD) camera during applying a fixed breakdown voltage (832±1 V). According to the results, the ratio of $[H_2]/[C_2H_2]$ is evaluated to 2.34±0.01. No interference is observed during introduction of at most 1000 folds excess of various foreign gas species such as CO, CO_2, He, N_2, O_2 as well as vapors of HNO_3, H_2SO_4, HCl, C_2H_4 and acetone to a fixed solution of acetylene (~5.0 ppm). Only a constant negative error to ~-5% is evaluated due to the multi-step reaction of hydride (H^-) as the ionization form of H_2 with C_2H_2. Kinetic mechanism of the reactions is also proposed according to the evidences such as infrared spectroscopy, hydride generation-atomic absorption spectroscopy and gas chromatography. Finally the reliability of the method is validated via analyses of two real samples.

Keywords: Gas phase titrametry; Breakdown voltage; Glow-discharge plasma; End point; Hydride generation.

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GC/MS analysis of the essential oil of *Ducrosia flabellifolia* and comparison with those of *Ducrosia assadi* and *Ducrosia anethifolia*

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Ducrosia species, normal flora in different countries including Iran, Iraq, Afghanistan and Pakistan belongs to Apiaceae family. *Ducrosia flabellifolia* has been reported that possesses anti-cancer activity [1]. The aim of the present study was to compare the content and composition of *Ducrosia flabellifolia* oil with those of *Ducrosia assadi* and *Ducrosia anethifolia* oils. The essential oil of the aerial parts of *Ducrosia flabellifolia* collected from Masjed solayman in province Khozestan, was isolated with hydro-distillation method using a Clevenger-type apparatus for 4.5 hours. The obtained oil was analyzed with capillary gas chromatography (GC) using flame ionization (FID) and mass spectrometry (MS) detection.

The yield of the oil was found to be 0.1% (w/w). Forty-eight compounds were identified in the oil of *Ducrosia flabellifolia*. Decanal was the main component accounting for 22.547% of total oil, followed by alpha-pinene (17.329%), dodecanal (9.02%) and decanoic acid (7.209%). Sixty three compounds were identified in the oil of *D. anethifolia* among which decanal was the main component accounting for 54.0% of total oil, followed by alpha-pinene (11.6%), terpinolene (3.2%), cis-chrysanthenylacetae (3.2%) [2]. Sixty one components were identified in the first oil of *D. assadi* with decanal (74.0%) as the main constituent. Fifty components were identified in the second oil of *D. assadi* [3] with decanal (35.2%) as the main component.

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Generation of Oxygen and Hydrogen Micro/Nanobubbles through the Electrochemical Process and its Influence on the Stoichiometry of Flame in Atomic Spectrometry

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In this research, micro/nanobubbles [1-3] were generated through electrochemistry and then adopted to study their influence in the stoichiometry of acetylene/air flame in atomic absorption spectrometry (AAS). For this purpose, the electrochemical cell was connected to the burner (pre-mixed burner) of anatomic spectrometry to generate both oxygen and hydrogen micro/nanobubbles during applying direct-current (DC) potential such as +8.50 V (for oxygen) and -8.05 V (for hydrogen)to a two-electrode system including a graphite rod and stainless steel inside an solution of 0.5 M NaCl as electrolyte. According to the results, introduction of each oxygen or hydrogen nanobubbles to the flame atomizer has strong influence in the stoichiometry (oxidizing and reducing behavior) as well as the temperature of an acetylene/air flame. In this study, formation of nanobubbles was evidenced via following the pressure flow rate of the acetylene/air during sequential generation and collapse of bubbles and also using surface tension of the electrolyte. According to the photographic images, blue and yellow flames are generated during introduction of oxygen and hydrogen micro/nano bubbles, respectively. This improvement not only has strong influence in controlling the stoichiometry of flame, but significantly prevents problems related to the flash back of the flame into the pre-mixed burner of the flame. Specially, higher sensitivity as well as more improved detection limit of the bubble-modified burner simply provided accurate and precise analyses of elements such as Pb(II), without need to use graphite furnace or hydride generation AAS The results also reveal that micro/nano bubbles also play role as both releasing and chelating agents during analysis of Pb(II). Finally, good agreements were also evaluated between bubble-modified AAS and Inductively coupled plasma (ICP) during analyses of Pb(II) in some rice samples.

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Genotyping of Single-Nucleotide Polymorphisms

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Development of techniques for mutation detection that may disrupt the gene functioning is of paramount importance in many disciplines of life science, particularly in genetics [1]. In this post-genomics era with the introduction of high-throughput technologies such as next-generation sequencing that can generate a flood of data; presence of a fast and simple procedure for the detection of single-nucleotide polymorphisms (SNPs) is lacking. These genetic polymorphisms are characterized by the substitution of one nucleotide for another at a specific locus in the genome. SNPs have been used for the study of various common diseases and individual differences in drug metabolism. [2, 3] Therefore, detection of SNPs is of paramount importance in the diagnosis and treatment of genetic disorders, drug screening and in forensic applications [4]. In this talk, we present some different protocols including electrochemical and electrogenerated chemiluminescence methods for the genotyping of single-nucleotide polymorphisms [5-9].

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Genotyping of Single-Nucleotide Polymorphisms

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Development of techniques for mutation detection that may disrupt the gene functioning is of paramount importance in many disciplines of life science, particularly in genetics [1]. In this post-genomics era with the introduction of high-throughput technologies such as next-generation sequencing that can generate a flood of data; presence of a fast and simple procedure for the detection of single-nucleotide polymorphisms (SNPs) is lacking. These genetic polymorphisms are characterized by the substitution of one nucleotide for another at a specific locus in the genome. SNPs have been used for the study of various common diseases and individual differences in drug metabolism. [2, 3] Therefore, detection of SNPs is of paramount importance in the diagnosis and treatment of genetic disorders, drug screening and in forensic applications [4]. In this talk, we present some different protocols including electrochemical and electrogenerated chemiluminescence methods for the genotyping of single-nucleotide polymorphisms [5-9].

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Gold Nanoparticles and L-Cysteine Double Layer Coated on Commercial Thin Layer Chromatography Plates as a New Substrate for Direct Resolution of Propranolol Enantiomers

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Propranolol is a sympatholytic non-selective beta blocker. Sympatholytic are used to treat hypertension, anxiety and panic. The (S)-propranolol is an important therapeutic against oxidative stress and its β -blocking potency is ca. 40 times greater than that of (R)-(+)-enantiomer in humans. So existence of simple, reliable and non-expensive method for control of its drug purity is important [1]. Gold nanoparticles (AuNPs) and L-cysteine (L-Cys), in order, were coated on the surface of a commercial thin layer chromatography and the assemble was used as a new chiral substrate for direct resolution of Propranolol enantiomers based on the ordinary TLC technique. The effect of concentrations of the involved chemicals, time periods of the required processes, pH of the sample solutions as well as the effects of different coating protocols on the resolution of the enantiomers, were investigated in order to find the optimized separation conditions. Since the association between AuNPs and L-Cys and also the interaction between L-Cys and the mobile phase (copper ion solution) depend on pH, it is important to optimize the pH values of both L-Cys and copper ion solutions. The results showed that 10 mmol L^{-1} copper(II) acetate, in 70% ethanol/water adjusted at pH= 6.3, was suitable for being used as the mobile phase. Concentration of the copper acetate played an important role in enantioseparation. The high concentration of copper ions could oxidize Au-S bonds causing collapses of the L-Cys layer as reported in literature [2]. The AuNPs with the average size of 15 nm and L-Cys solution with a concentration of 10.0 mmol L^{-1} and pH= 8.1 had been chosen for impregnating TLC plates. An R_s value of 4.8 was accessible for separation of Propranolol enantiomers by this method where R_s is defined as the distance between the resolved spot centers divided by the sum of their radii.

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Gold nanoparticles self- assembled onto silanizated glass surface as a novel sensitive spectrophotometric DNA biosensor

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Abstract

Sequence-specific detection of target DNA has become increasingly important in disease diagnostics, drug screening, epidemic prevention, and environmental protection [1,2]. Various techniques have been developed for such detection [3]. We have demonstrated a novel sensing strategy employing S-H labeled DNA modified glass surface to detect target ss-DNA by inexpensive spectrophotometric method. Firstly a glass surface functionalized with 3-(mercaptopropyl) trimethoxysilane (MPTS) and self-assembled with Au nanoparticles and then S-H labeled single strand DNA immobilized onto Au nano-particles. Methylene blue (MB) can interact with free guanine base of single strand DNA and absorbed onto glass surface and its absorbance can determined at λ_{max} of MB. After addition of target DNA, hybridization between probe and target DNA occurs and free guanine base decreases and hence less MB can absorbed onto glass surface and its absorbance decreases. This decrease in methylene blue absorbance was linear with addition of target DNA and can be viewed by naked eye and detected by spectrophotometric method. The linear range of the developed biosensor for determination of complementary ssDNA was from 1 to 300 nM with a detection limit of 0.6 nM. The developed DNA colorimetric biosensor possessed high sensitivity, selectivity and regeneration ability. The promising performance of the developed DNA colorimetric biosensor makes this methodological study and application attractive in DNA analysis.

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Gold nanoparticles supported on nanoporous stainless steel using galvanic replacement as a novel electrocatalyst design for dopamin electrooxidation

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Abstract

Stainless steel (SS) has novel characteristics such as low cost, excellent corrosion resistance, good mechanical properties and the commercial availability. Due to these advantages, SS is extensively used in biomedical devices, biomaterials researches and other applications involving contact with biologic compounds. Recently, several studies have reported the application of SS substrates in several electrochemical biosensors [1]. Moreover, it has been proved that the modification of stainless steel surface with nanostructures and catalysts such as nanopores and noble metals improves its physiochemical and electrical properties and introduces catalytic properties [2].

In this work, nanoporous stainless steel (NPSS) was used as an electrode material to support noble metal-based catalysts. Anodization as an inexpensive, simple and effective technique was used to form the highly ordered and size controlled nanopores over the macroscopic surface area of SS. Then, Cu was deposited into the pores of NPSS using pulsed electrodeposition, and followed by the galvanic replacement reaction between Cu and $HAuCl_4$ solution. The surface morphology of the prepared electrode was characterized using scanning electron microscopy (SEM), which showed that self-organized nanopores with an average size of about 77 nm were formed on the SS surface and were successfully filled with Cu. After galvanic replacement, a film composed of ordered Au nanoparticles covered the entire electrode surface, which resulted in a high active surface area with greatly reducing Au utilization. The electrocatalytic behavior of the electrode was investigated for dopamine (DA) electrooxidation using cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) and differential pulse voltammetry (DPV). The results showed that Au/Cu/NPSS electrode exhibits a considerably higher electrocatalytic activity and reproducibility toward the oxidation of DA compared with a flat gold electrode. The Au/Cu/NPSS electrode showed high sensitivity, low detection limit, high reproducibility and stability. Accordingly, the proposed electrode can be extended to construct other electrochemical sensors and biosensors for determination of biomolecules.

Keywords: Nanoporous stainless steel; Galvanic replacement; Gold nanoparticles; Dopamine;

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Gold Nanorods: A Promising Nanomaterial for Chemical Sensing and Cancer Therapy

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Gold nanorods(GNRs) have been receiving widespread attention owing to their tremendously attractiveapplications insurface-enhanced Raman scattering (SERS), chemical and bio-sensing, biomedical technologiesand optical devices [1].These anisotropic metal nanoparticles provide access to shape-dependentoptical properties which not seen in spherical NPs.One of these most striking features in the UV−Vis−NIRabsorption spectrum of a solution of GNRs is theemergence of multiple plasmon bands (transverse andlongitudinal). Unlike spherical AuNPswhose plasmonabsorbanceshave nearly similar energies across 4−100nm AuNPs diameters, the energy of the GNR longitudinalplasmon is strongly influenced by the aspect ratio (also shape; smooth or dogbone-shaped) of the goldnanorods. These shape-dependent properties havefrequently been taken advantage of over the past decade toenable sensing applications, plasmon-enhanced spectroscopies, biomedical imaging, and photothermal therapy for cancer [3].In this talk, we mainly focuson the recent advances in chemical sensing and cancer therapy using gold nanorods, with a primary focus on our own recent works [4].

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Gold Nanorods: A Promising Nanomaterial for Chemical Sensing and Cancer Therapy

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Gold nanorods (GNRs) have been receiving widespread attention owing to their tremendously attractive applications in surface-enhanced Raman scattering (SERS), chemical and bio-sensing, biomedical technologies and optical devices [1]. These anisotropic metal nanoparticles provide access to shape-dependent optical properties which not seen in spherical NPs. One of these most striking features in the UV−Vis−NIR absorption spectrum of a solution of GNRs is the emergence of multiple plasmon bands (transverse and longitudinal). Unlike spherical AuNPs whose plasmon absorbances have nearly similar energies across 4−100 nm AuNPs diameters, the energy of the GNR longitudinal plasmon is strongly influenced by the aspect ratio (also shape; smooth or dogbone-shaped) of the gold nanorods. These shape-dependent properties have frequently been taken advantage of over the past decade to enable sensing applications, plasmon-enhanced spectroscopies, biomedical imaging, and photothermal therapy for cancer [3]. In this talk, we mainly focus on the recent advances in chemical sensing and cancer therapy using gold nanorods, with a primary focus on our own recent works [4].

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Grafting of ion imprinted poly(vinylsulfonic acid) onto silica particles for preconcentration of thorium(IV)

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A ion imprinted poly(vinylsulfonic acid) coated silica gel sorbent has been prepared using the radical "*grafting from*" polymerization method through surface-bound azo initiators for selective thorium(IV) uptake [1]. The coated particles were characterized by IR spectroscopy and thermal analysis. The entrapped template ion was then removed from the polymeric matrix using 2 M HCl. The rebinding behavior of the thorium(IV) ion by the prepared sorbent was studied in the batch and column approaches. The influence of the thorium(IV) ion concentration, pH, treatment time, and elution flow rate on the sorbent affinity has been investigated after removal of template ion. Metal contents of the resulting solutions were determined by inductively coupled plasma atomic emission spectrometry (ICP-AES). Optimum pH for rebinding of thorium(IV) ion was pH 3.1. The rebinding behavior of the sorbent has been successfully described by the Langmuir-Freundlich isotherm as described by Umpleby et al. [2]. The maximum binding capacity obtained as 17.2 mg/g. The prepared sorbent was repeatedly used and regenerated by 1 M HCl without a significant decrease in the binding affinities. Analytical performance of the method was evaluated by a packed micro-column containing 200 mg of synthesized sorbent. Finally the sorbent was applied to the preconcentration of trace amounts of thorium(IV) ion in real samples.

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Graphene / ferric hydroxide nanocomposite for effective removal of lead(II)

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Graphene, a single layer of carbon atoms tightly packed into a two-dimensional honeycomb sp^2 carbon lattice, possesses a large surface area, open porous structure, flexibility, chemical stability, and very high electrical conductivity, which warrant it a good candidate for constructing graphene-based composite materials with metals [1, 2]. Integration of metals and graphene into nanocomposites has recently become a hot topic of research due to their new and/or enhanced functionalities that cannot be achieved by either component alone, and therefore holds a great promise for a wide variety of applications in catalysis, surface enhanced Raman scattering, biomedical fields, removal of contaminants from wastewater, etc. [3]. It is believed that hybrids of metals and graphene/graphene oxide would have better functionalities and performances in these applications.

In this study, the composite based on graphene cross-linked with ferric hydroxide was developed for effective removal of Pb(II) from aqueous solution. Graphene oxide, which was used as a supporting matrix, was firstly prepared from Staudenmaier method. Then, the ferrous compound cross-linked with graphene oxide was in situ oxidized to ferric compound by hydrogen peroxide, followed by treating with ammonium hydroxide. In next stage, graphene oxide as composite matrix reduced to graphene by sodium acetate. The morphology and composition of the composite was analyzed by IR, X-ray diffraction, scanning electron microscopy and X-ray photoelectron spectroscopy. The ferric hydroxide was found to be homogenously impregnated onto graphene sheets in amorphous form. The fabricated composite was evaluated as absorbent for lead removal from aqueous solution. For the solution containing Pb(II) with 5 ppm, more than 60% of lead was absorbed by 0.01 g of this composite. The optimum absorption of lead occurred in pH=6 and T=50 °C.

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Solid phase extraction based on graphene oxide nanosheets for separation and preconcentration of nicotine from aqueous solutions followed by gas chromatography-flame ionization detection

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Abstract

In the recent years, there has been an intense research interest in developing extraction/preconcentration techniques using graphene oxide (GO) as a new sorbent [1,2]. GO has a large surface area and huge capacity with various oxygenated functional groups on it that lead to good water solubility and binding with different compounds through electrostatic interaction, hydrogen bonding and π-π stacking dispersion forces [3]. In this research, GO was successfully synthesized and characterized by X-ray diffraction (XRD), Fourier Transform infrared (FT-IR) spectroscopy, transmission electron microscopy (TEM), Thermogravimetric analysis (TGA), and ultraviolet–visible (UV-Vis.) absorption spectroscopy. The prepared GO was then used as an effective adsorbent in a simple solid phase extraction for the isolation and preconcentration of nicotine from aqueous solutions. After preconcentration, the GO linked with nicotine was separated from the aqueous phase by centrifugation. Then, the analyte was desorbed and subsequently determined by gas chromatography-flame ionization detection (GC-FID) technique. Various experimental parameters affecting the extraction recovery including amount of GO, extraction time, pH value of the sample solution, salt concentration, and desorption conditions (type and volume of the desorption solvent, and desorption time) were investigated and optimized. Under the optimal conditions, the limits of detection (LOD, S/N = 3) and precision based on the relative standard deviation (R.S.D.) for nicotine were obtained equal to 3.1 ng mL^{-1} and 4.6 %, respectively. The efficiency of proposed method was evaluated by the measurement of nicotine in real biological and environmental samples. The results revealed that as a solid-phase extraction sorbent, the graphene oxide has a great potential for isolation and enrichment of nicotine from various samples.

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Graphene for preconcentration of trace amounts of Ni in water samples prior to flame atomic absorption spectrometry

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Abstract

This element was selected for evaluation of the procedure because Ni (II) is one of the principal heavy metals of analytical interest due to its extreme toxicity even at relatively low concentrations[1]. Ni has been considered as a human health hazard because it may cause kidney toxicity, neurological damage, paralysis, chromosome breakage and birth defects[2]. Ni itself and compounds are included in all lists of priority pollutants and different regulations and guidelines have been developed limiting their levels in water and sediments [3].A new sensitive and simple method was developed for the preconcentration of trace amounts of Ni using 1-(2-pyridylazo)-2-naphthol (PAN) as chelating reagent prior to its determination by flame atomic absorption spectrometry. The proposed method is based on the uti- lization of a column packed with graphene as sorbent. Several effective parameters on the extraction and complex formation were selected and optimized. Under optimum conditions, the calibration graph was linear in the concentration range of 5.0–240.0 µ g L^{-1} with a detection limit of 0.36 µg L^{-1}. The relative standard deviation for ten replicate measurements of 20.0 and 100.0 µg L^{-1} of Ni were 3.45 and 3.18%, respectively. Comparative studies showed that graphene is superior to other adsorbents including C$_{18}$ silica, graphitic carbon, and single- and multi-walled carbon nanotubes for the extraction of Ni. The proposed method was successfully applied in the analysis of four real environmental water samples. Good spiked recoveries over the range of 98% were obtained.

Keywords: graphene, preconcentration, Ni, water samples, to flame atomic absorption spectrometry (FAAS)

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Graphene–Nil blueNanocomposite for NonenzymaticDetection of Hydrogen Peroxide

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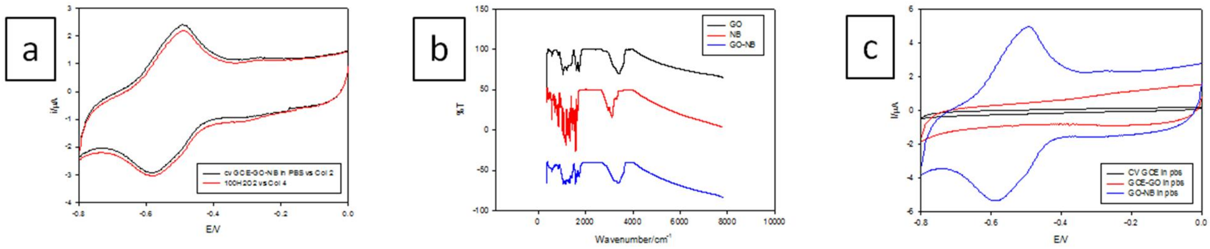
Abstract

Since reliable and fast determination of H_2O_2 is important in many areas such as medicine, food control, environmental protection,and a key factor in the development of efficient biosensors, the study of electrochemicalH_2O_2 sensor has attracted extensive attention [1].

However, there are several disadvantages of the enzyme-modified electrodes, such as instability, high cost of enzymes and complicated immobilization procedure. The activity of enzymes can be easily affected by temperature, pH value, and toxic chemicals. In order to solve these problems, considerable attention has been paid to develop nonenzymatic electrodes, for instance, noble metals, metal alloys, and metal nanoparticles [2].

Recently, graphenenanosheets (GNs) have aroused extensive interests in electrochemistry filed due to its high surface area, super conductivity and wide potential window [3].

In present work, the graphene/nile blue nanocompositeelectrode was prepared by spraying 10 µLGraphene oxide/Nile blue (GO/NB) homogeneous solution onto GC (0.03 cm^2) disk surface and dried. Then, the obtained GO/NB-GC electrode was scanned 14 cycles in KNO_3 N_2-saturated solution from 0.0 to −1.3 V at 20 mV s^{-1} and the resulting electrode was denoted as ER-GO/NB-GC. For comparison, the ER-GO/GC electrode was also synthesized by the same way..The catalytic performance of this sensor for H_2O_2 reductionis investigated using a cyclic voltammetry(CV) in PBS (pH=7) in the absence or presence of H_2O_2.These results indicated that the modification of NB on the surface of GO sheets significantly improved the electrocatalyticactivity towards the reduction of H_2O_2. Therefore, the catalytic nature of NB combined with the high surface area of GNstowards H_2O_2makes hold the promise for the development of nonenzymatic sensor at low cost.



a)CVs obtained atPBS in the absence or presence of H2O2, b)FTIR spectra, c) CVs obtained atPBS

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

Green biosynthesis of silver and gold nanoparticles using the leaf extracts from Eucalyptus *oleosa*

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ABSTRACT:

This paper reports a facile, rapid and environmental friendly process for the green synthesis of silver and gold nanoparticles using the leaf extracts from an indigenous Australia plant Eucalyptus *oleosa*. Synthesis process is carried out at the room temperature (25°C) and the Eucalyptus leaf extract acts as the both reducing agent and stabilizing agent. The synthesis process is clean, non-toxic and straight forward without requiring any complex processing equipment [1-3]. Presence of water-soluble organics in the plant materials is mainly responsible for the reduction of silver and gold ions to nano-sized Ag and Au particles. In order to synthesis of Ag and Au nanoparticles, $AgNO_3$ and $HAuCl_4$ were used as the sources of Ag^{I} and Au^{III} ions, respectively.

The color of the solution during the course of reaction was gradually turned from yellow to brick red and finally to dirty reddish brown in color and pink to reddish-pink for SNPs and GNPs, respectively at the end of 24 hrs. UV-Vib spectra for SNPs and GNPs were obtained at 420 and 552 nm to characterize the products based on the surface plasmon resonance. The synthesis conditions including leaf extract quantities, metal concentrations, reaction time and extraction temperature were evaluated by Taguchi robust design to find their effects on NPs synthesis. Spherical nanoparticles of silver and gold were prepared at the optimum conditions proposed by statistical optimization results. The produced nanoparticles were analyzed with dynamic light scattering (DLS), transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FTIR) and field emission scanning electron microscope (FESEM).

KEYWORDS: silver and gold nanoparticles, green synthesis, Eucalyptus oleosa

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Head space solid-phase microextraction of methadone prior to determination by gas chromatography-flame ionization detector

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Solid phase microextraction as a solvent free sample preparation technique invented by Pawliszyn and co-workers in 1990 [1], has attracted the eye ball of many researchers in different fields during the past few years. One of the main efforts is devoted to the introduction and application of novel fibre coatings. Ionic liquids is one of the concerned materials for this purpose [2].

Methadone is a synthetic opiate. In many countries it is used for substitution therapy of heroin addiction. As a result of the very liberal prescription practice it gets into the drug scene and is abused also by other persons leading to severe intoxications and methadone fatalities [3]. Methadone was first used to treat heroin addicts in 1965 in United States and in 1966 in Europe (Sweden), and it is still used in Iran as one of the main applicable therapy substituent. Hence its monitoring in biological or pharmaceutical samples is important considering the drug abuse as well as treatment processes. In the present work a newly synthesized ionic liquid abbreviated as $(Et)_2EMIMNTf_2$, is used as coating of a stainless steel wire(metal fibre)for extraction/preconcentration of methadone prior to its analysis by gas chromatography. The influence of experimental parameters such as initial sample basicity, agitation rate, temperature, salt concentration, and equilibrium time on the extraction efficiency was studied and optimized as 3M NaOH, 700 rpm, 50°C, 30%(w/v) NaCl, and 30 min, respectively. Under optimum conditions, the calibration graph was linear in the range of 10–50 and 200-1000 ng mL^{-1}. The detection limit and the relative standard deviation were obtained as 1.29 ng mL^{-1} ($3S_b/m$, n=6), and 5.6% (n=6, C=500 ng mL^{-1}), respectively. The technique was successfully applied for blood sample.

Keywords: Solid phase microextraction, ionic liquid, methadone

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Headspace thin layer chromatography plate modified with graphene quantum dots solid phase microextraction for extraction of nicotine

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Nicotine is the main component of tobacco and cigarette. It is absorbed by humans through the lungs and skin. Due to its toxicity, the nicotine suspected to contribute to various human diseases [1-3]. In this study, a very simple, inexpensive and sensitive sample preparation procedure “headspace TLC plate modified with graphene quantum dots solid phase microextraction (HS-GQD@TLC-SPME)” is proposed for the determination of nicotine as a model compound in biological samples using isocratic reverse phase high performance liquid chromatography (RP-HPLC) and UV detection. Briefly, a TLC plate modified with graphene quantum dots (GQD@TLC) was inserted above of the sample solution and analyte was extracted from the aqueous phase to GQD@TLC. The analyte was desorbed from GQD@TLC by ethanol. The good extraction efficiency and fast extraction time are due to the large surface area of GQD@TLC. The influence of several important parameters on the extraction efficiency of the analyte was evaluated. Under optimized experimental conditions, calibration graph was linear in the range of 10-400 µg/l with coefficient of determination of 0.9998. The limit of detection was 6 µg/l. This procedure was successfully applied with satisfactory results to the determination of nicotine in spiked biological samples. The relative mean recoveries ranged from 94.4 to 105.8%.

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Headspace-solid phase microextraction of acrylamide from heat treated foods using nanostructured polypyrrole fiber and analysis with ion mobility spectrometry

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In early 2002, Swedish National Food Administration (SNFA) and University of Stockholm together announced that certain foods, which are processed or cooked at high temperatures; contain relatively high levels of acrylamide which has potentially mutagenic and carcinogenic properties and neurotoxicity effect [1]. The most common methods for determination of acrylamide are GC-MS and LC-tandem MS which need complicate pretreatment steps, derivatization, using different cartridges, and the expensive equipment. In the past two decades, ion mobility spectrometry (IMS) has become a powerful technique for the rapid and sensitive detection of trace substances in different samples [2,3]. IMS is an ambient pressure ion separation technique that characterizes chemical substances using gas-phase mobilities of ions in an electric field. In this work the feasibility of headspace-solid phase microextraction (HS-SPME) by nano-structured polypyrrole (PPy) fiber coupled to IMS was investigated as a simple and rapid method for determination of acrylamide without derivatization in food samples. An ultrasound assisted extraction method was used for extraction of acrylamide presented in heat treated food samples following by the HS-SPME-IMS. Methanol was used as the extraction solvent. We have selected three parameters for the optimization of extraction of acrylamide from food samples, i.e. time of extraction, weight of sample and the amplitude of the ultrasonic waves.

The effect of various analytical parameters on microextraction procedure like extraction time, ionic strength, and temperature of the sample were also studied and optimized to obtain the best HS-SPME results. The HS-SPME–IMS method provided good repeatability (RSDs < 7 %) for synthetic samples. The calibration graph was linear in the range of 10–100 ng ml^{-1} ($R^2 > 0.99$) with a detection limit of 9 ng ml^{-1}. The proposed method was applied for determination of acrylamide in heat treated foods, such as potato chips samples. Validation of the analytical procedure was determined as percent of recovery and was confirmed by the add-found method in the real samples. The results obtained by the proposed method are in satisfactory agreement with the amounts added (96-105%).

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Heterogeneous photocatalytic degradation of tetracycline in aqueous solution using clinoptilolite nano particles containing copper(II) oxide

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Abstract

In this work, natural clinoptilolite was used for increasing the photocatalytic activity of copper(II) oxide in the photodegradation of tetracycline under UV irradiation. The nano particles of clinoptilolite were prepared by ball-milling mechanical method for 6 h [1]. Different photocatalysts were prepared using nanoclinoptilolite via ion exchange process in copper(II) aqueous solutions with different Cu(II) concentrations for 24 h. The Cu(II)-exchanged samples were calcined at 450°C for 12 h and characterized by FT-IR, XRD, DRS, SEM and TEM. Tetracycline (TC) capsule was prepared from Daru-Paksh company (Iran), and dissolved in sufficient water and filtered in 100 mL volumetric flask and diluted to mark. More diluter solutions were prepared from this stock solution using serial dilution method. In a typical photodegradation experiment, suspensions containing a definite amount of TC pollutant and catalyst were irradiated and at regular time intervals, the absorbance of the irradiated and un-irradiated solutions was recorded to calculate the degradation extent. The effect of different variables on the performance of photodegradation was investigated by examining the catalyst dosage, CuO loaded extent, pH and initial concentration of tetracycline solution. Maximal 68% photodegradation percent in the presence of 0.5 g L^{-1} CuO-NCP photocatalyst, pH 4.8, and 100 times diluted tetracycline solution. The degradation mechanisms were investigated via high performance liquid chromatography [2]. Using clinoptilolite particles significantly increased the photodegradation efficiency of CuO with respect to micronized clinoptilolite.

Keywords: Nanoclinoptilolite, Copper(II) oxide, Photocatalyst, Tetracycline

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Abstract

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High- throughput thin-film microextraction for the determination of penicillin G residue in biological products by liquid chromatography tandem mass spectrometry

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Abstract

Penicillin G is used in culture media preparation during vaccine production to inactivate certain organisms and also to prevent microbial growth in the product during storage and use. Regulatory control of these antibiotic is important, as it provides assurance that the biological product has been adequately preserved and that excessive amounts of its residue is not existed in final product [1]. Therefore, an analytical method for the determination of penicillin G residue in biological products is necessary. In this study a high-throughput thin-film microextraction (TFME) on 96-well plate together with high performance liquid chromatography coupled to electrospray ionization tandem mass spectrometry (LC-ESI-MS/MS) was developed for the determination of penicillin G in biological products. Pieces of cellulose acetate thin-film were precisely cut and then fixed on the end of stainless steel wires [2]. The prepared thin-films were positioned in a home-made polytetrafluoroethylene (PTFE)-based constructed ninety-six holes block to have the possibility of simultaneous immersion of the thin-films into the individual wells of 96-well plate. The performances of the TFME fibers, such as intra and inter-fibers reproducibility, were evaluated and the results showed a good similarity in extraction yields. The important parameters influencing the extraction process including desorption solvent, pH, salt addition, extraction time, desorption time, organic modifier, volume of organic modifier and volume of desorption solvent were optimized. The inter- and intra-day RSD% were found to be less than 15.6%. Limit of detection (LOD) and limit of quantification (LOQ) were 2 and 9µg L^{-1}, respectively. The coefficient of determination was satisfactory ($r^2 > 0.998$) for the studied analyte. The developed method was successfully applied to the monitoring of analyte in newcastel disease (ND) and infectious bronchitis, infectious bronchitis vaccine, newcastel disease vaccine (Lasota), newcastel disease vaccine (B1) and foot-mouth disease (FMD) vaccine.

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Highly luminescent carbon nanocrystals synthesized by microwave-assisted ionic liquid method

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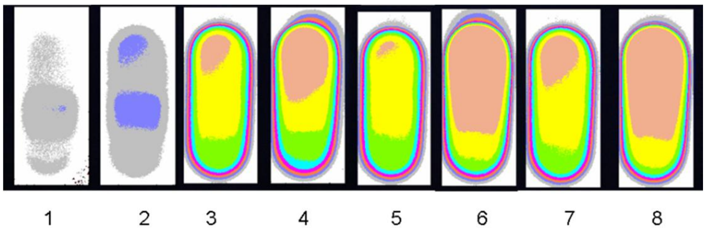
To obtain top-quality luminescent nano-crystals, the synthesis must be performed at elevated temperature to minimize lattice defects.[1] Microwave-assisted synthesis is generally characterized by the significant reduction of reaction times due to solvent superheating effect.

Herein, we combined the advantages of ionic liquid (IL) with microwave heating, MAIL method, to introduce a fast and environmentally friendly route for the fabrication of fluorescent carbon nanocrystals (CNCs)[2]. Because of their ionic character, ILs absorb microwave irradiation extremely well and transfer energy quickly by ionic conduction[3]. The ionic liquid of 1-butyl-3-methylimidazolium tetrafluoroborate ($BMIMBF_4$) was tested for the CNCs synthesis by MAIL method. In this work, IL acts not only as an excellent medium for absorbing microwaves, but also as a carbon source. In other words, starting materials for the CNCs synthesis is only ionic liquids without any additives. CCD camera and fluorescence instrument were used to investigate the fluorescence of CNCs fluorescence. Experimental conditions of CNCs synthesis such as power (P) and temperature (T) were optimized in order to have more intense brown color and thus higher yield of the products. Images were taken under UV exposure and the data were analyzed by the color det software (Fig. 1).



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Highly Selective and Sensitive Coated – Wire Silver Cation Selective Electrode

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Abstrac:

Potentiometric sensors based on ion-selective electrodes are especially suitedfor determination of several chemical species because they offer advantages such as selectivity, sensitivity, good precision, simplicity, and low cost.Nowadays, chemical sensors have attracted considerable attention because of their favourable portability, simple operation and suitability for on-sit monitoring. With the potentiometric transduction mode, polymeric membrane ion selective electrodes (ISEs) have found widespread use inindustrial, clinical and environmental analysis owing to their advantages such as good selectivity, low cost of preparation, rapidity and easiness of measurement and high reliability [1].

Ion-selective electrode is an electrochemical sensor based on thin films or selective membranes as recognition elements in aqueous solutions. Coated Wire Electrodes (CWEs), introduced by Freiser in the mid-1970s, are prepared by (Pt, Ag, Cu) or graphite-based with any conventional shape, such as wire or disk. The conductor is usually dipped in a solution of PVC and the active substance and the resulting film is allowed to air-dry. They are usually highly sensitive and very easy to use. An ion-selective membrane is the key component of all potentiometric ion sensors. The membrane establishes the preference with which the sensor responds to the analyte in the presence of various interfering ions [2]. Such a membrane is quite similar to liquid phase, because diffusion coefficients for dissolved low molecular weight ion-pairs or ion-associates are in the order of 10^{-7}-10^{-8}cm2 sec−1 [3]. The ionselectiveelectrode cell may be presented in conventional type or solid state coated wire types. Most ion-Selective Electrodes (ISEs) developed so far for determination of drugs arebased on the use of ion-exchange systems. If the drug involves compounds containing organic cation, different salts are used for their conversion to an electrode-active ion-associate .

In this study,a new coated wire electrode has been developed as a mercury ion-selective sensor. The optimized membrane incorporating DOP,PVC and IIP was directly coated on the surfact of a platinum wire electrode.The effects of the pH and possible interfering ions were investigated and the optimized conditions for electrode were evaluated. The electrode displayd very good selectivity, responsetime, and specially, lifetime.

Key word: Ion selective electrode, Nano Silver, PVC membrane ,Wire coated electrode

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Highly selective spectrofluorimetric determination of hazardous cyanide based on pure zinc sulfide quantum dot as luminescent probe

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Abstract

Semiconductor nanoparticles or quantum dots (QDs) have developed as luminescent probes for sensing events [1]. QDs, as a brand new class of fluorescent receptors, have the three dimensions confined to 1–8 nm length scale. QDs possess an excellent photostability, high quantum yields, and long fluorescence lifetimes [2]. However, due to the unique and novel properties of the QD based luminescent sensors such as low cost fabrication, reliability, reproducibility, accuracy, high sensitivity and selectivity; expanding applications of QDs to develop inorganic anions, cations, drugs, organic dyes, explosive compounds and biomolecular sensors is a topic of current interest [3].

In this research, a rapid and simple fluorescence method is presented for selective and sensitive determination of hazardous cyanide ion in aqueous solution based on functionalized zinc sulfide (ZnS) quantum dot as luminescent prob. The ultra-small ZnS QDs were synthesized using a chemical co-precipitation method in the presence of 2-mercaptoethanol (ME) as an efficient capping agent. The prepared pure ZnS QDs was applied as an optical sensor for determination of cyanide ions in aqueous solutions. ZnS nanoparticles have exhibited a strong fluorescent emission at about 424 nm. The fluorescence intensity of QDs is linearly proportional to the cyanide ion concentration in the range 2.44×10^{-6} to 2.59×10^{-5} M with a detection limit as 1.70×10^{-7} M at pH 11. The designed fluorescent sensor possesses remarkable selectivity for cyanide ion over other anions such as Cl^-, Br^-, F^-, I^-, IO_3^-, ClO_4^-, BrO_3^-, CO_3^{2-}, NO_2^-, NO_3^-, SO_4^{2-}, $S_2O_4^{2-}$, $C_2O_4^{2-}$, SCN^-, N_3^-, citrate and tartarate with negligible influences on the cyanide detection by fluorescence spectroscopy.

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Highly sensitive and simple metal-oxide nanocomposite based voltammetric sensor for dobutamine

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A simple and sensitive electrochemical sensor which was constructed with TiO_2/nafion/ carbon nanoparticle was first utilized to detect dobutamine (DBA). The electrochemical behavior of DBA at the TiO_2/nafion/ carbon nanoparticle modified electrode was investigated in detail by the means of cyclic and differential pulse voltammetry. At pH values lower than 2, a reversible oxidation process takes place involving two electrons and two protons corresponding to the oxidation of the two ortho-hydroxyl groups to stable o-quinone structure by an E mechanism. In these pHs the cyclization reaction of oxidation product is unfavorable. Also, another irreversible anodic peak was observed at more positive potentials that is attributed to oxidation of hydroxyl group of phenolic moiety of DBA structure which was disappeared in second scan. By increasing the pH of buffered solution, the mechanism of the electrode reaction changed to ECE. The results revealed that at basic pHs, the oxidation of DBA takes place according to the irreversible pH-dependent process that proceeds in an adsorption-controlled mechanism and results in the formation of an oxidation product, which adsorbs strongly on the surface of modified electrode. The adsorbed oxidation product presented reversible redox behavior, with the same electron and proton transfer. Under optimized condition, compared with previous reports [1, 2], the designed sensor presented lower detection limit (2 nM), higher sensitivity and more broad linear range (8.0 nM - 1 μM). This electrochemical platform offered a useful tool for the determination of DBA in pharmaceutical and clinical preparations.

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Highly Sensitive Determination and Selective Immobilization of Amoxicillin using Carbon Ionic Liquid Electrode

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Amoxicillin (AMX) is the phenolic compound belongs to the penicillin group. It is used in treatment of severe respiratory, gastrointestinal, urinary and skin infections [1]. Electrochemistry of AMX is widely investigated on various electrodes [2,3]. All electrodes use modifiers which not only make the electrode construction more complicated but also lower the performance of the electrodes.

In this work, carbon ionic liquid electrode (CILE) was used as a substrate for immobilization and determination of amoxicillin in phosphate buffer solution. Effect of experimental conditions on the electrooxidation of AMX and its adsorbed intermediates was investigated and possible mechanism for strong immobilization is suggested. The electrode response was linear in the concentration range of 5 to 400 µmol L^{-1} AMX with a detection limit of 0.8 µmol L^{-1} AMX. There was no serious interference from other β-lactam family antibiotics such as ampicillin and penicillin V. The electrode showed good selectivity as well as high sensitivity toward quantification of AMX in pharmaceuticals and urine samples. Enhancement in antibacterial activity of the immobilized AMX on CILE against two bacterial pathogens, *Escherichia coli* and *Staphylococcus aureus*, was observed when CILE-AMX was compared with both bare CILE and AMX.

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Highly Sensitive Electrochemical Aptasensor for Breast Cancer Detection using Glassy Carbon Electrode Modified with Chitosan-Carbon nanotube nanocoposite

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Abstract:

Breast cancer is the most common malignant cancer and main cause of deaths among the adult women. Overexpression of the Erbb2 (or Her2) receptor in breast cancers is associated with the most aggressive tumors. In this work a simple and sensitive electrochemical aptamer-based biosensor for the detection of breast cancer cells was developed. In this work, multiwalled carbon nanotubes/chitosan nanocomposite with large surface area and good electrical conductivity were synthesized. Aptamer molecules covalently attached on the surface of Carbon nanotube/Chitosan modified glassy carbon electrode. Methylene blue employed as an electrochemical probe for investigation of aptamer-breast cancer cell interaction. Electrochemical methods such as cyclic voltammetry, differential pulse voltammetry and electrochemical impedance spectroscopy used for aptasensor investigation. Under the optimized experimental conditions, the proposed aptamer biosensor exhibited excellent analytical performance for breast cancer cells detection, It was found that the calibration curve was linear up to 2.0×10^6cells mL^{-1} with a detection limit of 10 cells mL^{-1}. The biosensor showed high selectivity, good stability and reproducibility.

Keywords: Electrochemical DNA aptamer, Aptasensor, Erbb2 receptor, breast cancer

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Highly sensitive voltammetric determination of Efavirenz using carbon paste electrode modified with titanium dioxide (TiO_2)/Boehmitenanocomposite

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Abstract

An electrochemical sensor has been developed for the trace determination of efavirenz (EFV). This sensor was designed by incorporation of TiO_2 nanoparticles and Boehmite (γ-AlO(OH)) into the carbon paste matrix, which provides remarkably improved sensitivity and selectivity for the electrochemical stripping assay of EFV. The analytical performance of the modified electrode was evaluated with respect to the quantity of modifier in the paste, accumulation time, accumulation potential and other variables. The calibration graph was linear over the concentration range 0.01-40 nM with a detection limit of 0.006 nM. The proposed electrode was used successfully for EFV determination in biological samples.

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Highly sensitive voltammetric sensor based on self-assembly of bisphosphoramidate-derivative and quantum dots on multiwall carbon nanotubes modified gold electrode for the electrocatalytic determination of olanzapine

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Abstract

In the present paper, a new bisphosphoramidate derivative compound, Diethylbenzene- N,N'-bis(N-aryl-N-benzoylphosphoramidate) (EBABPP), was synthesized and used as a mediator for the electrocatalytic oxidation of olanzapine. The electro-oxidation of olanzapine was studied at the surface of the (EBABPP/quantum dots/carbon nanotubes (EBABPP/QDs/CNT) modified gold electrode. This sensor showed an excellent electrocatalytic oxidation activity toward olanzapine with a lower overvoltage, pronounced current response, and good sensitivity. The diffusion coefficient and kinetic parameters (such as electron transfer coefficient and the heterogeneous rate constant) were determined for olanzapine oxidation. Surface morphology and electrochemical properties of the prepared nanocomposite modified electrode were investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM), cyclic voltammetry and electrochemical impedance spectroscopy techniques.The hydrodynamic amperometry at rotating modified electrode at constant potential versus reference electrode was used for detection of olanzapine. Also it has short response time, low detection limit and high sensitivity,it can be used as an amperometric sensor for monitoring of olanzapine. The proposed method was successfully applied to the determination of olanzapine in both pharmaceuticals and human serum samples [1-3].

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان، ۶ الی ۸ اسفند ماه ۱۳۹۲

Hollow fiber-based microextraction of nalidixic acid in urine sample using Aliquat 336 as a carrier combined with high-performance liquid chromatography

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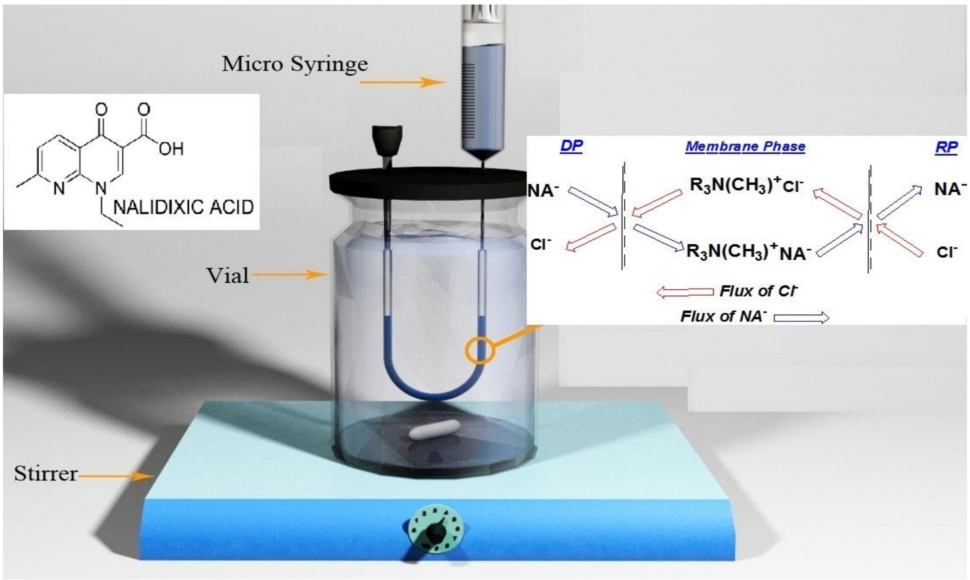
A simple and efficient hollow fiber liquid-phase microextraction technique in conjunction with HPLC is presented for extraction and quantitative determination of nalidixic acid in human urine sample. Nalidixic acid has acidic and basic functionalities. Therefore, to improve the permeability of polar drugs such as nalidixic acid through the organic liquid membrane, a carrier can be used. In this research, the drug was extracted from source phase (donor phase) into an modified organic phase with a Aliquat 336 (hydrophobic ion-pair reagent) as a carrier that impregnated in the pores of the hollow fiber followed by the back-extraction into a second aqueous solution (receiving phase) located in the lumen of the hollow fiber.



The effects of several factors such as the nature of organic solvent, composition of donor phase and receiving phase solutions, extraction time, and stirring rate on the extraction efficiency of the drug were examined and optimized. Under the optimum conditions, linearity of the method was observed over the range 0.05–2.0 µg L^{-1} with correlation coefficient R^2=0.9967. The proposed method has been found to have good sensitivity with a limit of detection aqual to 0.025 µg L^{-1}. A good enrichment factor (105) was obtained. The relative standard deviation (*n*=6) was 10.4% at concentration level of 0.8 µg ml^{-1}. This simple method has been successfully applied to analysis real urine sample with satisfactory results.

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Homogeneous liquid-liquid microextraction via flotation assistance followed by graphite furnace atomic absorption spectrometry as an efficient and sensitive technique for the determination of palladium in water samples

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Palladium is a metal, the output and use of which has more than doubled in the past 10 years. It is used in dental appliances, chemical catalysts, electrical appliances and jewellery. Thus, because of its increasing use, on the one hand, and the toxicity of Pd for mammals, fish and higher plants, on the other, all environmental analysis should include the determination of the amount of palladium [1]. Homogeneous liquid-liquid microextraction via flotation assistance (HLLME-FA) [2, 3] and graphite furnace atomic absorption spectrometry was presented for extraction and determination of palladium in water samples. Toluene at microliter volume level and acetone were used as extraction and homogeneous solvents, respectively. In this research, a special extraction cell was designed to facilitate collection of the low-density solvent extraction. No centrifugation was required in this procedure. The water sample solution with adjustment pH at 6 was added into the extraction cell, which contained an appropriate mixture of extraction and homogeneous solvents. Using air flotation, extraction solvent was collected at the conical part of the designed cell. Ammonium pyrrolidine dithiocarbamate (APDC) was used as chelating agent. The effects of different variables on the efficiency of the extraction such as kind and the volume of extraction and homogeneous solvents, pH, concentration of ligand, ionic strength and extraction time were studied and optimized. Under the optimum conditions, the calibration graph was linear in the range of 1.0 -100 $\mu g\ L^{-1}$ with detection limits of 0.3 $\mu g\ L^{-1}$. The relative standard deviation (RSD) for five replicate measurements of 20.0 $\mu g\ L^{-1}$ of palladium was 7.5%. HLLME-FA is a fast, simple and efficient method for the determination of palladium in water samples.

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HPLC determination of trace amounts of melatonin after dispersive liquid-liquid microextraction

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Melatonin is an important neurohormonal chemical that is responsible for regulating sleep. Melatonin dietary supplements are available and utilized to counteract the effects of jet-lag or to aid sleep. Due to its important changes in regulation there is an interest in developing rapid detection methods of melatonin, therefore different analytical procedures have been developed for determination of melatonin [2]. In recent years dispersive liquid-liquid microextraction (DLLME) has received great attention in analytical chemistry. Some of the advantages of the DLLME method are simplicity of operation, high speed and low consumption of extraction solvent (at µL level) [3].

There isn't any report for DLLME of melatonin therefore in the present work DLLME was used for preconcentration trace amounts of melatonin prior to HPLC determination. Rapid injection of a mixture containing 0.50 mL acetonitrile l (disperser solvent), 100.0 µL carbon tetrachloride (extraction solvent) into the 10.0 mL of 1 ng mL^{-1} melatonin sample. Various parameters (such as extraction time, centrifugation time, pH and etc.) that affect the efficiency of extraction were optimized. Under the optimum conditions the proposed method has good linearity of calibration and the results show DLME combined with HPLC is a selective, simple, sensitive, and effective analytical method for the preconcentration and determination of trace amount of melatonin. The proposed method was applied for preconcentration and determination of melatonin in different real samples.

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Hybridized ZnS-NiS incorporated onto nano-clinoptilolite particles for enhancement the photodegradation extent of 2-nitrotoluene

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Abstract

In this paper, photocatalytic degradation of 2-nitrotoluene (2-NT) aqueous solution was studied using ZnS-NiS-nano-clinoptilolite zeolite under UV irradiation. 2-nitrotoluene ($C_7H_7NO_2$) is one of compounds, which is used in different industrial processes like the explosives and lacquers production. It has long been recognized that volatile solutes are transferred to the atmosphere during mixing and aeration of wastewater. In heterogeneous photocatalysis degradation, excitation a semiconductor (such as: ZnS and NiS) produce electron-hole pairs which react in turn with water and oxygen molecules for producing hydroxyl and super oxide radicals as powerful oxidants for degradation of pollutant molecules to smaller fragments and finally to water and carbon dioxide [1-3]. Nano-powders of clinoptilolite (NCP) were prepared using a planetary ball mill mechanical method. The photocatalyst was prepared by ion exchange and precipitation procedures and was characterized by FT-IR, XRD, DRS and TEM methods. In a typical photodegradation experiment, suspensions containing a definite amount of pollutant and catalyst were irradiated and at regular time intervals, the absorbance of the irradiated and un-irradiated solutions was recorded to calculate the degradation extent. The Effects of various key operating parameters such as catalyst dosage, initial concentration of 2-NT, initial pH of the solutions, role of support and concentration of ion exchange solution were studied on the degradation efficiency. The COD and HPLC tests were also used to confirm the degradation extent of the pollutant [3]. The optimal operation parameters were found as follows: $ZnS_{7/1\%}$-$NiS_{2/7\%}$-NCP, pH=11, 0.075 g L^{-1} of catalyst and 55 ppm of 2-NT.

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Hydrogen evolution reaction at the surface of electrochemically fabricated Cu/Ni foam

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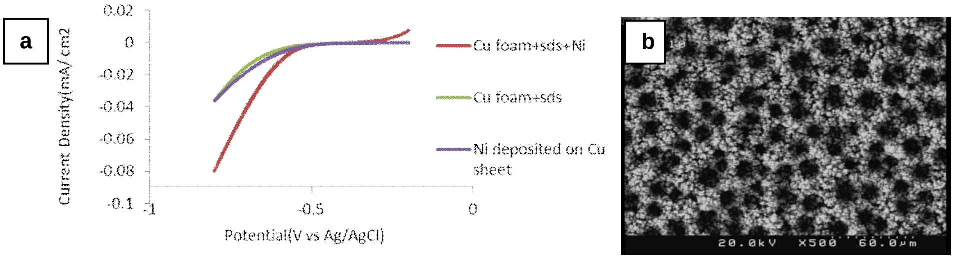
Abstract:

Hydrogen has been considered to be an ideal energy carrier, which can be efficiently converted into available energy forms without detrimental environmental effects with a practically unlimited supply. It can be produced from water electrolysis but with high energy consumption[1].The increase of electrode surface area and change in morphology, composition and structure of electrode are effective means for improving the electrocatalytic activity of electrodes toward HER.

In this work, copper foam was fabricated by electro deposition process by applying a constant current (as high as-0.56Acm^{-2}) for 28 s [2] from a solution of 0.4 M $CuSO_4$, 1.5 M H_2SO_4, and 11mM Sodium dodecyl sulfate(SDS).we attempted to use surfactant forincreasing surface area of copper foam. Then Cu-Ni foam was fabricated on as-prepared copper foam by applying a constant potential in 0.4M $NiSO_4$ solution. Ni was also deposited on copper sheet by applying a constant current of -0.86A from $NiSO_4$ solution. Morphology of as-fabricated Cu-Ni foam was examined by scanning electron microscopy (SEM), EDX,XRD.The effect of Ni deposition time on HER activity was examined. Our results indicate that under the deposition time of 2000 seconds onset potential for HER at the surface of Cu-Ni foam is about -0.48 V which is more positive than onset potential observed(about-0.58 V).at the surface of Cu foam and Ni deposited on Cu sheet. Also the current density was increased by three folds.



a) LSV for HER at Cu-Ni foam b)SEM images of as-fabricated Cu-Nifoam

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Identification and determination of the fatty acid composition of *Quercus Brantii* growing in southwestern of Iran by GC-MS

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Fatty acids, especially unsaturated fatty acids, are important as nutritional substances and metabolites in living organisms. Many kinds of fatty acids play an important role in the regulation of a variety of physiological and biological functions. Fatty acids are organic compounds formed by a hydrocarbonated chain and a carboxylic group that is normally bounded with glycerol forming acylglycerides (mono-, di- or triglycerides). Depending on the nature of the hydrocarbonated chain, fatty acids can be saturated or unsaturated, which in turn can be monounsaturated or polyunsaturated fatty acids (PUFA) [1-3]. The paper reports the fatty acid composition of the oil extracts from the fruits of *Quercus brantii* growing in three regions of Kohgiloye va Boyer Ahmad (province in southwestern of Iran). The fatty acid compositions of fruits of *Quercus brantii* were extracted with hexane in soxhlet apparatus and subsequently identified and determined by gas chromatography–mass spectroscopy (GC–MS). The results revealed that major fatty acid in three regions are oleic acid (52.99–66.14%), Linoleic acid (10.80-11.11%), palmitic acid (8.08–10.06%), stearic acid (0.74–1.57%), α-Linolenic acid (0.19–0.35%), erucic acid (0.12-0.15%) and arachidic acid (0.12-0.13%). The total unsaturation and saturation for the oil were 64.60-77.27 % and 9.17-11.75 %, respectively. Results indicate that the fruits of *Quercus brantii* contained 0.19-0.35 % omega-3, 10.92-14.77 % omega-6 and 53.14- 66.26 % omega-9. Therefore *Quercus brantii* can be introduce as rich sources of fatty acid in food dietary and medical health.

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Identification and quantitation of methamphetamine in oral fluid by Solid phase extraction and gas Chromatography-mass Spectroscopy

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The purpose of this study was to develope a qualitative and quantitative analytical method for determination of methamphetamine in oral fluid.Now-a-days Amphetamine–type stimulants (ATSs) drugs of abuse is one of the social problems in all around the word.

In the past few decades researchers have found some advantages for oral fluid over other traditional biologic fluids, like urine and blood for example, Oral fluid is ideal for detecting recent drug use[١].

Oral fluid samples was collected by spitting in test tube from drug free volunteers .
After spiking it was extracted by solid phase extraction procedure, Elution was (CH_2CL_2:Iso.Propanol:NH_4OH v/v 78:20:2)then Analyte was derivatized with HFBA and analyzed with GC.MS in SIM Mode[2].
The linearity range was 10-200 ng /ml. R^2 >0.99 and LOQ =10ng/ml ,LOD=3ng/ml and RSD in all concentrations in linear range was acceptable [3].
.

Key word: Methamphetamine ,Oral fluid ,Solid phase extraction ,Gas chromatography-Mass spectroscopy

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Identification of potential antimicrobial constituents in the essential oil of *Myrtus communis* L. using gas chromatography-mass spectrometry and multivariate calibration techniques

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Abstract

Nowadays traditional and herbal medicine for production of new drugs is under investigation by many scientists. Myrtus communis is an aromatic evergreen small tree or shrub that grows in Mediterranean area and distributed in many countries. Traditionally this plant in some countries have been used in cooking or as remedy for some disease such as stomachic, oral and cough diseases, appetizing, constipation. This plant grown up in different area, influences the quality of its essential oil, therefore the plant from different area have various influences in therapy [1]. Gas chromatography-mass spectroscopy (GC-MS) and chemometric methods were applied for quality control and also identification of various compounds in essential oil of this plant.

Partial least squares (PLS) regression is a flexible data analytical chemometric methods, which can be made even more versatile and useful by various modifications [2]. The orthogonal signal correction (OSC) method was used to improve and modify PLS method by removing systematic information in X not correlated to the modeling of Y [3]. In this study orthogonal projections to latent structures (OPLS) was applied for correlation between GC chromatogram of 15 Myrtus essential oil samples and minimal inhibitory concentration (MIC) of *Candidiaalbicans*. In this research, OPLS was preferred because of its better performing in indication of the potential antimicrobial active compounds in the Myrtus essential oil samples due to its repeatability and simplicity, and to remove the orthogonal information in the original data set that decreased model complexity contributing to an improved interpretability of the regression coefficients. OPLS results show that α-pinene, 1,8 cineole and Linalool are most effective compounds in antimicrobial properties of Myrtus essential oil.

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Identification, classification, characterization and synthesis procedures of ball valve sealing materials and collection of standard for the materials

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Ball valves are used for controlling of gas flow rate in the gas industry. In the present research, polymeric material used as sealing materials in ball valves were identified and classified. To identify the materials, infrared spectrometry, physical and mechanical properties of the polymers such as melting point, hardness, elongation at break and tensile strength were used. Comparison of the experimental results with available data of the polymers reveals that polytetrafluoroethylene (PTFE) and reinforeced polytetrafluoroethylene (RPTFE) are mainly used for sealing of class 150, 300 and 600 ball valves [1]. PTFE has excellent chemical stability and self-greasing capability, which are essential properties for polymers to be used as seat materials in ball valves. RPTFE used in ball valves is a kind of PTFE containing 15-25% carbon powder. Due to mechanical strength of nylon (polyamide), this polymer is used for class 600 ball valves [2]. Other sealing materials used in ball valves such as Vespel, PEEK, Develon, Viton and Hostaflon were also specified. All of the materials were classified and their application area was determined. Finally, standard specification of the seat materials for technical inspection was collected.

Keyword: Ball valve, Seat polymer, Mechanical properties, Teflon, Nylon

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Image Processing of Glow Discharge Plasma during Gas Ionization Process as a Novel Hydrogen Detection System at Parts per Billion Levels

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Abstract

Nowadays, H_2 is applicable in the design of fuel cells, in the welding and galvanic plating, in coal mines during methane or coal-dust explosions [1], in the hydrogen engine cars [2], etc. A small leakage of high concentration of hydrogen-containing gases is very dangerous [3]. Consequently, introduction of reliable, robust and accurate H_2 sensors with acceptable detection limit is seriously needed. For this purpose, in this study, a new hydrogen (H_2) detection system is introduced based on the image processing of the glow-discharge plasma of the ionization of hydrogen (H_2) at parts per billion (ppb) levels based on gas ionization process under vacuum condition (~8 torr). The system setup consists of a charge coupled device (CCD) camera as detection system. For this purpose, the blue component of the color related to ionization process image was analyzed during applying a 853-V direct current potential to a two-electrode system including Al disk as cathode (diameter: 2.4±0.1 mm) and multi-walled carbon nanotubes (MWCNTs)-modified disk (diameter: ~6.5±0.1 mm) as cathode with 700 µm inter-electrode distance. According to the results, formation of low temperature ionization systems such as non-thermal plasma (NTP) significantly reduces the temperature of the ionization system. Figures of merits of the fabricated sensors reveals two linear dynamic ranges between 5-36 and 36-180 parts per billion (ppb). The relative standard deviation of almost five replicate analyses during introduction of 90 ppb of H_2 standard solution (in air as solvent) was found as 2.29 %. Also, the detection limit was evaluated to ~2.25 ppb. No interfering effect was observed when introduction of at least 1000 folds excess (mass/mass) of foreign species such as Ar, He, CO_2, CO, O_2, acetone, and N_2 to 90 ppb of H_2 standard solution. Whereas, ~6% reduction was evaluated during introduction of ~1000 folds excel of acetylene to the H_2 standard solution. Reliability of the sensor was also evaluated via determination of H_2 in different industrial gas samples.

Keywords: Hydrogen sensor; Carbon nanotubes; Glow discharge plasma, Ionization sensor, Image processing.

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Imidazolium derivatives/copper as catalyst for Lucigenin chemiluminescence: Fluorescence quantum yields and application in H_2O_2 and Glucose Detection

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Abstract

The present study investigated the effect of six synthetic ionic liquids, on the Cu^{2+}-catalyzed Lucigenin chemiluminescence (Luc-CL) in the range of pH 6.5-11. In pH 9.0, the greatest enhancement on light emission was observed. The decrease at optimum pH of Luc-CL may be related to a strong interaction between Cu^{2+} and the imidazole rings in this pH [1]. As well as the formation of ionic liquid microdomains in the aqueous solution provides absolute solubility of N-methylacridone (insoluble emitter of Luc-CL), that can be very effective in Luc-CL intensity. To get consistent data for six imidazolium derivatives, fluorescence quantum yields were also measured under the same conditions by using time-correlated single photon counting and steady state fluorometer. The comparable data make it possible to suggest the mechanism that controls the Luc-CL properties in the presence of imidazole ring/copper. The mentioned findings encourage us to propose a novel, simple and sensitive method for the determination of glucose with the detection limits (LOD) of 6.5 μM which is based on the imidazole ring /Cu^{2+} catalyzed Lucigenin chemiluminescence measurement of enzymatically generated H_2O_2. Also, the method was satisfactorily applied for the determination of glucose in real serum.

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Impedimetric Detection of Uranyl Ions by using Fe_3O_4/HydroxyapatiteMagnetic NanocompositeModified Carbon Paste Electrode

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Abstract

Uranium is a toxic element that occurs naturally in most rocks, and in surface, ground and seawater [1]. Uranium, can, also, enter the environment as a consequence of several technogenic processes, such as burning of fossil fuels, nuclear weapons fabrication and testing, nuclear fuel cycle processes, radioisotopes production and application and nuclear accidents. Due to the uranium intake in human body, especially from water, vegetables, cereals and table salt, its monitoring in the environment is essential. Radiometric techniques [2], inductively coupled plasma-mass spectrometry (ICP-MS) [3] and neutron activation analysis [4] can be successfully used for the determination of uranium; however, these methods are not cost-effective and are, mostly, available in centralized laboratories. On the other hand, electroanalytical techniques are simpler in means of equipment and expenditure and yet are potent tools for the determination of uranium in water samples. Here, we developed a new impedimetry method for detection of uranium by using hydroxyapatite magnetic nanoparticles (Fe_3O_4/HAP), as a UO_2^{+2} sorbent, modified carbon past electrode (CPE/Fe_3O_4/HAP). Uranium was accumulated by heterogeneous complexation (30 min, pH 7.0) and then, it was detected in the presence of a Fe(CN)6]3-/4- and PBQ/H2Q, as a redox probe, by means of a electrochemical impedance spectroscopy (EIS). Affecting parameters like, pH, amount of adsorbent, accumulation time, and uranium concentration were investigated. In the optimized conditions, proposed method had linear relation with uranium concentration between 1×10^{-10} to 4×10^{-4} M UO_2^{+2} in the presence of both probes. Obtained results are presented and from which a new method for detection of uranium will be presented.

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In vitro antitumor activity of parent and nano-encapsulated $SiMo_{11}Co$ and its ctDNA binding properties: A combined spectroscopic study

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Abstract

The parent and nanosized starch and lipid encapsulated $K_6[SiMo_{11}O_{39}Co(H_2O)].nH_2O$ (abbreviated as SEP, LEP and $SiMo_{11}Co$, respectively), were synthesized and characterized by FT-IR spectroscopy, ICP, TG analysis, TEM and SEM images.

The results show that the $SiMo_{11}Co$ retains in its parent structure after encapsulated by starch and lipid nanoparticles. Antitumor activity tests of $SiMo_{11}Co$ and its encapsulated forms were carried out on two types of human cancer cells, MCF-7 and HEK-293 by MTT method. The encapsulated forms exhibit the higher antitumor activity respect to the parent $SiMo_{11}Co$.

However, this observed enhancement for lipid is more than starch nanocapsule that can be related to its smaller size. The calf thymus DNA (abbreviated as ctDNA) binding ability of $SiMo_{11}Co$ was also investigated, using UV-Vis absorption spectroscopy [1], fluorescence quenching [2] and fluorescence Scatchard plots[3]. Absorption spectral traces reveal 10.21% hyperchromism for $SiMo_{11}Co$. The value of 7.6×10^3 M^{-1} was obtained for association binding constant of $SiMo_{11}Co$ to ctDNA. The obtained results of absorption titration rejected the intercalating binding mode and suggest the groove or outside stacking binding for $SiMo_{11}Co$. These results were authenticated by fluorescence quenching experiments and scatchard plots.

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Indirect spectrophotometric determination of Epinephrine with chromium(VI) by flow-injection analysis

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Abstract

A simple, fast and sensitive FIA indirect spectrophotometric method for determination of Epinephrine (EP) is proposed. The method is based on a FIA indirect spectrophotometric determination of EP using the oxidation of sample by known excess amount of chromium(VI) in the presence of sulfuric acid. The remained chromium(VI) is then reacted with 1,5-diphenylcarbazid(DPC) to form a red-violet color which shows an absorption maximum at 542 nm. The absorbance is found to decrease linearly with increasing concentration of EP, which is corroborated by the calculated correlation coefficient value of –0.9998. The system obeys Beer's law for 0.6–45 μmol L^{-1} of EP. The molar absorptivity and RSD were calculated to be 3.1×10^4 l mol^{-1} cm^{-1} and 1.5%(*n*= 10), respectively. Flow injection and chemical variables such as chromium(VI) and sulfuric acid concentration were optimised for the proposed FI method. This study was carried out by altering each variable in turn while keeping the others constant. The proposed method was applied successfully to the determination of EP in pharmaceutical preparations and urine samples. The reliability of the assay was established by parallel determination by the standard iodimetric method and recovery studies.

Keywords: chromium(VI), FIA indirect spectrophotometric, Epinephrine.

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Chemical composition of the essential oil of *salvia chloroleuca* Rech.f and Aell. From Iran.

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The salvia of Iran comprises 58 species, including 17 endemics [1]. The known antioxidant, aromatic and antimicrobial properties of different members of the genus Salvia made it popular in the cosmetic industry, medicine and as food flavouring and preservation products since ancient times. The genus Salvia has widely used in traditional folk medicine [2].

In this investigation *salvia chloroleuca* Rech.f and Aell., Collected from razavi Khorasan Province, Iran. The plant material drayed in the shed and suitable air condition. The plant essential oil obtained by hydrodistillation using a Clevenger–type apparatus. The composition of the volatile oil from aerial parts of *salvia chloroleuca* was investigated by GC-FID and GC-MS. Kovat's retention indices were calculated using co-chromatographed standards hydrocarbons. The individual compounds were identify by MS and their identity was confirmed by comparing their retention indices relatives to C8 –C32 n-alkanes and by comparing their mass spectra and retention times with those of authentic samples or with data already available in the NIST library and literature Adams[3]. Thirty five components, representing of the total components were identified ٩٤,٣٩%. Sabinene with 14.35% was the main constituents in the oils of salvia chloroleuca. The othr main constituent of the oil were Germacrene-D, α-Pinene, trans-Caryophllene, β- Pinene, 1,8-Cineole, Bicyclogermacrene (١١,٥٩%, ٧,٢٣%, ٧,٧٦%, ٦,٩٧%, ٨,٤٥% respectively). A comparison of the chemical composition of volatiles of *salvia chloroleuca* from this study with previous studies on the same genus showed variation of the major components.

Keyword index:
Salvia chloroleuca, labiatae, Sabinene, trans- Caryophllene, α-pinene, 1,8- cineole, Germacrene-D.

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Indirect spectrophotometric determination of ascorbic acid with chromium(VI) by flow-injection analysis

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Abstract

A simple, fast and sensitive FIA indirect spectrophotometric method for determination of ascorbic acid (AA) is proposed. The method is based on a FIA indirect spectrophotometric determination of ascorbic acid using the oxidation of sample by known excess amount of chromium(VI) in the presence of sulfuric acid. The remained chromium(VI) is then reacted with 1,5-diphenylcarbazid(DPC) to form a red-violet color which shows an absorption maximum at 542 nm. The absorbance is found to decrease linearly with increasing concentration of ascorbic acid, which is corroborated by the calculated correlation coefficient value of –0.9994. The system obeys Beer's law for 0.3–30 μmol L^{-1} of ascorbic acid. The molar absorptivity and RSD were calculated to be 2.1×10^4 l mol^{-1} cm^{-1} and 2.5%(n= 10), respectively. Flow injection and chemical variables such as chromium(VI) and sulfuric acid concentration were optimised for the proposed FI method. This study was carried out by altering each variable in turn while keeping the others constant. The proposed method was applied successfully to the determination of ascorbic acid in pharmaceutical preparations and fresh fruit juices. The reliability of the assay was established by parallel determination by the standard iodimetric method and recovery studies.

Keywords: chromium(VI), FIA indirect spectrophotometric, Ascorbic acid.

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Influence of Erbium (III) chloride on folding pathway of Human serum albumin

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In order to understand the effect of Erbium ion on the structural stability of proteins, urea-induced unfolding transition of Human serum albumin (HSA) is characterized by Uv-Visible spectra. Solutions for experiments were prepared from stock solutions of protein and salt ($ErCl_3$) prepared in phosphate buffer (pH7.4). The concentration of protein and salt were 2 µM and 64µM, respectively. The complex solutions were incubated for enough time (>6 h) at 25ºc. On the chemical denaturation experiments, to a stock protein solution, different volumes of urea solution (6M) were added to obtain the desired concentration of denaturant. Our results provide evidence that the Erbium ion alter the urea-induced unfolding pathway of HSA. Furthermore, the change of free energy (ΔG_{H2O}), the midpoint of chemical denaturation (C_m) and the solvent-exposed surface (m) during the urea-induced unfolding process have also been obtained to characterize the effects of Erbium ion on the conformational stability of HSA [1-3]. The value of m is increased in the presence of Erbium ion, indicating that the solvent-exposed surface of the HSA molecules in the unfolding process is more than that of HSA without Erbium ion. The C_m and ΔG_{H2O} for the HSA with Er^{3+} ion [C_m = 1.94 / M and ΔG_{H2O} =11.18 $kJ.M^{-1}$ respectively] in the unfolding process are all increased with respect to that of HSA without Erbium ion [C_m .=1.58 / M and ΔG_{H2O} =7.02 $kJ.M^{-1}$] this is shown that this ion unstable tertiary structure of HSA.

Keywords: Human serum albumin, Erbium chloride, Urea , UV spectroscopy.

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Influence of Nano-Bio Adsorbent on the Cadmium Removal from Industrial Wastewaters

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Magnetic nanoparticles are a class of nanoparticle which can be manipulated using magnetic field. Some particles commonly consist of magnetic elements such as iron, nickel, cobalt and their chemical compounds. Many industrial wastewaters such as metal working, mining operation, refining process, batteries and electronic manufacturing and the paint industry are contaminated with heavy metals [1,2]. The easy separation through applying a magnetic field due to very large surface to volume ratio caused to magnetic nanoparticles have a good potential for treatment of contaminated water [3]. In this study, Fe_3O_4 magnetic nanoparticles (MNP) was successfully modified into an agricultural corn silk powder (CSP) by co-precipitating method for remove of the cadmium from industrial wastewaters. Some of the parameters such as pH of the solution, and contact time as effective parameters were investigated, each one at a time. To this end, pH values were set at 2, 4, 6, 8, and 10; also, contact times were adjusted at 10, 30, 50, 70, and 90 minutes. In addition, amount of the CSP-MNP as adsorbent was set at 0.5 gr. The remaining of the Cd into the solution was measured by atomic absorption system (AAS). Meanwhile, scanning electron microscopy (SEM) was used for investigation of the surface morphology of the CSP, MNP and CSP-MNP. Results show that, best elimination was happened at the pH=8 and 10 minutes as contact time.

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Inhibition effect of Lactuca sativa seed extract on the corrosion of aluminium in 1.0 M NaOH solution

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ABSTRACT

Recently, due to environmental regulations, plant extractants have become important because they are environmentally acceptable, readily available and renewable source for a wide range of needed inhibitors [1].

In this study the inhibition effect of Lactuca sativa seeds extract on the corrosion behavior of commercial aluminum in 1.0 M NaOH as corrosion medium was investigated by potentiodynamic polarization and electrochemical impedance measurement techniques. EIS measurement was carried out in the 100 kHz–10 mHz frequency range at OCP. The linear Tafel segments of the anodic and cathodic curves were extrapolated to corrosion potential (E_{corr}) to obtain the corrosion current densities (I_{corr}). Nyquist plots present two capacitive semicircles at higher and lower frequencies separated by an inductive loop at intermediate frequencies. The results show that inhibition efficiency increased with an increase in extract concentration that increase is due to physical adsorption and efficiency of inhibition depends on the mechanical, structural and chemical characteristics of the adsorption layers formed under particular conditions [2, 3]. The present study shows that Lactuca sativa seeds extract inhibit the corrosion of aluminum in NaOH solution and up to 50% inhibition efficiency was obtained at range 0.1-2g/l. The potentiodynamic polarization results reveal that the inhibitor act as a mixed type inhibitor and Lactuca sativa seeds extract can be added to alkaline solution as a non-toxic (green) inhibitor.

Key Words: Lactuca sativa seed, corrosion behavior, aluminum, electrochemical impedance.

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In-situ surfactant-based solid phase extraction for preconcentration trace amounts of silver prior to its FAAS determination without chelating agent

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The widespread use of silver compounds and silver-containing procedures in industry, medicine and jewellery has resulted in an increasing silver content of environmental samples [1]. The recent information about the interaction of silver with essential nutrients, especially selenium, copper and vitamins E and B12, has focused attention on its potential toxicity [1]. Consequently, determination of trace amounts of silver is important for many areas of chemical analysis. Flame atomic absorption spectrometry (FAAS) is the most commonly used techniques for the determination of trace silver. In many cases the determination of trace silver in environmental samples by FAAS is notably difficult due to both the low levels of these metals in the samples and the high complexity of the sample matrices. It is usually necessary to carry out a preconcentration/or separation step prior to the analysis. Recently Shemirani and Yousefi [2] demonstrated a surfactant-based SPE mode termed in-situ surfactant-based solid phase extraction (ISS-SPE). In the present work, we report an ISS-SPE procedure for preconcentration of silver from water samples without addition of any chelating agent. In this method a cationic surfactant with a proper alkyl group chain is dissolved in the aqueous sample and then a proper ion-pairing agent is added. Due to interaction between surfactant and ion-pairing agent, very fine solid particles are formed. In the other hand, Ag(I) ions was react with CO_3^{2-} ion and as a result Ag_2CO_3 was formed. After this, Ag_2CO_3 was adsorbed on surface of fine solid particles quickly. After centrifugation, the solid particles are sedimented at the bottom of the centrifuge tube and the aqueous phase is removed by simply decantation of the centrifuge tube. The sedimented sorbent was dissolved in 2.0 mL of 1.0 mol L^{-1} HNO_3 in ethanol. The final solution was aspirated directly into the flame of AAS. In order to obtain the best conditions, several parameters affecting the performance of the ISS-SPE such as volume of cationic surfactant, ion-pairing agent and Na_2CO_3, pH and potentially interfering ions were optimized. The calibration curve exhibited linearity over the range of 0.02 - 1.5 µg $mL^{-1}$with a correlation coefficient of 0.998 (V=12.0 mL). The eight replicate measurements of 250.0 ng mL^{-1} of silver gave a relative standard deviation ±2.1%. Limit of detection based on three times the standard deviation of the blank divided by the slope of analytical curve ($3S_b/m$) was 8.3 ng mL^{-1}. Finally, the proposed method has been successfully applied for the determination of silver in the real water samples.

Keywords: In-situ surfactant-based solid phase extraction; Silver determination;

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Integrating Molecular Docking approaches for a series of Proline-Based 2,5-Diketopiperazines that act as Novel αβ-Tubulin Inhibitors

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Abstract

Microtubules, are the basic components of cell structure, which take part in chromosome segregation during cell division. Many chemically diverse compounds bind to microtubules and arrest the cell cycle by interfering with microtubule assembly. These molecules constitute an important class of antitumor agents [1].

2,5-Diketopiperazines (2,5-DKPs) are an interesting class of cyclic peptides which are often found in a variety of natural products from fungi, bacteria, the plant kingdom, and mammals. Since the 2,5-DKP core found in the potent and selective microtubule binding agents, proline-based 2,5-Diketopiperazines have therefore; become attractive candidates for anticancer drug design [2].

Several experimental studies have been reported on synthesis and biological activity of the 2,5-Diketopiperazines. However, there is not any computational study on the interactions between proline-based DKPs and αβ-tubulin in the literatures. Hence, it would be of interest to do molecular modeling studies on the structure–activity relationship (SAR) of these compounds to obtain insight in to their inhibition properties with a certain level of accuracy.

In this study More than five hundred of proline-based 2,5-DKPs were chosen from the ZINC free database to investigate their binding modes within the binding site of αβ-tubulin. Molecular Docking results revealed that a group of 20 compounds among them exhibit strong anti-tubulin activity and they show suitable binding energies ranging from -11.49 to -14.05 kcal/mol.

By taking into account the results of the first part, the most active analogous of TPS-A was chosen to perform a 25 ns molecular dynamics (MD) simulation to more validate the proposed binding mode and interactions of this compound.

The binding modes between αβ-tubulin and proline-based 2,5-DKPs described here may be helpful for the design of new αβ-tubulin inhibitor.

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Interaction of Pyrocatechol violet with Gold Nanoparticles, Spectrophotometric and Spectrofluormetric studies

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Abstract

Gold nanoparticles (GNPs) with special physical and chemical properties are important nanomaterials which have drawn worldwide attention in recent years and are preferred candidate materials for the fabrication of smart sensing devices for detection of special biomolecules [1]. Pyrocatechol is an important chemical industry intermediate product with wide and different uses. Pyrocatechol can be used as rubber harden-idant, electroplate additive, skin preservative as well bactericide, dye hair, photographic developer, color photographic. Dihydroxy benzene and its derivatives are important environmental pollutants because they are toxic to humans and difficult to degrade. Hormozi Nezhad et al studied on the use of hydroquinone, catechol and pyrogallol as active reducing agents for the generation of GNPs. The optical properties of the generated nanoparticles enable the quantitative analysis of the different phenolic compounds by spectrophotometric detection [2]. Dong research group reported in the presence of tyrosinase, the fluorescence intensity of GNPs was quenched by catechol which could be employed to develop a new method for monitoring catechol [3].

In this contribution, the interaction between pyrocatechol violet with citrate capped gold nanoparticles was studied. The measurements performed using resonance light scattering (RLS) spectroscopy and absorbance spectroscopy. The size and surface characteristics of pyrocatechol violet capped GNPs also investigated by DLS and TEM techniques. The average diameters of synthesized GNPs were 19.0 nm and their surface potential were -56.3 mV. Interaction of the GNPs and pyrocatechol violet leads to increase of their sizes and surface potential. In the optimum condition in terms of pH and ionic strength, this system was used for determining of pyrocatechol violet. Under the optimum conditions it was found that the LDR range was 7×10^{-7} to 2×10^{-4} M for pyrocatechol violet with RSD less than 5% and DL of 7.6×10^{-8} M.

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Interaction of tyrosine with DNA studied by DNA/CNT based electrochemical biosensors and spectroscopy

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The study of the interaction between DNA and biomolecules or drugs is currently the object of many studies [1, 2]. Many proteins bind specific DNA sequences localized in the gene promoter region to regulate genome expression and maintenance. The interaction of Tyrosine (Tyr) with double-stranded DNA was studied by cyclic voltammetry at DNA-SWCNT modified electrodes, fluorescence emission and UV-Vis spectroscopy. The presence of DNA results in a decrease of the current and a positive shift of the electrode potential of the oxidation peak current of Tyr, indicating the intercalative interaction. The corresponding heterogeneous rate constant (k_s) and the electron transfer coefficient (α) were calculated for the bound Tyr-DNA according to the Laviron equation as 0.84 s^{-1} and 0.43. The binding behavior of Tyr with DNA was measured by recording the cyclic voltammograms of Tyr at various concentrations of Tyr and calculated 6.55×10^{3} mol^{-1} L by the Langmuir equation. The UV spectroscopic data confirmed the interaction between Tyr and DNA is intercalative with the binding constant of 3.98×10^{3} mol^{-1} L. Furthermore, the fluorescence emitted by the Tyr at 303 nm could be quenched in B-R buffer solution (pH=7, 0.04 M) by DNA increasing. The mechanism of DNA-Tyr interaction was discussed and the binding constant was obtained as 3.37×10^{3} mol^{-1} L, respectively from the Stern-Volmer plot of Tyr fluorescence quenching.

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Intrinsically conductive polymers and electrochemically assisted ion exchange

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This paper summarizes the various aspects of using electrochemically assisted ion exchange techniques in analytical chemistry with focus on recently our works in sample clean-up, drug delivery, electrochromic and electro-switching sensors using intrinsically conductive polymers (ICP).

Ref.

-*Food chemistry*, *155 (2014) 186-191.*

-*Electrochimica Acta, 114 (2013) 409-415.*

-*Analytica Chimica Acta, 707 (2011) 62-68.*

-*Microchimica Acta 180 (2013) 783-789.*

-*Analytical and Bioanalytical Chemistry, 405 (2013) 5333-5339.*

-*Analytical Biochemistry, 428 (2012) 99-106.*

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Journal of Separation Science, 36 (2013) 1797-1804.

Intrinsically conductive polymers and electrochemically assisted ion exchange

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Introducing a novel chiral surface through modification of commercial thin layer chromatographic plates by NiO nanoparticle and Histidine layers for resolution of enantiomers of propranolol

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Abstract: Propranolol, a drug widely used in the treatment of hypertension, angina cardiac arrhythmias and thyro toxicosis, is almost completely metabolized in human [1]. This compound is an amino alcohol and belong to the commonly known group of β-blockers (or β-adrenergic antagonists) and β-agonist. The (S)-propranolol is an important therapeutic against oxidative stress [2], and its β-blocking potency is ca. 40 times greater than that of (R)-(+)-enantiomer. The assembly of NiO nanoparticles (NiONPs) with histidine provided a chiral selector for resolution of propranolol enantiomers. This research introduced a novel chiral surface through modification of commercial thin layer chromatographic (TLC) plates by histidine-coated NiO nanoparticles. The technique was used for improving the enantiomeric resolution of propranolol from its racemic mixture. In this method, NiONPs were synthesized using a simple coordination precipitation procedure as previously reported by Kashani Motlagh et al. [3]. The appropriate size of the nanoparticles used in this work was 50±2 nm. For developing the TLC plates, they were floated in an ethanolic solution containing NiO nanoparticled for 40 sec. After drying, the plates were floated in a dish containing certain concentration of histidine for 20 sec and were dried afterward. Both (±)-propranolol and (S)-propranolol were individually spotted side by side on the developed TLC plates based on the procedures regularly performed in laboratories when deal with TLC plates. The mobile phase was copper acetate solution in ethanol/water (70/30 v/v) as solvent. The separation of the enantiomers was obtained with a resolution of 4.4 defined as the distance between the resolved spot centers divided by the sum of their radii.

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Introducing modified carbon ionic liquid electrode by palladium nanoparticles for investigating the electrocatalytic oxidation of isoniazid

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Abstract

Isoniazid (INH) or pyridine-4-carboxylic acid hydrazide, is one of the drugs that widely used for treatment of tuberculosis. The poisoning, even death, have sometimes happened due to over dosage taking of isoniazid [1]. Therefore, the control of isoniazid level in human body fluids is very important in clinical chemistry. On the other hand, small concentration difference between effectively therapeutic and toxic dosages makes it necessary to develop a rapid and specific method for determining isoniazid level in body fluids to aid the diagnosis of isoniazid intoxication.

In this research, palladium nanoparticles deposited on the carbon ionic liquid electrode, and electrocatalytic oxidation of isoniazid was investigated with this electrode in phosphate buffer (pH 7.0). The surface morphology of electrode was investigated by scanning electronmicroscopy and average diameter of palladium nanoparticles was observed as 30 nm. The cyclic voltammetry was employed for the electrochemical determination of isoniazid. Using this electrode, a good electrochemical response was obtained at a potential of 0.34 V *vs.* Ag/AgCl reference electrode. The anodic transfer coefficient was calculated as 0.47. Two linear relationships were established between peak current and the concentration of isoniazid in the ranges of 5.0-100.0 and 150.0-2600.0 μmol L^{-1} with a detection limit 0.47 μmol L^{-1}. The constructed electrode exhibited good reproducibility, repeatability, and storage stability. The method was successfully applied for analysis of isoniazid in human blood serum and tablet samples.

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Introduction of a new material as a methanizer catalyst and new method to recover the poisoned-catalyst in gas chromatography

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Gas chromatography is one of the analytical methods that can be used for many liquid and gas analysis. In gas analysis, determination of carbon monoxide and carbon dioxide is very important to control power transformer. In methanizer, a catalytic converter is used for determination of carbon monoxide (CO) and carbon dioxide (CO_2). The methanizer is a column containing Ni-catalyst that can catalyze the reduction reaction of CO and CO_2 with H_2 at 350°C and convert them into methane. Then, a flame ionization detector is used to detect the produced methane. The produced signal is proportional to the CO and CO_2 concentration. The Ni-catalyst should be replaced after fixed number of analysis because of the catalyst poisoning.

Here, we introduced a new and novel practical and valuable method to recover of the methanizer catalysts. In this method all of the compounds occupied the surface of the Ni-catalyst sites were oxidized and removed by use of oxygen gas passage at appropriate temperature. The recycled Ni-catalyst activity was evaluated by analysis of reference materials containing known amounts of CO and CO_2 concentrations in ppm range. The results confirmed the proficiency of the recovered methanizer. This method is a laboratory-developed method that validated according to ISO/IEC 17025.

In the second work, we have introduced a new low cost material catalyze the reduction reaction of CO and CO_2 with H_2 at 350°C and convert them into methane. The prepared catalyst activity was also evaluated by analysis of reference materials containing known amounts of CO and CO_2 concentrations. The results confirmed the proficiency of the recovered methanizer.

Keywords: Recovery, Catalyst preparation, Gas chromatograph, Methanizer.

References:
1-This method is a laboratory-developed method that validated according to ISO/IEC 17025.

In-tube solid phase microextraction followed by high performance liquid chromatography for determination of parabens in water samples

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Todays, in parallel to the development of technology, the emergence of environmental pollutants is a serious treat for living beings. Therefore, fast and precise determination of trace amount of different analytes has received major attention around the world. This situation has propelled the research community to develop new sample preparation techniques reaching higher sensitivity and better accuracy of analysis. Modern trends in analytical chemistry are moving toward simplified and miniaturized sample preparation methods providing sample clean-up and analyte preconcentration, simultaneously. Among these, solid phase microextraction (SPME) is a fast alternative technique for the conventional extraction systems which can be used for both portable sampling (field analysis) and sample preparation aims [1, 2]. In this work, a simple and fast in-tube solid phase microextraction (IT-SPME) method was developed for preconcentration of trace amount of parabens in water samples followed by high performance liquid chromatography [3]. Several important factors affecting extraction efficiency such as type of SPME coating fiber, extraction and desorption time as well as their flow rates, pH of sample solution and ionic strength were investigated and optimized. Under the optimal conditions, the limits of detections (LODs) were in the range of 5.0-9.0 ng L^{-1}. This method showed good linearity for parabens in the range of 50-10000 ng L^{-1}, with correlation coefficients above 0.9990. The intra- and inter-assay precisions (CV%, n = 3) were in the range of 5.9-7.0% and 4.4-5.7% at three concentration levels of 2, 10 and 20 ng L^{-1}, respectively. The extraction recoveries for the spiked samples were in the acceptable range of 72.4-78.9%. The validated method was successfully applied for the analysis of methyl-, ethyl-, and propyl paraben in some environmental water samples.

Keywords: Solid phase microextraction, Paraben, Water sample, Liquid chromatography.

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Investigating Gas Permeability of Composite Membranes Fabricated Based on Porous Polyacrylonitrile (PAN) Membrane

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Gas separation using polymer based membranes is an emerging technology and displays attractive advantages over conventional methods due to its good manufacturability, low cost, ease of operation, high energy efficiency and environmental friendliness. [1] Mostly dense symmetric polymeric membranes have been studied for gas separation. However, these membranes show low mechanical strength and relatively high resistance to gas flow which limits the application of these films for gas separation in industrial scale. [2] Due to high gas flux, composite membranes consisting of a very thin selective layer and a porous support are more preferred. Polydimethylsiloxane (PDMS) is well known for gas separation. [3] In order to develop high performance composite membrane with superior mechanical stability and resistant against compaction at high pressures compared to pure membranes, in this study, PDMS solutions were coated on polyacrylonitril (PAN) porous substrate using dip coating, spin coating and spraying techniques. The effect of applied coating technique, PDMS solution concentration, application of water as pre-wetting agent on the gas permeability of PDMS/PAN composite membranes were investigated. PDMS solution concentration was varied from 2wt% to 16wt%. The morphology and pore size distribution of the fabricated composite membranes was studied using scanning electron microscopy (SEM). The gas permeability of these membranes was measured using single gas permeation at ambient temperature and different pressures. SEM images confirmed the penetration of PDMS selective layer into PAN support pores at low concentrations of PDMS solution. Experimental results indicated that the undesirable penetration during the dip-coating stage could be avoided by increasing the viscosity of PDMS by increasing concentration of PDMS solution or changing the ratio of two component of PDMS. It was also found that on application of a higher concentration of PDMS solution a thicker selective layer is formed at the top surface of PAN support which limited the gas permeability of the fabricated composite membrane. Amongst above-mentioned coating techniques, spin coating was preferred since a more homogenous pore size distribution was introduced in the coated selective layer structure.

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Investigating the effect of SWCNTs on the permeability and selectivity of polymeric nanocomposite membrane for gas separation

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Many studies have reported that the efficiency of gas separation process using polymeric membranes depends significantly on the permeability of these membranes to the gas feed flow. Attempts to increase the gas permeability of the polymeric membranes have revealed that compositing of these materials with inorganic nanofillers such as zeolites, carbon molecular sieves and activated carbon is the best possible method to achieve this goal. polymer-inorganic hybrid membranes may combine the advantages of polymers including flexibility and processibility with the thermal stability and the selectivity of nanofillers. Solution blending is the most common way to fabricate polymer–inorganic nanocomposite membranes. A polymer solution is first prepared by dissolving the polymer in a suitable solvent and then inorganic nanofillers are dispersed by stirring or ultrasonic bath. The nanocomposite membrane is cast by removing the solvent. In the present work, polymer-inorganic nanocomposite membranes were fabricated by loading of SWCNTs as an inorganic disperse phase into PEBAX as a polymer matrix. Carbon nanotubes (CNTs) have been considered as proper fillers for preparation of hybrid membranes due to their outstanding mechanical, thermal, and electrical properties and their high aspect ratio[1]. poly (amide-6-b-ethylene oxide) (PEBAX), which is classified as a rubbery polymer has been considered as a promising candidate for gas separation due to its unique properties such as; mechanical strength, high permeability and the high selectivity for polarizable/nonpolar gas pairs [2]. However its inappropriate gas permeability restricts its application in these processes. Researchers have shown that for sufficient enhancement of the selectivity and permeability of the hybrid membranes, the dispersion of SWCNTs in the polymer matrix should be very fine and homogenous to achieve the maximum surface interaction between the nanofiller and the polymer matrixes. Thus, nanocomposite membranes based on PEBAX containing different weight percentage of SWCNTs were prepared using DMAc as solvent by solution casting method. The effect of different weight percentage of SWCNTs, time and energy of the sonication step (for dispersing the SWCNTs in the solvent) on the morphology, permeability and selectivity of the obtained membranes were investigated. It was found that PEBAX/SWCNT nanocomposite membranes had an increased CO_2 permeability, retaining similar CO_2/CH_4 selectivity compared to the pure polymer membrane. The CO_2 permeability increased with increasing SWCNT content up to 5 wt %. SEM images showed that the morphology of the hybrid membrane underwent significant changes by altering the time and energy of sonication which theses changes were in good agreement with the observed permeability enhancement for the corresponding nanocomposite membranes.

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Investigating the Parameters Affecting Fabrication and Electrochemical Properties of nanosize Pt-Impregnated Membrane Electrode Assembly (MEA) for PEMFCs

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Membrane electrode assembly (MEA) is the heart of proton exchange membrane fuel cells (PEMFCs) and it consists of a proton exchange membrane (PEM), anode and cathode electrodes (catalysts layers) made of the electrocatalyst supported on carbon and PEM material and gas diffusion layer (GDL).[1] Catalyst layer provides sites for the electrochemical oxidation of hydrogen and reduction of oxygen, channels for proton, electron, gas and water diffusion through PEMFCs. [1] electrocatalyst is the most effective parameter on the PEMFC’s performance, cost and durability. Platinum or platinum – based catalysts are mainly applied in fabrication of PEMFCs. MEAs are mainly fabricated through a direct spraying technique or coating of the catalyst slurry by a doctor blade method on the pre-swollen Nafion membrane. Since the catalyst layer is coated via a physical method and it does not penetrate into the surfaces of the Nafion membrane it shows poor mechanical and thermal stability and so affects the efficiency of electrochemical reactions at the interface of the polymer and the catalyst layer. Impregnation reduction method can overcome the above- mentioned problems since the catalyst layer is coated at the sub-surfaces of the membrane through an electrochemical reaction. In tthis work, Nafion membrane was impregnated by platinum nanoparticles at its both surfaces through two steps; absorption of platinum salt (Pt $(NH_3)_4Cl_2$ and its reduction using hydrazine as reducing agent. The effect of Pt salt solution concentration and pH and the concentration of reducing agent on the catalyst layer morphology, pore size distribution, the crystallinity of the fabricated catalyst coated membrane(CCM) and the charge transfer resistance was studied using scanning electron microscopy (SEM), x-ray diffraction (XRD) technique and impedance spectroscopy, respectively. SEM results showed that on decreasing the Pt salt solution concentration from 9.0×10^{-3} M to 2.5×10^{-3} M and lowering Pt solution pH from 9 to 4 the most uniform catalyst layer was formed at the top surface and sub-surfaces of the Nafion membrane in depth of 2 μm with the average Pt size of 30 nm. XRD results proved that the crystalinity of the prepared CCM increased by decreasing Pt salt solution pH to 4. The lowest charge transfer resistance was observed for the mentioned CCM according to the impedance spectroscopy results.

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Investigating the phase transitions in nano Al_2O_3 by infrared spectroscopy and multivariate models

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Aluminum oxide (alumina) is known as one of the most famous ceramic materials due to its various physical properties. Usually, alumina exists in 7 different polymorphs, among which, 6 are semi-stable (i.e. μ, θ, γ, δ, κ and x), have their own mechanical, physical and chemical properties this polymorphs are called transition alumina and the phase, called corundum (α-alumina), is the most stable one. During high temperature oxidation of alumina forming alloys, various transition aluminas are formed before reaching the most stable α-Al_2O_3 structure. Several researches have been conducted to obtain the knowledge about the structure and the features of transition alumina through crystallography tests, but it is not easy to determine the structure of these polymorphs using x-ray diffraction (XRD) [1]. In this work, a novel approach has been introduced based on diffuse reflectance infrared spectroscopy (DRIFTS) coupled with chemometric pattern recognition techniques to evaluate different structural phases of alumina. To reach this aim, different phases of nano alumina were synthesized with sol-gel technique at 400, 600, 900, 1000 and 1200 °C. The morphology and particle size of synthesized powders were characterized by a SEM and XRD [2]. In order to provide a new and rapid measurement technique for evaluation of different structural phases of alumina, the diffuse reflectance FTIR spectra of the powder samples were obtained in 400–4000 cm^{-1} spectral region. Raw spectra data were evaluated for classification models using different multivariate classification techniques, including principal component analysis (PCA), hierarchical cluster analysis (HCA) and linear discriminant analysis (LDA). Finally, the best classification model was obtained using LDA technique with classification correctness percentage (cc %) of 88.33%. These results confirm the capability of DRIFTS coupled with pattern recognition techniques to evaluate the different structural phases of nano alumina [3].

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

Investigation of atmospheric-pressure chemical ionization reaction products of explosive samples using SIMPLISMA - GWFA on gas chromatography-ion mobility spectrometry data

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Fast screening measurements are essential in the detection of the explosives and narcotics samples. Gas chromatography-ion mobility spectrometry (GC-IMS) offers numerous advantages that improve its applicability as a screening procedure compared to other methods. Some of these advantages are low detection limits, rapid response, high sensitivity, portability, and real-time monitoring capabilities. SIMPLe-to-use Interactive Self-modeling Mixture Analysis (SIMPLISMA) was developed as an initial estimation of the data [1]. In addition, method referred to as window target-factor analysis (WTFA) was proposed for verifying the presence of target analytes in complex spectrochromatographic applications [2]. Harrington et al. demonstrated that SIMPLISMA simplifies the complexity of the atmospheric-pressure chemical ionization (APCI) processes of IMS data and overcomes the false negative problem [3]. This method was coupled with new Gaussian window factor analysis method (GWFA) to resolve the overlapping APCI product peaks and increasing of detection ability of IMS.

In SIMPLISMA the components of the mixture must vary in concentration during the measurement period by GC. The SIMPLISMA algorithm locates pure variables (PVs) in the data set. The PV intensities are used to estimate the concentration profiles of the analytes. The drift time points are evaluated to find the points that have the largest purity and are used to model the concentration profiles. GWFA was used for precise investigation of model.

Coupling SIMPLISMA- GWFA with GC-IMS allowed sequential changes to be exploited instead of lost which could occur when averaging and simplify GC-IMS data complexity resulted by APCI (atmospheric-pressure chemical ionization) processes and new detection procedure.

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Investigation of Extractive Properties of N_2O_2-Type Schiff Base Ligands Towards Cu(II) Ions

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Copper is an industrially important metal ions and its separation, recovery and recycling from a variety of sources are important. Among the methods used to achieve these aims, liquid-liquid extraction is known as most industrially important method [1-2]. The success of this method depends crucially on the selected extracting agent. Schiff bases are known as a class of powerful extractants, applied in various metal separation and purification investigations [3]. In this study, two N_2O_2-type Schiff base extractants, bis(2-hydroxybenzaldehyde)-1,2-ethylendiamin (L_1) and bis(2-hydroxyacetophenone)-1,2- ethylendiamin (L_2), have been synthesized by the reaction of 2-hydroxybenzaldehyde and 2-hydroxyacetophenone with 1,2-ethylendiamin in ethanol. Distribution study of the prepared ligands between sodium chloride aqueous phase and dichlomethane organic solvent shows their suitability for using in solvent extraction process. The results of competitive solvent extraction of heavy metal ions reveal a significant selectivity of both ligands towards Cu(II) ions in comparison with Ni(II), Co(II), and Pb(II) ions. Both ligands transfer copper ions into the dichloromethane organic phase by a cation exchange mechanism. Analysis of the extraction data reveals that Cu^{2+} ions are extracted as the complexes with a 1:1 metal to ligand ratio by both extractants. Extraction efficiency and the influence of pH, amount of ligand, and the influence of solvent were investigated and discussed.

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Investigation of inhibitory action of self-assembled garlic extract on the corrosion protection of copper

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Abstract

The corrosion of the metals and alloys is a serious problem at industry. Among the existing techniques to prevent or minimize corrosion, the use of corrosion inhibitors is most common. Because of strict environmental regulations for using from friendly environment and safe corrosion inhibitors, the use of some inhibitors, such as chromates because of toxicity and the environmental hazards they create, has been forbidden [1]. Therefore, there is a great need to find a nontoxic replacement that is compatible with current industrial technologies. Consequently, because vegetable and plants wastes are an important source of eco-friendly corrosion/scaling inhibitors and biodegradable, available and nontoxic they have been attracted the opinion of the many researchers [2]. At this work has been used garlic as green inhibitor for protection of the copper by self-assembling method. The self-assembling is simple process that self-assembled monolayers the highly ordered molecular assemblies is formed spontaneously by chemisorptions on the copper surface [3]. The inhibitory effect of garlic extract for improving the inhibition efficiency on the copper corrosion in sodium chloride 3.5% has been investigated using impedance, Tafel, scanning electron microscopy, and energy dispersive X-Ray methods. Rresults show the inhibition efficiency increases with increasing garlic extract concentration and self-assembling time. After immersion copper for 4.0 h in 1000 ppm extract, the monolayer was able to protect the copper against corrosion 99%. The effect of temperature on the function of inhibition indicates that the inhibition efficiency increases by increasing the temperature.

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Investigation of Some Barbiturate derivatives as corrosion inhibitors for mild steel in acidic media

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Abstract:

The use of acid solution during pickling and industrial cleaning leads to corrosive attack on mild steel. The corrosion of mild steel is of fundamental academic and industrial concern that has received a considerable amount of attention. Among efficient corrosion inhibitors use to prevent the deterioration of mild steel are heterocyclic organic compounds consisting of a π-system and/or O, N, or S heteroatoms [1]. Barbituric acid and its derivatives exhibit a wide range of biological activities such as antibacterial and anti-hypertensive agents. The corrosion inhibition efficiency of four Barbiturates (namely 5-(3-phenylallylidene) pyrimidine-2,4,6-trione (PPT), 5-(2-hydroxybenzylidene) pyrimidine-2,4,6-trione (HPT), 5-benzlidenepyrimidine-2,4,6-trione (BPT) and 5-(4-nitrobenzylidene) pyrimidine-2,4,6-trione (NPT)) of carbon steel in hydrochloric acid have been shown through electrochemical impedance spectroscopy (EIS), Tafel polarization and weight loss, and found that all the examined Barbiturate compounds reduce the corrosion of carbon steel with the order of inhibition efficiency being PPT >HPT >BPT> NPT [2]. The effect of the molecular structure on the chemical reactivity has been subject of great interest in several disciplines of chemistry. The quantum chemical calculations have been widely used to study the reaction mechanisms and to interpret the experimental results as well as to solve chemical ambiguities. The correlation between the quantum chemical parameters and inhibition efficiency using quantitative structure activity relationship (QSAR) of these compounds was investigated using DFT/B3LYP and HF calculations. The inhibition efficiency of the inhibitor are closely related to the quantum chemical parameters, the highest occupied molecular orbital (EHOMO), energy of lowest unoccupied molecular orbital (ELUMO), HOMO–LUMO energy gap (ΔE_{H-L}), the hardness (σ), the softness (η) and the fraction of electrons transferred (ΔN) for the neutral inhibitors and no significant relationship was found with parameters, dipole moment (μ) and the total energy (TE).

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Investigation of Uncertainty in Quantitative Analysis Using MCR-Methods: Augmentation Type Effect

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Abstract Multivariate curve resolution (MCR) has been shown to be a powerful tool for the qualitative and quantitative analysis [1], in particular, for the investigation of chemical systems where little or no previous knowledge about the system exists [2]. Alternating least squares (ALS) is an excellent algorithm to accomplish the goals of MCR. The key issue in the quality assessment of the results obtained by any chemometric method [3], is how experimental errors and noise are propagated from experimental data to parameter estimations.

The present work describes the effect of augmentation type on uncertainty in soft modeling quantitative analysis, where both the column-wise and row-wise augmentation are possible experimentally. In the quantitative analysis by second-order calibration, the measured matrix of the mixture was augmented with the measured matrix of one of the analytes (standard matrix) and simultaneously analyzed. Dependent on the type of augmentation of standard and unknown data matrices, either resolved concentration profile, or absorbance profile, could be used to evaluate the quantitative information. In column-wise and row-wise augmentation, quantitative information is obtained from resolved concentration profiles and absorbance profile, respectively. The simulated data was used to investigate the error propagation effects in second-order calibration. Data were generated under a kinetic model, and artificial noise of known size was added. High numbers of replications were generated using new noise generations. The statistical parameters, standard deviation (s) and relative standard deviation (RSD) were used to compare the uncertainty on quantitative results of column-wise and row-wise augmented data matrix. The results indicated that, uncertainty is minimum in column-wise augmentation, for data having more selectivity in absorbance profile, and in row-wise augmentation for data having more selectivity in concentration profile.

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Investigation the potential of electrospray ion mobility spectrometry in negative mode for determination of different deoxynucleotides and oligonucleotides

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Abstract

Oligonucleotides are polymeric sequence of DNA and RNA molecules. These polymers are used in different biological research such as pharmacogenomic studies using microarrays, hybridization and as an aptamer for selective separation of an antigen. There are a variety of methods for analysis of these polymers [1]. In this work, for the first time the capability of electrospray ion mobility spectrometry (ESI-IMS) in negative mode was investigated for the determination of deoxynucleotides and oligonucleotides. The negative ion mode was chosen for this study since these compounds can easily form negative ions because of presences phosphate groups in their structures and acidic proton in phosphodiester bonds [2-3]. Parameters affecting on the electrospray performance were optimized. The results showed that methanol with flow rate of 7µL/min were the best conditions for the electrospray.

The reduced mobility values of deoxyadenosine monophosphate, deoxyguanosine monophosphate and thymidine monophosphate were calculated to be 1.10, 1.13 and 1.16 $cm^2\ V^{-1}\ S^{-1}$respectively. The detection limits of these compounds were obtained 10.3, 12.1 and 1.9 pmol respectively. Furthermore, oligonucleotides with different sequences, but with the same molecular mass were also investigated. In these cases multiple ion peaks due to multiple phosphate groups in these sequences with the reduced mobility values of 1.31, 1.43, 1.52 and 1.76 $cm^2\ V^{-1}\ S^{-1}$ were recorded. The mass spectra of these compounds were also obtained to compare the results of ESI-IMS with MS. The MS assigned the ion peaks in ESI-IMS. Finally, the advantages and limitations of IMS were demonstrated for the determination of the oligonuclides with the same molecular mass but with different nuclotide sequences.

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Ion imprinted polymeric nanobeads based on dicyclohexyl 18C6 for highly selective flame photometric determination of potassium ions in water samples

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Ion imprinted polymers (IIPs) represent a new class of materials possessing high selectivity and affinity for the target ion. Ion imprinting is a technique for creating template shaped cavities in synthetic rigid polymer matrices with memory of the template ions [1]. IIPs are prepared by copolymerizing of functional and cross-linking monomers in the presence of a template. Owing to their mechanical and chemical stability, low cost of preparation, ease of mass production and fitting for wide range of operating conditions, IIPs have been developed in wide fields, such as solid-phase extraction, chromatographic separation, catalysis and biosensor [2].

This work reports the first application of the ion imprinting technology for determination of potassium ion by precipitation polymerization method. Ion imprinted polymeric (IIP) nanoparticles were prepared by using dicyclohexyl 18C6 (DC18C6) as a K^+ ion selective crown ether, in the acetonitrile- dimethylsulfoxide (3:1; v/v) mixture as porogen. The imprint potassium ion was removed from the polymeric matrix using 0.5 M HNO3. The scanning electron microscopy (SEM) micrographs showed colloidal nanoparticles of 60-90 nm in diameter and slightly irregular in shape. The obtained ion-imprinted particles for K^+ showed selective recognition with rapid adsorption and desorption process. It was found that imprinting results in increased affinity of the material toward K+ ion over other competitor metal ions with the same charge and/or close ionic radius. The synthesized IIP nanobeads were shown to be promising for solid-phase extraction coupled with flame photometry for determination of trace K^+ ion in different water samples.

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Ionic liquid–based dispersive liquid–liquid microextraction method for preconcentration of trace amounts of copper, cobalt and zinc in environmental waters prior to flame atomic absorption spectrometry determination

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A new, rapid and sensitive procedure for simultaneous preconcentration of copper, cobalt and zinc in water samples, based on IL-DLLME as a prior step to their determination by FAAS has been developed. N,N´-bis(2-salicylaldiminato)-1,8-diamino-3,6-dioxaoctane(H_2L) was chosen as the complexing agent. In the proposed approach 1-hexyl-3-methylimidazolium hexafluorophosphate [Hmim][PF_6] and acetone were used as extraction and dispersive solvents, respectively. In order to obtain optimum conditions, the effect of different parameters such as pH, concentration of chelating agent, amounts of IL, type and volume of disperser solvent, concentration of salt, extraction time and coexisting ions were optimized.

Results and discussion

The influences of the pH on extraction recovery values of Cu, Co and Zn ions were investigated at the pH range of 3.0 –9.0. The results shown at the pH< 6.5 values the chelating agent is protonated so the extraction efficiencies are decreased, while increase in pH facilities the metal ions to hydrolyse which decreases the extraction recovery. Therefore, the pH of 6.5 was selected for the following experiments. To evaluate the effect of the extraction solvent amount, different amounts of [Hmim][PF_6] in the range of 40-120 mg were examined. Therefore, amount of 90 mg was chosen as optimum value. For selection of the dispersive solvent, various solvent such as acetone, methanol, acetonitrile and ethanol were selected. The experiments showed that the best disperser solvent was acetone. The effect of the volume of acetone on the extraction recovery was also studied in the range of 0-800 µL. By using low volume of acetone, the cloudy state of solution was not formed completely. Thus, a volume of 400 µL of acetone was used in the subsequent experiments. The influence of the ionic strength of the solution was evaluated by adding $NaNO_3$ (0.0–0.8 mol L^{-1}) into the sample solution. Based on these results, no salt was needed for further study. Table1 summarizes the analytical characteristics for the determination of copper, cobalt and zinc by IL-DLLME FAAS.

Table 1 Analytical characteristics of IL DLLME-FAAS for determination of Cu(II),Co(II) and Zn(II)

Parameter	**Cu(II)**	**Co(II)**	**Zn(II)**
Linear range(µg L^{-1})	8-400	6-500	6-150
R^2	0.9992	0.9981	0.9993
Equation	Y=0.0013x+0.0065	Y=0.0013x+0.0086	Y=0.0056x+0.0078
Enrichment factor	50	50	50
RSD% n=6	3.78 Cu Concentration: 200µgL^{-1}	3.10 Co Concentration:200 µgL^{-1}	1.76 Zn Concentration:100 µgL^{-1}
LOD(µg L^{-1}) n=10	1.90	4.42	2.45

The proposed method was applied to determine of cobalt, copper and zinc in water samples. The proposed method offers the advantages of simplicity, rapidity, and sensitivity.

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Ionic liquid-based ultrasound-assisted surfactant-emulsified microextraction as a new and novel microextraction method for simultaneous determination of para-anisaldehyde, trans-anethole and its isomer (estragole) in plant extracts and urine samples using HPLC

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Fennel, anise, and basil extracts have countless therapeutic properties that can be pointed as muscle relaxant, carminative, reducer the blood sugar, expectorant antinociceptive and antiseptic [1-3] effects. The prominent components of these extracts are para-anisaldehyde (ANA), trans-anethole (TAN) and its isomer estragole (EST), which due to close relations between their functionality and mechanism in the human body, have significant effects on the healthcare. Besides, due to highly usability of these compounds in the world as flavoring additives (for foods, medicinal and perfumery industry) and dose dependent properties of some of them (estragole as a suspected cancer agent), development of a green, rapid and effective method for their simultaneous determination in the real samples is necessary.

In this way, for the first time, a novel, efficient and environmentally friendly method termed ionic liquid-based ultrasound- assisted surfactant-emulsified microextraction (IL-USA-SE-ME) was developed for simultaneous determination of ANA, TAN and EST in different plant extracts (fennel and basil) and urine samples using HPLC. Under the optimum conditions (including extraction solvent: 90 µL of [C_6MIM][PF_6]; disperser: 5 mg of N-dodecylbenzenesulfonic acid sodium salt; sample pH: 3; sonication time: 5 min; and centrifuging time: 5 min), limits of detection, limits of quantification, and recoveries were in the range of 16 - 22 ng mL^{-1}, 49 - 67 ng mL^{-1}, and 94.31 % - 101.1 %, respectively.

Furthermore, due to their similar structures and characteristics, these compounds (especially trans-anethole and estragole) usually coexist and interfere with each other, so their simultaneous separation was also examined and a convenient HPLC-UV method was developed. The results showed that IL-USA-SE-ME coupled to HPLC can be a good choice as an efficient microextraction method for simultaneous determination of these compounds in liquid matrices.

Keywords: Ionic-liquid based ultrasound-assisted surfactant-emulsified microextraction, para-anisaldehyde, trans-anethole, estragole, urine, plant extract.

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Ionic liquid-based ultrasound-assisted surfactant-emulsified microextraction as a new and novel microextraction method for simultaneous determination of para-anisaldehyde, trans-anethole and its isomer (estragole) in plant extracts and urine samples using HPLC

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Kinetic and equilibrium study of biosorption of Cr(VI) by *Sargassum ilicifolium*, application of response surface methodology

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Biosorption is an innovative technology that employs inactive and dead biomass for the recovery of heavy metals from aqueous solutions. The major advantages of the biosorption technology are its effectiveness in quickly reducing the concentration of heavy metals ions to very low levels with high efficiency and the use of inexpensive biosorbent materials. These characteristics make biosorption an ideal alternative for treating high volumes of low concentration complex wastewaters. Biosorption can be considered a collective term for a number of passive, metabolism independent, accumulation processes and may include physical and/or chemical adsorption ion exchange, coordination, complexation, chelation and micro precipitation [1, 2]. In this study, effects of operational parameters on biosorption of Cr(VI) by brown macroalga *Sargassum ilicifolium* were studied using response surface methodology. Removal process was influenced significantly by the variation of pH. Optimum conditions were found to be initial pH 2, biosorbent concentration 0.2 g/l and initial Cr(VI) concentration 200 mg/l at room temperature and contact time of 7h. The maximum uptake capacity for Cr(VI) was found to be 203 mg/g. The thermodynamic and kinetic parameters were also evaluated from the biosorption measurements. The abundant and economic biomass *Sargassum ilicifolium* could be used for removal of Cr(VI) from wastewater.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

Kinetic spectrophotometric insight into the pathway leading to cyclic phosphorus ylide formation in the presence of 3-chloropentane-2,4-dione: stopped-flow technique

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Monitoring the zwitterionic intermediates of the synthesized phosphorous ylide compounds for the kinetics and mechanism studies were a challenge [1]. The stopped-flow technique reduces the manipulation involved in the determination step and intermediate formation and allows measurements to be made instantly after mixing [2]. Herein, spectral analyses of the formation and decay, and effect of the zwitterionic intermediate on the mechanism of the reaction between triphenylphosphine and dimethyl acetylendicarboxylate in the presence of 3-chloropentane-2,4-dione has been investigated as a model. Pro-K analyzer confirmed the existence of 6 species assigned in consistent of the proposed mechanism of the reaction. In the solvent with upper dielectric constant, because of the ionic structure of intermediate, the rate of forward step (k_1) was increased and the rate of backward step (k_{-1}) was contrary decreased. Moreover the order of reaction in fast step of the reaction was found to be first order with respect to each reactant. Experimental kinetic data (k_1, k_{-1}) along with activation parameters ($\Delta G^{\neq}$, $\Delta S^{\neq}$ and $\Delta H^{\neq}$) were recognized for the fast step reaction [3].

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Kinetic study on removal of Perchlorate from aqueous solutions using stabilized zero-valent iron nanoparticles

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Perchlorate (ClO_4^-) is used as an oxidizer in flares, pyrotechnics, explosives and the recent detection of environmental contamination has primarily been associated with its use in rocket propellants and missile motors. Perchlorate is a concern because it can interfere with iodide uptake by the thyroid gland. Because iodide is an essential component of thyroid hormones, perchlorate exposure may result in decreased thyroid hormone production. It is highly water soluble, non-complexity, non-volatile and chemically stable for its unique chemistry, it has been highly challenging to remove perchlorate from water by traditional water treatment approaches[1]. In recent years, various treatment technologies have been developed and tested, including biological reduction ion exchange, tailored activated, carbon sorption, filtration and chemical reduction. However, these technologies are limited by some critical technical and economic drawbacks such as slow degradation kinetics and production of large volumes of concentrated process waste residuals. In recent years, zero-valent iron (ZVI) has attracted increasing interest for abiotic dechlorination of chlorinated organic compounds removal of nitrate, reduction of chromate, and sorption of arsenic). ZVI was also tested for reduction of perchlorate. It has been reported that decreasing the size of ZVI particles to the nano scale can greatly enhance the reaction rates for perchlorate a stabilizer are actually agglomerates of ZVI particles in the micron scale[2].

In this work, starch solution was used to modify particle size (dispersion those better and avoid getting lumps) and stability is preserved. The results of the analysis XRD (X-ray diffraction), DLS (dynamic light scattering) and TEM (transmission electron microscopy) show that ZVI nanoparticles synthesized with particle size distribution of 28-54 nm. Under optimum conditions (0.45 g/lit of NZVI, pH=5-6, time= 50 s and $T=65^{\circ}C$), removal of perchlorate was nearly complete. The activation energy of perchlorate reduction using ZVI was obtained 39.83 kJ/mole and rate constant increase from 0.0147 to 0.057 min^{-1} at different temperature.

Keywords: Kinetic study, Removal of Perchlorate, Stabilized zero-valent iron nanoparticles.

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Laccase immobilization on the electrode surface to design a biosensor for the detection of phenolic compounds such as catechol

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Laccases belong to the largest subgroup of multicopper oxidases [1]. Laccases can catalyze the oxidation of various aromatic substrates as phenol. Laccases have been employed for the design of biosensors, the detection of phenols in wastewaters or in food industry applications [2]. The identification and quantification of phenolic compounds are important for environmental monitoring because many of them showing harmful effects on plants, animals and human health. Laccase biosensors can make ideal sensing systems to monitor the effects of phenolic compounds on the environment [3].Catalytic activity of enzymes varies with different environmental conditions such as pH. We work at different pHs for free laccase. The kinetic constants, K_m and V_{max}, were obtained 755 μM/min and 95.9 μM, respectively, for laccase from Trametes versicolor in phosphate buffer pH 3. A method was designed to construct an electrochemical biosensor for detection of phenolic compounds based on covalent immobilization of laccase onto polyaniline electrodeposited onto a glassy carbon electrode via glutaraldehyde coupling. After immobilization of laccase onto the surface of modified electrode and construction of biosensor, we used this biosensor for detection of catechol. Linear range, sensitivity, and detection limit were obtained 3.2×10^{-6} to 19.6×10^{-6} M, 0.7067 μA/μM, 2.07×10^{-6} M respectively. Atomic force microscopy (AFM) was also used for the topographic characterization of the laccase immobilized onto polyaniline by glutaraldehyde. From AFM data we concluded that laccase was immobilized onto the surface of the modified electrode.

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Large Scale Synthesis of Heterogeneous Nanocatalyst-based carbon Nanocomposite through Three-Dimensional formation of C-C Bonds for Kinetic and Catalytic Study on the Gas Phase reaction of Some Light Organic Compounds

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Growth on transition metal substrates is becoming a method of choice to prepare large area C-C nanocomposite, as a method for delivering the best compromise between cost and quality [1]. This method used to prepare nickel support (catalyst of nickel) based on C-C nanocomposite (graphene) by naphthalene as procedure. The nanocatalyst prepared by using chemical vapor deposition (CVD) and/or high isostatic pressure (HIP) methods [2]. CVD or other methods used to bring carbon atoms on or into the nickel films. The prepared nanocatalyst was characterized by scanning electron microscope (SEM). Then; it was used in a flow system to study the effect of heterogeneous nanocatalysts for thermal decomposition (pyrolysis) of some light organic compounds [3]. The products have been detected by gas chromatography (GC). The GC result exhibited a sharp peak for methane test. The reaction carried out at a temperature of 300 °C and pressure of 100 atm. In this study, kinetic relationships were evaluated in detail in the presence and absence of C-C composites on the surface of Ni support. The results reveal that modification of Ni support with C-C composites significantly promotes the gas phase reaction of light organic compounds such as methane. Consequently, using C-C nanocomposite on Ni support provides proper site for oxidation of methane and provides feasibility of catalyst recovery and possibility of reaction at lower temperatures while increasing the reaction efficiency.

Keywords: C-C nanocomposite, Nanocatalyst, Gas phase reaction, Oxidation.

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Lead ion removal from polluted waters by walnut skin nanosorbent

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Walnut skin nanoparticles are prepared by an innovative vibratory ball mill (Spex). The innovated spex consists a stainless steel cylindrical tank with 10 cm inner diameter and 20 cm inner height. 100 g walnut skin powder with together 200 hard chromium electroplated balls are filled in the tank and vibrated by 1200 rpm for 10 h. SEM and TEM images show that the milled powder includes uniform nanoparticles with 70 nm average diameters. The prepared walnut nanopowder is used as a new nanosorbent for removal of lead ions from polluted waters. Experimental data shows that the adsorption of lead ions on the surface of walnut skin nanoparticles is acceptably fitted to Langmuir isotherm model. Based on the Langmuir isotherm, a maximum adsorption capacity of 17 $mg.g^{-1}$ is achieved for adsorption of lead ions on the surface of walnut skin nanoparticles. The experimental conditions to remove lead ions from waters are optimized by the "one at a time" method. The optimum conditions for lead ions removal includes pH=4, 500 ml sample volume, 50 mg sorbent, retention in room temperature, retention time lower than 5 s. The adsorbed ions are easily eluted by 1.5 ml 0.5 M HNO_3 solution. Under the optimal conditions, preconcentration factor of 200, relative standard deviations (%RSD) of 4.32% and the detection limit of 0.10 ppb are obtained. The effects of some ions such as Cu^{2+}, Ni^{2+}, Fe^{2+}, Zn^{2+}, Mg^{2+} and Na^{+} on the removal efficiency of lead ions. The presented method is successfully applied for removal and determination of lead ions from real samples.

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Ligand-less rapidly synergistic cloud point extraction as an efficient method for the separation and preconcentration of trace amounts of lead from food and water samples

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Recently, chemical pollution with heavy metals has been one of the serious problems for sustainable environment. Lead is one of the most abundant heavy metals in the environment and its toxic effects have been recognized since a long time. Lead is released into the environment through several processes. The World Health Organization (WHO) has released the guidelines for drinking water quality containing the guideline value 0.01 mg Pb L^{-1} [1]. Consequently, the accurate determination of lead at trace levels in environmental samples is a subject of great concern. Recently, rapidly synergistic cloud point extraction (RS-CPE) was introduced by Wen and coworkers [2, 3]. It is based on the use of a synergic reagent for decreasing the cloud point temperature of surfactants in traditional CPE method. Triton X-114 was one of the most applied surfactants in CPE for its lower cloud point temperature (30 °C). In this method, octanol functioned as cloud point revulsant of Triton X-114 and synergic reagent for extraction. Rapid cloud point extraction was realized at room temperature (about 20 °C) without heating units or adding salts. Thus, traditional CPE pattern was considerably simplified and accelerated. Only 1 min was needed for the improved extraction compared with traditional CPE (about 40 min). Compared to traditional CPE method, the proposed method is low time-and labor-consuming. There is no need to heat and/or cool solutions for separating phases which, as a result, avoids any possible losses due to volatilization and adds greater stability to phases once they have been separated. In this study, a simple RS-CPE procedure has been developed for the separation and preconcentration of trace amounts of lead from food and water samples by flame atomic absorption spectrometry (FAAS). Non-ionic surfactant Triton X-114 was used as extractant and octanol worked as cloud point revulsant and synergic reagent which lowered the cloud point temprature of Triton X-114 and assisted the subsequent extraction process. Various factors affecting extraction efficiency were evaluated and optimized. Under optimized conditions, an enhancement factor of 40 could be obtained, and the detection limit (LOD) for lead was 1.6 μg L^{-1}. Relative standard deviation for ten replicate determinations of the standard solution containing 100 μg L^{-1} lead was 2.1%. The proposed method was successfully applied for the determination of lead in food (spinach, rice, and black tea bag) and water samples (tap, river, spring, mineral, and sea water) with satisfactory results.

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Low-density solvent-based dispersive liquid–liquid microextraction for the fast determination of ethion in grape and underground water by negative corona discharge ion mobility spectrometry (NCD-IMS)

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In this research, coupling of dispersive liquid–liquid microextraction and negative corona discharge-ion mobility spectrometry (DLLME-NCD-IMS) was employed for preconcentration and determination of ethion in grape and underground water. The most commonly used solvents in DLLME method are chlorinated ones which are dangerous for human and environment [1]. On the other hand, chlorinated solvents have a high electron affinity, resulting in complication of mobility spectrum in addition to diminution in sensitivity. To overcome these problems, cyclohexane was used as the extraction solvent which is lighter than water and therefore, the extraction solvent containing analyte can be accumulated in upper layer of aqueous phase. In this method, dispersive solution containing 500 µL disperser solvent (methanol) and 40 µL extraction solvent (cyclohexane) was injected rapidly into an aqueous solution contains ethion in a special vessel with a syringe. After centrifuging of solution at 3000 rpm for 5 min, the fine droplets of cyclohexane were collected from the upper area of the extraction vessel. Then, 2 mL distilled water was injected through the septum located in the bottom of extraction vessel, so that the organic phase could be easily collected using a syringe [2]. 5 µL of the collected phase was injected into the NCD-IMS to analyze the extracted ethion. Some important parameters such as the kind of extraction and dispersive solvents and their volume, salt concentration, and sample pH were investigated. Under optimized conditions, linear range (0.2-100.0 µg/L), detection limit (0.075µg/L), and RSD (7%) were obtained. In analysis of spiked samples, the enrichment factor and extraction recoveries were 60 and 68-92%, respectively. In addition, some samples of underground water and grape were also analyzed and the satisfactory results reveal the capability of DLLME-NCD-IMS for convenient and very rapid alalysis of real samples.

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LSPR-based gold nanobiosensor for determination of thyroid stimulating hormone(TSH)

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Recently, LSPR-based nanobiosensors were developed as tools for highly sensitive, and flexible sensing techniques for the detection of biomolecular interactions [1]. Localized surface plasmon resonance (LSPR) is induced by incident light when it interacts with noble metal nanoparticles that have smaller sizes than the wavelength of the incident light. LSPR is a non-specific technique and the specificity is only achieved through biomolecular recognition elements such as antibodies. The unique properties of gold nanoparticles have stimulated the increasing interest in the application of GNPs in interfacing biological recognition events with signal transduction and in designing biosensing devices exhibiting novel functions [2]. Thyroid stimulating hormone (TSH) is a hormone that stimulates the thyroid gland to produce thyroxine (T4) and the triiodothyonine (T3) which stimulates metabolism of almost every tissue in the body. The determination of serum or plasma levels of TSH is recognized as a sensitive method in the diagnosis of primary and secondary hypothyroidism [3]. In this work, GNPs were synthesized by sonochemical procedure. The size of the particles was about 15 nm. The GNPs were modified by anti-TSH antibodies. TSH can be bond to bioconjucated GNPS and resulted in wavelength shift observed from localized surface plasmon resonance. The modified GNPs were used as a sensitive and selective tool for determination of TSH hormone.

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LSPR-based gold nanobiosensor for determination of thyroid stimulating hormone(TSH)

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Recently, LSPR-based nanobiosensors were developed as tools for highly sensitive, and flexible sensing techniques for the detection of biomolecular interactions [1]. Localized surface plasmon resonance (LSPR) is induced by incident light when it interacts with noble metal nanoparticles that have smaller sizes than the wavelength of the incident light. LSPR is a non-specific technique and the specificity is only achieved through biomolecular recognition elements such as antibodies. The unique properties of gold nanoparticles have stimulated the increasing interest in the application of GNPs in interfacing biological recognition events with signal transduction and in designing biosensing devices exhibiting novel functions [2]. Thyroid stimulating hormone (TSH) is a hormone that stimulates the thyroid gland to produce thyroxine (T4) and the triiodothyonine (T3) which stimulates metabolism of almost every tissue in the body. The determination of serum or plasma levels of TSH is recognized as a sensitive method in the diagnosis of primary and secondary hypothyroidism [3]. In this work, GNPs were synthesized by sonochemical procedure. The size of the particles was about 15 nm. The GNPs were modified by anti-TSH antibodies. TSH can be bond to bioconjucated GNPS and resulted in wavelength shift observed from localized surface plasmon resonance. The modified GNPs were used as a sensitive and selective tool for determination of TSH hormone.

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Magnetic chitosan nanoparticles as solid phase extractor for spectrophotometeric determination and separation of carmoisine

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Magnetic nanoparticles have been recognized as a new adsorbent with large surface area and small diffusion resistance. Iron oxide nanoparticles (IONPs) have been studied extensively due to their wide range of applications in ferrofluids, high-density information storage, magnetic resonance imaging (MRI), biological cell labeling and sorting, separation of biochemicals, targeting, and drug delivery. For many of these applications, surface modification of IONPs is a key of challenge. In general, surface modification can be accomplished by physical and/or chemical adsorption of the desired molecules in order to coat the surface, depending on the specific applications. Several reports have been published on the using of magnetic nanoparticles for separation and determination of metals ions, organic compounds and dyes.

In this research, new synthesized chitosan-coated IONPs was employed for the determination of carmoisine in water samples. IONPs were prepared from the $FeCl_3\cdot 6H_2O$ and $FeCl_2\cdot 4H_2O$ and coated with chitosan. The particles were ageing in the solution under mechanical stirring, decanted by magnetic settling, and washed three times with distillated water.

The effects of various parameters such as pH, type and concentration of eluent and its volume, eluting agents, time of sttiring, amount of adsorbent, sample volume, PZC were studied in details. The calibration graph was linear in the range of 20-150 ng mL^{-1} for carmoisine. The limit of detection based on 3Sb was 6.43 ng mL^{-1}. The relative standard deviation (R.S.D) for ten replicate measurements of 40 and 120 ng mL^{-1} was 3.80 and 2.18%, respectively. The method was applied to the determination of carmoisine in water sample.

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Magnetic Fe_3O_4 nanoparticles modified with N^2,N^6-di(thiazol-2-y1)pyridine-2,6-dicarboxamide and their application for removal of Cd(II) & Zn(II) from wastewater samples

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Abstract:

A novel magnetic nano-adsorbent has been synthesized by the covalent immobilization of N^2,N^6-di(thiazol-2-y1)pyridine-2,6-dicarboxamide on the surface of Fe_3O_4 nanoparticles. Size, structure, magnetic property and porosity of the prepared magnetic nanoparticles (MNPs) were studied by SEM, AFM, TEM, VSM, FT-IR and XRD analysis [1]. The ability of the prepared MNPs for removing heavy metals ions (Zn^{2+} and Cd^{2+}) from industrial wastes was studied and the effects of different affecting parameters such as metal ion concentration, background electrolytes, contact time, temperature of solution and pH of solution, on the heavy metal ion uptake capacity of the prepared nanoparticles were explored [2]. The maximum adsorption capacities of Zn^{2+}, Cd^{2+} were found to be 119.04 and 55.86 mg g^{-1}, respectively. Complete removal of Zn^{2+} and Cd^{2+} from solution was performed less than 1 h. the optimum pH values for removal of Zn^{2+} and Cd^{2+} were 3 and 7, respectively. Excellent adsorption capacity of the modified nano-adsorbent together with other advantages such as reusability, easy separation, environmentally friendly composition and freedom of interferences of alkaline earth metal ions make them suitable adsorbents for removal of heavy metal ions from environmental and industrial wastes. The obtained results showed that surface modified Fe_3O_4 magnetic nanoparticles could be used as a cheap and efficient adsorbent material for removal of cationic ions form aqueous solutions [3].

The obtained results indicated that surface modified Fe_3O_4 magnetic nanoparticles could be used as a cheap and efficient adsorbent material for removal of cationic ions form aqueous solutions. Excellent adsorption capacity of the modified nano-adsorbent together with other advantages such as reusability, easy synthesis, easy separation and more specially freedom of interferences of alkaline earth metal ions make them suitable alternatives to the well-known adsorbents for removal of heavy metal ions from industrial wastewaters.

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Magnetic Fe_3O_4 nanoparticles modified with novel polyamic hydrazide and their application for removal of Cd(II) & Pb(II) from wastewater samples

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Abstract:

A novel magnetic nano-adsorbent has been synthesized by the covalent immobilization of polyamic hydrazide on the surface of Fe_3O_4 nanoparticles. Size, structure, magnetic property and porosity of the prepared magnetic nanoparticles (MNPs) were studied by SEM, AFM, TEM, VSM, FT-IR and XRD analysis. The ability of the prepared MNPs for removing heavy metals ions (Pb^{2+} and Cd^{2+}) from industrial wastes was studied and the effects of different affecting parameters such as metal ion concentration, background electrolytes, contact time, temperature of solution and pH of solution, on the heavy metal ion uptake capacity of the prepared nanoparticles were explored. The maximum adsorption capacities of Pb^{2+}, Cd^{2+} were found to be 119.04 and 55.86 mg g^{-1}, respectively. Complete removal of Pb^{2+} and Cd^{2+} from solution was performed less than 1 h. the optimum pH values for removal of Pb^{2+} and Cd^{2+} were 5 and 6, respectively. Excellent adsorption capacity of the modified nano-adsorbent together with other advantages such as reusability, easy separation, environmentally friendly composition and freedom of interferences of alkaline earth metal ions make them suitable adsorbents for removal of heavy metal ions from environmental and industrial wastes. The obtained results showed that surface modified Fe_3O_4 magnetic nanoparticles could be used as a cheap and efficient adsorbent material for removal of cationic ions form aqueous solutions.
The obtained results indicated that surface modified Fe3O4 magnetic nanoparticles could be used as a cheap and efficient adsorbent material for removal of cationic ions form aqueous solutions. Excellent adsorption capacity of the modified nano-adsorbent together with other advantages such as reusability, easy synthesis, easy separation and more specially freedom of interferences of alkaline earth metal ions make them suitable alternatives to the well-known adsorbents for removal of heavy metal ions from industrial wastewaters.

Keywords: Magnetic nanoparticle; Polyamic hydrazide; Lead; Cadmium; Heavy metal;

Fe_3O_4.

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Magnetic Fe_3O_4@C nanoparticles as a new adsorbent for preconcentration of copper prior to its determination by AAS

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Recently, many research groups have developed various nanomaterials for sample preaparation in biological and chemical analysis and among them magnetic nanoparticles (MNPs) have attracted great attention due to its rapid magnetic separablity from matrix. Like other adsorbents when certain special functional ligands with affinity for target ions are bound onto these magnetic nanoparticles, selective preconcentration of target ions from complex environmental matrices can be obtained. In this study, a new type of magnetic Fe_3O_4@C core–shell nanoparticles have been synthesized by a simple strategy and it was used as adsorbent for preconcentration and determination of trace copper in various matrices after its modification with 1-(2'-thiazolylazo)-2-naphthol (TAN). There are different methods for binding of these ligands to nanomaterial surfaces. In this study, the carbon shell around MNPs has been used as mediator for binding of TAN as a ligand. Due to the high surface area and excellent adsorption capacity of this sorbent satisfactory concentration factor and extraction recoveries can be produced with only 0.03 g Fe_3O_4@C@TAN core–shell nanoparticles. The main analytical factors affecting this extraction procedure such as: TAN amount, pH, agitation time for complete sorption, mass of adsorbent, desorption of metal ions from adsorbent and sample volume were studied by flame atomic absorption spectrometry. The linear range of calibration curve for Cu(II) was 0.01 -0.4 μgmL^{-1} with a correlation coefficient (R^2) of 0.9983 and the detection limit was obtained 4.4 $ngmL^{-1}$.

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Magnetic nanoparticles based dispersive micro-solid-phase extraction as a novel technique for coextraction of acidic and basic drugs from biological fluids and waste water

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The coextraction of acidic and basic drugs from different samples is considerable and disputable in sample preparation methods [1]. In this study, simultaneous extraction of acidic and basic drugs with magnetic nanoparticles based dispersive micro-solid phase extraction followed by high performance liquid chromatography-ultraviolet detection was introduced for the first time. Cetyltrimethyl ammonium bromide-coated Fe_3O_4@decanoic acid as an efficient sorbent was successfully applied to adsorb diclofenac (DIC) and diphenhydramine (DPH) as acidic and basic model compound, respectively. First, appropriate amount of synthetic Fe_3O_4@decanoic acid nanoparticles was added to aqueous solution of drugs. Then pH of the solution was adjusted and afterwards cetyltrimethyl ammonium bromide (CTAB) was added to the mixture, while solution was stirred at a constant rate. After the drugs adsorption and decantation of supernatant with a magnetic field the sorbent was eluted with methanol by fierce vortex. It was shown that the application of an appropriate sorbent and method, the drugs extraction of different classes proved to be possible. The effective parameters on the extraction efficiency were optimized and obtained as: pH of the sample = 9, concentration of CTAB = 0.2 mmol L^{-1}, amount of sorbent = 10 mg, extraction time = 5 min, no salt addition to sample, type and volume of the eluent = 50 µL methanol, desorption time = 1 min. Under the optimum conditions, percent of extraction recovery, detection limits, linear dynamic ranges, and relative standard deviations (n = 5) were achieved in the range of 47.3-60, 1.8-3.0 µg L^{-1}, 5-1500 µg L^{-1}, 5.2-9.0 for DPH and 64-76.7, 1.5-3.5 µg L^{-1}, 5-1500 µg L^{-1}, 5.1-5.8 for DIC respectively for various samples. Ultimately, the applicability of the method was successfully confirmed by extraction and determination of the DIC and DPH in human urine, plasma and waste water samples in the range of microgram per liter and satisfactory results were obtained.

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Magnetic nanoparticles functionalized with sodium dodecyl sulfate for the preconcentration and determination of Atenolol

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Abstract:

Magnetic nanoparticles (MNPs) have many potential for biomedical applications such as magnetic separation, cell labeling, targeted drug delivery, hyperthermia treatment of solid tumors and contrast agents for magnetic resonance imaging [1,2]. Recently hemimicelles and admicelles have been used as novel sorbents for SPE of organic compounds. Moreover, several SPE methods based on surfactant-coated Fe_3O_4 MNPs have been reported [3,4]. Atenolol is a beta-adrenoreceptor antagonist, a beta-1 blocker, because it is cardioselective and mainly affects the heart, competing for receptor sites on the cardiac muscle. This slows down the strength of the heart contractions and reduces its oxygen requirements and the volume of blood it has to pump. So, determination of its concentration in biological fluids is very important.

In this study, a novel extraction/preconcentration method based on sodium dodecyl sulfate coated Fe_3O_4 nanoparticles was employed for preconcentration of Atenolol from aqueous and urine samples followed by their determination by spectrophotometric method. Various parameters affecting the adsorption of the analyte on magnetic nanoparticles, such as surfactant type, surfactant amount, desorption conditions, extraction time, amount of asorbent and sample volume were studied and optimized. Under optimal conditions, the enrichment factor of 190 was obtained. Recoveries of analyte from urine samples were in the range of 92 to 98% and relative standard deviation was lower than 1.8 %.

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Magnetic nanoparticles-based solid phase extraction and preconcentration of trace amount of Al^{3+} in biological sample

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Abstract:

Aluminum is a non-essential element to which humans are frequently exposed. During recent years, much interest has been raised by the toxicity and biological effect of aluminum. Some studies suggest that aluminum may be accumulated in the brain via different routes (drinking waters, food, and medicines) and interfere with the normal activities of nervous system. This metal ion has been considered as a possible cause of renal osteodystrophy, Parkinson disease and Alzheimer’s disease [1]. In the past decade, the synthesis of magnetite nanoparticles has been intensively developed not only for its great fundamental scientific interest but also for many technological applications in targeted drug delivery, bio-separation and separation and preconcentration of various anions and cations, due to their novel structural, electronic, magnetic and catalytic properties [2-4].

In this work, an SPE method which employed 3-mercaptopropionic acid modified 3-aminopropyl triethoxysilane coated Fe_3O_4 nanoparticles as the absorbent was proposed for separation and preconcentration of aluminum prior to its determination by ET-AAS. Several experimental variables affecting the adsorption of metal ion on magnetic nanoparticles were investigated in detail. Under optimal conditions, maximum recovery was 96% with enrichment factor of 192. The detection limit was obtained 0.21 ng mL^{-1} and relative standard deviation was 2.1%. The proposed method has been applied for the determination of aluminum in biological samples with satisfactory results.

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Magnetic Solid Phase Extraction of Sulfur Contaminants by Fe_3O_4 Magnetic Nanoparticles Modified by Polythiophene

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Crude oil and its distillates contain significant amounts of polycyclic aromatic sulfurated hydrocarbons (PASHs) such as, thiophene, benzothiophene, dibenzothiophene and their alkylated derivatives [1]. In the recent years, these compounds have been of concern because they constitute a major class of ubiquitous environmental contaminants found in both air and water. As the concentration levels of PASHs in environmental samples are low, a pre-treatment step is usually needed. Liquid-liquid extraction (LLE) and solid phase extraction (SPE) techniques have been widely used for this purpose.

In the present work, Fe_3O_4/Polythiophene magnetic nanoparticles was successfully synthesized in aqueous media. The prepared material was characterized by scanning electron microscopy, X-Ray diffraction patterns, Fourier transform infrared spectroscopy and thermogravimetric analysis. Then it was used as a new sorbent for magnetic solid phase extraction. The extraction behavior of the synthesized nanoparticles towards thiophen, benzothiophen and di-benzothiophen, was examined.

To achieve the best performance, affecting parameters on adsorption and desorption processes such as, adsorption and desorption time, salt effect, desorption solvent type and volume, were optimized. In the optimum conditions, repeatabilites and reproducibilities were between 2.5-5.9 and 3.0-6.2 % respectively. Linear ranges were varied in the range of 0.05-2.5 μg/mL for thiophene and 0.05-5 μg/mL for benzothiophene. Detection limits were between 5-10 ng/mL. Recoveries were found in the range of 98-105%. As real samples, two well water samples were studied.

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Magnetically Assisted Uranyl Removal by Using Magnetic Hydroxyapatite Nanocomposite

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Abstract

Uranium is likely to occur as a contaminant in the environment as a result of the combustion of coal and other fuels, natural weathering of igneous rocks and nuclear industry. Hydroxyapatite is a calcium phosphate mineral that is chemically similar to the components of bones [1]. It is an ideal material for long term containment of contaminants such as uranium, strontium, and other heavy metals due to high sorption capacity, low water solubility, high stability under reducing and oxidation conditions, availability and low cost [2]. However, due to hard task of isolating the suspended fine solids from aqueous its application has limit. In order to improve its applicability for the purification of contaminated water, several composite materials which combine HAP with polymers and magnetite (Fe_3O_4) have been reported [3]. In this study, nanocomposite absorbents, HAP/Fe3O4, with different weight ratio was prepared for removing uranyl ions from aqueous solution. Prepared nanocomposites were characterized by XRD, SEM, FTIR and VSM techniques. In addition, sorption capability of prepared HAP/Fe_3O_4 to uranyl ions was investigated and affecting parameters on adsorption efficiency like, adsorbent dosage, solution pH, shaking time, ionic strange and coexistent interferes were investigated. It is remarkable that using the HAP/Fe_3O_4 absorbent, more than 97% of uranyl can be removed under desirable experimental conditions. The maximum adsorption capacity for HAP/Fe_3O_4 is 47 mg/g. Mechanistic and thermodynamic studies also were performed and obtained results will be presented here. Finally, prepared HAP/Fe_3O_4 successfully tested in wastewater samples.

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Magnetically responsive polycaprolactone nanoparticles for progesterone screening in biological and environmental samples using gas chromatography

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Abstract

Measurement of serum or plasma progesterone levels is used as an index to monitor ovulation and investigate luteal function. Plasma concentrations of progesterone are approximately 4-20 ng/ml during the follicular phase and luteal phase of the menstrual cycle. Currently, progesterone is measured using costly methods such as enzyme immunoassay techniques. In this research, a new Fe_3O_4/ poly-ϵ-caprolactone (PCL) magnetite microspheric material have been successfully developed as the magnetic-mediated solid-phase extraction nano sorbent in dispersion mode (MM-SPE-NS) for the determination of progesterone hormone in biological and environmental matrices, via quantification by GC-FID. The magnetite Fe_3O_4 core of this nano sorbent was fabricated by co-precipitation of ferrous (Fe II) and ferric (FeIII) aqueous solution in the presence of ammonium hydroxide [1,2], then nanomagnetite particles were coated with poly-ϵ-caprolactone (PCL) by emulsion polymerization[3]. The resultant material was characterized by scanning electron microscopy, X-ray diffraction and FTIR. This magnetic microspheric material can be easily dispersed in aqueous samples and retrieved by the application of external magnetic field via a small piece of permanent magnet. The MM-SPE-NS process for progesterone assay involved the dispersion of the sorbent in feed samples with sonication, followed by magnetic aided retrieval of the sorbent and solvent (methanol) desorption of extracted progesterone for GC-FID analysis. In order to obtain high enrichment and extraction efficiency of the analyte using this novel sorbent, the main parameters were optimized. Under the optimized extraction conditions, this work demonstrates the usefulness of MM-SPE-NS in the rapid and highly sensitive monitoring of trace amounts of analyte. For progesterone, LOD were 1.00 ng/mL and LOQ were 3.3ng/mL, respectively. The relative recoveries in real samples were quite good. Linearity was observed over a wide range of 2.2–10000 ng/mL and 10-70 mg/L. The results obtained suggest that this method can be an alternative to the other time consuming and expensive progesterone assay methods already used in this field.

Keywords: progesterone, Fe_3O_4/ poly-ϵ-caprolactone, HPLC

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Malva sylvestris flower as low cost sorbent for the removal of Pb (II) ions from wastewaters

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The use of natural materials as low cost and green sorbents for the removal of heavy metal ions from aqueous solutions has recently received increasing attention. The present work proposes the use of Malva sylvestris flower as a natural adsorbent for removal of toxic Pb(II) ions from wastewaters. The anodic stripping voltammetry (ASV) method was used for quantitative determination and study of the ion Pb(II) adsorption on the solid phase.

Effective parameters in this study such as solution pH, biosorbent particle size and dosage, contact time, initial metal ion concentration, temperature and agitation speed were optimized by Taguchi experimental design method. Using this method the optimum conditions was obtained at pH 4.5, with 0.2 g of adsorbent, 60 ppm lead ion concentration, at temperature 55 °C, with 15 min contact time, sorbent size of mesh number 30 (595 µm) and 500 rpm agitation speed.

Pb^{2+} biosorption by Malva sylvestris was found to be significantly better fitted to the Langmuir isotherm (R2 = 0.995). According to the evaluation using the Langmuir isotherm, the maximum biosorption capacities of Pb^{2+} onto Malva sylvestris was 25.64 $mg.g^{-1}$.

The effect of some common cations (250 ppm of Na^+, K^+, Mg^{2+}, Ca^{2+} and Fe^{2+}) in 60 ppm Pb^{2+} solution on the biosorbent capacity was investigated, which there were any significant interference for the mentioned cations.

Also kinetic studies showed that the adsorption reaction fitted (R^2 =1) with a pseudo-second order equation with the initial adsorption rate constant of 208.33 $mg.g^{-1}.min^{-1}$. The change of adsorption with time showed that about 80% of process is completed in the first 3 min.

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Measuring Selenium in solid samples by hydride generation flam atomic adsorption spectroscopy

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Selenium as a nonmetallic chemical element, has received High attention of biologists because of its dual role as an essential trace nutrient and a toxic element. This dual effect has been recognized for many naturally occurring chemical determinations of these elements at such ranges needs a relative sensitive technique with enough low detection limit [1]. Atomic absorption spectrometry methods provide simple and low cost techniques for such measurements. However, their detection limits in simple instrumentations lies in ppm ranges[2]. Direct solid sample spectrometry is important because it decreases time consumption for preparation of sample solutions and use of contaminants reagents like acids, for analysis of samples.

In the present work Selenium is determined by hydride generation flame atomic absorption spectrometry[3].Selenium as a heavy metal element is determined directly by atomic absorption spectrometry in solid samples. In this method sample digestion and preparation steps are eliminated. To achieve the best results, first a slotted quartz tube is fabricated as an atom trapping device. This device is then, employed for determination of Nano and microgram amounts of selenium in solid samples. The powder of solid samples and viscose solutions after vaporization of their solvents can be analyzed by this method directly.RSD for 4 times measurement is 5.44% and LOD is 5 ppm.

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Mercapto-nanoporous fructose modified carbon paste electrode for simple and sensitive detection of omeprazole in biological samples

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Abstract

Omeprazole is a substituted benzimidazole that inhibits gastric acid secretion by covalently binding to the proton pump (H^+/K^+ ATPase) at the surface of gastric parietal cells, and inhibiting the final step in secretion of the hydrogen ions into the gastric lumen [1]. Only few methods have been used for determination of OM in pharmaceutical and biological systems, such as high-performance liquid chromatography (HPLC) coupled with UV detector [2].

We are introducing mercapto-nanoporous fructose modified carbon paste electrode (mercapto np-F-CPE) as a new biosensor for trace determination of omeprazole (OM) in biological samples. The electrochemical response characteristic of the modified-CPE toward OM was investigated by cyclic and differential pulse voltammetry (CV and DPV). The proposed biosensor displayed a good electro oxidation response to the OM, its linear range is 0.25 nM to 25 µM with a detection limit of 0.04 nM under the optimized conditions. The prepared modified electrode shows several advantages such as high sensitivity, long-time stability, wide linear range, ease of preparation and regeneration of the electrode surface by simple polishing and excellent reproducibility.

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Methanol Fuel cell Performance by Use of Pt-Ru Anod catalyst on Carbon Nanotube support

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In this study, more than 100 articles related to anode catalysts for the direct methanol fuel cell (DMFC) are reviewed, mainly focusing on the three most active areas:

1. progress in preparation methods of Pt–Ru catalysts with respect to activity improvement and utilization optimization,
2. preparation of novel carbon materials as catalyst supports to create highly dispersed and stably supported catalysts,
3. exploration of new catalysts with low noble metal content and non-noble metal elements through fast activity methods such as combinatorial methods. Suggested research and development (R&D) directions for new DMFC (anode catalysis) are also discussed.

In this study, we investigate the influence of Pt:Ru atomic ratio on the electrochemical activity of PtRu/carbon nanotube (CNT) catalysts in the electrooxidation of methanol. Bimetallic PtRu alloy nanoparticles were embedded onto the CNTs by chemical impregnation, followed by the refluxing of ethylene glycol. Four types of catalysts, namely $Pt_{100}Ru_0$, $Pt_{75}Ru_{25}$, $Pt_{50}Ru_{50}$, and $Pt_{25}Ru_{75}$, were synthesized for the investigation of the compositional effect. The crystalline size of PtRu nanocatalysts generally decreased with the Ru atomic ratio, i.e., from 4.34 to 2.77 nm. The measurement of electrooxidation of methanol was carried out in 1 M H_2SO_4 electrolyte containing 0.5 M CH_3OH with PtRu/CNT, catalysts using cyclic voltammetry (CV) and AC electrochemical impedance spectroscopy (EIS). CV analysis revealed that the $Pt_{50}Ru_{50}$/CNT electrode had the highest electrochemical activity, owing to its lower onset potential and higher ratio of the forward to reverse anodic peak current. EIS combined with equivalent circuit reflected that after cycling, $Pt_{50}Ru_{50}$/CNT electrode has not only a much lower charge-transfer resistance, but also higher capacitance than $Pt_{100}Ru_0$/CNT. This enhancement of electrochemical activity can be ascribed to the presence of Pt–Ru atomic pair sites in the bimetallic alloys, which play an important role in regenerating the inactive $Pt–CO_{ads}$ sites, according to the bifunctional theory.

Keywords: PtRu nanoparticles, Carbon nanotubes, Methanol oxidation, Fuel cells, Carbon support

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[1] N. Kakati, J. Maiti, S. H. Jee, S. H. Lee and Y. S. Yoon, *Hydrothermal synthesis of PtRu on CNT/SnO_2 composite as anode catalyst for methanol oxidation fuel cell*. J. Alloys Compd. 509 (2011), 5617.

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Methanol Fuel cell Performance by Use of Pt-Ru Anod catalyst on Carbon Nanotube support

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In this study, more than 100 articles related to anode catalysts for the direct methanol fuel cell (DMFC) are reviewed, mainly focusing on the three most active areas:

1. progress in preparation methods of Pt–Ru catalysts with respect to activity improvement and utilization optimization,
2. preparation of novel carbon materials as catalyst supports to create highly dispersed and stably supported catalysts,
3. exploration of new catalysts with low noble metal content and non-noble metal elements through fast activity methods such as combinatorial methods. Suggested research and development (R&D) directions for new DMFC (anode catalysis) are also discussed.

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Microwave assisted functionalization of CNT: optimization and infrared spectrometric investigation

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One of the effective carbon nano tubes (CNTs) functionalization methods during recent years has been microwave (MW) irradiation under oxidative condition [1]. This method is preferred compared to other current methods due to high output, low concentration of reagents and trustable control of temperature and pressure [2]. On the other hand, it is a simple, fast, environmentally friendly, and green approach. Usually, the use of MW facilitates and accelerates reactions, often improving relative yields. In case of MW assisted functionalization of CNTs, irradiation is desired to reduce the reaction time and give rise to products with higher degrees of functionalization than those obtained by the conventional thermal methods. MW irradiation was used as the energy source for nitric acid assisted functionalization of CNT. It is obvious that effective parameters in functionalizing process i.e. the concentration of acidic solution, temperature and the time of process not only make a direct effect but also influence the output of the process due to mutual interactions. Simultaneous analysis based on designed monitoring of the influencing factors is a desirable strategr to optimize a process. The functionalization process was optimized using response surface method by (RSM) determination of reciprocal effect between process variables (acid concentration, reaction time, temperature and MW power) and considering quantitative measure of functional groups,created in the structure of CNT. A quadratic model was selected to create a relationship between response and process variables. The form of response function depended on the relationship between independent and response variables multi-term high grade equation. According to the experimental results and analysis of response surface, the optimized condition was obtained as C_{acid}: 14 N, temperature: 100 °C, reaction time: 25 min and MW power: 800 w.

Quantitative amount of functionalization was set as the output of procces using the Bohem titration method. Considering the overall situation of titration analysis, its difficulties and disadvantages, infrared (IR) spectroscopy was utilized to determine the quantitative amount of functionalized CNT is MW treated samples. IR spectra data were modeled by a PLS to predict the amount of reaction yield quantitatively. This could be an approach for saving more power and chemicals during such as a process, avoiding the useless experimental activity.

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Mixed micellar liquid chromatography as a toxicity screening method of psychotropic drugs

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Abstract

The knowledge of drug toxicity is necessary for risk assessment and ranking of drugs according to their hazard potential [1]. Better prediction of safety risk of drug candidates remains a major challenge in order to prevent late stage attrition. In this study, the capability of biopartitioning micellar chromatography (BMC) using the pure polyoxyethylene (23) lauryl ether (Brij35) solution and the mixed micellar system of Brij35/sodium dodecyl sulfate ($Brij35/SDS_{85:15}$) as a green mobile phase respectively, has been studied to predict the acute toxicity (lethal dose, LD_{50}) of psychotropic drugs. The relationships between the BMC retention data of 13 basic psychotropic drugs and their LD_{50} parameter were studied in two different mobile phases and the predictive ability of models was evaluated. A better statistically model was obtained using $Brij35/SDS_{85:15}$ retention data (R^2 = 0.936, F=73.715, SE = 277.33, R^2_{CV} = 0.913). The superiority of $BMC_{Brij35/SDS}$ is due to the fact that the mixed micellar mobile phase can simulate the resting membrane potential and the conformation of the long hydrophilic polyoxyethylene chains remains unchanged [2].The application of the developed model to a prediction set demonstrated that the model was also reliable with good predictive accuracy. The external and internal validation results showed that the predicted values were in good agreement with the experimental value.

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MnO_2 nanorodes coated on multi-walled carbon nanotubes ($NMnO_2$/MWCNTs) for oxidative removal of amaranth from food samples

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Industrial activity is responsible for generating a large volume of hazardous effluents [1]. Colour is one of themost important hazards in industrial effluents, which needs to be treated [2]. This study presents a simple route for the fabrication of manganese dioxide nanorodes coated multi-walled carbon nanotubes nanocomposites. The composite was characterized by transmission electron microscopy, scanning electron microscopy Fourier-transform infrared spectroscopy, X-ray diffraction and thermal gravimetric analysis. $NMnO_2$/MWCNTs composite is more effective adsorbent for oxidative removal of amaranth dye compared with other materials. Amaranth adsorption by $NMnO_2$/ MWCNTs composite is strongly pH dependent and best results was obtained in acidic media. The Langmuir isotherm model is consistent with the experimental data at different temperatures. The maximum adsorption capacity was determined to be $q_{max} = 500$ mg g^{-1}. Comparison of the COD value of the initial dye solution with solution after oxidative removal indicates the COD value is substantially reduced. It is observed that the COD value shows a significant decrease from 580 to 2.8 mg/L indicating good potential of employing technique. A contact time of different initial concentrations (50, 80, 100, 120 mg L^{-1}) was about 30 min to attain adsorption equilibrium. The kinetic adsorption of different initial concentrations can be described by the pseudo-second-order rate equation. The overall rate process was apparently influenced by external mass transfer and intraparticle diffusion. Moreover, the thermodynamic parameters indicated that the adsorption process was spontaneous and endothermic, and that the adsorption mechanism included both the physical and the chemical adsorption mechanisms. After adsorption, $NMnO_2$/MWCNTs can be conveniently and quickly separated from the media by centrifuge, and adsorption capacity can remain up to 98% after five times of usage. Thus, $NMnO_2$/ MWCNTs are good candidate for efficient removal from wastewater and for the deep-purification of food aqueous solutions.

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Model-based analysis of coupled equilibrium-kinetic processes: calculation of thermodynamic and kinetic parameters from the kinetic data

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Model-based least squares fitting analysis or hard modeling is a specific part of chemometrics, which is based on mathematical relationships for describing the measurements [1]. Furthermore, model-based analysis of coupled equilibrium-kinetic processes is the computation of the concentrations of all chemical species as a function of the progress of an intertwined kinetic-equilibrium chemical process. This means equilibrium processes have been directly incorporated into the rate laws [2]. In this study, we demonstrate several models, that they can be applied to numerous chemical mechanisms. There are several different types of intertwined kinetic-equilibrium chemical processes that can be modeled; common examples, modeled in this work, include charge transfer complex formation reactions, pH_dependent degradation of chemical compounds, kinetic of complexation reaction at variable pH and Tautomerization kinetics in micellar solutions. Actually, we have restricted ourselves to chemical reactions where both equilibrium and kinetic procedures are linked with each other. The goal is to demonstrate how the concentrations of all reacting species can be computed as a function of time for any reaction mechanism using the kinetic data. It is relatively straightforward to incorporate an appropriate algorithm into a general non-linear least-squares routine for the investigated data. The calculations are based on the known initial concentrations of the components as well as all estimated rate and equilibrium constants. After the data fitting process, the optimal parameters together with an estimate of their standard deviations have been obtained. Such models allow the fitting of the rate as well as the equilibrium constants. Model-based analysis together with the possibility of calculating and incorporating the equilibrium and kinetic parameters into the fitting algorithm has allowed the complete analysis of complex reaction mechanisms. This is the first comprehensive study of all of these linked reactions, their kinetic constants, and thus their equilibrium constants. This study opens up a promising new avenue for obtaining equilibrium and kinetic constants, simultaneously, using the same kinetic data.

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Modeling of response surface and kinetics of a cationic dye adsorption on magnetic multi-wall carbon nanotube nanocomposite

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Response surface modeling is an empirical statistical technique that uses quantitative data obtained from appropriately designed experiments to determine optimum operating conditions [1,2]. In this study, response surface method was employed to optimize the removal of Methyl Violet (MV) from aqueous solutions using magnetic multi-wall carbon nanotube nanocomposite as adsorbent. The nanocomposite was synthesized by mixing commercial multi-wall carbon nanotube with a solution containing ferric and ferrous chlorides in highly basic media at 80^{o}C [3]. Properties of the magnetic nanocomposite were characterized by scanning electron microscopy, Fourier transform infrared spectrometry and X-ray diffraction pattern. The experiments were carried out based on a central composite design with four input variables including adsorbent dose (0.4-1.2 g/L), contact time (10-42 min), pH (3–9) and ionic strength (0.02-0.1). Dye concentration of 10 mg/L was taken as a fixed input parameter. Regression analysis showed good fit of the experimental data to a second-order polynomial model with coefficient of determination value (R^2) of 0.972 and Fisher ratio of 28.7. Adequacy of the model was verified by analysis of variance, lack of fit test and residual analysis, and the model was used for prediction of optimum condition of dye removal. Good agreement between the predicted and experimental values of dye adsorption confirmed high efficiency of response surface method in modeling MV removal from aqueous solutions using magnetic nanocomposite. Kinetic study showed that the pseudo-second-order rate equation was able to provide realistic description of adsorption kinetics of MV on magnetic multi-wall carbon nanotube nanocomposite.

Keywords: Adsorption; Dye removal; Multi-wall carbon nanotube; Magnetic nanocomposite; Response surface method; Kinetic study

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Modeling of stratum corneum partition coefficient of some chemicals from their molecular structural descriptors

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The partition coefficients of 67 hydrophilic solutes to the stratum corneum (Ksc/w) were modeled and predicted from their theoretical molecular descriptors.These molecular descriptors encodes electronic and topological aspects of interested molecule which important ones were selected by using stepwise multiple linear regression. To develop quantitative structure-activity relationship(QSAR) models, the methods of multiple linear regression (MLR), and artificial neural network(ANN) were used. The standard errors in the prediction of logKsc/w for training, and test sets, are;0.281 and 0.228 for MLR model and are; 0.151 and 0.196 for ANN model, respectively. The leave-many-out cross validation test was used to further investigate the prediction power and robustness of models, which lead to the statistics of Q^2=0.9903 and SPRESS=0.0601 for ANN model, and Q^2=0.6935 and SPRESS=0.0919 for MLR model. Moreover the chance correlation among data matrix was studied by y-scrambling test and the applicability domains of developed models were investigated .The results obtained indicated the applicability of QSAR methodology in prediction of partition coefficient of solutes to stratum corneum.

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Modified Au nanoparticles-imprinted sol-gel, multiwall carbon nanotubes pencil graphite electrode used as a sensor for ranitidine determination

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A new, simple, and disposable molecularly imprinted electrochemical sensor for the determination of ranitidine was developed on pencil graphite electrode (PGE) via cyclic voltammetry (CV). The PGEs were coated with MWCNTs containing the carboxylic functional group (f-MWCNTs), imprinted with sol-gel and Au nanoparticle (AuNPs) layers (AuNP/MIP-sol-gel/f-MWCNT/PGE), respectively, to enhance the electrode's electrical transmission and sensitivity. The thin film of molecularly imprinted sol-gel polymers with specific binding sites for ranitidine was cast on modified PGE by electrochemical deposition [1, 2]. The AuNP/MIP-sol-gel/f-MWCNT/PGE thus developed was characterized by electrochemical impedance spectroscopy (EIS) and CV. The interaction between the imprinted sensor and the target molecule was also observed on the electrode by measuring the current response of 5.0 mM $K_3[Fe(CN)_6]$ solution as an electrochemical probe[3]. The pick currents of ranitidine increased linearly with concentration in the ranges of 0.05 to 2.0 µM, with a detection limit of (S/N=3) 0.02 µM. Finally, the modified electrode was successfully employed to determine ranitidine in human urine samples.

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Modified carbon ceramic electrode by La_2CuO_4 nanocrystals for simultaneous determination of dopamine, uric acid and xanthine

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Perovskite materials exhibit many interesting and intriguing properties from both the theoretical and the application point of view. Colossal magnetoresistance, ferroelectricity, superconductivity, charge ordering, spin dependent transport, high thermo power and the interplay of structural, magnetic and transport properties are commonly observed features in this family. These compounds are used as sensors and catalyst electrodes[1] Dopamine (DA) is one of the excitatory neurotransmitters that play an important role in several physiological events such as the mammalian central nervous system [2] Uric acid(UA) is a product of the metabolic breakdown of purine nucleotides. High blood concentrations of uric acid can lead to gout. Xanthine (XA) (3,7-dihydro-purine-2,6-dione), is a purine base found in most human body tissues and fluids and in other organisms. A number of stimulants are derived from xanthine, including caffeine and the bromine [3].

In this study, a voltammetric sensor for simultaneous determination of DA, UA and XA by carbon ceramic electrode modified by La_2CuO_4 is reported. The electrocatalytic behavior of the La_2CuO_4 modified CCE were studied in pH 1.0 choloracetate acid buffer solutions by cyclic voltametry (CV) in the presence of DA, UA and XA. Due to the excellent electrocatalytic activity the simultaneous determination of EP, UA and XA with three well-defined peaks was achieved at the La_2CuO_4 modified CCE. The catalytic peak current obtained, was linearly dependent on the DA, UA and XA concentrations in the range of 16.0 – 712, 8-356 and 4-224µM, respectively. The detection limits for DA, UA and XA were 2.7, 1.0 and 0.6 µM. The proposed sensor was successfully examined for real sample analysis urine sample and revealed stable and reliable recovery data.

Keywords: La_2CuO_4 nanocrystals, Carbon ceramic electrode, Dopamine, Uric acid, Xanthine

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Modified magnetite nanoparticles for selective trace determination of diphenylamine

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Diphenylamine (DPA) is one of the most used pesticides worldwide. It is used as a pre- or postharvest scald inhibitor for some fruits include apples and pears. Its anti-scald activity is the result of its antioxidant properties, which protect the fruit skin from the oxidation products of alpha-farnesene during storage. Therefore, residues of DPA are often found in agricultural crops. However, the presence of pesticide residues in foods can be considered as a hazard to human health [1]. Therefore, qualitative and quantitative determination of DPA in materials is of biological and environmental importance.

In this work, a new preconcentration method has been developed for trace determination of diphenylamine. A N-Lauryl sarcosine sodium salt modified magnetite nanoparticles adsorbent was prepared in-situ as an extractant. Diphenylamine was coupled with 3-methyl-2-benzothiazolinone hydrazone in the presence of Fe(III) as an oxidant. The produced blue derivative of diphenylamine then was extracted by the magnetite based adsorbent, then was eluted by 0.6 mL of ethanol and determined by spectrophotometry. The condition of reaction, extraction and elution were optimized, and the important analytical characteristics were investigated. Beer's law was obeyed in the ranges of 0.2-150.0 ng mL^{-1} of diphenylamine. The relative standard deviation and recovery as percent for 5.0 ng mL^{-1} diphenylamine were 3.3 and 98.3, respectively. The limit of detection was 0.02 ng mL^{-1}. The selectivity of the method was evaluated and the method was successfully applied to the determination of diphenylamine in various environmental samples.

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Molecular docking studies on the binding properties of TR-14 peptide to angiotensin converting enzyme

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Abstract

There are many causes of hypertension; angiotensin converting enzyme (ACE) is well-identified for its important physiological roles in the regulation of blood pressure. ACE converts angiotensin-I, an inactivedecapeptide, to angiotensin-II, a potent vasoconstrictoroctapeptide [1].Nowadays, peptides have been found to possess many physiological functions such as antioxidant, antimicrobial, antithrombotic and antihypertensiveactivities [2]. The objective of the present study was to *insilico* analysis of the ACE-inhibitory activity of TR-14 peptide obtained from ostrich serum hydrolysate. The interaction between TR-14 peptide and N- domain (4APJ)as well as C- domain (3L3N) of ACE was stimulated using MVD software.The chemical structure of TR-14 was constructed by MOE software. Furthermore, Energy minimization of peptide was performed by GizMOE minimizer method.ACE domainswere tested by means of different softwaressuch as PROCHECK and ERRAT [3].Following simulation process, the best configuration of TR-14 binding into N and C domains of ACE was selected and the results (**table 1**) used for further analysis. Our results demonstrated that the inhibiting constant of TR-14 - N-domain complexwas very smaller than that of the TR-14 - C-domain complex.Therefore, TR-14 peptide has more affinity binding to C-domain with Moldockscore of -104.483. In conclusion, the results of docking simulation can help us in the interpretation of*in vitro* results.

Keywords: Angiotensin converting enzyme, TR-14 peptide, Molecular docking, Molegro Virtual Docker

Energy overview	MDS.N Domain	MDS.C Domain
Total Energy	-33.869	-104.483
Protein - Ligand interactions	-114.123	-184.736
Hydrogen bonds	-6.788	-12.825
LE1	-0.284611	-1.4391
LE3	-0.0562346	0.606804

Table 1.The molecular docking energy overview of TR-14 with N and C domain of ACE

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Molecular self-assembled monolayers of new 1,3-dithiolan derivative on nanoparticle of gold for determination of isoprenaline

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Self-assembled monolayers (SAMs) have recently become very important due to their potential applications to biosensors, nanotechnology and biomolecular electronics. Using SAMs to functionalize noble metal surfaces provides a simple route to functionalize electrode surfaces by organic molecules containing free anchor groups such as thiols, disulfides, amines, acids or silanes for improved stability of biomolecules. Thiols on gold make the strongest bond and are known to be very stable [1]. In this paper, the use of a gold electrode modified with gold nanoparticls and 1,3-dithiolan derivative selfassembled monolayer, for the sensitive voltammetric determination of isoprenaline was described. A pair of well-defined quasi reversible redox peaks of 1,3-dithiolan derivative was obtained at the modified electrode by direct electron transfer between the 1,3-dithiolan derivative and the gold electrode. The apparent charge transfer rate constant, k_s, and transfer coefficient, α, were calculated. In the second part of the work, the mediated oxidation of isoprenaline at the modified electrode was described. The oxidation peak potentials of isoprenaline at modified electrode in cyclic voltammogram was occurred around 180 mV (at pH 7.0) while this peak potential at unmodified electrode was appeared around 400 mV at the same scan rate. The values of electron transfer coefficients (α), catalytic rate constant (k), diffusion coefficient (D) and detection limit were calculated for isoprenaline, using electrochemical approaches [2]. Finally, the modified electrode was successfully applied for isoprenaline measurements in the pharmaceutical preparations.

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Molecularly imprinted-solid phase extraction combined with dispersive liquid-liquid microextraction for selective extraction and preconcentration of triazine pesticides from aqueous samples

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In this study, a developed technique was reported for extraction and preconcentration of triazines from ground water using molecularly imprinted-solid phase extraction (MISPE) [1] along with dispersive liquid-liquid microextraction (DLLME) [2]. Molecularly imprinted microspheres used as sorbent in SPE procedure were synthesized using precipitation polymerization with atrazine as the template. Aqueous solution of the target analytes was passed through MAMP-MIP cartridge and the adsorbed analytes were then eluted with methanol. The collected eluate was mixed with carbon tetrachloride which served as the extraction solvent. The mixture was immediately injected into de-ionized water. After centrifugation, 1 µL of the settled organic phase was injected into gas chromatography-flame ionization detection (GC-FID). Various experimental parameters that affect the performance of both of the steps (MISPE and DLLME) were thoroughly investigated. The extraction of atrazine, simazine, cyanazine, ametryn, prometryn and terbutryn using the proposed extraction method and followed by GC analysis, resulted in detection limits of 1, 15, 21, 28, 32 and 42 ng mL^{-1}, respectively. The relative standard deviation (RSD%) obtained for six repeated experiments (75 ng mL^{-1} of atrazine) was below 5.5 %. The relative recoveries obtained for the analytes in water samples, spiked with different levels of each analyte, were within the range of 80-92%. Finally, the proposed method was successfully applied to the analysis of triazine pesticides in different aqueous samples.

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Monitoring of five sulfonamide antibacterial residues in animal origin foods by three-phase hollow fiber liquid phase microextraction combined with HPLC-DAD

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The antibiotic sulfonamides (SAs) are commonly used as antibacterial drugs in both human and veterinary medicine to fight infectious diseases, and in animal feed to promote livestock and birds growth. The continual overuse of sulfonamides, much attention has been paid to their potential carcinogenesis and the worry of the development of antibacterial resistance. To ensure animal origin food safety for the consumers, the European Commission (EC), America and other countries, for example China, have adopted a maximum acceptable limit of residual sulfonamide in foods of animal origin, for example meat, milk and eggs. For example, the maximum residue limit (MRL) in the European Union countries and the United States for SAs in animal muscle tissue is 100 ng g^{-1}, while in Japan it is 20 ng g^{-1} [1]. Therefore, the development an analytical methodology for the separation and determination of these drugs becomes essential.

In the present research, hollow fiber based liquid phase microextraction (HF-LPME) prior to HPLC-DAD was developed for simultaneous determination of five sulfonamide antibacterial residues, which are commonly used as veterinary medicines. On the basis of this method, no toxic solvents were used, and simple, rapid, and sensitive analysis was accomplished in the meanwhile. The extraction involved filling an 7.6 cm length of hollow fiber with 25 μL of acceptor solvent (1 mol L^{-1} NaOH solution) using a microsyringe, followed by impregnation of the pores in the fiber wall with 5% aliquat-336 in *n*-octanol. The fiber was then immersed in 18 mL of aqueous sample solution. Following this microextraction, 20 μL of acceptor phase was injected into HPLC-DAD. Separation was performed on C18 (250 mm × 4.6 mm, 5 μm) using gradient elution by mobile phase containing methanol and phosphate buffer pH 4.5. In order to obtain high extraction efficiency, the parameters affecting HF-LPME such as pH of donor and acceptor phase, percentage of aliquat-336 in membrane solvent, HF length, extraction time, salt addition and stirring rate are optimized using the some advanced experimental design methods such as simplex, central composite design and pattern search. Finally, the applicability of the proposed method was successfully confirmed by extraction and determination of drugs in water and various animal food samples. Comparing to the traditional methods, the proposed method exhibits high sensitivity and high preconcentration factors as well as good precision.

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Monitoring the zwitterionic intermediate of reaction between triphenylphosphine and dimethyl acetylendicarboxylate by applying a stopped-flow technique

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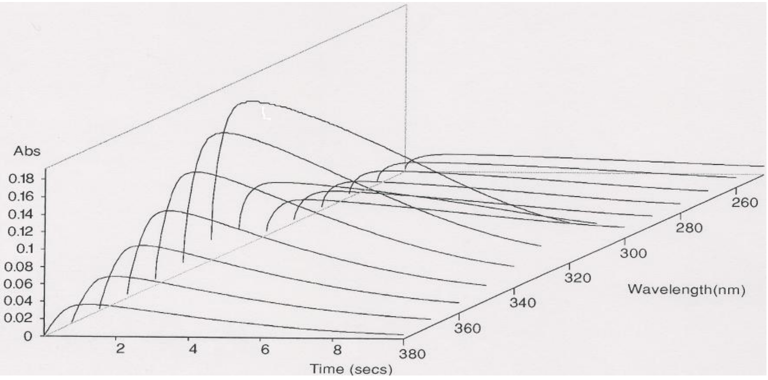
Evidence of formation and decay, and effect of the zwitterionic intermediate on the reaction mechanism of the reaction between triphenylphosphine and dimethyl acetylendicarboxylate has been investigated in aqueous-organic solvents by applying a stopped-flow technique [1, 2]. Spectral analyses on the two-component reaction was confirmed the existence of the zwitterionic intermediate. Step **1** of the reaction involving the formation and decay of the zwitterionic intermediate (ZI) was recognized as a fast step. Also, Pro-K analyzer was confirmed the existence of 3 species assigned as TPP, DMAD and ZI and an equilibrium reaction model (A+B<>C) for the step **1** of the two-component reaction. Experimental kinetic data (k_1, k_{-1}) along with activation parameters ($\Delta G^{\neq}$, $\Delta S^{\neq}$ and $\Delta H^{\neq}$) of the reaction were recognized [3].



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mPEG Coated Magnetite Nanoparticles as an Adsorbent for Removal of Diazinon from Aqueous Solution.

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The widespread occurrence of pesticides in the environment water such as surface water, groundwater and drinking water is a recognized problem owing to their widely applied in agriculture and hygiene throughout the world. Organophosphorus pesticides (OPPs) are one group of the most frequently encountered pesticides [1]. High diazinon, an organophosphorus pesticides, residues have been found in urban waterways and effluents from sewage treatment plants [2]. Therefore the removal of diazinon seems necessary.

The object of this study was to evaluate the efficiency of diazinon removal with application of mPEG coated magnetite nanoparticles (Fe_3O_4@SiO_2-mPEG) as adsorbent. Adsorption has been found to be superior to other techniques for removal of colors, odor, oils, and organic pollutants from process or waste effluents treatment in terms of initial cost, simplicity of design and ease of operation [3].

Magnetic nanoparticles (MNPs) were prepared via co-precipitation process, and then were coated by methoxy polyethylene glycol (mPEG). Preparation of magnetite nanoparticles with average particle size of 27 nm was proved by SEM and bonded mPEG on the surface of MNP was confirmed by FTIR spectroscopy.

Adsorption of diazinon on Fe_3O_4@SiO_2-mPEG nanoparticles was investigated by UV spectrophotometry at 236 nm, through batch experiments. Tests were done to evaluate the effects of adsorbent dosage, solution pH, contact time, initial concentration of diazinon; solution temperature and water impurities on adsorption of diazinon onto MNPs. Results showed that the maximum removal efficiency was 80%. Finally evaluations were also made for kinetic, isotherm, and thermodynamic of diazinon adsorption on modified MNPs. The experimental adsorption data had the best fitness with the pseudo-second-order model (R^2>0.99) and equilibrium adsorption data could be better fitted with the Freundlich isotherm (R^2>0.99). Furthermore thermodynamic analysis suggests the van der Waals interactions, as the main mechanism of adsorption.

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Multi Walled Carbon Nanotube-Modified Screen Printed Electrode for Anodic Stripping Voltammetric Detection of Uranyle

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The use of chemically modified electrodes (CMEs) in anodic stripping voltammetry is an expanding area of investigation. To date, some kinds of CMEs have been reported for the determination of Uranyle [1-3].
This paper reports the interest of the novel multi walled carbon nanotube screen printed electrodes (MWCNT-SPE) for sub-nanomolar analysis of uranium in water samples. Electrodes were easily prepared via casting of the MWCNT-nafion ink on the screen-printed electrodes. The stability of the grafted layer has been clearly demonstrated. Uranium detection was then achieved by immersing the casted In a pH 4.4 NaOAc-HOAc buffer containing 0.02 $\mathrm{molL^{-1}}$ $\mathrm{Mg(NO_3)_2}$, $\mathrm{UO_2^{+2}}$ first was adsorbed onto the surface of a MWCNT-SPE and then reduced at -0.40 V vs. Ag. During the positive potential sweep, at the reduced uranium was oxidized, and a well-defined stripping peak was appeared at +0.20 V vs. Ag. Low concentrations of $\mathrm{Mg^{+2}}$ significantly enhance the stripping peak currents since it induces $\mathrm{UO_2^{+2}}$ to adsorb at the electrode surface. Deposition time and potential were investigated so as to find the best compromise between analysis time, repeatability and reproducibility. Limit of detection and quantitation reached 7×10^{-10} and 2×10^{-9} mol $\mathrm{L^{-1}}$, respectively. Moreover, interference study was conducted with Zn(II), Cd(II), Pb(II) and Cu(II); no major interference was established. MWCNT-SPEs were finally applied for uranium determination in estuarine water demonstrating the convenience of these electrodes for environmental analysis.

Keyword: Screen Printed Electrode; MWCNT, Impedimetric; Uranyle.

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Multivariate adaptive regression splines (MARS) for QSAR study on 5-(2-methylbenzimidazol-1-yl)-N-alkylthiophene-2-carboxamide derivatives as *P. falciparum* dihydroorotate dehydrogenase (*Pf*DHODH) inhibitors

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The search for new antimalarial agents is necessary as current drugs in the market have become vulnerable due to the emergence of resistant strains of *Plasmodium falciparum* (*Pf*). The enzyme dihydroorotate dehydrogenase (*Pf*DHODH) is a validated target for development of antimalarial agents. *Pf*DHODH is a crucial enzyme in the de novo pyrimidine biosynthesis pathway and is essential for the growth of the parasite [1,2].

MARS is a local modeling technique, dividing the data space in several possible overlapping region and fitting truncated spline functions in each region. A MARS analysis generally consists of three steps. The first step consists of a forward stepwise procedure which selects the best spline functions in order to improve the model and the second step in the MARS methodology consists of a pruning step. A backward elimination procedure is applied in which the basic functions with the lowest contribution to the model are excluded. Eventually, the selection of the optimal model is performed in the third step [3].

In this study the inhibitory activity of 5-(2-methylbenzimidazol-1-yl)-N-alkylthiophene-2-carboxamide derivatives (35 compounds) were investigated. After structural optimization, different descriptors (1497 descriptors) were calculated using Dragon software and the best of them (5 descriptors) selected using stepwise method and then MARS was applied for modeling.

Determination coefficient (R^2) and root mean square error (RMSE) for MARS model were obtained as 0.9514 and 0.2401, respectively.

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Multivariate curve resolution with non-linear fitting of porphyrin binding to human telomeric DNA

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G-rich sequences in which could generate G-quadruplex are located in telomeres as a repeated unit and elsewhere in the genome. It has been believed that G-quadruplex regions may be directly involved in the gene regulation at the level of transcription. Furthermore, promoter regions are significantly enriched in quadruplex motifs. Additionally promoter quadruplex regions are associated with nuclease hypersensitive sites. Investigation of drugs and nuclease hypersensitive sites interaction is a way to artificially regulate gene expression, thereby generating medicinal benefits such as anticancer activity.

In this research, equilibrium study of oligonucleotide sequence AGGGTTAGGGTTAGGGTT AGGG (H22) and porphyrin-based ligand were investigated using molecular absorption spectroscopy. We are aimed at characterizing the nature of interaction between H22 sequence and porphyrin-based ligand in the presence of different ionic strength of sodium and potassium ions. Two different porphyrin-based ligand including ([Cu(2,3-tmtppa)]4+) and ([Cu(3,4-tmtppa)]4+) were considered in this study.

For each ligand at specific ionic strength spectra recorded in an inverse titration during each step and were arranged in a data matrix D. The dimensions of this matrix were n rows × m columns, where n represented spectra recorded in each step of titration and m represented the number of wavelengths measured. In order to full resolution of data matrix for each experiment, hard modeling strategy was applied. Hard modeling involves the fitting of a defined mathematical relationship to measured data. Unambiguous recovery of profiles is distinguishable advantages of hard modeling compared with soft methods. Using non-linear least-squares fitting, equilibrium constants, concentration and spectral profiles were obtained. Close inspection of results revealed that in the presence of potassium ions, a two-step complexation should considered ($M + L \leftrightarrows ML$ & $ML + L \leftrightarrows ML_2$) while for the medium containing sodium ions a single step complexation is sufficient ($M + 2L \leftrightarrows ML_2$). By considering the fact G-quadrplex is more stable in the presence of potassium ions our results are satisfactory.

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Multivariate kinetics study of electron donor–acceptor complexes using hard-modelling approach and rank annihilation factor analysis

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Abstract

Kinetics of electron donor–acceptor (EDA) complex formation between procaine (an antiarrhythmic drug) with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) and iodine has been investigated spectrophotometrically in some organic solvents by using hard-modelling approach and rank annihilation factor analysis (RAFA). Rank annihilation factor analysis is an efficient chemometric technique based on rank analysis for two-way spectral data matrices. The principle of this method is that the rank of two-way biliner matrix of pure compound is one [1]. For two-way kinetic spectral data the contribution of this pure matrix can be removed when the rate constant of the process act as an optimizing object and simply combined with the pure spectrum of reactant. This results in decreasing the rank of original data matrix by one [2]. In hard-modelling approach, multivariate kinetic absorption data monitored spectrophotometrically has been fitted to proper kinetic model that correctly describes the chemical process. So, the nonlinear parameters (rate constants) as well as the linear parameters (spectral profiles) were obtained [3]. Small values of the residual standard deviation (RSD) and sum of squares of the residuals (ssq) obtained showed the proper selection of the models. The results of the hard-modelling approach and RAFA have been compared. Variations in the kinetic constants were in the kinetics of the interaction of procaine with the acceptors is dependent on the solvent polarities.

Keywords: Electron donor–acceptor complex; chemometric technique; RAFA; Rate constant determination; Two-way kinetic-spectral data; Hard-modelling;

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Multivariate wavelet de-noising: Useful approach to improve the classification capabilities in NMR spectral data

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In vivo NMR signals contain noise besides the signals of interest. Most of this noise originates from the receiver circuitry [1]. However, some of the noise is also a consequence of subtle instabilities during long measurements. The Fourier transform has the disadvantage that it convolutes noise with the spectrum [2]. The work describes the application of the multivariate wavelet de-noising approach for better statistical diagnosis of patterns via evaluation of NMR spectral data, focusing on the output of unsupervised classification techniques. The methods are illustrated by an application in the different stages of biological area of gliomas brain tumour nuclear magnetic resonance (NMR) data acquired from a set of 50 human brain tumour samples. In this regard, untreated spectra of understudy cases were processed with and without wavelet de-noising, and then the spectra were evaluated with principal component analysis (PCA) and Silhouette methods to realize the probable effect of pre-processing. Silhouette refers to a method of interpretation and validation of clusters of data. The technique provides a succinct graphical representation of how well each object lies within its cluster. The multivariate wavelet de-noising problem deals with models of the form $X(t) = F(t) + e(t)$, where the observation X is p-dimensional, F is the deterministic signal to be recovered and e is a spatially correlated noise signal. This kind of model is well suited for situations in which such additive, spatially correlated noise is realistic. Also mathematical results such as average of distances and sum of the total distances have been evaluated. The results show, application of wavelet de-noising to the data can improve the outputs of classifier. Sum of distances and mean of distances for preferred method were 3.48 and 0.54 respectively which were reasonable in comparison with 6.53 and 0.4 for untreated spectra. The results of PCA showed that the captured variances for first 4 PCs of data set after treatment are more than 98% which could give good classification power to the data.

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Multiwalled carbon nanotubes/polypyrrole/SiO_2 composite prepared by electrodeposition technique as a new solid phase microextraction sorbent for preconcentrationon of BTEX

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Abstract

BTEX is the collective name of benzene, toluene, ethylbenzene, and the xylene isomers (p, m, and o-xylenes), all being harmful volatile organic compounds (VOCs). Human exposure to these compounds can have serious damaging effects on the kidneys, heart, lungs, and the nervous system [1]. BTEX are emitted to the environment from an extensive variety of sources including combustion products of wood and fuels, industrial paints, adhesives, degreasing agents and aerosols [2]. Exposure to BTEX can occur by ingestion, inhalation or absorption through the skin. Maximum contamination levels (MCLs) according to the U.S. Environment Protection Agency (EPA) for benzene, toluene, ethylbenzene, and xylenes are 0.005, 1, 0.7, and 10 mg L^{-1}, respectively, and concentrations above MCL in air and water seriously affect human health [3]. There is, therefore, a vital need to develop simple and cheap analytical methods with low detection limits for the quantification of BTEX. A novel polypyrrole-carbon nanotubes-Silicon dioxide (PPy-CNT-SiO_2) composite film coated stainless steel wire, was fabricated through electrochemical deposition and used for the extraction of BTEX compounds, followed by gas chromatographic analysis. The new PPy-CNT-SiO_2 fiber coating showed better analytical performance than PPy-CNT fiber. Under the optimized conditions, the detection limits (S/N=3) were in the range of 0.005–0.02 ng mL^{-1} and the limits of quantification (S/N=10) between 0.01–0.06 ng mL^{-1}. The relative standard deviations (RSDs) for one fiber (repeatability) (n=5) were obtained from 3.9 up to 6.4% and between fibers or batch to batch (n=3) (reproducibility) for one fiber in the range of 6.0–8.5%. The proposed method was successfully applied for determination of BTEX compounds in water sample, and the recoveries were from 91–106.7%.

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Murexide-Fe_3O_4 nanoparticles as a novel magnetic sorbent for solid phase extraction of Cd(II) and Pb(II) with the aid of experimental design methodology

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Heavy metal such as lead and cadmium are toxic to living organisms, even in trace levels, which can cause serious disorder. Therefore, determination of these metals in the environment is crucial. Recently, SPE has been adopted as one of the most efficient methods for removal of toxic metal ions from environmental. This method is simple, efficient, selective and cost-effective. In this procedure, full recovery and high enrichment factor is achieved by using proper sorbent [1]. Nanometer-sized materials are one of these sorbents which are widely applied due to their unique chemical, physical and biological properties. Magnetic Fe_3O_4 nanoparticle is a special kind of Nano-sized materials which has magnetic property besides the general features of nanometer-sized materials. However, naked Fe_3O_4 nanoparticles are tended to aggregate, and are not selective and suitable for complex samples.Therefore, the surface of these magnetic nanoparticles has been modified with a specific ligand which makes them to be a selective and appropriate sorbent. This work describes the synthesis and application of murexide-Fe_3O_4 nanoparticles as a magnetic sorbent for preconcentration of trace amounts Pb(II) and Cd(II) ions and its determination by FAAS. A Box–Behnken design was used to find the affecting parameter on the preconcentration procedure through response surface methodology and experimental design. Three variables including extraction time, amount of magnetic sorbent, and pH were selected as affecting factors for uptake step. The optimized values were 3.5 min, 10.5 mg and 6, respectively. Four parameters including type, volume, concentration of eluent, and elution time were studied in elution step. The optimized values were H_2SO_4, 2.6 mL, 1.5 mol L^{-1} and 6 min, respectively. The limit of detection (LOD), the relative standard deviation and the recovery of the method were 2.3 and 0.9 ng mL^{-1}, 4.2% and 5.1%, 98.2% and 97.5, for lead and cadmium respectively. The method was validated using certified reference material, and has been applied for the determination of trace Pb(II) and Cd(II) in different real samples with satisfactory results.

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Nano-composite Based Carbon Paste Electrode for Selective Determination of Fe (III) Ion

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Potentiometric sensors have shown to be very effective tools for analysis of a wide variety of metal ions. Among them, carbon paste electrodes (CPEs) have attracted interest as a result of their improved renewability, stable response, and low ohmic resistance compared to membrane electrodes [1]. Di-butylazodicarboxylate (TBADC) seemed to be a suitable ion carrier for metal ions due to the presence of hard donating O and intermediate donating N atoms and even the existence of electron rich double bound in its structure [2]. Preliminary solution study shows the selectivity of (TBADC) toward Fe(III) ions in comparison of other common cations, including alkali, alkaline earth, transition and heavy metal ions. To have a long-term stable electrode for potentiometric uses, (TBADC) was applied as ionophore in preparation of a nano-composite carbon paste electrode. The carbon paste was made based on a new nano-composite including multi-walled carbon nanotube (MWCNT), nanosilica (NS), graphite powder and paraffin oil as binder. The carbon paste electrode composed of 3% MWCNT, 0.3% NS, 4% TBADC, 30% paraffin oil, and 62.7% graphite powder showed the best response. The proposed sensor exhibits a Nerstian slope of 19.9±0.5 mV decade^{-1} toward Fe^{3+} ions in the range of $1.0\times10^{-9} – 1.0\times10^{-2}$ M and detection limit of 8.0×10^{-10} mol L^{-1}. The response of the sensor was found to be stable in the pH range of 1.6 – 4.3. It displayed a relatively fast response time, in the whole concentration range (~ 5 s), and it can be used for at least 12 weeks. The application of the TBADC-based sensor to analysis of Fe(III) ion in a pharmaceutical formulation, and also to the monitoring of Fe(III) ion in mixtures of two, three and four different ions showed satisfactory results.

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Nano-crystalline clinoptolite modified by bis(2-hydroxybenzaldehyde)-1,2-ethylenediamine; adsorbent for thorium(IV) and lanthanides(III) ions

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Extraction and recovery of lanthanide and actinide metals from nuclear and metal-containing industrial waste streams is important both environmentally and economically [1]. This provokes the separation of these metal ions to be of great interest. As a part of such studies, adsorption techniques are investigated and used [2,3]. Zeolites are crystalline hydrated aluminosilicates. Their structure is built up of tetrahedral SiO_4 and AlO_4 units bridged by oxygen atoms generating secondary building units. The trivalent aluminum in the structure generates a negative charge in the framework that imparts the ion exchange properties to the material. Zeolites are known to function as ion exchangers. There are numerous natural zeolite minerals in Iran. One of them is clinoptolite zeolite found in Semnan province. This type of Zeolite was selected and modified for removal of and separation of La(III), Eu(III), Er(III) and Th(IV) ions from aqueous phase. Adsorption of these ions on the surface of natural zeolite shows the pH dependency. The results show that natural clinoptilolite at pH=4 can remove quantitatively Er(III) and Th(IV) ions, and at pH=5 can remove La(III) and Eu(III) ions from aqueous phase. The selectivity of adsorption varies as Th(IV)>Er(III)>Eu(III)>La(III). The higher selectivity presented towards thorium ions towards lanthanide ions and erbium ions towards europium and lanthanium can be attributed to the high charge to size ratio of these ions. It is noteworthy that with the modification of nano-crystalline zeolite's surface by bis(2-hydroxybenzaldehyde)-1,2-ethylenediamine ligand**,** a shift in the extraction uptake versus pH values of aqueous phases (~0.5 unit) was observed. The other parameters affecting on their adsorption properties such as the initial concentration of studied ions, contact time, mass of sorbent and temperature have been investigated and discussed. The investigation of experimental data with kinetic models has shown a good agreement with second order equation. Adsorption isotherm studies signify that Langmuir isotherm model match to experimental data for all studied ions. The influence of temperature on the adsorption of these ions was studied. The thermodynamic parameters such as free energy (ΔG^o), enthalpy (ΔH^o) and entropy (ΔS^o) changes showed that the adsorption of metal ions was endothermic.

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Nano-crystalline clinoptolite modified by bis(2-hydroxybenzaldehyde)-1,2-ethylenediamine; adsorbent for thorium(IV) and lanthanides(III) ions

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Nanomagnetic solid phase microextraction for determination of lanthanum by spectrophotometry UV-Vis in real sample

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The main restrictions in determination of the inner transition elements in environmental samples come from the high content of the matrix and the extremely low concentration levels of the elements in those samples, which are below the detection limits of the conventional instrumental techniques[1]. Therefore, an efficient separation and preconcentration technique is frequently required for determination of these elements in complex matrices. In this project, a new facile, rapid, inexpensive, and sensitive method based on magnetic solid phase microextraction was developed for determination of lanthanum in environmental water samples. In SPME technique, selection of an appropriate sorbent is an important strategy in the elaboration of analytical procedure, and nanometer sorbents have proved to be especially effective as a result of its high specific surface areas, highly active surface sites and the absence of internal diffusion resistance in the separation process. Magnetic nanoparticles (MNPs), as a new kind of nanometer material, have gained more attention, easy control and fast magnetic separation under an extra magnetic field makes them excellent candidates for many important applications [2]. The magnetic separation technique based on magnetic iron oxide nanoparticles (MNPs) has potential applications in adsorption and purification[3] . Fe_3O_4 nanoparticles(iron oxides magnetic nanoparticles are synthesized by co-precipitation method from aqueous Fe^{3+}/Fe^{2+} salt solution. The effective parameters on extraction efficiency and desorption conditions were investigated and optimized. After microextraction and complex formation with appropriate ligand, was measured at 500 nm by spectrophotometry using a micro cell. Under optimized conditions, detection limit (DL, S/N = 3) and limit of quantification (LOQ, S/N = 10) of the method were 0.001and 0.010 μg L^{-1}, respectively. The calibration curve of lanthanum showed linearity in the range of 0.01–0.10 μg L^{-1}.

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Nanomagnetic solid phase microextraction for determination of lanthanum by spectrophotometry UV-Vis in real sample

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Nanoparticle-functionalized Aptamer-based Biosensor for Electrochemical Detection of Leukemia Cancer Cells

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Abstract:

In this study, a strategy for highly sensitive and specific detection of leukemia cells using silver nanoparticle-functionalized aptamer based biosensor has been investigated. Whole-cell aptamers are oligonucleotide strands that can bind to the specific cells with high affinity and selectivity [1]. They are selected using a cell-based SELEX (systematic evolution of ligands by exponential enrichment) procedure [2,3].

Herein, for the construction of the biosensor, the thiol terminated anti-leukemia aptamer (sgc8c) was self-assembled onto the gold electrode surface as recognition probe. The immobilization of the aptamer was monitored using differential pulse voltammetry (DPV) and electrochemical impedance spectroscopic (EIS) techniques. Subsequently, the aptamer-modified electrode was incubated in various concentrations of leukemia cancer cells (CCRF-CEM) suspended in PBS solution. By attaching the cancer cells onto the immobilized aptamers on the electrode surface, the electron transfer resistance to $[Fe(CN)_6]^{3-/4-}$ on the biosensor surface was increased. Finally, the cells on the surface were treated with the secondary aptamer that was modified with silver nanopaticles (AgNPs). The electrochemical oxidation signal of AgNPs has been followed as an analytical signal for the detection of the cancer cells. The biosensor is capable to detect the leukemia cancer cells as low as 10 cells/mL.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

Nanostructure coating prepared by electrospinning technique for headspace solid-phase microextraction of BTEX from environmental samples

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ABSTRACT

Nanomaterials are nowadays considered as a base for solid-phase micro extraction (SPME) fibers. Electrospinning is a method for preparing nano-dimensional sorbents. Electrospinning involves applying a high electric field between a viscous polymeric solution and a conductive collector. This method can be used for preparation of fiber coating in SPME. On the other hand applying high temperature in SPME for desorbing analyte from fiber causes limitation of using polymeric coating with low melting point. Poly dimethylsiloxane (PDMS) is one of the most popular coating fibers in SPME because of its high thermal stability and enhanced solute diffusion capabilities. PDMS has a very low glass transition temperature and as a result, this material cannot be electrospun under normal conditions [1]. Some investigations have been performed on the electrospinning of block copolymers [2] and graft copolymers of PDMS [3].

In this study sol-gel and electrospinning techniques were applied with each other for preparation of PDMS coated fiber. The scanning electron microscopy (SEM) images of this coating showed a diameter range of 34–61 nm with a porous surface structure.

The applicability of this fiber was assessed for the headspace SPME of benzene, toluene, ethylbenzene and xylenes (BTEXs) from water sample followed by gas chromatography–mass spectrometry (GC–MS). Effects of different parameters such as extraction condition, desorption condition, agitation rate and salt effect were investigated and optimized. Under the optimized conditions, Limit of detections and limit of quantifications of 30 μg L^{-1} and 50μg L^{-1} were respectively obtained. The method showed linearity in the range of 50–5000 μg L^{-1} with correlation coefficient of >0.99. The relative standard deviation was less than 8.47%.

Keywords: Polydimethyl siloxane, Electrospinning, Solid- phase microextraction, BTEXs, gas chromatography-mass spectrometry

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Naphthalene modified with 2-(3, 4, 5-trimethoxybenzylidene) malononitrile as a new sorbent for selective removal of lead and zinc ions from aqueous solutions

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Water pollution by toxic heavy metal ions occur globally. Strict environmental regulations on the discharge of heavy metal ions and rising demand for clean water with extremely low level of heavy metal ions make it greatly important to develop different efficient technologies for heavy metal ions removal. The conventional technologies for the removal of heavy metal ions from aqueous solution include chemical precipitation, ion exchange, reverse osmosis, electrochemical treatment and adsorption [1]. Among the different treatments described above, adsorption technology, on the other hand, is one of the most recommended physicochemical treatment processes that is commonly used and applied for heavy metal ions removal from water samples and aqueous solutions.

This work presented the preparation of a novel adsorbent naphthalene modified with 2-(3,4,5-trimethoxybenzylidene) malononitrile. It was characterized by FT-IR. Batch method was applied for testing of adsorption behavior. Adsorption experiments showed, the new sorbent has high selectivity and good adsorption for lead and zinc from aqueous solutions. Lead ions are retained on the sorbent in the pH>6 and and zinc ions in the pH=8. Equilibration time was 5 min for zinc and 30 min for lead. There was no significant effect in removal of the ions with increasing of the salt. Langmuir and freundlich isotherms models were applied to analyze, the adsorption and experimental data of Pb(II) and Zn(II). The maximum adsorption capacity for Pb(II) and Zn(II) was 34.2 and 38.9 mg g^{-1}, respectively. The thermodynamic studies indicated that the adsorption was spontaneous and exothermic for Pb(II)adsorption and endothermic for zinc**.**

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New Method for the Analysis of Aluminumic Oxygen Cleanser Used in Iron Making

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Oxygen cleansers are materials added to iron slag for reducing FeO and MnO by eliminating the oxygen content. Oxygen comes out as slag after the reaction. Also oxygen cleansers may improve the fluidity of melt. Brikets contain metallic aluminum pleats those are one of relatively new oxygen cleanser material. Because of mixing of metallic aluminum with its oxide forms in uneven matrix of this material, aluminum speciation is a key parameter, since just metallic aluminum can clean the FeO and MnO from oxygen.

For analysis of this real sample, it is necessary to random sampling from brikets packages. All of brikets crashed and quartered to about 150 g. Then it was grinded to 60 mesh with a high power and speed Herzog miller to make a good laboratory sample. Using acid digestion, the metallic aluminum was dissolved and separated from the sample. The undissolved part was used for determination of Al_2O_3 and SiO_2. Other mineral analytes was determined in the case of acid dissolved or undissolved. In this studied, flame atomic absorption spectrometry, X-Ray fluorescence and pH metric titration are used and our studied showed that this various methods have comparable results. In addition, Leco CS300 carbon sulfur analyzer was used to determine the carbon and sulfur content using infrared absorption after combustion.

Key words: Oxygen cleanser, aluminum, speciation

References:

1-This method is a laboratory developed method validated according to ISO/IEC 17025.

New pincer type palladium complexes as a photosensitizes in dye sensitized solar cells

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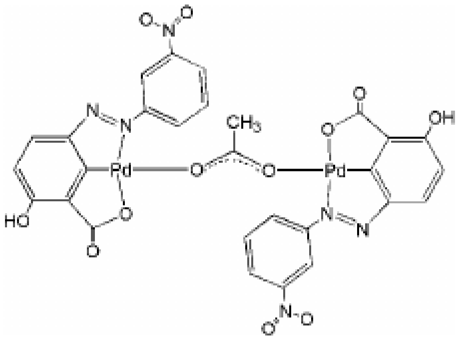
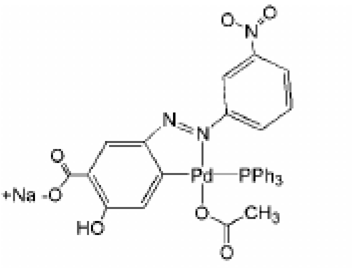
Increasing energy demands and concerns over global warming have led to a greater focus on renewable energy sources in recent years. Dye-sensitized solar cells (DSSCs) have emerged as an important cheap photovoltaic technology. The search for novel dyes in the context of dye-sensitized solar cells (DSSCs) has gained much impetus since the first report in 1991 by O’Regan et al. [1]. As a key part of DSSCs, dyes play a crucial role in high solar-to-electricity conversion efficiency and have been in high solar-to-electricity conversion studied in depth by many researchers. In this research, we investigated new palladium complexes as a sensitizer:





Complex A (left) is a binuclear complex. After treated A with triphenylphosphine, Complex B (right)was formed. Half wave and zere-zero potentials for these complexes obtained with cyclic voltammetry and absorbtion and emission spectra intersection. The results were used to estimation HOMO and LUMO energy levels. Finally photoanode was maked with these complexes and was used to fabricate DSSC. The same procedure was done for Di-tetrabutylammonium cis bis(isothiocyanato)bis(2,2’-bipyridyl-4,4’ dicarboxylato) ruthenium(II) (N719) as standard sensitizer. The results shows most efficient solar-to-electricity conversion efficiency of the dyes under simulated AM 1.5 G solar irradiation (100 mW cm-1). For complex A: Jsc=12.65 mA cm-2, Voc=675 mV, ff=0.63 and for complex B: Jsc=16.87 mA cm-2, Voc=574 mV, ff=0.59.

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New solid-phase microextraction coating based on ionic liquid-mediated poly(dimethylsiloxane) grafted carbon nanotube prepared by sol-gel technique for head-space solid-phase microextraction of methyl tert-butyl ether using Gas chromatography

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Abstract

In the present work, head-space solid-phase microextraction method was developed for the preconcentration and extraction of methyl tert-butyl ether. For the first time, an ionic liquid mediated multi-walled carbon nanotube-poly(dimethylsiloxane) hybrid coating that was prepared by covalent functionalization of multi-walled carbon nanotubes with hydroxyl-terminated poly(dimethylsiloxane) using the sol-gel technique [1, 2], was used as solid-phase microextraction adsorbent. This innovative fiber exhibited high porous surface structure, high thermal stability (at least 320 °C) and long lifespan (over 210 times). Potential factors affecting the extraction efficiency were optimized. Under the optimum conditions, the method detection limit (S/N=3) was 0.007 ng mL^{-1} and limit of quantification (S/N=10) was 0.03 ng mL^{-1}. The calibration curve was linear in the range of 0.03 to 200 ng mL^{-1}. The relative standard deviation for one fiber (repeatability, n=5) at three different concentrations (0.05, 1 and 150 ng mL^{-1}) were 5.1, 4.2 and 4.6% and for the fibers obtained from different batches (reproducibility, n=3) were 6.5, 5.9 and 6.3%, respectively. The developed method was successfully applied to the determination of methyl tert-butyl ether in different real water samples in three consecutive days. The relative recoveries for the spiked samples with 0.05, 1 and 150 ng mL^{-1} were between 94-104%.

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New Strategy for Synthesis of Three-Dimensional Cooperite (PtS)–Nanoparticles on Reduced Graphene Oxide Surfaces Through Surface Functionalization Using Diazonium Chemistry: Application for Electrocatalytic Oxidation of Methanol

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Abstract

A new functionalization method has been introduced to achieve controlled deposition of Pt nanoparticles (NP) on reduced graphene oxide (rGO). On the contrary, we observe heterogeneous deposition of nanometer sized Pt islands on rGO subjected to oxidation prior to deposition in the same conditions. We demonstrated that functional groups act as nucleation sites for Pt–NP nucleation, and that homogeneous nucleation of small particles can be achieved by combining surface functionalization with diazonium chemistry and appropriate stabilizers in solution [1]. Here we have used 4-aminothiophenol modified rGO as a support decorated with Pt nanoparticles ($Pt^{@}SC_6H_4$–rGO) for preparation of a suitable and effective electrochemical mediator for the oxidation of methanol and reduction of oxygen. The surface morphology, structure and composition of $Pt^{@}SC_6H_4$–rGO and $Pt^{@}$rGO as well as their activity toward methanol oxidation and oxygen reduction were investigated by atomic force microscopy, scanning electron microscopy, X–ray diffraction, cyclic voltammetry and electrochemical impedance spectroscopy. The results from FE–SEM and AFM showed that decoration of Pt nanoparticle on functnalized EGO leads to three-dimensional (3D) platinum nanostructures. In addition, the XRD results on functionalized rGO showd that the lattice has lower defects compare with unmodified rGO. Therefore, the electrical and mechanical properties of the functionalized rGO are severely better then unmodified rGO. In addition, decoration of Pt on the surface of functionalized rGO fabricated a crystalline tetragonal of PtS (Cooperite). The electrochemical performances of the synthesized $Pt^{@}$rGO and $Pt^{@}SC_6H_4$–rGO electrocatalysts were also showed that $Pt^{@}SC_6H_4$–rGO has better electrocatalytic activity to methanol oxidation *vs.* $Pt^{@}$rGO.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

NH_2 functionalized KIT-6 mesoporous silica magnetite nanoparticles as an efficient solid phase for preconcentration of anionic surfactant

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Abstract

In this study, mesoporous silica magnetite nanoparticles (Fe_3O_4@SiO_2@KIT-6) functionalized with NH_2 were synthesized and introduced as a magnetic solid phase for preconcentration of sodium dodecyl sulphate (SDS) in aqueous samples. For this purpose, magnetite nanoparticles (MNPs) with particle size lower than 21 nm and KIT-6 mesoporous-coated MNPs were synthesized via chemical precipitation methods. The surface of bare MNPs was coated with SiO_2 and then was coated by a highly ordered large pore mesoporous of KIT-6. After that, amide groups were conveniently loaded on the surface of mesoporous MNPs via chemical. Scaning electron microscopy (SEM), powder X-ray diffraction (XRD), Fourier transform infrared spectrophotometery (FT-IR) and Nitrogen adsorption-desorption isotherms confirmed the magnetic mesoporous structure and regularity of the synthesized functionalized mesoporous magnetite nanoparticles (MMNPs). The applicability of these synthesized nanoparticles for solid phase extraction of SDS from aqueous samples was investigated via Taguchi method. To optimize different parameters which influence the extraction efficiency, a Taguchi L_{16} (4^5) orthogonal array experimental design was used. In order to have the highest extraction yield, the effect of various experimental parameters (five factors at four levels) including volume of desorption solvent, amount of adsorbent, solution pH, ionic strength and stirring time were studied and the optimum values were selected. Concentration of eluted analytes was measured by UV-Vis spectrophotometry after ion pair formation of SDS with methylene blue and its further extraction into $CHCl_3$. Dynamic linear range (DLR) was observed in the range of 2-200 µg L^{-1} with correlation coefficients more than 0.99 and the limits of detection was obtained as 0.55 µg L^{-1}. Finally, the applicability of the synthesized nanoparticles for extraction and determination of SDS from real wastewater samples was successfully investigated and satisfactory results were obtained.

Keywords: Mesoporous magnetite nanoparticles, KIT-6, preconcentration, SDS

NH_2 functionalized KIT-6 mesoporous silica magnetite nanoparticles as an efficient solid phase for preconcentration of anionic surfactant

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Keywords: Mesoporous magnetite nanoparticles, KIT-6, preconcentration, SDS

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Nitrite optical chemical sensor based on its catalytic effect on sulfonazo (III) of bromate red-ox system spectrophotometric

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Abstract

Nitrite is a naturally occurring ion that is part of the nitrogen cycle. Nitrite can be formed chemically in distribution pipes by Nitrosomonas bacteria during stagnation of nitrate-containing and oxygen-poor drinking-water in galvanized steel pipes or if chloramination is used to provide a residual disinfectant and the process is not sufficiently well controlled [1]. A rapid, simple, and most economical spectrophotometric method is presented for the determination of nitrite in water samples. The method is based upon catalytic effect of nitrite on the reaction of immobilized Sulfonazo(III) dye and bromate in sulphuric acid medium [2]. The decolorization of the dye was monitored as analytical signal spectrophotometrically at 586 nm. The method was optimized for effected of concentrations of Sulfonazo(III), Bromate, sulphuric acid, time and temperature. The limit of detection was obtained 0.15 mgL^{-1} (S/N=3). Linear dynamic range of 0.05-2 µg mL^{-1} was obtained in the optimized condition. The effect of possible interfering ions on the determination of nitrite is also studied and revealed that no major interference exist for nitrite detection in water samples. The color of the immoblized Sulfonazo (III) dye correspond to shelf life of sensor was found to be stable for about1 month. The accuracy and validity of the proposed kinetic method was established by studying recovery of spiked nitrite ion in some real water samples. The method was successfully applied to the determination of nitrite in some drinking and tap water.

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NMR and NQR study of Ge-doped on zigzag models of BNNTs

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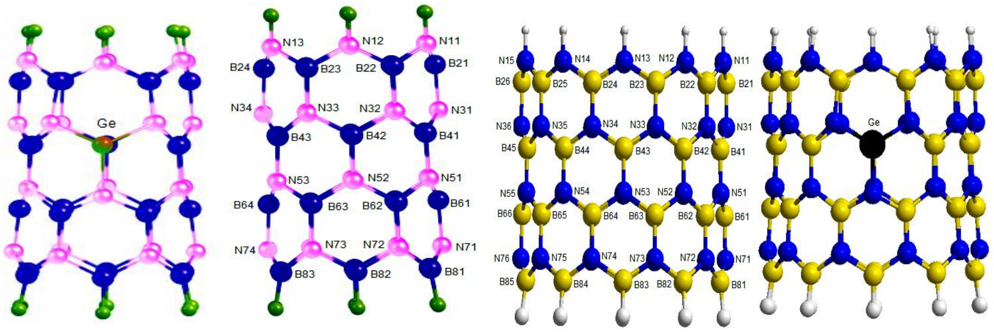
Over the last decade, a significant effort has been made to form BN nanotubes (BNNTs) because of their high potential in future electronic and mechanical devices. In fact, BNNT s have been predicted to be wide band-gap semiconductors independent of radius, chirality and the number of tubular shells. Boron nitride (BN) nanotubes were successfully synthesized [1] shortly after prediction in theory [2], which are known to be wide gap semiconductors with band gaps about 5.5 eV, and in their native state are essentially electrical insulating. Like carbon nanotubes, BN nanotubes also possess high surface area, unique physical properties, and morphology. In this work NMR and NQR parameters of Ge-doped on the (6, 0) and (10, 0) zigzag models of BNNTs are calculated from optimized structures by using DFT theory.

The results show that with doping Ge the CSI parameters of B42 and B44 of two-nanotube increase and at N33, N34 and N53 sites decrease significantly from original values due to donor electron effect of Ge. The CQ parameters at layers one and two increase and other layers decrease.



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Nonylphenol removal from aqueous solutions using ß-CD modified silica supported iron oxide nanoparticles

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Abstract

Nonylphenol (NP) is product of anaerobic degradation of a large group from non-ionic surfactant nonylphenol poly ethoxylate (NP_nEO) [1]. NP_nEO is used in much household and industrial activity. It s environmental effects include feminization of aquatic and decreased breeding in males, reduced seed germination in plants and water adsorption. In human, it cause cancer ,be side it s action on disrupting the productive system of males by readucing sperm conunts. How evere remediation of these contamints is necessary so differen method have been applied for removal of nonylphenols from aqueous solution. The aim of this reaserch was to identified the ability of beta cyclo dextrin (ß-CD), ß-CD@Fe_2O_3@Si nanoparticles in NP removal from aqueous solution. To do so, effect of the important parameters on its removal were investigated by response surface mythology (RSM) using Box Behnken desing(BBD). All concentration were measured after SPE separation on C_{18} catridge using HPLC equipped detected with photo diod ary. At optimum conditions, pH=9.2,contact time =11min, NP 10ppm, and 4.4 mg ß-CD@ Fe_2O_3@Si %97removal was obtained. Different isotherms namely, Langmuir, Freundlich, Dubinin-Radushkevitch, Temkin were examined, It was concluded that adsorption obey Freundlich isotherms (R^2 =0.971) .The adsorption kinetic was proved to be psedue first order.The method was applied for the removal of NP from different surface and tap water, obtainig removal in the range of %89 to %97 percent.

Keywords : nonyl phenol, ß-CD modified silica supported iron oxide nanoparticles, surfactant(Np_nEO)

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Novel Method for Simultaneous Production and Immobilization of Nano-Ferric Oxide on the Agarose Membrane

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In this work, we proposed a simple and low-cost procedure for production of iron oxide nanoparticles via mild decomposition of iron(III) complex with Schiff base ligand N,N'-disalicylidenetrimethylenediamine (Salpn) in aqueous solutions at 45 °C. The proposed method has no problems about fuel consumption and environmental pollution. In addition, due to the mild condition used, the method can be used for immobilization of obtained iron oxide nanoparticles on biocompatible and transparent supports such as agarose. Because the obtained metal oxide nanoparticles can be simply immobilized on transparent membranes, the proposed method offers a simple technique for recording the UV-vis spectra of the metal oxides. The phase, size and morphology of the obtained iron oxide nanoparticles were characterized by scanning electron microscopy, IR spectroscopy and X-ray diffraction. The scanning electron microscopy (SEM) image of the obtained Fe_2O_3 nanoparticles indicated that the average particle size of nanoparticles is 45 nm. The peak of XRD pattern indicates that the prepared iron oxide presents in a well crystalline rhombohedral hematite (Fe2O3) phase. The result of AFM image of the immobilized Fe2O3 nanoparticles on the agarose membranes together with size distribution curves have been depicted that the diameters of the Fe2O3 nanoparticles are about 45 nm and Fe2O3 nanoparticles have homogeneous size.

Novel modified carbon nanotubes as a selective sorbent for preconcentration and determination of trace copper ions in fruit samples

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In this work, multiwalled carbon nanotube (MWCNT) was reacted with N-(3-(triethoxysilyl)propyl)isonicotinamide (TPI) to produce pyridine group on the surface of CNT. This novel sorbent was characterized by infrared spectroscopy (IR), thermal analysis (TG/DTA), and elemental analysis (CHN) and scanning electron microscopy (SEM) and it was applied for preconcentration and determination of copper ions using flame atomic absorption spectrometry (FAAS). SPE procedure has been used for separation and preconcentration of trace Cu(II) due to their simplicity, flexibility, more environments friendly (due to consumption of small volumes of organic solvent), high enrichment factor, being costless, and short extraction time for sample preparation [1-2]. Various parameters such as sample pH, flow rate, eluent type and concentration, and its volume were optimized. The optimized values were: sample pH, 7; sample and eluent flow rate, 18 and 6 mL min^{-1}; eluent type, HCl; eluent concentration, 1 mol L^{-1}; eluent volume, 3 mL. Under optimal experimental conditions the limit of detection (LOD), the relative standard deviation and the recovery of the method were 0.65 ng mL^{-1}, 3.2% and 99.4%, respectively. After validating method using standard reference materials, the new sorbent was applied for extraction and determination of trace Cu(II) ions in fruit samples.

To the best of our knowledge, this is the first report on the use of carbon nanotubes functionalized with N-(3-(triethoxysilyl)propyl)isonicotinamide (TPI) as a sorbent in the solid phase preconcentration and determination of metal ions. The high selectivity, high thermal stability, high pyridine loading, low LOD are the advantages of this sorbent.

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A Comparative Survey upon the Corrosion Behavior of the Platinum/Indium Oxide Nanocomposite Coatings Obtained from the Pulse Electro-Deposition Method

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Research in the scope of nanocomposite coatings has been paid attention by many researchers particularly within the recent 3 decades. the coating process and the surveying on the enterance of particles into coating and optimization of the mechanical & technical specifications are some samples of this case. In this research , the coating method of platinum along with indium oxide nano particles using the pulse electro deposition method is investigated. In addition ,we studied the morphology of two coating sample: 1-nano composite, 2-the usual composite, by using a "SEM". The morphological test was performed by using the vicrose method and their abrasive behavior was investigated by "pin on plate" method . Then , the corrosion behavior of nanocomposite coatings was studied by "Impedance" method. The mechanical desirable characteristics can be obtained by establishing the desirable conditions exactly and studying the method of sedimentation of particles in coating . Also , we can modify and optimize the corrosion behavior in comparison with the net platinum coating by using the nanocomposite corrosion and abrasion mechanisms. In electro deposition bathing-tub, some substances such as carborendum(SiC),chromium sulfide(Cr_2S_3), molybdenum sulfide were used. In this research, as precating procedure, a copper-foil set with dimensions: 40*11*0.2 cm was prepared and the preparation related works such as polishing with emery, removing the grease by using the 30% industrial grease remover specific solution in 75^0C,washing with dilute sulfuric acid and distilled water and making dry was done. Then, the nano indium oxide powder with mean dimension which was 100 nm was added to bathing-tub 24 hours after the preparation of electro deposition specific solution and stirred in 90 minutes with an ultrasonic stirrer. The bath temperature was 65^0C.As a final result, we observed that coatings have the higher resistance versus corrosion in comparison with the net platinum coatings[1,2].The comparative aspect related upon the circuits is shown in Figure1.

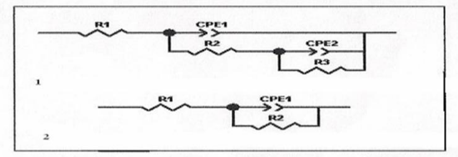

Figure1. The related circuit for 1-the cracked platinumized coatings, 2-The platinum-indium nanocomposite coatings.

Key words: coatings, corrosion, electro deposition, nanocomposite

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NQR study of CO adsorption on (4,4)armchair of Boron nitride nanotube

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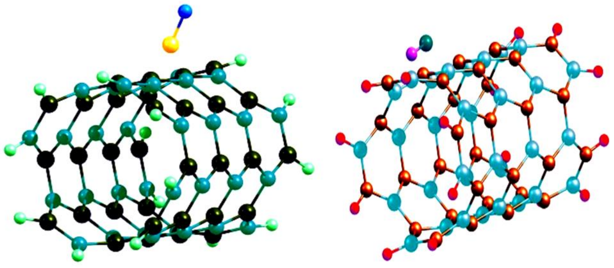
In the last years, much investigation has been done to find new sensors materials for carbon monoxide (CO). Carbon monoxide is a harmful gas for the human body and is also a main cause of air pollution. Therefore, many investigations have been undertaken to develop rapid, simple, and sensitive methods for detecting and adsorbing CO [2–3]. In this project we considered the (4,4)armchair of (BNNTs) as a candidate for this propose. The structural and electrical properties of adsorption of CO on outer surface of (4, 4) armchair (see Fig. 1) are investigated at B3LYP level of theory using the Gaussian 03 set of programs. The calculated EFG tensor eigenvalues in the principal axis system are converted into measurable NQR parameters (C_Q), (η_Q)) using bellow equations. The standard Q values (Q (^{11}B) = 40.59 mb) reported by Pyykkö.

$$C_Q(MHZ) = e^2 Q q_{zz} h^{-1}$$

$$\eta_Q = \left|(q_{xx} - q_{yy}) / q_{zz}\right| \quad 0 \prec \eta_Q \prec 1$$

The comparison results show that CQ values in all study models in the first layer are bigger than other layers. This layer plays important roles in determining the electronic behaviour of these nanotubes, because the geometrical properties of this layer are different from the other layers.



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Nucleic Acid Based Fluorescent Nano-thermometer

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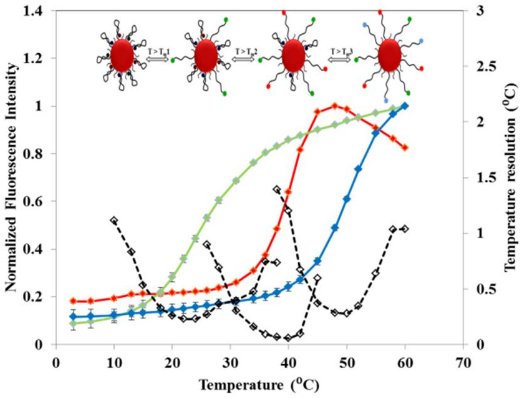
Accurate thermometry is important in micro and nano-biotechnologywhere traditional methods are insufficient. A shortcoming of the current molecular beacon (MB)-based thermometers, as one of the suggested probes for micro and nano scale, is that they rely on only one DNA hairpin structure with a limited temperature sensing function [1]. In this study, we present a novel nano-bio assembly that can function as a sensitive fluorescent thermometer for the measurement of a wide range of temperatures at nanoscopic scale.

The proposed thermometer is made from three different MB probes, each labeled with a different fluorophore, which are attached onto the surface of gold nanoparticles (AuNPs). Wide range temperature sensing of this sensor is a result of different melting behaviors of MBs as a function of the sequence/length of their stem regions. At the lowest temperature, the MBs are all in closed form; bringing the fluorophores near the AuNP surface, where they are quenched. By increasing the temperature, the MBs start to open one by one regarding their melting points and as a result, their fluorescence emission will increase. The overall result will be a sensitive fluorescence method, which depends on the current temperature in the system.

In this study we used three MBs with melting points (T_m) equal to 23, 38, 49 °C labeled with FAM, Texas red, and Atto425 respectively. The MBs were first designed virtually by the help of Mfold web server [2] to avoid multiple secondary structures. The optimum structures with the fluorophore at the 5′ end and the thiol linker at the 3′ were synthesized. After activation, the MBs were incubated with 13 nm AuNPs to self-assemble through the Au-S bond [3].

In Figure, the response of the probe composed of three probes to the temperature is shown. Such a probe can be used for sensitive and high resolution temperature sensing from less than 15 °C to temperatures higher than 50 °C for many applications at micro and nanometer scale. The proposed thermometer performed well in bulk solutions and also used for temperature mapping by fluorescence microscopy.



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Optical spectroscopy studies of the complexation of one new azo-azomethine derivative with some transition metal ions

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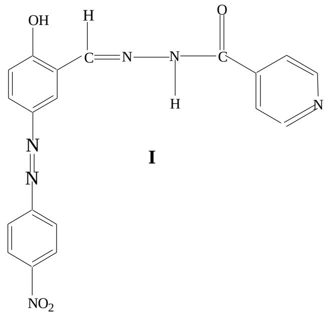
Azo ligands containing salicyaldimine-based ligand as side chains can be used in the production of chemical sensors because they show a significant change of color of the solution and maxima of the absorption band when they interact with transition metal ions and anions [1-3]. Therefore among the wide range of materials that may be suitable for the electronic and optical applications, salicyaldimine-based ligands are of particular interest. In this work, azo Schiff-base derivative **I** was synthesized and characterized with FT-IR, ^{1}H NMR and elemental analysis techniques. The optical response of azo groups of **I** towards Ni^{2+}, Co^{2+}, Cu^{2+}, Hg^{2+}, Zn^{2+} and Cd^{2+} metal ions was studied in DMSO by UV-vis spectroscopy. The absorption spectra of **I** with cations showed marked changes. In solution, azo Schiff-base **I** produced a cation indued 95 nm blue shift for Cu^{2+} ion from 555 nm to 460 nm with remarkable color change from red to yellow. Furthermore, Job's plot indicated 1:1 binding-stiochiometry for **I** with Cu^{2+} ion and Benson-Hilderbrand plot was used for the determination of its association constant. It was revealed receptor **I** is highly specific for cupper ions in DMSO solution.



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Optimization of arsenic species determination in environmental, food and biological samples using new modified aluminum oxide nanoparticles

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Abstract

Arsenic is known to be one of the most toxic elements and has serious effects on plants, animals and human health [1]. It is very well known that toxicity depends not only on total concentration but also on chemical species in which this analyte is present. Toxicity of arsenic depends on its chemical form; inorganic arsenic species are more toxic than their organic counterparts and inorganic trivalent form [As (III)] is more toxic than the pentavalent one [As(V)][2,3]. In the present study, an efficient solid phase extraction method based on new functionalized aluminum oxide nanoparticles for preconcentration, speciation and determination of trace amounts of arsenic in different type of environmental, food and biological samples was developed. The effective parameters on arsenic species determination were optimized using two optimization methods: fractional factorial design (FFD) and central composite design (CCD). At pH 1.5, As (III) was selectively extracted and As(V) retained in the sample solution, but at pH 4.5 both As(III) and As(V) were extracted from sample solution. Thus, the total As(III, V) was determined without need for oxidation or reduction process. The concentration of As (V) in the sample solution was calculated easily by subtracting As (III) from total arsenic. Based on three SD of the blank, the detection limits were 1.81 ng $L^{-1}$and 1.97 ng $L^{-1}$for As (III) and total As respectively.

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Optimization of Dipyridamole hollow fiber-liquid phase microextraction from water and human urine samples using experimental design

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Dipyridamole (DIP), 2,6-bis(diethanolamino)-4,8-dipiperidinopyrimido-[5,4d] pyrimidine is widely used for the treatment of several cardiovascular diseases because of its vasodilating and antiplatelet properties [1]. Unfortunately, it is consumed fraudulently in certain sports to improve efficiency and decrease tiredness. Nevertheless, the uncontrolled usage of this drug could cause loss of mental power and serious secondary effects with grave danger for health [2]. Therefore, the development of simple and sensitive analytical methods is needed for the determination of DIP in pharmaceutical and biological samples. Recently many research groups, have developed hollow fiber-liquid phase microextraction (HF-LPME) for sample extraction in biological and chemical analysis [3]. In this work, HF-LPME with high performance liquid chromatography and experimental design was developed for preconcentration and determination of DIP in water and urine samples. Octanol and methanol were selected as the extraction solvent and desorption solvent, respectively. The effects of different factors on the extraction efficiency were studied simultaneously using fractional factorial design. Chromatographic separation was achieved on a C18 column using a mobile phase consisting of methanol and ammonium acetate. The uv-vis detector was performed at a 280 nm. The results showed that the stirring rate, salt amount and desorption time were ineffective. Therefore, optimum conditions was include: volume of sample; 20 mL, extraction time; 60 min, stirring rate; 300 rpm, desorption time; 5 min, and pH; 12.

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بیستمین کنفرانس شیمی تجزیه ایران
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Optimization of electrocoagulation process for removing lead from urbane drinking water by Taguchi method

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Expansion of urban areas exacerbated the situation of water resources[1].Lead was hazardous metal due to its high toxicity[2].The coagulant was produced insitu by electrolytic oxidation of a consumed anode[3].The aim of this applied-analytical study was to investigate lead removal from urban drinking water by batch reactor electrocoagulation with aluminum electrodes.Various operating variables were tested.Lead removal efficiency was examined in different operating cases of the variables current density(0.8-2.4 mA/cm^2),initial concentration(75-150 mg/l),pH(4-9),and time(5-80 min).It was found that theoptimum initial pH to remove Pb^{+2} ions was 9.Also, increasing the currentdensity from 0.8 to 2.4 mA/cm^{2}and reaction time from 5 to 80 min had a positive effect on the Pb^{+2} removal efficiency.Pb^{+2} removal rate enhanced with increasing the initial concentration of lead.Results showed that,at a batch mode with an initial concentration of 150 mg/l,a pH of 9,a reaction time of 20 min,and a current density of the anode of 2.4 mA/cm^2,Pb^{+2} reduction was 100%.The results of lead removal efficiency by Taguchi model showed that reaction time was the most important variable.Thus,batchexperiments indicated that the electrocoagulation reactor could be efficient in removinglead from drinking water and considered as a promising technology for treating lead-polluted drinking water.

Keywords: Drinking water; Electrocoagulation; Lead; Metal.

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Optimization of electrocoagulation process for removing lead from urbane drinking water by Tachuchi model

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The population growth and expansion of urban areas exacerbated the situation of water resources [1]. Lead was hazardous metal due to its high toxicity [2]. There was necessity for new technologies in water treatment. Electrocoagulation was an innovative technology, and low operational and maintenance costs. The coagulant was produced in situ by electrolytic oxidation of a consumed anode [3]. The aim of this applied-analytical study was to investigate lead removal from urban drinking water by batch reactor electrocoagulation with aluminum electrodes. Various operating variables were tested for their effects on lead removal; these included current density, initial concentration, pH, and time. Lead removal efficiency was examined in different operating cases of the variables current density (0.8 to 2.4 mA/cm^2), initial concentration (75 to 150 mg/l), pH (4 to 9), and time (5 to 80 min). Lead was determined according to procedure detailed in standard methods. It was found that the optimum initial pH to remove Pb^{+2} ions was 9.0. Also, the results indicated that increasing the current density from 0.8 to 2.4 mA/cm^2 and reaction time from 5 to 80 min had a positive effect on the Pb^{+2} removal efficiency. Also, the findings of our study indicated that Pb^{+2} removal rate enhanced with increasing the initial concentration of lead. Results showed that, at a batch mode with an initial concentration of 150 mg/l, a pH of 9.0, a reaction time of 20 min, and a current density of the anode of 2.4 mA/cm^2, Pb^{+2} reduction was 100%. The results of lead removal efficiency by Tachuchi model showed that reaction time was the most important variable. Thus, batch experiments indicated that the electrocoagulation reactor could be efficient in removing lead from drinking water and could be considered as a promising technology for treating lead-polluted drinking water.

Keywords: Drinking water; Electrocoagulation; Lead; Metal.

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Optimization of electrocoagulation process for removing lead from urbane drinking water by Tachuchi model

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The population growth and expansion of urban areas exacerbated the situation of water resources[1].Lead was hazardous metal due to its high toxicity[2]. There was necessity for new technologies in water treatment.Electrocoagulation was an innovative technology, and low operational and maintenance costs. The coagulant was produced insitu by electrolytic oxidation of a consumed anode[3]. The aim of this applied-analytical study was to investigate lead removal from urban drinking water by batch reactor electrocoagulation with aluminum electrodes. Various operating variables were tested for their effects on lead removal; these included current density, initial concentration, pH, and time.Lead removal efficiency was examined in different operating cases of the variables current density (0.8 to 2.4 mA/cm^2), initial concentration (75 to 150 mg/l), pH (4 to 9), and time (5 to 80 min).Leadwas determined according to procedure detailed in standard methods.It was found that theoptimum initial pH to remove Pb^{+2} ions was 9.0. Also, the results indicated that increasing the currentdensity from 0.8 to 2.4 mA/cm^{2}and reaction time from 5 to 80 min had a positive effect on the Pb^{+2} removal efficiency. Also, the findings of our study indicated that Pb^{+2} removal rate enhanced with increasing the initial concentration of lead. Results showed that, at a batch mode with an initial concentration of 150 mg/l, a pH of 9.0, a reaction time of 20 min, and a current density of the anode of 2.4 mA/cm^2, Pb^{+2} reduction was 100%.The results of lead removal efficiency by Tachuchi model showed that reaction time was the most important variable. Thus, batch experiments indicated that the electrocoagulation reactor could be efficient in removinglead from drinking water and could be considered as a promising technology for treating lead-polluted drinking water.

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Optimization of removing of lead ion by magnetic nano particles modified by chitosan from real samples

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Abstract

In this study the possibility of removing lead metal ion with Fe_3O_4 magnetic nano particles from Aquent samples and measuring the remained solution was investigated. Chitoson was used for this purpose. Chitosan is a hyphophilic and cationic polymer that obtained from removing acetil citin in basic solutions and use as a absorptive to removing heavy metals. Chitosan was first carboxymethylated and then covalently bounded onto the surface of Fe_3O_4 nanoparticles via carbodiimide activation. SEM micrograph of magnetic chitosan particles provides information on their size and morphology. The experiments of this investigate was carried out in Discrete system and room temperature. pH, commence concentration of metal, time were optimized by experimental design. The optimum condition for pH, the amount of sorbent, commence concentration of metal and time were 4.14, 0.06gr, 60mg/l and 66 respectively. Recover of sorbent by EDTA and multi usages are important merit of this sorbent.

Keywords: Lead, Magnetic nano particles, Chitosan, Experimental design

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Optimization of Separation of Phenolic acids and Flavonoids in Low-Submicellar Liquid Chromatography using Empirical Retention Models and Desirability Function

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Low-submicellar liquid chromatography (LSLC) is a rather uncommon mode of surfactant mediated reversed phase liquid chromatography (RPLC) which employs hydro-organic solution of surfactants under critical micellar concentration (CMC) as mobile phase [1]. In LSLC, mobile phase contains a small amount of free surfactant monomer while, on the stationary phase, the alkyl chain of the adsorbed surfactant monomer associated to the alkyl-chains bonded to the silica support, and the polar head groups oriented away from the surface [2]. At these conditions, hydrophilicity of stationary phase was increased, as a consequence significant changes in the chromatographic behavior of solutes can be occurred. In this study, separation optimization of five phenolic acids (galic, chlorogenic, caffeic, p-coumaric and ferulic acid) and four flavonoids (myricetin, morin, quercetin and kaempferol) in LSLC was performed using interpretive optimization strategy and Derringer's desirability function [3]. Prediction of the retention of solutes was carried out through 16 hyperbolic and logarithmic retention models using a procedure that utilizing the retention data of nine mobile phases of sodium dodecyl sulfate (SDS) (0.001, 0.004, 0.007 M) and methanol (35, 45, 55% v/v) at 0.2% of H_3PO_4. Among the all investigated models the hyperbolic retention model "$1/k = c_0 + c_1\varphi + c_{11}\varphi^2 + c_2[S] + c_{12}\varphi[S]$" showed the best prediction capability and used to predict the solutes retention factors. The mean relative error of the prediction of retention factors of nine target phenolic compounds in the whole variables space was lower than 2.8%. A grid search program was used to calculate the retention times of each solute for all combination of SDS and methanol concentrations in the variables space (by numerical solution of the retention model). Two different chromatographic criteria, analysis time and retention differences between adjacent peaks were evaluated simultaneously using Derringer's desirability function for each of the 273 different mobile phase composition in the grid search. A sigmoid function was used to transform the optimization criteria to desirability values (ranging between 0 and 1). The efficiency of this optimization strategy was evaluated by performing the experiment under the predicted optimal conditions. At the optimal condition (0.007 M SDS, 35% methanol at 0.2% of H_3PO_4), full resolution of target phenolic compounds was achieved in 70 min. However it should be mentioned that an appropriate resolution between target compounds in the hydro-organic RPLC using isocratic elution (35% methanol at 0.2% of H_3PO_4) was achievable at 250 min.

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Optimization of temperature-controlled ionic liquid homogenous microextraction combined with high performance liquid chromatography for analysis of diclofenac and mefenamic acid in urine sample

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Diclofenac and mefenamic acid as anti-inflammatory drugs (NSAIDs) usually are widely used as ananalgesic, anti-inflammatory and anti-arthritic agent. Nowadays, determination of pharmaceutical drugs in biological fluids has found noticeable importance in medical sciences, due to increasing of medicine consuming among people. However, determination of drugs and metabolites in biological fluids such as plasma and urine is still a very challenging task [1, 2]. In this work, temperature-controlled ionic liquid homogeneous microextraction (TCIL-HME) [1-2] followed by HPLC-UV was applied for preconcentration and determination of diclofenac and mefenamic acid in urine samples. 1-butyl-3-methylimidazolium hexafluorophosphate ([C4mim][PF6]) was used as the optimum extraction solvent. The effect of different variables on the extraction efficiency was simultaneously studied using an experimental design. The Plackett-Burman design was employed for screening to determine the variables significantly affecting the extraction efficiency. Then, the significant factors were optimized by using a central composite design (CCD) and the response surface equations were developed. The optimal experimental conditions obtained from this statistical evaluation included: extraction solvent volume of 105 µL; centrifugation time of 5 min; heating time of 2 min; solution temperature of 40 °C and 1.2 g addition of salt. Under optimum conditions, the preconcentration factors of 45 and 80 were obtained for diclofenac and mefenamic acid, respectively. The calibration curves were linear in the range of 10 to 1000 ng mL^{-1}. The intra- and inter-assay precisions (CV%, n = 3) were in the range of 3.5-8.0% and 6.5-8.5% at concentration level of 100 ng mL^{-1}, respectively. The validated method was successfully applied for the analysis of target analytes in some urine samples.

Keywords: Microextraction, Ionic liquid, Diclofenac, Mefenamic, Urine sample.

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Optimized dispersive liquid-liquid microextraction for determination of melatonin by HPLC-UV in water and plasma samples

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Melatonin (N-acetyl-5-methoxytryptamine), an indoleamine synthesized from an essential amino acid, tryptophan, is a unique molecule which involved in the regulation of circadian rhythm (sleep–wake cycle) and the alleviation of sleep disorders, such as insomnia due to jetlag and shift work [1, 2]. Melatonin may also act as a protective agent in ocular diseases and headache disorders. Furthermore, it mitigates neurodegenerative diseases, such as Alzheimer's and Parkinson's diseases and also acts as an anticancer agent. It also has a possible role in metal regulation in central nervous system and could form complexes with aluminium, cadmium, copper, iron and other metals. A rapid and simple method for the extraction of melatonin in water and samples using dispersive liquid–liquid microextraction (DLLME) using chemometric was developed. Influence variables such as volume of extracting (CCl_4) and dispersing solvents (ACN), pH and ionic strength, extraction time and centrifugation time were screened in a 2^{6-2} fractional factorial design was investigated. The significant variables were optimized by using a Central Composite Design (CCD) combined with Desirability Function (DF) [3]. At optimum conditions values of variables set as pH = 6.0, 1.5 mL acetonitrile and 140 μL CCl_4. At optimum conditions method has linear response over 0.005-5 μgmL^{-1} with detection limit between 0.0015-0.0025 μgmL^{-1} with relative standard deviations (RSDs) less than 6.0 % (n=3).

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Optimizotion of dispersive solid phase extraction combined with dispersive liquid-liquid extraction for the determination of BTEX in soil samples using Aco- Ann

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Abstract

Dispersive liquid-liquid extraction (DLLE) is a preconcentration method, which used a ternary system of solvents. The advantages of this technique are simplicity, fast, low cost, good efficiency and high enrichment factor. DLLE is a green extraction technique, due to the consumption of organic solvent is reduced to the microliter [1-3]. In this study, dispersive solid phase extraction (DSPE) combined with DLLE has been developed for the extraction of benzene, toluene, ethylbenzene and xylenes isomers (BTEX) in soil samples prior to gas chromatography-mass spectrometry. The BTEX were extracted from soil sample into acetonitrile by DSPE method, and the extract was then used as dispersive solvent in DLLE procedure. Ant colony optimization - artificial neural network (ACO-ANN) have been employed to develop the model for simulation and optimization of this method. The volume of dispersive solvent, volume of extraction solvent, extraction time and ultrasonic time were the input variables, while the multiple response function (R_m) of analytes was the output. The optimum operating condition was then determined by ACO method. The optimium condition were as follows: the volume of dispersive solvent 1.5 mL, the extraction time 8.0 min, the ultrasonic time 14.8 min and volume of extraction solvent was 100 µL. At the optimum conditions, the preconcentration factors of 281.5-328.1, and the limit of detections of 0.12 - 0.75 ngg^{-1} were obtained for the BTEX. The developed procedure was then applied to the extraction and determination of BTEX in the soil samples.

Key Words: Dispersive solid phase extraction; Dispersive liquid-liquid extraction; Gas chromatography-mass spectrometry; Artificial neural network - Ant colony optimization; Soil samples.

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Palladium(II) adsorption onto the $Fe(OH)_3$/graphene nanocomposite layer

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The heavy metals released to environment were also detected in the river water and the sediment accumulated to be managed as hazardous materials [1]. Adsorptive removal of aqueous heavy metal ions has been widely applied using such a lot of materials as activated carbon, ash, zeolites, metal oxides, chitosan and agricultural by products [2]. Graphene, a new kind of carbon material, is a single layer of carbon atoms with a hexagonal arrangement in a two-dimensional lattice. Its unique structural and excellent physical, chemical and mechanical properties make it promising for potential applications in many technological fields, such as nanoelectronics, sensors, nanocomposites, batteries, supercapacitors, and adsorbents. This features make graphene an attractive choice as the matrix for nanocomposites. Since combination of graphene and functional nanoblockes may lead to materials with interesting properties for a variety of applications, considerable efforts have recently been made to fabricate various graphene nanocomposites with metal, metal oxide and sulfide [3].

This work reports a general approach for the preparation graphene oxide, deposition of iron(III) hydroxide onto surfaces of graphene oxide by chemical deposition method and reduction of graphene oxide matrix to graphene. The as-synthesized composites were characterized by X-ray diffraction (XRD), Fourier transform infrared (FT-IR) spectroscopy, Raman spectroscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS). It was shown that the as-formed $Fe(OH)_3$ nanoparticles were densely and homogeneously deposited on the surfaces of the graphene sheet. Ferric hydroxide, which was cross-linked with graphene sheets, caused to enhance the efficiency and capacity of palladium removal. The optimum absorption of palladium using 0.01 g of $Fe(OH)_3$/graphene was observed in conditions of pH = 4, T = 50 °C and Conc. 5 ppm. It could be concluded that the used composite is a promising material for palladium removal.

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Particle size effect of nano Co_3O_4 on their catalytic activity of the thermal decomposition of Ammonium perchlorate

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Ammonium perchlorate (AP) has long been of chemical interest as oxidizers, and has found excellent application in propellants, explosives, and pyrotechnics. Many investigations have been carried out on the catalytic improvement of thermal decomposition properties of AP particles by different nano catalysis additives such as metals powders, metal oxides and Spinels. However, there is no report on the effect of particle size of Co_3O_4 nano Spinels on the heat of decomposition, kinetic parameters and thermal decomposition temperature of AP particles. The present investigation is intended to study the effect of the particle size of nano Co_3O_4 on the thermal decomposition of AP particles. Co_3O_4 nano particles with different particles size (10-30, 30-50 and higher than 50 nm) were used to prepare AP/Co_3O_4 nanocomposites by a novel solvent evaporation method. The preliminary evaluation of results revealed that a significant variation in the catalytic activity was observed according to the range of the particle size used. The results showed that, as the particle size of Co_3O_4 increased, the thermal decomposition temperature of AP/Co_3O_4 nanocomposites and the decomposition activation energies ranges enhanced. The results revealed that smallest nano-sized Co_3O_4 with 10-30 nm diameters shows strong catalytic property for AP thermal decomposition. Adding 5% of Co_3O_4 with 10-30 nm to AP, decreased the exothermic peak temperature of thermal decomposition by 130 °C and increased the heat of decomposition from 880 to 1260 J g^{-1}. Also, the values of $\Delta S^{\#}$, $\Delta H^{\#}$, and $\Delta G^{\#}$ for thermal decomposition processes of pure and treated AP particles have been estimated by non-isothermal methods that proposed by Friedman and Ozawa.

Keywords: Ammonium perchlorate, Thermal decomposition, Particle size of Co_3O_4 nano, Catalytic activity, non-isothermal methods.

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Performance evalaution of nano additive doped detergent powders by infrared spectrometry and pattern recognition techniques

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New generation of detergents are complex mixtures containing different ingredients such as surfactants, builders, bleaches, enzymes and auxiliaries. Performance improvement and meeting the commercial and environmental criteria are the main aims of research and development in detergent industry. Utilization of nano additives have been considered as a route to meet theses aims. Different phases of nano alumina were synthesized at different temperatures with sol-gel technique [1]. In the next step, regarding the effect of nano alumina in detergent powder, various formulations of detergent powder were prepared with a determined amount of different structural phases of nano alumina [2]. According to ISO 4319, the performance of detergents washing powders is tested. In order to provide a new and rapid measurement technique to evaluate the efficiency of powder samples, the diffuse reflectance FT-IR spectra of detergent and nano alumina powders were also obtained. Then different multivariate classification methods such as principal component analysis (PCA), hierarchical cluster analysis (HCA) and linear discriminant analysis (LDA) were utilized for diffuse reflectance spectra to evaluate the classification approach. The best model was built by LDA, with correct classification rate (%CC) of 93.55%. Furthermore, sensitivity and specificity for test set were 0.90 and 0.95, respectively. These results confirm the capability of diffuse DRIFTS coupled with chemometric techniques for estimating the performance of detergent powder formulations containing nano alumina.

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Performance evaluation of natural zeolite – supported zero valent iron nano particles for nitrate removal from aqueous solution

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Abstract

High nitrate concentration has become a serious common water quality problem. The nitrate has high chemical stability, especially at low concentrations, which can lead to several health treats to human such as cancers. In recent years, nanoscale Zero-Valent Iron (nZVI) has been widely studied as an environmentally friendly strong reducing agent, due to its small size, high specific surface area and suitable redox potentials. However, iron nanoparticles expose vigorous propensity to agglomerate into larger solid particles which reduces the effective surface area and thereby decreasing removal efficiency [1]. Furthermore, the separation of non-supported iron nanoparticles from the aqueous phase is still a difficult undesirable task [2]. The present study reports the synthesis and characterization of nano scale zero-valent iron in the presence of natural zeolite as a stabilizer. This novel adsorbent (Ze-nZVI) was synthesized by the sodium borohydride reduction method. The nature and morphology of adsorbent were characterized by Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and Brunauer–Emmett–Teller (BET-N_2) analysis. The SEM images revealed that the stabilized nZVI particles were uniformly dispersed across the zeolite surface without obvious aggregation. The specific surface areas as measured by the BET-N_2 method, for Ze-nZVI and the natural zeolite were 49.65 and 17.82 (m^2 g^{-1}), respectively. Using zeolite as a support bead leads to an increase in the specific surface area of the Ze-nZVI. The XRD pattern of Ze-nZVI, displayed obvious peaks of Fe^0 at $2\theta=44.8$ and $2\theta=65.1$ which confirmed its existence in freshly prepared Ze-nZVI [3]. The synthesized Ze-nZVI was then tested for the removal of nitrate from aqueous solution. Batch experiments indicated that the removal of nitrate is depended on the initial pH of the solution, initial nitrate concentration and sorbent dosage. The adsorption capacity decreased with increasing the Ze-nZVI dosage and initial pH value of the solution but increased with the increase in the initial concentration of nitrate. Reduction of nitrate using Ze-nZVI was in accordance with the pseudo second order kinetic model, moreover, the equilibrium data fitted well to the Langmuir model, representing a monolayer adsorption. The maximum adsorption capacity of Ze-nZVI for nitrate was 22.94 ($mg\ g^{-1}$). Natural zeolite is a low-cost clay mineral, thus, this material could be an effective and promising stabilizer, and dispersant for supporting nano zero valent iron due to its porous structure and high resistance in the environment.

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Performance Study for Platinum and Non-Platinum Cathode Nanocatalysts in Direct Borohydride Fuel Cell

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Abstract

A PEFC that utilizes a borohydride compound, usually sodium borohydride in aqueous alkaline medium, directly as a fuel is termed as direct borohydride fuel cell (DBFC). A DBFC employs an alkaline solution of sodium borohydride ($NaBH_4$) as fuel and oxygen as oxidant. DBFC has some attractive features such as high open circuit potential, low operational temperature and high power density. The DBFC is a promising power system for portable applications.Sodium borohydride (NaBH4) is a good alternative to methanol and hydrogen as a fuel[1].

In the recent years, extensive research work is being carried out and reported on improving the performance of nanocatalysts for both anode and cathode of fuel cells[2,3]. In this study the single cell performance of carbon supported non-platinum electrocatalyst (Hypermec^{TM}K$_{14}$) as cathode material was used in alkaline electrolyte borohydride fuel cells and compared to that of 10% Pt/C (commercial). The structural and electrochemical aspects of the Hypermec^{TM} K$_{14}$ and 10% Pt/C (commercial) electrocatalysts were further investigated. X-ray diffraction (XRD) indicates the Pt and K$_{14}$ particles average size is lesser than 20 nm. Scanning electron microscopy (SEM) shows the spherical shape of Pt and K$_{14}$ particles and distributed homogeneously across the substrate matrix. The Hypermec^{TM}K$_{14}$ cathode showed superior performance to that observed using the 10% Pt/C cathode, e.g. power density up to 138 mWcm^{-2} for DBFC with Hypermec^{TM}K$_{14}$ as cathode. Cyclic voltammetry data exhibit the better borohydride tolerance for K$_{14}$ and Electrochemical impedance spectroscopy (EIS) analyses demonstrated that the cell with K$_{14}$ nanocatalyst showed not only low ohmic resistance but also small charge and mass transfer resistances. The difference in performance can be attributed to variations in activity towards oxygen reduction reaction and in borohydride tolerance among the cathodes.

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Permanganate-bromide-silver nanoparticles as a new chemiluminescence system and its analytical application

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Chemiluminescence, production of light generation through a chemical reaction, is a powerful tool in many fields of analytical chemistry. In recent years, metal nanoparticles have been favorably utilized as an active unit (luminophor, catalyzer or energy receiver) in the chemiluminescence assays to improve their sensitivity [1, 2]. In this study, a novel chemiluminescence system based on the oxidation of bromide by permanganate is introduced. In sulfuric acid medium, Br^- oxidizes by $KMnO_4$ and a weak chemiluminescence emission produce. It observed that silver nanoparticles have a catalytic effect on this reaction and cause an enhanced emission. It was demonstrated that spherical silver nanoparticles with average size of 18 nm had a most remarkable catalytic effect on this reaction. Chemiluminescence emission wavelengths and UV–vis spectra were used to characterize the system and propose a possible mechanism. Furthermore, it was found that glutathione inhibits the action of nanoparticles and decreases the intensity of chemiluminescence. This decrease is proportional to glutathione concentration and, based on this phenomenon, a new chemiluminescence method was developed for the determination of glutathione in the 2.0×10^{-8} to 5.0×10^{-7} mol L^{-1} concentration range with a detection limit of 5.0 nmol L^{-1}. The method was successfully applied to the determination of glutathione in human urine samples.

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Perpetration of reference material containing specific elements such as Ti and Nb to intermediate control of spark optical emission spectrometer

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1. Mobarakeh Steel Company, Isfahan, Iran

Spark analyzer (quantometer) device is used in steel making factory for chemical analysis of elements in steel samples. Intermediate control by reference materials in specified periods is a general requirement in all of the standard test methods, like ASTM, to verify the accuracy of the recalibration. High prices and deficit or absence of such elements like Nb and Ti in common commercial reference material is the problem. Thus, this project tried to prepare domestic steel reference material with favorite chemical composition in Mobarakeh Steel Company, to compensate deficit or absence of some elements in addition of saving a lot of money.

For this purpose a number of steel block samples with special shape and chemical composition were prepared. The quantometer was recalibrated with commercial certified reference materials and after chemical analysis; calculations were done according to standard test method, ASTM E826, to ensure the chemical composition homogeneity of the prepared samples. Then, using standard ASTM E178 the outlier results were eliminated. The uncertainties of the element concentration were also determined. Finally these reference materials were analyzed in different laboratories (out of Mobarakeh Steel Company labs). The results of the inter-laboratory studies confirm that the reference materials have been successfully completed and they can be used as standard samples for the calibration of the instrument.

Keywords: Reference material, Quantometer, Steel, Spark, Optical Emission Spectrometry, Chemical analysis.

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Photocatalytic activity of NiO –clinoptilolite nano-particles towards cefuroxime

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Abstract:

The present study describes photocatalytic behavior of NiO/nano-clinoptilolite (NiO/NCP) as a photocatalyst in the photodegradation process of aqueous solution of cefuroxime pharmaceutical capsule under UV irradiation and the effects of parameters affecting the degradation extent. For this goal, the prepared ball-mill nano-zeolite was ion exchanged with nickle(II) chlorid aqueous and the obtained exchanged form was cacined at 450 °C to obtain NiO/NCP catalyst. All raw and modified samples were characterized by XRD, UV–Vis DRS, TEM and FT-IR methods. The kinetics of the photodegradation process obeyed from Langmuir–Hinshelwood model. The results showed that the optimum catalyst amount for degradation of cefuroxime is 0.025 g L^{-1}. The decrease in the initial rate beyond the catalyst dosage of 0.025 g L^{-1} may be attributed to the screening effect of excess catalyst particles in solution which decreases the light penetration and hence reduces the photodegradation rate [1]. In addition, the role of NCP in the efficiency of photocatalytic degradation was evaluated by performing some degradation experiments using micronized and nano particles of clinoptilolite and our results were showed the importance of the zeolitic bed due to super adsorption capability of the zeolite. NiO dispersion on the surface of the nano-clinoptilolite increases the effective surface area of the NiO which in turn increases the formation of hydroxyl radicals [2].

Keywords: Clinoptilolite, NiO, Photodegradation, Cefuroxime, Nano-particles.

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Photocatalytic degradation of benzophenone by ZnS–CuS doped on the nanoclinoptilolite in aqueous solution

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Abstract:

The catalyst ZnS–CuS doped onto nanoclinoptilolite with different weight ratios were prepared. The prepared catalyst was characterized by XRD, TEM, FT-IR and UV–Vis DRS. XRD patterns show that the crystalline structures of nanoclinoptilolite (NCP) can be stable after ion exchange. Benzophenone was used to estimate the photocatalytic activity of the samples using UV light. The effects of operational parameters such as the amount of photocatalyst, pollutant concentration and pH on photo mineralization were studied. The mineralization of benzophenone was confirmed by HPLC and COD measurements. The results of photodegradation of benzophenone using prepared photocatalyst indicated that the effective degradation was increased in the presence of hybrid catalysts in comparison with that of unhybrid CuS/NCP and ZnS/NCP catalysts. Hybrid catalysts can prevent the recombination of holes and electrons, thus decrease the photocatalytic activity [1-3]. Supported semiconductor onto nano-particles of clionoptilolite increases the efficiency of $ZnS_{1.39}$–$CuS_{6.30}$. Nanoclinoptilolite can distribute excited electrons in conductance band of semiconductors into its network structure hence prevent to recombination of electron–hole pairs [2] which is a desirable phenomenon in photocatlytic processes. The photodegradation process of benzophenone was fitted to first-order kinetics. The maximum efficiency was achieved at pH 7.5, 0.10 g L^{-1} of $ZnS_{1.39}$–$CuS_{6.30}$/NCP and benzophenone concentration of 30 mg L^{-1}.

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Photodegradation of phenol in an industrial wastewater using FeO-nano clinoptilolite

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Abstract

Phenols are pollutants of high priority concerns because of their toxicity. Various methods have been proposed for the treatment of wastewaters containing organic and inorganic pollutants [1]. In this study, photocatalytic degradation of phenol in a wastewater was investigated by the advanced oxidation process (AOP) and using FeO-nano Clinoptilolite as a photocatalyst. With this method complete oxidation of organic compounds to harmless products such as CO_2 and H_2O that appears to be the most emerging technology recently [2,3]. Nano clinoptilolite particles were obtained by mechanical ball-milling method of the pretreated of micro-size clinoptilolite. FeO-nano Clinoptilolite was prepared by ion exchange method and calcined at 250°C for 4 h and it was characterized by XRD, IR and SEM. The wastewater sample was prepared from Esfahan steel factory. The effects of experimental parameters such as catalyst dosage, initial concentration of phenol, initial pH of the solution and effect of the presence of hydrogen peroxide and potassium bromate were studied on the degradation efficiency. The operation optimum values were: 0.3 mgL^{-1} of the photocatalyst dosage, 96 mgL^{-1} of phenol concentration and pH 6.7. Also increasing the concentration of H_2O_2 and $KBrO_3$ increased phenol degradation percent. The FeO particles out of the zeolite framework did not show significant degradation efficiency.

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Photodegradation of Tartrazine by composite of nanomolecularly imprinted polymer-nanophotocatalyst

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Abstract

One of the major concerns in the world today is water pollution. Water-soluble dyes are commonly used as coloring agents in a variety of products, even at very low concentrations, are highly visible and Because of their complex molecular structure and large size most of the dyes are considered non-oxidizable by convectional physical and biological treatments. Thus their decolourization or removal of them, is necessary processes in wastewater treatment [1-2]. In this research a new composite of nano molecularly imprinted polymer (MIP) - nanophotocatalyst for tartrazine with high-efficiency and non imprinted polymer (NIP)-nanophotocatalyst were synthesized by precipitation polymerization in aqueous medium as a green method and were characterized by TGA, SEM, TEM and FT-IR. The effects of pH, time, and composite dosage were studied.

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Polypyrrole Molecularly Imprinted Modified Glassy Carbon Electrode for the Recognition of Dapsone

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Despite the increasing number of applications of biosensors in many fields, the construction of a steady biosensor remains still challenging. Molecular imprinting is a technique that generates, inside a polymer matrix, selective binding sites that recognize a particular molecule. The high selectivity and stability of molecularly imprinted polymers for the template molecule make them ideal alternatives as recognition elements for sensors. Molecularly imprinted polypyrrole is presented as an electrosynthesized coating for the development of selective biosensors. A glassy carbon electrode has been modified with this covering by cyclic voltammetry in the potential range from -0.4 to +0.95 V (versus Ag/AgCl) in 0.1M HCl aqueous solution obtaining a biosensor able to recognize the template molecule (dapsone). The proposed method was successfully utilized for the determination of dapsone in various dosage forms without interference from matrix. The DPV peak current showed a linear dependence on the dapsone concentration and a linear calibration curve was obtained in a certain concentration range. Several important parameters controlling the performance of the molecularly imprinted polymer (MIP) modified sensor, such as the effect of pH, the monomer concentration, the electropolymerization cycles and interferents, were investigated in detail. The molecularly imprinted polypyrrole-modified glassy carbon electrode showed a stable and reproducible response, without any influence of interferents commonly existing in pharmaceutical samples.

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Polypyrrole/montmorillonite nanocomposite as a new solid phase microextraction fiber combined with gas chromatography–corona discharge ion mobility spectrometry for determination of organophosphorus pesticides

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Abstract

A novel solid phase microextraction (SPME) fiber was prepared and coupled with gas chromatography corona discharge ion mobility spectrometry (GC–CD–IMS) based on polypyrrole/montmorillonite nanocomposites for the simultaneous determination of pesticides. The nanocomposite polymer was coated using a three–electrode electrochemical system and directly deposited on a Ni–Cr wire by applying a constant potential [1]. The scanning electron microscopy images revealed that the new fiber exhibited a rather porous and homogenous surface. The thermal stability of the fabricated fiber was investigated by thermogravimetric analysis. The effects of different parameters influencing the extraction efficiency such as extraction temperature and time, salt addition, stirring rate, the amount of nanoclay, and desorption temperature were investigated and optimized. The method was exhaustively evaluated in terms of sensitivity, recovery, and reproducibility. The linearity ranges of 0.05–10 and 0.08–10 μg L^{-1}, and the detection limits of 0.020 and 0.035 μg L^{-1} were obtained for diazinon and fenthion, respectively. The relative standard deviation values were calculated to be lower than 5 and 8% for intraday and interday, respectively. Finally, the developed method was applied to determine the diazinon and fenthion (as model compounds) in cucumber, lettuce, apple, tap and river water samples. The satisfactory recoveries revealed the capability of the two–dimensional separation technique (retention time in GC and drift time in IMS) for the analysis of complex matrices extracted by SPME.

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Polypyrrole/sol-gel composite as a solid phase microextraction fiber coating for the extraction and determination of diazinon in underground water and grape juice samples

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A method based on the direct immersion solid-phase microextraction coupled with corona discharge ion mobility spectroscopy (CD-IMS) was used for extraction and determination of diazinon (as an organophosphorus pesticide) in underground water and grape juice samples. The solid-phase microextraction (SPME) fiber coated with polypyrrole/sol–gel composite was prepared using a three-electrode electrochemical system and directly deposited on a stainless steel wire by applying a constant potential (1.2 V for 1000 s) [1]. Effect of different parameters including salt concentration, extraction time, temperature, PH, stirring rate, desorption time and desorption temperature were investigated and optimized. The calibration curve was plotted at the optimized conditions, indicating a dynamic range of 0.2-200 μg L^{-1} with determination coefficient (R^2) of 0.9932. Using the standard definition of S/N = 3, the detection limit of 80 ng mL^{-1} was determined for this compound. The relative standard deviation lower than 9% was achieved for the standard solutions of 5, 10, and 50 μg L^{-1}. The obtained relative recoveries using the proposed method in analysis of spiked samples were in the range of 71% to 98%. The quantitative results obtained in this work are promising for development of this method as a powerful analytical tool for detection of diazinon in various real samples.

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Polysiloxane nanofiber as a novel solid phase microextraction coating for determination of chlorobenzenes by gas chromatography and electron capture detection in sludge, sediment and water samples

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In this study, a new solid phase microextraction (SPME) coating based on polysiloxane nanofiber was introduced for determination of chlorobenzenes in water, sludge and sediment samples. SPME is a microextraction and preconcentration technique that used for trace analysis of compounds in different samples. Although, commercial solid phase microextraction coatings such as polydimethylsiloxane(PDMS), divinylbenzene(DVB), Carboxen(CAR), polyacrylate(PA) and Carbowax (CW) widely used for determination of different compounds, they have some disadvantages like fragility, memory effect and high cost [1,2]. Nowadays, nanostructure materials were successfully used as solid phase microextraction coatings because of their unique chemical and physical properties like thermal stability, high surface area and thus good extraction efficiency. In this work, polysiloxane nanofibers were synthesized with liquid phase method and used for determination of trace amounts of chlorobenzenes(1,4-dichlorobenzene, 1,2-dichlorobenzene, 1,2,4-trichlorobenzene and 1,2,3,4-tetrachlorobenzene) in sediment, sludge and water samples. The influence of important factors such as stirring rate, salt concentration, equilibrium time, extraction time, extraction temperature, desorption time and desorption temperature were studied and optimized. The intra- and inter-day relative standard deviations were below 5% for all analysis under optimized conditions. The method shows good linearity in the range of 0.0005-24 μg/L and detection limits in the range of 0.15-80 ng/L for these chlorobenzene compounds. This novel coating shows better extraction efficiency compared to commercial fiber and sol-gel coating.

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Potentiality of multivariate chemometric methods in characterization of potential antioxidant compounds in the essential oil of Thyme

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Abstract

Thyme species are used in traditional medicine throughout the world. This plant is known as an antiseptic, antispasmodic and antitussive. Also essential oils of Thyme have many properties that one of the interesting is antioxidant activity. It is aspired to indentify from chromatogram of gas chromatography (GC) and antioxidant activity of essential oils, the peak potentially responsible for antioxidant activity. Therefore the chemical components of hydrodistilated essential oil of thyme species from different zone were analyzed by gas chromatography (GC). Then antioxidant activity of essential oils was measured by DPPH radical scavenging test. Combining chromatographic data and antioxidant activity with chemometric methods, allows indicating the peaks potentially responsible for given activities. Several linear multivariate calibration techniques with different preprocessing were applied to chromatogram of essential oil of thymes to indicate the peaks responsible for the antioxidant activity. These techniques were applied on data both before and after alignment of chromatograms with correlation optimized warping (COW). In this study, orthogonal projection to latent structures (OPLS) was found to be the better performing technique to indicate the potential antioxidant active compounds in the thyme oil due to its simplicity and repeatability, and to remove the orthogonal information in the original data set. OPLS resulted in a decreased model complexity contributing to an improved interpretability of the regression coefficients. Finally we used GC-MS to indicate the structure of chemical compounds that are responsible for antioxidant activity. Results show that thymol, para cymene, γ terpinene and carvacrol are the most effective compounds in antioxidant properties of Thyme essential oil.

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Potentiality of multivariate chemometric methods in characterization of potential antioxidant compounds in the essential oil of Thyme

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Abstract

Thyme species are used in traditional medicine throughout the world. This plant is known as an antiseptic, antispasmodic and antitussive. Also essential oils of Thyme have many properties that one of the interesting is antioxidant activity. It is aspired to indentify from chromatogram of gas chromatography (GC) and antioxidant activity of essential oils, the peak potentially responsible for antioxidant activity. Therefore the chemical components of hydrodistilated essential oil of thyme species from different zone were analyzed by gas chromatography (GC). Then antioxidant activity of essential oils was measured by DPPH radical scavenging test. Combining chromatographic data and antioxidant activity with chemometric methods, allows indicating the peaks potentially responsible for given activities. Several linear multivariate calibration techniques with different preprocessing were applied to chromatogram of essential oil of thymes to indicate the peaks responsible for the antioxidant activity. These techniques were applied on data both before and after alignment of chromatograms with correlation optimized warping (COW). In this study, orthogonal projection to latent structures (OPLS) was found to be the better performing technique to indicate the potential antioxidant active compounds in the thyme oil due to its simplicity and repeatability, and to remove the orthogonal information in the original data set. OPLS resulted in a decreased model complexity contributing to an improved interpretability of the regression coefficients. Finally we used GC-MS to indicate the structure of chemical compounds that are responsible for antioxidant activity. Results show that thymol, para cymene, γ terpinene and carvacrol are the most effective compounds in antioxidant properties of Thyme essential oil.

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Potentiometric Detection of Mercury Ion(II) Using Carbon Paste Electrode as a Selective Sensor Based on Multi-walled carbon nanotubes (MWCNTs)

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A new modified carbon paste electrode (CPE) in potentiometric mode as a selective and sensitive tool for determining of mercury in real samples is introduced. To create selectivity in CPE, a new ligand, 1, 4 bis (4pyridyl)-2, 3-diaza-1, 3 butadiene (4-bpdb) as an excellent carrier is applied [1-3]. To improve the sensor response, the electrodes are modified with multi walled carbon nano tubes (MWCNTs) because of their high conductivity and low ohmic resistance properties . The construction of this electrode caused to significantly enhanced selectivity toward mercury over a wide concentration range of 1.0×10^{-6} to 1.0×10^{-2} M. The lower detection limit of the electrode is equal to 10^{-6} M and there is a Nernstian slope of 33.3 mVdecade−1 for mercury activity. The electrode has a short response time (10s) with long term stability. Finally, the proposed electrode is successfully used as an indicator for potentiometric determination of mercury ions in real samples.

Keywords: Potentiometry, mercury, carbon paste electrode, multi-walled carbon nanotubes.

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Potentiometric detection of trace Cr(III) ions based on carbon paste electrode modified with functionalized nanoporous silica gel

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The determination of trivalent chromium is important in a variety of fields such as food [1], clinical and biological sciences [2], and also in environmental and industrial applications [3,4]. Here, a chemically modified carbon paste electrode based on newly designed functionalized nanoporous silica gel (FNSG) has been developed for the determination of Cr^{3+} in real samples.
The performance of this electrode was investigated using potentiometric measurements. The developed sensor exhibits a good linear response with a slope of 20.69(±0.3) mV per decade over the concentration range of 1.0×10^{-6}–1.2×10^{-1} mol L^{-1}. It has a very low limit of detection, 8.0×10^{-7} mol L^{-1}, with response time of 10 s. The proposed electrode could be used in pH range of 4.0–6.0. Moreover, the selectivity coefficients measured by the match potential method, were investigated. The electrode presented very good selectivities with respect to alkali, alkaline earth and some transition metal ions and good long-term stability (more than 6 months). Also the electrode was applied for direct determination of copper in different water samples.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

Potentiometric determination of sulfate anion by membrane electrode containing modified nano-clinoptilolite particles with tetradecylthrimethyl ammonium bromide

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Abstract

Clinoptilolite nanoparticles were prepared using ball-milling the natural Iranian clinoptilolite [1] and its cation exchange property was changed to anion exchanger by modification of the nanopowder with tetradecylthrimethyl ammonium bromide (SMZ-NCP) [2]. A selective membrane electrode based on the modification of PVC with the modified powder as an ionophore exhibited good behavior with a Nernstian response of 29.9 ± 0.9 mV per decade of activity for sulfate ions and a working concentration range of 1.0×10^{-7}-1.0×10^{-1} mol L^{-1} sulfate with a detection limit of 7.0×10^{-8} mol L^{-1} sulfate. The sensor showed a fast response time of 7–10 s and can be used for at least 8 weeks without any divergence in potential. The electrode was used in the pH range of 3–10. The sensor was highly selective for sulfate over a wide variety of other anions and exhibited a rapid response time of <10 s after immersing the electrode in sulfate solution with good reproducibility. A successful application of sulfate determination in real sample of a pharmaceutical zinc sulfate capsule, using direct potentiometry was presented. The practical utility of the sensor was demonstrated by using it successfully as an indicator electrode in the potentiometric titration of SO_4^{2-} with Ba^{2+}. The obtained results were validated by statistical “ t ” and “ g ” tests [3].

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Potentiometric Study of DAP/pydc Proton Transfer and Complexation with Cu^{2+} and Zn^{2+} Using Hyperquad Program and Comprising with Solid State Results

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The protonation constant of pyridine 2,6-dicarboxylic acid(pydc) and complexation of this acid with Cu^{2+} and Zn^{2+}ion was in our focus previously [1]. In this work we pay attention to the system containing pydc and phenazine-2,3-diamine (DAP).

In present work the protonation constants of DAP and pydc, the equilibrium constants for DAP-pydc proton transfer system and the stoichiometry and stability constants of binary complexes including each of ligands (DAP or pydc) in presence of Cu^{2+} and Zn^{2+} ion, ternary complexes including both of ligands (DAP-pydc) in presence of metal ion were calculated in aqueous solutions.

The potentiometric pH titration curves of DAP(Q), pydc(L) and their corresponding 1:1 mixture in the presence of Cu^{2+} and Zn^{2+} ion were fitted by Hyperquad2008 program which is as a new version of the older one[2]. The corresponding distribution diagrams were depicted using Hyss2009 as a new version of Hyss program [3]. The results show that, the most likely species for DAP-pydc with Cu^{2+} and Zn^{2+} ternary system are: CuLQ, CuL_2QH, ZnLQ, and ZnL_2QH_2. It is interesting to note that the stoichiometries of the some of the most abundant ternary complexes, such as CuL_2QH, and ZnL_2QH_2 existing in aqueous solution, are very similar to that reported for the corresponding isolated complexes in the solid state. Structural effects of the ligands on the stability of binary and ternary complexes were discussed.

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Precocentration and determination of Zn using [4-(2-pyridylazo)resorcinol] on active carbon by solid phase extraction- Flame atomic absorption spectrometry.

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In this project a preconcentration method was developed for determination of trace amount of Zn(II) using a 4-2-pyridylazo resorcinol (PAR) by solid phase extraction.

The method is based on the retention of their PAR complexes on active carbon adsorbent . The adsorbed were eluted with nitric acid and determined by flame atomic absorption spectrometry.

Several parameters such as pH, acid concentration and the effect of diverse ions were investigated . preconcentration factor of the method was 82.

The calibration curve and detection limit were 1-25 and 0.12 μg L^{-1} respectively.

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Preconcentration of U(VI) based on nanocomposite solid phase microextraction and determination by spectrophotometry UV-Vis in environmental water sample.

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Uranium is a toxic metal that can be absorbed by humans and other organism and its occurrence in the environment is very harmful for all living organism. Uranyl (UO_2^{2+}) ions, such as uranium trioxide or uranyl nitrate and other hexavalent uranium compounds, have been shown to cause birth defects and immune system damage in laboratory animals. Duo to the higly toxic nature of this element the determination of such low level in an aquatic environmental is of great importance. There are various techniques for the determination of uranyl [1,2]. These techniques are very expensive and suffer from many complicated processing separation and are frequently recognized as time consuming with low reproducibility. Since the amount of uranium in the samples is very low, it is necessary to preconcentrate the low contents before its determination. The aim of this study was to develop a sensitive and reliable method for extraction and determination of uranyl from the aqueous samples via complexation with 4-(2-pyridylazo) resorsinol and which upon solid phase microextraction (SPME). For the synthesis of nano-composites , chitosan was coated on Fe_3O_4 nanoparticles. The excellent adsorption behavior of chitosan for heavy metal removal is attributed to high hydrophilicity due to the large number of hydroxyl groups, primary amino groups with high activity, and the flexible structure of polymeric chain to make a suitable configuration for adsorption of metal ions [3]. After microextraction complex was measured by spectrophotometry using a micro cell. The characterization of the synthesized Fe_3O_4-chitosan was performed by scanning electron microscope (SEM), transmission electron microscope (TEM) and X-ray diffraction methods (XRD). In order to achieve higher sensitivity, all the parameters influencing the complex formation, microextraction, and determination were studied and optimized. At the optimum conditions, the LOD and RSD for determination were 0.001 μg mL^{-1} and 2.2 % respectively. The calibration curve was linear in the range of 0.002 - 0.01 μg mL^{-1} and the. The feasibility of the proposed method was successfully confirmed by the extraction and determination of uranyl in water samples such as sea water, river water and suitable results were obtained.

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Preconcentration and determination of patulin from apple juice using dispersive liquid - liquid microextraction coupled with high - performance liquid chromatography

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ABSTRACT

Patulin is a mycotoxin naturally found in fruits, including apples. Its occurrence as a natural contaminant of fruit juices is indicative of fruit quality in production [1]. Due to its mutagenic and teratogenic nature and possible health risks to consumers, many countries have regulations to reduce level of patulin in apple products to as low as practically possible. The European Union has set the maximum content of patulin in 50 μg L^{-1} for fruit juices [2]. In this paper, dispersive liquid–liquid microextraction (DLLME) based on supramolecular solvent, has been proposed for the extraction and preconcentration of patulin in apple juices, followed by its determination by High - performance liquid chromatography (HPLC) with UV detection. In DLLME, an appropriate mixture of the extraction solvent and the disperser solvent is rapidly injected into the aqueous solution of sample and a cloudy solution is then formed as a result of the formation of fine droplets of the extraction solvent which disperse in the sample solution [3]. After centrifuging a small drop of extraction solvent is collected on the surface of solution. Tetrahydrofuran and decanoic acid used as dispersiver and extraction solvent, respectively. Different DLLME parameters influencing the extraction efficiency such as percent of THF, percent of decanoic acid, pH and extraction time were investigate. Under the optimal conditions the relative standard deviation range(RSD) was obtained from 1-3.3% (n = 5), correlation coefficient was 0.9995 and detection limit was 0.1 μg/L. Patulin extraction recovery values were in the range of 98-101%. Finally the proposed method was successfully applied for the analysis of patulin in apple juice samples and obtained satisfactory results.

Keyword: Dispersive liquid- liquid microextraction, Patulin, Apple juice, High-performance liquid chromatography

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Preconcentration and determination of β-carotene nanoparticles in fruit and juice samples based on micellar mediated system

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Abstract

β-carotene is a precursor of vitamin A and plays an important role in the metabolism and human health. However, β-carotene can be destroyed in food processing method at high temperature and it does not dissolve in water well. However, the nanomaterial form of β-carotene could protect it from heat and make β-carotene be easily dissolved in water. Over the last few years β-carotene nano dispersions have found a wide range of applications in food, pharmaceutical and chemical industries as well as agriculture and environmental engineering [1,2]. Cloud point extraction (CPE) has become one of the most outstanding extraction techniques for determination of analytes. CPE is based on the separation of a homogeneous micellar aqueous solution into two isotropic liquid phases [3]. This paper presents a simple, green and low cost analytical method for preconcentration and determination of β-carotene nanoparticles content in some fruit and juice samples. The proposed method for preconcentration and determination of β-carotene nanoparticles is based on the enrichment of β-carotene nanoparticles in non-ionic surfactant phase with cloud point extraction followed by UV-vis spectrophotometric determination at λ=468 nm. The influence of chemical variables such as pH of the sample solution, type and volume of the buffer, electrolyte type and concentration, surfactant concentration, temperature and incubation time was studied. Under the optimum conditions the method yielded a linear calibration curve in the range of 0.05-5 ng mL^{-1} for β-carotene nanoparticles (R=0.9992). For robustness, samples considered for analysis with this analytical method were: carrot, potato, watermelon, tomato fruits and cherry and orange juices. The recoveries of the analyte in spiked samples were in the range of 96-105%, with relative standard deviation (RSD) of 3.2% and 5.6%.

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Preconcentration and speciation of trace amounts of inorganic and organic selenium in mushroom samples with cloud point extraction and spectrophotometry

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Selenium is an essential, trace element in environmental and biological systems. It plays an antioxidant and chemo-preventive role in cancer and other illnesses like heart disease and arthritis [1]. This element would exist in various samples such as vegetables, foods, biological matrixes in different chemical forms: inorganic species (selenite , SeO_3^{2-} ; selenate , SeO_4^{2-}) or in the forms of organic species (selenoamino acids ; selenomethionine and very complex selenoproteins). Inorganic forms are more toxic than organic forms and the toxicity of selenite is more than selenate for humans and most of other mammals [2].

A new micelle mediated speciation and preconcentration method was developed for trace amounts of inorganic and organic selenium prior to spectrophotometric determination .The method is based on cloud point extraction (CPE) of Se(IV) ions with polyethylene glycol tert-octylphenyl ether (Triton x-114) in the presence of 2,3-diaminonaphthalene (DAN) as a chelating agent .Se(IV) ions react with DAN in a surfactant solution yielding a hydrophobic complex at pH 3.The phase separation was accelerated by centrifugation for 20 min at 3500 rpm .The calibration graph obtained from Se(IV)-DAN complexes was linear in the concentration range of 0.005-0.4 $\mu g\ L^{-1}$ with detection limit 0.009 $\mu g\ L^{-1}$ .The relative standard deviation (RSD) was 1.6% in determination of 0.08 $\mu g\ L^{-1}$ Se(IV) .The proposed method was successfully applied to the speciation and determination of selenium species (Se(IV),Selenomethionine) in walnut samples with satisfactory results .

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Pre-concentration and spectrophotometric determination of trace amounts of ciprofloxacin by flotation using congo red

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Ciprofloxacin is one of the most widely used fluoroquinolones for the treatment of urinary tract infections, respiratory tract infections and chronic bacterial prostatitis [1]. Many spectrophotometric methods have been reported for the determination of CPF [2]. In this study a simple floatation method using congo red as a floating agent has been developed for pre-concentration and determination of trace amounts of ciprofloxacin using spectrophotometry. In a mild acidic medium (pH 4), ciprofloxacin and congo red produce a floated layer in the presence of small amount of chloroform. The layer was floated between aqueous and organic phases. Small amounts of squalene was added to adsorbed the floated layer and facilate its separation by centrifugation. Formed layer was separated using a syringe and dissolved in small volume of NaOH solution. The pre-concentration conditions, including the effect of pH, buffer and its volume, amount of adsorbent, ionic strength, composition and volume of dissoving solvent, accepted tolerable volume, time of adsorption and desorption of ciprofloxacin and influences of interferences on the pre-concentration process were investigated. The calibration curve was linear in the range of 0.5-8.00 µg mL^{-1} with a correlation coefficient of 0.995. The limit of detection (LOD) and relative standard deviation (RSD, for 5.0 µg mL^{-1}) were 0.15 µg mL^{-1} and 4.5 %, respectively. The proposed method was successfully applied to the determination of the amounts of ciprofloxacin in 500 mg tablet samples.

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Preconcentration and trace determination of cadmium in spinach and various water samples by temperature-controlled ionic liquid dispersive liquid phase microextraction

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The pollution of natural waters by heavy metals such as cadmium is a great concern nowadays due to their potentially toxic effects on living organisms. Urbanization, industrial development, and heavy traffic lead to contamination of waters by heavy metals [1]. Cadmium is a very toxic metal which is not regarded as essential to human life and is known to damage organs including the kidneys, liver and lungs. According to World Health Organization (WHO) the upper permissible level of cadmium in drinking water is 3.0 µg L^{-1} [2]. Consequently, the determination of this element, at trace level, in food and water samples is of great interest. More recently, temperature-controlled ionic liquid dispersive liquid phase microextraction (TC-IL-DLPME) was developed by Zhou et al. [3]. The mechanism of this method is similar to that of DLLME, but the dispersion of extraction solvent into the water sample is realized by high temperature rather than the use of expensive and toxic organic disperser solvent which decreases the extraction efficiency to some extent. It is based on the temperature change that permit ILs to completely disperse in the aqueous phase and increase the mass transfer of the target analytes into the IL phase. Due to the environment friendly behavior and good extraction capability of ILs, and the avoidance of disperser solvent, TC-IL-LPME has many benefits such as simplicity, environmental friendliness and high extraction efficiency. In this study, a sensitive and selective method for the preconcentration and separation of cadmium ions in real samples is described. The developed method is based on TC-IL-DLPME of cadmium using the 1-hexyl-3-methylimidazolium bis (trifluoromethylsulfonyl) imide (ionic liquid) as an extractant followed by flame atomic absorption spectrometry determination. The extraction of cadmium ions from the aqueous solution into the fine droplets of IL was performed with dithizone as the chelating agent. Some predominant factors affecting the preconcentration of cadmium ions were evaluated and optimized. Under the optimum conditions, the calibration graph was linear over the concentration range from 0.6-20.0 µg L^{-1} for cadmium. The limit of detection (LOD) of the developed method was 0.2 µg L^{-1} of cadmium. The enrichment factor was found to be 25. The relative standard deviation for ten replicate determinations of 10 µg L^{-1} of cadmium was 1.5 %. The developed method was successfully applied to the determination of cadmium in spinach and water samples.

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Preconcentration of bromate ion from aquatic samples using CPC-MAC-NC followed by spectrophotometric determination

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Bromate ion is one of important oxidants which can oxidize many inorganic and organic compounds [1]. This ion is a powerful oxidant and has been shown to cause kidney and possibly other tumors in laboratory animals [2]. United States Environmental Protection Agency (US-EPA) has classified bromate as a possibly carcinogenic substance to humans by the oral route of exposure. In children, serious poisonings have been reported following the ingestion of 60–120 mL of 2% bromate. Other toxic effects of bromate include nausea, vomiting, abdominal pain, anuria and diarrhoea, varying degrees of central nervous system depression, haemolytic anemia and pulmonary edema. In view of the above harmful effects of bromate on human health, US-EPA and American Water Works Association (AWWA) have set bromate maximum contaminant level (MCL) in drinking water as 10 μgL^{-1} [3].

In this study, first, Magnetic iron oxide-activated carbon nanocomposite (MNC-AC) was prepared and was characterized by transmission electron microscopy (TEM), thermogravimetric analysis (TGA) and vibrating sample magnetometer (VSM). Then, the preconcentration of bromate was accomplished by utilization of micelle system. The effect of various parameters on the recovery of bromate from CPC-modified MAC-NC surface such as eluent (type, pH, volume and concentration), pH of solution, amount of adsorbent, contact time and breakthrough volume, were studied and optimized. Under optimized condition, the calibration curve was linear in the range of 25-500 μgL^{-1} with $R^2 = 0.997$ .the limit of detection (LOD) for bromate determination was 4.8 μgL^{-1}. Also, relative standard deviation (R.S.D) was lower than 4.4% and an enrichment factor was obtained as large as 6. In this study, all of determinations were carried out by UV-Visible in $\lambda_{max} = 350$ nm.

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Preconcentration of gadolinium by nano-iron oxide-encapsulated chitosan and spectrophotometry determination in real sample

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Abstract

Because of the increase in the industrial use of gadolinium compounds as well as their enhanced discharge , determination of gadolinium has recently been of an increasing concern. Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS), mass spectrometry, neutron activation analysis, X-ray fluorescence spectrometry, Isotope dilution and Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) are the available methods used for low-level determination of rare earths [1-3]. These methods are either time consuming, involve multiple sample manipulations, or are too expensive for most analytical laboratories. The aim of this study was to develop a sensitive and reliable method for extraction and determination of Gadolinium from the aqueous samples via complexation with 4-(2-pyridylazo) resorsinol(PAR) and which upon solid phase microextraction(SPME). The present study investigates the utility of composite beads of nano- particles of iron oxide and chitosan for absorb Gd (III) ions from aqueous solution . The analyte ions quantitatively were adsorbed on magnetic nanocomposite in the acidic pH. Gadolinium (III) loaded on nanoparticles could be separated easily from the aqueous solution by simply applying an external magnetic field. The Gd ions can be eluted from the composite magnetic particles using 0.01 mol L^{-1} HNO_3 as a desorption reagent, finally measured at 500 nm by spectrophotometry using a micro cell. In order to achieve higher sensitivity, all the parameters influencing the complex formation, microextraction, and determination were studied and optimized. At the optimum conditions, the LOD and RSD for determination were 0.0008 µg mL^{-1} and 2.8 % respectively. The calibration curve was linear in the range of 0.001to 0.01 µg mL^{-1}. This method was applied for determination of gadolinium in real sample.

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Preconcentration of nickel in waste water by ultrasound-assisted ionic liquid cold-induced aggregation dispersive liquid–liquid microextraction and determination by flame atomic absorption spectrometry

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Dispersive liquid–liquid microextraction (DLLME) technique based on ionic liquids (ILs) was developed and has become increasingly popular as a successful model of LPME. Another useful method which was termed cold-induced aggregation microextraction (CIA-ME) based on the use of ILs for homogeneous liquid–liquid microextraction (HLLME), was developed by Baghdadi and Shemirani in 2008, and modified in 2010 [1,2]. The mechanism of this method is very similar to that of DLLME, but the dispersion is accomplished by temperature-dependent dissolution instead of injection. In this method, a suitable temperature is used to fully dissolve the IL in the sample solution without the use of disperser solvent [3]. In present work we consider cold-induced aggregation microextraction combined with flame atomic absorption spectrometry for the preconcentration and determination of trace levels of nickel in waste water samples. 2-hydroxy-1-naphthaldehyde oxime (HNAO), and N-octylpyrdinuim hexaflourophosphate [OPY][PF$_6$] were used as chelating agent and extraction phase respectively. The IL mixed completely with the sample solution at 70 ˚C, and the solution was cooled with ice-water for a selected period of time. The IL and aqueous phases were then separated out by centrifugation. Experimental conditions were optimized by experimental design method in order to find the significant factors and their interactions. Linearity range with coefficients of correlation 0.9994 was observed in the concentration range of 2.5–2000 ng mL^{-1}. The limit of detection (LOD) was 0.4 ng mL^{-1}. while the relative standard deviation (RSD) was 3.6% (at 0.5 mg L^{-1} Ni level and n = 7).

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Preconcentration of trace amounts of vanadium using dispersive liquid-liquid microextraction followed by spectrophotometric determination

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Vanadium is a rare and soft element used mainly for manufacturing certain alloys such as titanium and aluminium. Vanadium alloys are also used in nuclear reactors because of its low neutron-adsorption abilities. Vanadium is also used as a steel additive .Vanadium oxide (V_2O_5) is employed as a catalyst in manufacturing of sulfuric acid and maleic anhydride. In biology, vanadium is an essential component of some enzymes performance.[1]

Several methods have been employed for preconcentration and determination of vanadium in various samples mainly using spectrophotometery and atomic absorption spectrometery. [2,3] in this work a dispersive liquid-liquid microextraction hase been developed for pre-concentration and determination of trace amount of vanadium by UV spectrophotometry. vanadium in nearly neutral pHs with PAR in the presence of an appropriate surfactant forms an ion pair which is the basis of pre-concentration of vanadium. Different parameters affecting the extraction process such as pH, types and volumes of dispersing and extracting solvents, time and speed of centrifuge, salt effects, and the effects of interfering ions were thoroughly investigated .The LOD for the proposed method was 1.5 μgL^{-1} . The RSD for 5 replicate analysis of 10 μgL^{-1} vanadium was 4.5% . The enrichment factor based on the ratio of slopes after and before the extraction was calculated to be 90 .This method was successfully applied for determination of vanadium in real samples and reference material.

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Preconcentration trace amounts of cobalt, copper and manganese ions using in-situ surfactant-based solid phase extraction prior to its FAAS determination

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Potential sources of heavy metal ions pollution are various effluents emanating out of industries, domestic activities and erosion of natural deposits [1]. However, if these metal ions are continuously released into the biosphere, they may be changed to one of the major sources of pollution.

Flame atomic absorption spectrometry (FAAS) is now probably the most used technique for determination of heavy metals in different samples. In many cases the determination of trace heavy metals in environmental samples by FAAS is notably difficult due to both the low levels of these metals in the samples and the high complexity of the sample matrices. It is usually necessary to carry out a preconcentration/or separation step prior to the analysis. Recently Shemirani and Yousefi [2] demonstrated a surfactant-based SPE mode termed in-situ surfactant-based solid phase extraction (ISS-SPE). In the present work, we report an ISS-SPE procedure for preconcentration of cobalt, copper and manganese from water samples using 5-(4-chloro-benzylidene) 1,3-dimethyl-pyrimidine-2,4,6 trione as chelating agent. In this method a cationic surfactant with a proper alkyl group chain is dissolved in the aqueous sample and then a proper ion-pairing agent is added. Due to interaction between surfactant and ion-pairing agent, very fine solid particles are formed. After centrifugation, the solid particles are sedimented at the bottom of the centrifuge tube and the aqueous phase is removed by simply decantation of the centrifuge tube. The sedimented sorbent was dissolved in 3.0 mL of 1.0 mol L^{-1} HNO_3 in ethanol. The final solution was aspirated directly into the flame of AAS. In order to obtain the best conditions, several parameters affecting the performance of the ISS-SPE such as volume of cationic surfactant, ion-pairing agent and chelating agent, pH and potentially interfering ions were studied and optimized.

Under optimized conditions, calibration curves were constructed for determination of the analyte ions. Linearity for cobalt, copper and manganese ions in the original solution (35 mL) were maintained between 0.01 and 1.5, 0.007 and 2.0 and 0.005 and 1.0 µg mL^{-1}, respectively. The detection limits of the method based on $3S_b/m$ ($n = 7$) for cobalt, copper and manganese ions were 2.3, 1.9 and 1.3 ng mL^{-1} in the original solution, respectively. Eight replicate determinations of a mixture of 0.5 µg mL^{-1} cobalt, copper and manganese ions in the original solution gave relative standard deviations of 2.1%, 1.9% and 1.8%, respectively. Finally, the proposed method has been successfully applied for the determination of the analyte ions in the real water samples.

Key words: In-situ surfactant-based solid phase extraction; Heavy metal determination

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Preparation and characterization of chitosan- mesoporous silica nanocomposite as a new material for adsorption and efficient removal of sunset yellow from food samples

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SBA-15 mesoporous silica was synthesized under a moderately condition by sol gel methodes. These mesoporous materials were characterized with X-ray diffraction pattern, scanning electron microscopy, transmission electron microscopy, and nitrogen adsorption-desorption measurements. The three-dimensional microporosity of the prepared mesoporous silica having ordered hexagonal mesoporous structure was evidenced by the well-defined XRD patterns combined with TEM photographs. The inverse phase technique [1-2] was used for immobilization of chitosan on the SBA-15 mesoporous silica and preparation of nanocomposite. The adsorption of sunset yellow dye onto SBA-15/chitosan nanocomposite has been investigated. The composite was found to have excellent dye adsorption capacity than the SBA-15 and chitosan. The adsorption experiments were carried out in batch mode to optimize various parameters such as, contact time, pH, initial dye concentration, and temperature that influence the adsorption. The points of zero charge (PZC) for the nanocomposite was obtained and was about 6. The maximum adsorption capacity of adsorbent at pH=5 and contact time of 60 minutes was 113.64 mg.g^{-1}. Langmuir, Freundlich, temkin and Dubinin-Radushkevich adsorption isotherm models were applied to describe isotherm constants. Equilibrium data agreed well with the Freundlich isotherm model. Thermodynamic studies revealed that the nature of sorption is spontaneous and endothermic. The obtained results showed that the this nanocomposite adsorbent can be reused for least 5 adsorption–elution cycles without any decreased in the efficiency. The removal efficiency of this nanocomposite toward sunset yellow dye is above 95% in distilled water, tap water, river water, wastewater and also food samples .

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Preparation and Characterization of a Novel Nanocomposite Hydrogel Film Based on Chitosan, Cellulose Nanofibers and Ag NPs

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Hydrogels are three-dimensional networks of hydrophilic polymers held together by crosslinks of covalent bonds or ionic bonds and secondary forces in the form of hydrogen bonds or hydrophobic interactions that are used for a variety of applications in industry, analysis process and medicine [1, 2]. One of the most common applications of hydrogel in medicine is wound dressings. In this work a novel pH sensitive chitosan-based nanocomposite hydrogel was reported for using in the healing burn wounds. Graft copolymerization of D, L-lactic acid (LA) with chitosan (CS) was attempted in the presence of different percentage of cellulose nanofibers. The structures of prepared film were investigated by Scanning Electron Microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy. Gel Fraction and kinetic of swelling and dehydration of graft copolymers were studied in varied conditions of pH and temperature. The experimental results showed that with increasing the percentages of cellulose nanofibers *vs*. chitosan for each type of the samples, the gel fraction values are decreased which demonstrate that the chitosan biopolymer is responsible of the most cross-linking bonds in polymeric networks and the cellulose nanofibers improve the mechanical stability of the prepared nanocomposite hydrogel. Meanwhile, these initial experimental studies indicates that the excellent properties of chitosan like as biocompatibility, low toxicity and biodegradability together with inherent mechanical and strength properties of cellulose nanofibers makes this prepared chitosan based nanocomposite hydrogel film as an desired candidate for using in biomedical applications. In next step the effect of well characterized and widely applied nanoparticles including silver nanoparticles [3] were loaded in the structure of films by precipitation of Ag NPs from its colloidal solution with chitosan. The test of antimicrobial activities revealed salient development of antibacterial activity and wound healing of proposed nanocomposite films which may have potential application as antimicrobial materials such as wound dressings.

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Preparation and characterization of graphene ceramic composite electrode (GCCE) for electrocatalytic and storage applications

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Abstract

In this study, a new graphene ceramic composite electrode (GCCE) is prepared based on the hydrothermally reduced graphene oxide grafted in acid catalyzed sol-gel derived silica [1, 2]. The fabricated GCCE was characterized by field emission scanning electron microscopy, Fourier transform infrared spectroscopy, x-ray diffraction, thermogravimetric analysis, cyclic voltammetry, and galvanostatic charge/discharge techniques. The GCCE shows good mechanical, chemical, and thermal stability and was shown to be an efficient material for electrocatalytic and storage applications. In order to demonstrate the electrocatalytic performance of the fabricated electrode, the electrocatalytic oxidation of ascorbic acid was studied as a model analyte. The GCCE responds effectively to the electro-oxidation of ascorbic acid in the concentration range 3 – 84 µM with a sensitivity of 6.06 µA μM^{-1} cm^{-2} and detection limit 0.82 µM (S/N=3). Due to the unique configuration, large specific surface area, and high electrical conductivity of the graphene nanosheets, the resultant GCCE showed high specific capacitance of 428 F g^{-1} at current density of 1 A g^{-1} and good cycling stability in Na_2SO_4 solution using a three electrode system [3, 4]. Having good mechanical and chemical stability, ease of fabrication, reproducible preparation process, and appropriate electrocatalytic and storage properties, GCCE can be used as a universal renewable electrode in analytical electrochemistry and storage devices.

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Preparation and Characterization of graphene- nile blue nanocomposite using electrochemical method

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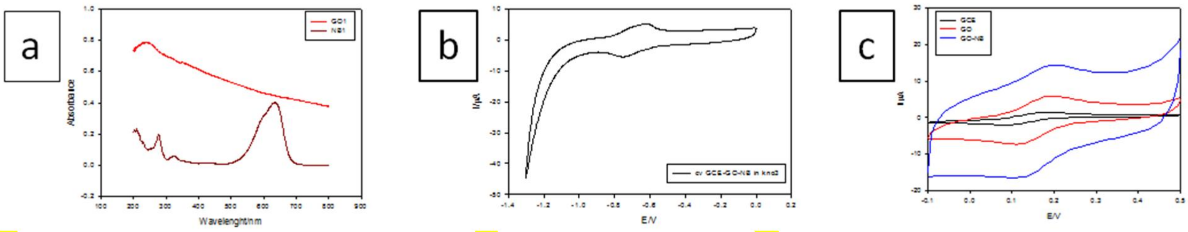
Abstract:

Graphene nanosheets (GNs), a novel generation of carbon-based nanosheets with a sp2 hybrid carbon network, has inspired much theoretical and experimental application compared to other carbon materials due to its exceptional electronic, optical, high charge mobility and large surface area properties [1]. These superb properties of graphene offer many potential applications ranging from, nanoelectronics, composite materials, and sensors to electrochemical electrodes [2]. Noticeable progress has been made in the utilization of GNs and GNs-based composites in recent years.

There are a number of reasons for developing graphene-based composites. The first and most obvious reason is that addition of filler with such impressive mechanical properties would be expected to lead to a significant improvement in the mechanical properties of the host polymer matrix. It is also found, however, that changes to the mechanical properties are also accompanied by modification of functional properties such as electrical conductivity, thermal conductivity and barrier behaviour.By virtue of the outstanding physical and chemical properties, graphene based nanocomposites are increasingly attractive and have been designed towards applications in electrochemical biosensors [3].

In this study Nile blue (NB)/GNs nanocomposite was synthesized via a facile two-step electroreduction process and was characterized with various techniques. The electrocatalytic activity of the as-prepared NB/GNs nanocomposite was studied.

Grapheme oxide (GO) is mixed with a certain proportion of NB then is put in an ultrasonic bath for 2h. Then, The GO–NB nanocomposite was first modified on the surface of glassy carbon electrode (GCE), and then, drying at room temperature. GNS/NB nanocomposite is produced by applying a certain potential then its electrochemical behavior is investigated. Scanning electron microscope (SEM), ultraviolet–visible (UV–vis) spectrometry and cyclic voltammetry (CV) were employed to characterize the biofunctionalized NB–GNs nanocomposite.



a) UV–Vis absorption spectra of GO and NB, b) CVs obtained at KNO_3, c) CVs obtained at $FeCN_6$

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Preparation and evaluation of polymethacrylate capillary columns for liquid chromatography

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Preparation methods for porous materials as separation bed in chromatography has achieved a significant scientific and technical leap in the past few decades [1,2]. In this work, two kinds of organic polymer monolithic capillary columns were prepared in fused-silica capillaries by radical co-polymerization of ethylene dimethacrylate and butyl methacrylate or methylmethacrylat monomers with azobis isobutyronitrile as initiator of the polymerization reaction in the presence of various amounts of porogenic solvent mixtures and different concentration ratios of monomers and 1-propanol, 1,4-butanediol, and water.

This monolithic column was successfully characterized using Fourier transform Infrared spectroscopy (FT-IR), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) results. Finally capillary column was assembled on ultra pressure liquid chromatography (UPLC) instruments with PDA detector and 1μL loop. The chromatographic properties of these columns were tested with tryptophan, tyrosine, phenylalanine at flow rate 1 μL min^{-1} with acetonitrile and acetate buffer contains 0.01% TFA (pH= 4.6, 30:70 v/v) as the mobile phase at λ= 218 and 280 nm and several parameters related to column efficiency were calculated.

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Preparation of a polar monolithic stir bar based on ethylene glycol dimethacrylate for the sorptive extraction of benzodiazepines from aqueous sample and plasma

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Abstract

Stir bar sorptive extraction (SBSE) is a sample preparation technique that was introduced by Baltussen and coworkers in 1999 [1] and commercialized by Gerstel (Mülheim, Germany) under the name “Twister”. This technique is widely used for enrichment of compounds from various aqueous matrixes using polydimethylsiloxane Twister or home-made polar sorptive phases [2, 3]. Up to now several synthetic techniques have been developed to prepare stir bar sorptive phase. One of those approaches is using monolithic method which has become popular in recent years due to their high permeability, high absorbance capacity and simplicity in preparation.

In this study a novel monolithic polymer sorptive phase based on copolymerization of N-vinylpyrrolidone and ethylene glycol dimethacrylatepoly was prepared in the presence of dodecanol and toluene as porogen solvent. After primary experiments, 11 stir bars with different percentages of cross-linker and functional monomer were made. Various techniques such as scanning electron microscopy (SEM) and infrared (IR) spectroscopy were used to characterize the monolithic coating. Then, these stir bars was applied to extraction of several benzodiazepines such as diazepam, clonazepam nordiazepam and lorazepam, with different polarities. Liquid desorption by methanol and high performance liquid chromatography was used to final analysis using C_{18} column. The mobile phases for analysis of diazepam, clonazepam, nordizaepam and lorazepam were acetonitrile/water (65:35v/v), acetonitrile/methanol/water (60:20:20v/v/v), acetonitrile/buffer (40:60v/v, pH= 4) and methanol/water (65:35v/v), respectively. In all analysis flow rate was 1 mL min^{-1}. Retention times of these target analyts were obtained 6, 5, 11 and 7 min, respectively. Based on the area of each peak and calculated enrichment factors the best stir bar was containing 88% cross-linker and 12% functional monomer.

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Preparation of antibacterial electrospun Trimethoprim/Polyvinylpyrrolidone nanofibers as oral fast-dissolving delivery system

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Electrospinning(ES) has been shown to be an effective and simple method for the production of structured polymer fibers with a wide range of diameters from micrometers to nanometers. This technique is relatively new in the field of medical industry and ES is quite slightly known in pharmaceutical technology, where most of the relevant papers deal with sustained drug release [1]. Fast-dissolving drug delivery systems (FD-DDSs) are in demand to meet the increasing needs of developing new forms of treatment. Difficulty in swallowing medicines is often encountered by patients, which can result in ineffective treatment. Not only are FD-DDSs able to address the problem but also they have additional advantages such as enhancing drug solubility, and extending the half-life in the body drugs, inter alia [2].

Formation of non-woven tissues of the nanofibers, owing to their huge surface area, is a promising way to develop oral fast-dissolving dosage forms and the electrostatic spinning can be a capable technology for manufacturing such formulations. The wide versatility of the polymers available for this purpose supports this idea. Papers dealing with immediate release from electrospun mat applied organic solvents during their electrospinning process [3].

The aim of this work was to investigate applicability of organic solvent free electrostatic spinning method to produce orally dissolving formulation using a water insoluble model drug. ،Trimethoprim (TMP), administered against Urinary tract infection and Acute Bronchitis, was chosen. Polyvinylpyrrolidone(PVP) of high molecular weight was found to be the most appropriate for this purpose. Morphology of the prepared nanofibers loaded TMP was characterized by scanning electron microscope(SEM) as a function of viscosity and drug content, FT-IR and NMR. Diameters of the fibers were between 300 and 500 nm with narrow distribution. In vitro drug release of the webs was immediate (less than 10 s) after immersion independently of their drug content owing to the formed huge surface area, while cast films with the same compositions needed 5 minutes or more for complete dissolution. Also the quality of the cast films looks coarse and fragile. Additionally, according to the study it is specified that nanofibers hold more drugs compared to films. Looking at the bioactivity studies it was specified that the antibacterial properties of TMP are preserved in nanofibers form.

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Preparation of chitosan nanoparticles by two different methods of Ionic gelation and Electro-spray and comparison their properties

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Abstract

Chitosan is a natural biopolymer. It is suitable for many applications [1]. In this research, chitosan nanoparticles were prepared based on the ionic gelation and electro-spray methods and then compared together. In ionic gelation method [2], effects of parameters such as chitosan concentration, tripolyphosphate concentration, and reaction time on the particle size distribution were investigated. Therefore, the main object was to investigate the effect of some parameters on particle size distribution and determine the optimum conditions for preparing chitosan nanoparticles by two different methods, followed by evaluation of the physicochemical and structural properties. The optimum chitosan concentration, tripolyphosphate concentration, and reaction time were found to be 1.0 mg/ml, 1.0 mg/ml, and 60 min, respectively. In electro-spraying method, chitosan was dissolved in TFA at a concentration of 2% w/v before being sprayed onto the target by electrostatic charge and at a feeding rate of 0.33 ml/hr and an applied voltage of 13-14 kV. The distance between the spraying nozzle and the mat was 10 cm. The physicochemical properties of the chitosan nanoparticles were studied using SEM, TEM, XRD pattern, FTIR, UV–Vis, TGA, and NMR spectra. The average particle size of chitosan nanoparticles prepared by ionic gelation method is less than the ones that prepared by electro-spray method. Chitosan nanoparticle prepared by ionic gelation method has a narrow particle size distribution.

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Preparation of copper- manganese oxide nanocomposite coated with walnut skill and removal behavior of direct blue -15 dye

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Dyes are aromatic organic compounds applied in various industries such as textile, pharmaceutical, paper, tanning, cosmetics and food industries. Dyes, especially AZO dyes such as direct Blue-15 color distort ecosystem balance and pollute waters and aquatic creatures. In such industries, so dyes must be eliminated from wastewaters before being discharge into the environment.

Copper- manganese oxide nanocomposite was prepared using a co precipitation procedure. The solution of manganese and copper chloride in cethyl trimethyl ammonium bromide media was heated at 80 ° C. The solution of sodium carbonate was added to the mixed Chloride solution with stirring to adjust at pH 8.9. The precipitate was filtered, washed several time with warm distilled water and dried at 120 ° C for 16 h. Finally precipitate was calcined at 500° c for 6 h[1]. The product was mixed with walnut skill powder (ratio 1:2 respectively) in ultrasonic homogeneizer and precipitate was dried 50°C/5 h. The obtained copper- manganese oxide nanocomposite and in walnut skill were characterized by FT-IR spectroscopy, XRD and SEM.

These two compounds were used to remove direct blue-15 dye from simulated wastewater. Study of the removing was done in the maximum wavelength (595 nm) of dye by UV-Vis spectroscopy. The influence of the process variables such as pH, dye concentration, absorbent value, temperature and contact time were studied. The results of different experiments in various conditions specified Cu-Mn oxide nanocomposite with walnut skill showed high performance more than 90 % in the removal of direct blue -15 color.

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Preparation of copper- manganese oxide nanocomposite coated with walnut skill and removal behavior of direct blue -15 dye

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Dyes are aromatic organic compounds applied in various industries such as textile, pharmaceutical, paper, tanning, cosmetics and food industries. Dyes, especially AZO dyes such as direct Blue-15 color distort ecosystem balance and pollute waters and aquatic creatures. In such industries, so dyes must be eliminated from wastewaters before being discharge into the environment.

Copper- manganese oxide nanocomposite was prepared using a co precipitation procedure. The solution of manganese and copper chloride in cethyl trimethyl ammonium bromide media was heated at 80 ° C. The solution of sodium carbonate was added to the mixed Chloride solution with stirring to adjust at pH 8.9. The precipitate was filtered, washed several time with warm distilled water and dried at 120 ° C for 16 h. Finally precipitate was calcined at 500° c for 6 h[1]. The product was mixed with walnut skill powder (ratio 1:2 respectively) in ultrasonic homogeneizer and precipitate was dried 50°C/5 h. The obtained copper- manganese oxide nanocomposite and in walnut skill were characterized by FT-IR spectroscopy, XRD and SEM.

These two compounds were used to remove direct blue-15 dye from simulated wastewater. Study of the removing was done in the maximum wavelength (595 nm) of dye by UV-Vis spectroscopy. The influence of the process variables such as pH, dye concentration, absorbent value, temperature and contact time were studied. The results of different experiments in various conditions specified Cu-Mn oxide nanocomposite with walnut skill showed high performance more than 90 % in the removal of direct blue -15 color.

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Preparation of modified multi wall carbon nano tube with dithiooxamide for preconcentration and determination of trace amounts of cobalt ions in real samples

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Abstract

A fast, sensitive and simple method using multi wall carbon nano tube (MWCNT) coated with dithiooxamideas as sorbent has been ssuccesfully developed for extraction and preconcentration of trace amounts of cobalt ions prior to their determination by flame atomic absorption spectrometry (FAAS). These modified nanocomposites can be easily dispersed in aqueous samples. It was prepared by immobilization of dithiooxamide on multi wall carbon nano tube (MWCNT). Nano sorbent was synthesized by the reaction of dithiooxamide with functionalized multi walled carbon nanotube. Initially, the surface of the multi-walled carbon nanotube oxidized by a mixture of nitric and sulfuric acid and then functionalized using thionyl chloride. The ligand has been attached to the multi-walled carbon nanotubes in a somewhat shorter time and lower temperature than previous reported methods [1-2].The properties of sorbent were characterized by scanning electron microscope (SEM) and FT-IR. A microsample introduction system was employed for the nebulization micro-volume of diluted solution into FAAS. Some effective parameters on extraction and complex formation, such as pH, concentration and volume of the chelating agent, adsorbent dosage, eluent concentration and volume, extraction time, salt effect and sample volume have been optimized. The calibration graph was linear range between 30-800 ng mL^{-1} with detection limit of 11.4 ng mL^{-1}. Relative standard deviation (RSD) for Co ions were 2.2%. The proposed method has been applied to the determination of cobalt ions at ng mL^{-1} levels in real samples sush as tomatoes, potato, wheat flour, red beans, oat, tap water, river water and sea water with satisfactory results.

Keywords: Solid phase extraction, Preconcentration, Cobalt, MWCNT.

Reference

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[2] K. Yang, M. Gu, Y. Guo, X. Pan, G. Mu, Carbon. 47(2009)1723.

Preparation of modified multi wall carbon nano tube with pyrrolidine dithiocarbamate for extraction and determination of trace amounts of cadmium ions in food and natural water samples

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Abstract

A new method was developed for the preconcentration and separation of cadmium ions in food and water samples with using solid phase extraction (SPE) followed by flame atomic absorption spectrometry (FAAS). Modified multi wall carbon nano tube was used as sorbent. Nano sorbent was synthesized by the reaction of pyrrolidine dithiocarbamate with functionalized multi walled carbon nanotube. Initially, the surface of the multi-walled carbon nanotube oxidized by a mixture of nitric and sulfuric acid and then functionalized using thionyl chloride. The pyrrolidine dithiocarbamate has been attached to the multi-walled carbon nanotubes in a somewhat shorter time and lower temperature than previous reported methods [1-2].The properties of sorbent were characterized by scanning electron microscope (SEM) and FT-IR. A microsample introduction system was employed for the nebulization micro-volume of diluted solution into FAAS. Some effective parameters on extraction and complex formation, such as pH, concentration and volume of the chelating agent, adsorbent dosage, eluent concentration and volume, extraction time, salt effect and sample volume have been optimized. The calibration graph was linear range between 10-600 ng mL^{-1} with detection limit of 3.8 ng mL^{-1}. Relative standard deviation (RSD) for Cd ions were 2.7%. The proposed method has been applied to the determination of Cadmium ions at ng mL^{-1} levels in real samples such as tomatoes, potato, wheat flour, red beans, oat, tap water, river water and sea water with satisfactory results.

Keywords: Solid phase extraction, MWCNT, Cadmium, Separation.

Reference

[1] H. Tahermansouri, Y. Aryanfar, Bulletin of the Korean Chemical Society. 34(2013)149.
[2] K. Yang, M. Gu, Y. Guo, X. Pan, G. Mu, Carbon. 47(2009)1723.

Preparation of molecularly imprinted polymer for the extraction of antioxidant TBHQ from edible oil by gas chromatography

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In this work, a simple and selective method was successfully developed for the determination of TBHQ in edible oil using molecularly imprinted polymer (MIP) as sorbent. The MIP has been prepared by using TBHQ as template molecule, methacrylic acid as functional monomer, ethylene glycol dimethacrylate as crosslinking agent [1]. For achieving optimal preconcentration and reducing non-specific interactions, various parameters affecting the extraction efficiency such as extraction solvent, the amount of MIP, extraction time, washing solution and eluting solution were evaluated. Using the optimized conditions, the method presented a linear response in the concentration range of 2-10 $\mu g ml^{-1}$ for TBHQ ($r = 0.995$) with detection limits of 0.05 $\mu g ml^{-1}$, respectively. The relative standard deviations 3.4% are obtained. The proposed method was successfully applied to determine TBHQ in edible oil samples.

Keyword : TBHQ , MIP, gas chromatography, antioxidant

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Preparation of molecularly imprinted xerogel HPLC column for chiral separation of L & D arginine amino acids

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Chirality remains a major topic in the chemical and biological sciences [1]. Concerning the recently developed techniques [2,3], ability of a molecularly imprinted xerogel was evaluated through imprinting L-arginine into the 3(trimethoxysilyl)propylmethacrylate (TMSPMA) structure. In the presence of TFA and H_2O hydrolysis and condensation of the precursor, TMSPMA, was accomplished during the sol-gel process while methacrylate moiety acts as the functional monomer to imprint the selected amino acid. Aniline was added to the prepared sol to reduce the long gelation time of the sol-gel transition duration and therefore improve the processability of the imprinting. In-situ electro-polymerization of aniline was performed to prepared polyaniline (PANI) into the silica network. The mild conditions used in this process enabled the incorporation of organic species, PANI, into silica particles without degradation. There is a relationship between the gelation time and the conductive polyaniline loading. The gelation time in some cases was more than one month for pure TMSPMA and it was sharply decreased to a few days while PANI was added to the solution. The main reason for the acceleration in the gelation process of TMSPMA lies in the fact that the conductive polyaniline is weakly acidic, which is beneficial for accelerating the hydrolysis and condensation of the selected precursor. The prepared material was washed with methanol:acetic acid (100:1) and then ground into the powders, followed by packing into the HPLC column.

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Preparation of nanocomposite (Co, Mn) doped-ZnO for photodegradation of azo dye in wastewater

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ZnO is a self-excitation semiconductor, one of the most important promising materials which have been applied to many fields such as transparent conductive contacts, solar cells, laser diodes, ultraviolet lasers, thin film transistors, optoelectronic and piezoelectric applications, surface acoustic wave devices and photocatalyst [1]. Recently, micro- and nanostructured ZnO has been obtained by using various physical and chemical techniques [2]. Also owing to high photocatalytic activity, low cost and environmentally friendly feature, ZnO has been widely used as a photocatalyst. However, due to a wide band gap of 3.37 eV, poor photon absorption of ZnO limits its application in visible light photocatalyst [3]. In order to shift the optical absorption of ZnO into the visible region, one possible approach is to dope transition metal ions into ZnO photocatalyst. Metal-doped ZnO nanomaterials permit tuning of chemical and physical properties by the incorporation of the dopant in lattices of ZnO [4].

In this work, transition metals (Mn, Co) doped ZnO nanopowders prepared by a hydrothermal method. The phase and morphology studies have been carried out by X-ray diffraction, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) respectively.All the samples of the present investigation are found to have hexagonalwurtzite structure and crystallite sizes are found to vary from 25 nm to 30 nm. Different molar ratios of metal cation doping in ZnO structure is tested. The photocatalytic activity of M-doped ZnO was examined on the degradation of the Methyl Orange (MO). Login cations in ZnO lattice structure change in the energy band gap. This causes the use from visible light instead of UV light for the ZnO photocatalytic activity for degradation of dyes in wastewater. $ZnO_{0.9}Co_{0.1}O$ show 99% degradation in 60 min.

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Preparation of nano-sized Pb^{2+} imprinted polymer and its application as an electrochemical sensor for toxic lead determination in different real samples

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Abstract

High toxicity of lead is due to chronic poisoning resulting from trace exposure and leads to several organs disruption particularly kidneys and nervous system [1]. The European Union has set the maximum allowable concentrations in food to be from 0.02 to 1mgL^{-1} and World Health Organization has set 10 mg L^{-1} for drinkable water [2].

In this work, a new nano-structured ion imprinted polymer (IIP) was synthesized. A carbon paste electrode modified with IIP-nanoparticles was used for fabrication of a Pb^{2+} sensitive electrode. Differential pulse stripping voltammetry method was applied as the determination technique, after open circuit sorption of Pb^{2+} on the electrode and its reduction to metallic form. The IIP modified electrode showed a considerably higher response, compared to the electrode embedded with non-imprinted polymer (NIP). This indicated that the suitable recognition sites were created in the IIP structure in the polymerization stage. Various factors, effective on the response behavior of the electrode, were investigated and optimized. The introduced sensor showed a linear range of 1.00×10^{-10} to 1.00×10^{-8}M and detection limit of 3.30×10^{-11}M (S/N=3). The sensor was successfully applied for the trace lead determination in different samples.

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Pulsed electromembrane extraction of mefenamic acid and diclophenac in body fluids followed by high-performance liquid chromatography

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In the present work, pulsed electromembrane extraction (PEME) was performed for extraction and quantification of mefenamic acid and diclophenac in body fluids followed by high-performance liquid chromatography. PEME was introduced using a simple and inexpensive electronic device, which creates pulsed voltage in combination with common DC constant power supplies [1-3]. This new concept of EME offers an alternative to overcome the difficulty of analysis of real samples and the instability of the extraction system. In the samples with high concentration of ions, increasing of the ion transportation across the liquid membrane results in Joule heating during the extraction process which causes bubble formation due to electrolysis reaction and sparking in some cases. Nonsteroidal anti-inflammatory drugs such as diclofenac and mefenamic acid, have been used to reducing the pain, swelling and stiffness in Lrzhyt, gout and other rheumatic diseases such as headaches and muscle pain. In this paper, the effective parameters such as extraction time, applied voltage and the duration of pulse and outage period are optimized. Preconcentration factors in the range of 80-120 and recoveries in the range of 80-100 were obtained. The linear dynamic ranges of 40-250 ng mL^{-1} and limits of detection of 5.0 ng mL^{-1}.The figures of merit of PEME were compared with the results from conventional EME, which proves the advantages of the proposed technique.

Keywords: Pulsed electromembrane extraction, mefenamic acid, diclophenac

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PVC-Membrane Modified with 1-(2-Pyridylazo)-2-naphthol for Preconcentration and Determination of Ag(I) in Water Samples

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The quick determination of minute quantities of ionic species by simple methods is of special interest in analytical chemistry [1]. Silver can enter the environment via industrial water because it is often an impurity in copper, arsenic and antimony industries and silver has been recognized as a toxic element in marine environments [2]. The development of 1-(2-pyridylazo)-2-naphthol (PAN)-modified polymeric membranes (Fig. 1) for the effective batch preconcentration and determination of Ag(I) traces with flame atomic absorption spectrometry (FAAS) is described. The method is based on the chemical bonding of the metal species with a suitable ligand, which has been immobilized into a water-insoluble cellulose acetate (CA) membrane followed by simple rinsing of the chelating agent–metal complex with an acidified methanolic solution. The latter is directly aspirated to the nebulizer of a FAA spectrophotometer without any other treatment. As an analytical demonstration, trace concentrations of Ag(I) were successfully detected in real samples, such as seawater, river and lake water, wastewater as well as in a reference material, without any laborious and time-consuming treatment. Several working parameters were investigated. A pre-concentration factor of 125 was achieved by simple immersing of a circular piece of the CA–PAN membrane (0.6 cm diameter) in the tested samples for 30 min at room temperature. The analytical curve was rectilinear up to 40 µg L^{-1} Ag(I) with detection limit of 0.9 µg L^{-1}, a quantitation limit of 2.9 µg L^{-1} and a relative standard deviation lower than 3.3%.

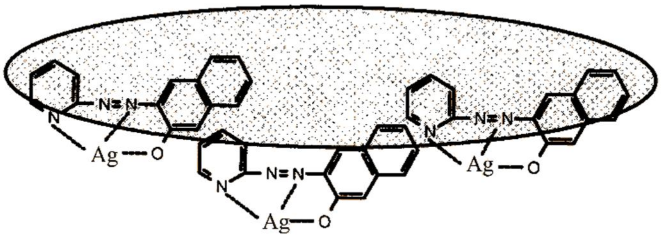

Figure 1. Schematic representation of the proposed extraction

References:
[1] M. Masrournia, et al. J. Chil. Chem. Soc. 53, Nº 5 (2009).
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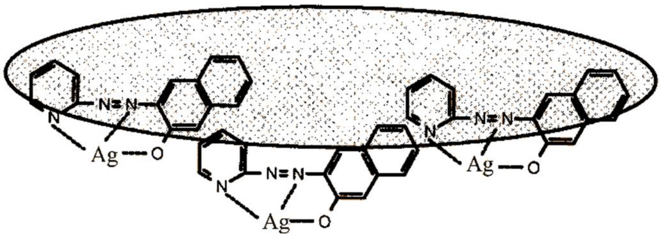

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Pyridine-functionalized nano silica as an efficient adsorbent for the removal of Th^{4+} and UO_2^{2+} ions

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One of the important processes in the management of nuclear wastes is the separation of lanthanides and actinides [1]. In addition to their importance in production of nuclear energy, thorium and uranium are known as strategic elements in a variety of industries. Application of various adsorbents such as organic chelating resins, silica gel, activated carbon, alumina, zeolites and microcrystalline materials [2] are among the techniques used for separation of these ions. Nowadays, nanometer materials have become more important owing to its special physical and chemical properties. The size, surface structure and inter particle interaction of nanomaterials determine their unique properties and the improved performances and make their potential application in many areas. In this study we report on the application of nanoparticles silica as sorbent for removing Th^{4+} and UO_2^{2+} ions from aqueous solutions. The adsorption efficiencies of these ions were found to be pH dependent. At pHs 4 and 4.5 thorium and uranium ions, respectively, were quantitatively removed from aqueous media. In view of this point that the adsorptive properties of nanoparticles can be improved [3], modification of the nanoparticle’s surface by a Schiff base was done. The Schiff base pyridylmethylidenepropyltriethoxysilane ($(EtO)_3SiCH_2CH_2CH_2N{=}C{-}C_5H_5N$) was synthesized by aminopropyltriethoxysilane and pyridine-2-carbaldehyde in methanol. Refluxing this product with silica nanoparticles in toluene results modified nanoparticles. This product has been characterized by using its ^1H-NMR, ^{13}C-NMR and FT-IR spectra and elemental analysis. The results show that by using modified adsorbent, pHs of the quantitative adsorption of studied metal ions shifts to the lower pH values. The effect of other various parameters such as contact time, metal concentration and amount of adsorbent on the process was investigated. A quantitative removal of the studied metal ions (100% for Th^{4+} and UO_2^{2+}) was achieved by the modified adsorbent disperse in 20 ml of the sample containing the metal ions (initial ion concentration 20 mgl^{-1}).

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Pyridine-functionalized nano silica as an efficient adsorbent for the removal of Th^{4+} and UO_2^{2+} ions

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QSAR model for prediction the lifetime of some halocarbons by using multiple linear regression and support vector machines

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Abstract:

In this work, the lifetime of 45 halocarbons were estimated from their theoretical derived molecular descriptors by applying quantitative structure-activity relationship (QSAR) methodology. In order to calculate molecular descriptors in the first step the structures of the molecules were drown and optimized by the AM1 semi empirical method by Hyperchem 7.0 pakage. Then 1497 molecular descriptors for each molecule were calculated by the Dragon program and the most relevant descriptors were selected by stepwise multilinear regressions. The selected descriptors are X2sol, DISPe, RDF020u, E3s, H0e, HTp and R1u, which encode topological and electronic aspects of studied molecules. These descriptors were used to developing linear and nonlinear models by using MLR and support vector machines (SVM), respectively [1, 2]. In MLR model the R^2 and standard error for training set are 0.977 and 0.253 and for test set are 0.966 and 0.356 respectively. These statistical parameters for SVM model are R^2_{train}=0.979, R^2_{test}= 0.979, SE_{train}=0.220 and SE_{test}=0.26. The built SVM model was fully assessed by leave one out cross-validation (Q^2= 0.928, SPRESS=0.479) and Y-randomization test (R^2=0.222 for 25 trail). The obtained results indicate the applicability of QSAR base SVM model in prediction of lifetime of halocarbons.

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QSAR study on tubulin inhibitors using adaptive neuro-fuzzy inference system (ANFIS)

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Tubulin is one of several members of a small family of globular proteins. The most common members of the tubulin family are α-tubulin and β-tubulin, the proteins that make up microtubules. Microtubules are assembled from dimers of α- and β-tubulin. Microtubules are essential components of cell structure and are involved in many important cellular processes including mitosis, morphogenesis, intracellular transport, and secretion [1].

The synergism of fuzzy logic (FL) systems and neural networks (NN) has produced a functional system capable of learning, high level thinking, and reasoning. Fuzzy inference system (FIS) is a knowledge representation where each fuzzy rule describes a local behavior of the system. The network structure that implements FIS is referred to as an adaptive neuro-fuzzy inference system (ANFIS) and employs hybrid learning rules to train a Sugeno-style FIS with linear rule outputs. The goal of ANFIS is to find a model or mapping that will correctly associate the inputs (descriptors) with the target (property) [2].

In this study 44 compounds as tubulin inhibitors were investigated using ANFIS. The 3-D structures of these compounds were optimized using HyperChem software. After optimization, descriptors were generated using Dragon software. For the ANFIS modeling, data set was divided into three groups: training (24), test (10) and validation (10) sets. The model was trained after 30 epochs. Finally, to evaluate the predictive power of ANFIS model, the optimized model was applied to all dataset (training, test and validation sets). RMSEs of 0.16, 0.34 and 0.17, were obtained for the training, test and validation sets, respectively.

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QSPR modeling of abiotic degradation rate constants of aromatic hydrocarbons by using genetic algorithm partial least squares

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Aromatic and polycyclic aromatic hydrocarbons (PAHs) have dangerous effects on human health and are known as mutagen and carcinogen materials [1]. One of the most important characteristics of pollutants, which determine their lifetimes, is their degradation rate constants with hydroxyl radical. Experimental determination of degradation rate constants of chemicals is time consuming, expensive and instrument dependent. Therefore, development of theoretical models for prediction of degradation rate constants of chemicals is highly beneficial. Among these methods, quantitative structure property relationship (QSPR) is an appropriate tool for construction of predicting models [2, 3]. The aim of present study was to develop a statistically acceptable QSAR model, which could be able to predict the values of degradation rate constants of several polycyclic aromatic hydrocarbons whose experimental values are not reported previously. Data set is including the abiotic degradation rate constants of 46 aromatic hydrocarbons. In the first step their molecular structural descriptors were calculated by using Dragon software. Genetic algorithm partial least squares was used for feature selection and stepwise multivariate linear regression was used for model generation. Finally, a five-parameter model utilizing the molecular descriptors: PJI3, Mor28u, DDr12, RDF010u, and R3e was constructed. The proposed model was thoroughly validated by means of statistical tests such as internal validation, external validation, and cross validated leave-one-out tests. The statistics of 0.96584 for R^2, 0.94929 for Q^2, 0.97312 for r^2_{pred}, 2.05153 for predicted residual sum of squares, and 0.2089 for residual mean square error of prediction confirmed the high predictive capacity of the model. Finally, the degradation rate constants of several aromatic hydrocarbons that their values were not determined experimentally estimated from optimal developed model.

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QSPR study of binary solvents mixtures dielectric constants: multivariate calibration and feature selection using genetic algorithm and jackknifing for validation of descriptors

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Quantitative structure-property relationship (QSPR) models are used to find a mathematical relationship between the molecular descriptors (molecular structure coded information) and desired physicochemical properties of molecules. Indeed, QSPR models are useful to characterize the solvent important properties such as dielectric constant (ε, DC) as a physical property of chemical compounds which is very vital and influential in the solubility and synthesizing of drugs based on the theories and principles of pharmacy [1].

In the present study, the dielectric constants of 3207 diverse binary solvents mixtures were modeled using 23 calculated experimental-theoretical descriptors including solvent fractions (f_1 and f_2) and individual solvents dielectric constants for each mixture (dc_1 and dc_2), various temperatures, Abraham and Hansen solvation parameters.

A QSPR model for DCs was constructed using multiple linear regression (MLR) as a simple multivariate calibration technique. To avoid the over-fitting problems and building of an efficient model, a combination of genetic algorithm (GA) (as an evolutionary intelligence algorithm) for feature selection [1] and jackknifing [2] method for validation of the selected descriptors were implemented. For evaluation of model performance, around 70% out of all of the samples (2245) were randomly implemented for calibration (as training set) and rest samples were used for prediction process (as test set) and the model accuracy parameters as criteria of prediction ability of the model such as R^2 and $Q^2_{(F3)}$ (independent parameter from size of test set samples) [3] were calculated. Finally, 4 descriptors including f_1, f_2, dc_1 and dc_2 were used to build the optimal model. Implementation of jackknifing on data set involving the selected descriptors, exhibits that *p*-value of the selected descriptors are equal to 0. It manifestly demonstrates the significance of selected descriptors and confirms their validity from sign change point of view. The R^2 and $Q^2_{(F3)}$ values for cross validated and predicted (test) samples are equal to 0.925 ± 0.001 and 0.922 ± 0.001, 0.873 ± 0.003 and 0.862 ± 0.004 (for 100 observation times), respectively.

It can be concluded that the selected descriptors superbly confirm the main theoretical equation to calculate the experimental dielectric constants of binary solvents mixtures ($dc_m = f_1dc_1 + f_2dc_2$). The selected descriptors were properly verified by jackknifing technique. In addition, the constructed model accuracy is clearly acceptable.

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Qualitative and quantitative characterization of the organo–arsenical chemical warfare agents and thiols interaction via liquid chromatography electrospray ionization tandem mass spectrometry

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Trivalent arsenic species are not easily detected using electrospray ionization due to their poor ionizability, but they are known have to high affinity for the sulfhydryl groups of biomolecules, such as cysteine and glutathione [1]. Lewisite 1 (2-chlorovinyldichloroarsine) and lewisite 2 (bis(2-chlorovinyl)chloroarsine) are organo–arsenical chemical warfare agents which causes a vesicant with attendant toxicities due to its ability to combine with thiol groups which are essential for activity of a variety of enzymes [2]. 2-chlorovinylarsonous acid (CVAA) and bis(2-chlorovinyl)arsinous acid (BCVAA) are as hydrolysis products and urinary metabolites of lewisite exposure in aqueous and urine samples [3]. There have been no reports of the interaction of CVAA, BCVAA, phenylarsine oxide (PhAsO) and diphenylaminechloroarsine (Adamsite, DM, sternutator agent) with cysteine (Cys), homocysteine (Hcy) and reduced glutathione (GSH) in vitro, so we were investigated these interactions using liquid chromatography electrospray ionization mass spectrometry (LC-ESI-MS). CVAA, PhAsO could bind with two thiols and BCVAA and DM could bind to only one thiol group. The mass spectra showed a covalent bond between arsenic species and thiol compounds after incubation in aqueous solution with a stoichiometry of 1:1. The intense molecular ions at m/z 256 ($[M+H-H_2O]^+$) and 318 ($[M+H]^+$) attributed to the CVAA-Cys and the BCVAA-Cys conjugates, respectively. The results showed that a mixture of mono and two GSH conjugates of CVAA with m/z 442 ($[M+H-H_2O]^+$) and 749 ($CVAA-(GS)_2$, $[M+H]^+$) were formed, respectively. For the DM, no interactions with sulfhydryl groups could be detected. In particular, Hcy showed much less reactivity than cysteine. To elucidate the structures of the arsenic species conjugates, collision induced dissociation (CID-MS/MS) analyses were performed. The As-thiol compounds were stable in the range of pH 2-6 at room temperature for two weeks. The method provided limits of detection in the range of 10-25 $\mu g\ L^{-1}$ in selected reaction monitoring (SRM) for arsenic-thiol complexes.

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Quality assessment of the saffron samples using second-order spectrophotometric data assisted by three way chemometric methods via quantitative analysis of synthetic colorants in adulterated saffron

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Saffron is valuable culinary spice derived from the flower of crocus sativus, commonly known as the saffron crocus. The quality of saffron depends on the concentration of its three important and major metabolites that are responsible for supplying the distinctive colour and flavour to the stigmas [1]. These components are crocin, picrocrocin and safranal present in commercial saffron. Saffron types are graded by quality according to laboratory measurement of these components present in commercial saffron. Although such attempts on the quality control of saffron sample have been done, but adulterated saffron is selling. Addition of synthesis colorants to saffron is the most common way for adulteration [2]. The active components present in saffron such as safranal and alfa-crocin have antioxidant, anti-depressant, and anti-cancer properties. So saffron is used as a therapeutically plant and adulterations make it completely harmful. The method of saffron quality control is UV–Vis spectrophotometry. This method enables to adequately discriminate the genuine saffron from the adulterated one [3].

This study is focused on simultaneous determination of the two colorants tartrazin (TA) and sunset yellow (SY), in adulterated saffron. Due to the presence of unknown interferences, three way chemometric methods such as parallel factor analysis (PARAFAC), alternating penalty trilinear decomposition (APTLD) and self-weighted alternating trilinear decomposition (SWATLD) on the three-dimensional absorbance spectra–pH gradient data are used. The data is rank deficient and unique results cannot be found. To overcoming above problem, three-way variation array V was generated by subtracting the first pH spectrum from each spectrum at each pH. This allows the extraction of extent reaction profile and mixture reaction spectra profiles, as well as the relative concentrations of the analytes.

The analytical applicability of the proposed methods in saffron samples collected from field was demonstrated through predicting the analyte concentrations in complex matrix.

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Quantitative determination of allopurinol in pharmaceutical formulation and biological samples using 2,3–dichloro–5,6–dicyano–p–benzoquinone (DDQ) as π–acceptor reagent

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Allopurinol (1H-pyrazolo [3,4–d] pyrimidin–4–ol) is a commonly used drug in the treatment of chronic gout or hyperuricaemia associated with leukaemia, radiotherapy, antineoplastic [1]. It is a structural isomer of hypoxanthine (a naturally occurring purine in the body) and acts to inhibit xanthine oxidase. In the presence of xanthine oxidase, allopurinol will be converted to alloxanthine after that the formation of uric acid from xanthine and hypoxanthine will be inhibited [2].

In this work a simple, rapid and reliable spectrophotometric method is described for the determination of allopurinol via charge-transfer formation using 2,3–dichloro–5,6–dicyano–p–benzoquinone (DDQ) as an π–acceptors. The proposed method can be carried out at room temperature without solvent extraction step or pH control, and can be achieved within 30 min to give highly colored complexe at λ max 440 nm in metanol with a good accuracy and precision. The effect of different parameters such as solvent, indicator concentration and time of complexation was investigated and calibration curve was drawn in optimum conditions. Beer᾽s law was obeyed in concentration range of 1–70 $\mu g\ mL^{-1}$with correlation coefficient ≥ 0.9939. The molar absorbtivity and limit of detection (LOD) was found to be 2.128×10^{3} L mol^{-1} cm^{-1} and 0.567$\mu g\ mL^{-1}$, respectively. Results of analyses were validated statistically and through recovery studies. The proposed method was successfully applied for the determination of this drug in pharmaceutical preparations and human blood serum samples.

Key Words: Allopurinol, Spectrophotometry, Charge transfer.

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Quantitative determination of flavonoids in micro liter volume of citrus fruit juice samples by drop-to-drop solvent microextraction coupled to HPLC

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Flavonoids are one of the largest classes of naturally occurring aromatic secondary plant metabolites and biologically active compounds in vegetables, citrus fruit and their juices. Citrus fruits stand out among the most common flavonoid sources, and the most significant cropped citrus fruits are oranges, tangerines, grapefruits, and acid citrus fruits, namely, lemons and limes [1]. These fruits typically contain more than 60 flavonoids, and reports indicate a wide range of apparently beneficial properties, such as anti-inflammatory, antitumor activity, inhibition of blood clots and reduction of chronic diseases [2]. Due to the importance of flavonoids as contributors of beneficial health effects of fruit and citrus fruit juice consumption, their determination plays an important role in many areas of science [3].

In the present study, for the first time, drop-to-drop solvent microextraction (DDSME) as a simple, rapid and environmentally friendly method -coupled to high performance liquid chromatography (HPLC)- was developed for the quantitative determination of hesperidin, diosmin and naringin in micro liter volume (30 µL) of citrus fruit juice (orange, tangerine and lemon) samples. All parameters, such as type and volume of extraction solvent, extraction time, pH and ionic strength of the sample solution- which affect the separation and preconcentration of the analytes- were investigated. Under the optimum conditions, the limits of detection (LODs) of the method and the relative standard deviations (RSDs) for determination of the analytes were in the rage of 19-38 ng mL^{-1} and less than 5.4 %, respectively. Recoveries of the analytes in the citrus fruit juice samples were found to be 91.8-102.4 %, which showing good reliability of this method. The obtained results showed that DDSME method could also be applied as a good choice for the analysis of different analytes in small volume of biological samples, like plasma, saliva and urine, where the availability of samples are limited.

Keywords: Drop-to-drop solvent microextraction, citrus fruit juice, flavonoids, HPLC

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Quantitative determination of vanadium in biological and environmental samples using kinetic spectrophotometric method

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Abstract

Vanadium determination due to its increasing importance in biological and environmental studies has gain many attentions. This metal is widely distributed in the earth's crust constituting 0.02% of the elements. Trace amounts of vanadium are a vital factor for growth of the cells. However, higher concentrations of vanadium are toxic and hazardous for human being and environment [1]. Therefor, accurate and precision determination of vanadium in sample with different matric can be all active the researchers. Many methods have been used for the determination of vanadium, such as including atomic absorption spectrophotometry [2], inductively coupled plasma atomic emission spectrometry [3]. In this study, a simple, rapid and sensitive kinetic sepectrophotometric method was developed for determining trace amounts of vanadium. Orange-G was oxidized by bromate in acidic media and vanadium showed catalytic effect. The reaction was followed sepectrophotometrically at λ_{max} (478.5 nm). Under optimum experimental conditions (H_2SO_4, 6.2×10^{-1} mol.L^{-1}; Orange–G, 7.9×10^{-4} mol.L^{-1}, bromate, 6.5×10^{-3} mol.L^{-1}, 25 ˚C and 360 s), vanadium can be determined in concentration range of 0.05 – 8.1 µmol.L^{-1} also the limit of detection was 0.014 µmol.L^{-1} of vanadium. The proposed method was successfully applied for the determination of vanadium as vanadate in various samples including human blood and hair as biological samples, rice as food samples, tobacco as plant samples and sea water as environmental samples that satisfactory results obtained.

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Quantitative determination of vanadium(V) in biological, food and environmental samples using kinetic spectrophotometric method

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Vanadium determination due to its increasing importance in biological and environmental studies has gain many attentions. This metal is widely distributed in the earth's crust constituting 0.02% of the elements. Trace amounts of vanadium are a vital factor for growth of the cells. However, higher concentrations of vanadium are toxic and hazardous for human being and environment [1]. Therefore, accurate and precise determination of vanadium in samples with different matrices can be all active the researchers. Many methods have been used for the determination of vanadium such as atomic absorption spectrometry [2] and inductively coupled plasma atomic emission spectrometry [3].

In this study, a simple, rapid and sensitive kinetic sepectrometry method was developed for determining trace amounts of vanadium. Orange-G was oxidized by bromate in acidic media and vanadium has catalytic effect. The reaction was followed sepectrometry at λ_{max} (478.5 nm). Under optimum experimental conditions (H_2SO_4, 6.2×10^{-1} mol L^{-1}; Orange–G, 7.9×10^{-4} mol L^{-1}, bromate, 6.5×10^{-3} mol L^{-1}, 25 °C and 360 s), vanadium can be determined in concentration range of 0.05 – 8.1 μmol L^{-1} also the limit of detection was 0.014 μmol L^{-1} of vanadium. The proposed method was successfully applied for the determination of vanadium as vanadate in biological, food and environmental samples with satisfactory results.

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Quantitative spectrometric data-activity relationship (QSDAR) study on radical scavenging activity of antioxidants

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Free radicals can damage membranes, proteins, enzymes and DNA [1]. The scavenging of free radicals play a notable part in the antioxidant activity of phenolic compounds [2]. The main aim of this work was investigation the applicability of using ^{1}H NMR, ^{13}C NMR chemical shifts and IR fingerprint region frequencies data as molecular descriptors for developing of quantitative spectrometric data-activity relationship (QSDAR) based model. Therefore simulated ^{13}C and ^{1}H NMR and IR spectra have been employed as 3D spectral descriptors for modeling and prediction of Trolox equivalent antioxidant capacity (TEAC) of 96 phenolic antioxidants. Y sorting method was used for choosing test series from the entire data set. Accordingly 19 molecules considered as test set and the rest of them (77 molecules) as training set. For each type of simulated spectrum (IR, NMR), a separate array was generated. These spectra were presented as a list of peak positions that accompanied by their corresponding intensities. The application of the ‘break point’ algorithm to select the most relevant spectral region led to the conclusion that the best model had 8 descriptors (spectral interval) that were a combination of ^{1}H NMR ^{13}C NMR and IR. The developed multiple linear regressions model had statistic of $R = 0.888$, $SE= 0.92$ and the leave-one-out (LOO) cross-validated R^2 (Q^2) was $Q^2 = 0.68$. Also the Y-randomization test obtained $R_r= 0.26$ which indicated that there is not any chance correlation among data set. Appearance of selected spectral descriptor in the developed QSDAR model indicate the role of electronic and topological aspects of molecules (that encoded by their NMR and IR spectra) on the radical scavenging activities of chemicals.

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Free radicals can damage membranes, proteins, enzymes and DNA [1]. The scavenging of free radicals play a notable part in the antioxidant activity of phenolic compounds [2]. The main aim of this work was investigation the applicability of using ^{1}H NMR, ^{13}C NMR chemical shifts and IR fingerprint region frequencies data as molecular descriptors for developing of quantitative spectrometric data-activity relationship (QSDAR) based model. Therefore simulated ^{13}C and ^{1}H NMR and IR spectra have been employed as 3D spectral descriptors for modeling and prediction of Trolox equivalent antioxidant capacity (TEAC) of 96 phenolic antioxidants. Y sorting method was used for choosing test series from the entire data set. Accordingly 19 molecules considered as test set and the rest of them (77 molecules) as training set. For each type of simulated spectrum (IR, NMR), a separate array was generated. These spectra were presented as a list of peak positions that accompanied by their corresponding intensities. The application of the 'break point' algorithm to select the most relevant spectral region led to the conclusion that the best model had 8 descriptors (spectral interval) that were a combination of ^{1}H NMR ^{13}C NMR and IR. The developed multiple linear regressions model had statistic of R = 0.888, SE= 0.92 and the leave-one-out (LOO) cross-validated R^2 (Q^2) was Q^2 = 0.68. Also the Y-randomization test obtained R_r= 0.26 which indicated that there is not any chance correlation among data set. Appearance of selected spectral descriptor in the developed QSDAR model indicate the role of electronic and topological aspects of molecules (that encoded by their NMR and IR spectra) on the radical scavenging activities of chemicals.

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Quantitative structure-property relationship (QSPR) prediction of solvation Gibbs energy of bifunctional compounds by support vector machine (SVM)

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In this work, we apply a quantitative structure-property relationship (QSPR) study based on support vector machine (SVM) [1] to the prediction of the standard Gibbs energy of solvation in water of mono- and polyfunctional organic compounds.

Semi-empirical quantum chemical calculations were used to find the optimum 3D geometry of the studied molecules and different descriptors were calculated by the Dragon software. Then the selected descriptors by stepwise method were applied for model development. The powerful modeling method of SVM was employed to investigate the possible nonlinear relation between the selected descriptors and the standard Gibbs energy of solvation. The proposed model is able

to directly take as input a structured representation of the molecule and to model a direct and adaptive relationship between the molecular structure and the target property.

A data set of 337 mono- and polyfunctional acyclic compounds including alkanes, alkenes, alkynes, alcohols, ethers, thiols, thioethers, aldehydes, ketones, carboxylic acids, esters, amines, amides, haloalkanes, nitriles, and nitroalkanes was considered [2]. The data set (337 molecules) was randomly divided in two groups, training set and test set consisting of 250 and 87 compounds, respectively. The training set was used for the model generation and the test set was used for the evaluation of the generated model. The root mean square error (RMSE) and correlation coefficient (r) for the training set were 3.4 and 0.953, respectively, while the root mean square error and correlation coefficient for the test set were 2.7 and 0.970, respectively.

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Quenching effect of some amino phenol derivatives on peroxyoxalate chemiluminescence of 7-amino-4-methylcoumarin (C120) and 7-amino-4-(trifluoromethyl)coumarin (C151)

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A peroxyoxalate chemiluminescence (PO-CL) method has been widely utilized in environmental, pharmaceutical and biomedical analyses owing to its high sensitivity and a need of simple instrumentation without a light source[1,2]. cumarin derivatives are of fundamental practical interest. Many of them are widely used as optical dyes and brighter for natural and synthetic materials, as well as in lasers.

7-amino-4-methylcoumarin (C120) and 7-amino-4-(trifluoromethyl)coumarin (C151) emit blue and green like blue lights in methanol solvent under uv lamp respectively.The quenching effect of some amino phenol derivatives on strong chemiluminescence of bis-(2,4,6-trichlorophenyl)oxalate–H_2O_2 system in the presence of coumarins (C120 and C151) was studied. The effects of fluorescer, TCPO, hydrogen peroxide, sodium salysilate and the surfactant of Brij 35 concentration on the light intensity were investigated. The highest light intensity was obtain at concentrations 1.67 × $10^{-3,}$ 33/3 × 10^{-3} , 8/81 × 10^{-3} , 1.11 × 10^{-3} for fluorescer, TCPO, hydrogen peroxide, sodium salysilate and the surfactant of Brij 35 respectively. Based on the proposed mechanism, and from steady-state kinetics calculations, the chemiluminescence intensity for fluorescer (F) in the absence of quenchers (Q) can be written cas:

$$I_0 = k\,[C_2O_4][F] \qquad (1)$$

where $k = k_2k_4/(k_4 + k_5)$. In the presence of quencher Q, the chemiluminscence intensity is reduced from I_0 to I, which is given by:

$$I = k\left(\frac{k_2[F]}{k_2[F] + k_{2a}[Q]}\right)[C_2O_4][F] \qquad (2)$$

The ratio of I_0/I will thus result in a Stern–Volmer type expression as [3]:

$$\frac{I_0}{I} = 1 + \left(\frac{K_{2a}}{K_2\,[F]}\right)[Q] = 1 + K_Q[Q] \qquad (3)$$

Thus, according to equation [3], the concentration of amino phenols used (Q) can be determined by quenched chemiluminscence.

These systems resulted in Stern–Volmer plots in the quencher concentration range of 1.67 × 10^{-5} to 1.67 × 10^{-4} M with k_Q values of 1880/0, 1228/0, 319/8, and 71.6 for 4-amino-3-methyl phenol, 5-amino-2-methyl phenol, 2-amino-4-nitro phenol and 2-amino-4-chloro phenol respectively for C151 and 785/8, 317/9, 259/2, and 165/5 for 4-amino-3-methyl phenol, 5-amino-2-methyl phenol, 2-amino-4-chloro phenol and 2-amino-4-nitro phenol Respectively for C120.

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Quenching effect of some amino phenol derivatives on peroxyoxalate chemiluminescence of 7-amino-4-methylcoumarin (C120) and 7-amino-4-(trifluoromethyl)coumarin (C151)

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Rank Annihilation Factor Analysis of tensorized simple first order NMR data

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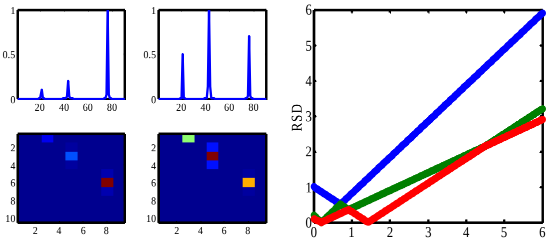
Determining the rank of data matrices, and as a result the number of components, using singular value decomposition (SVD) is common for chemometricians. The maximum possible rank for a matrix is the minimum of numbers of matrix rows and matrix columns. In this way, the maximum estimated number of components for the matrix of three first order 1D-NMR spectra is three. Also, for a matrix including two 1D-NMR spectra the maximum possible estimated number of components is two, even if the actual number of components in each of the samples is higher than two. One way of obtaining the estimated number of components higher than one for a vector (a spectrum) is by matricizing it. Applying SVD, ranks higher than one can be estimated from the obtained matrix. In this report, a number of simple 1D-NMR data sets were simulated from two samples, including three narrow peaks. In some of them all three peaks were from one component. In some others the three peaks were from two or three components. Applying rank annihilation factor analysis (RAFA) [1] on the tensorized data the number of components in the two samples was successfully determined [2,3]. Effect of tensorized data, dimensionality and peak widths on the obtained rank were utilized to extract the information content from the obtained two matrices from two vectors. Actually from the number of minimums in RSD plot, obtained from RAFA, the number of independent principal components can be estimated easily. In this way, the estimated rank can be more than two. For example, for two 1D NMR, with three peaks related to two components, after matricizing them and applying RAFA, RSD plots for the 1st, two 1st and three 1st singular values are in Figure 1, on the right and two 1D NMR data and their related matrices after tensorization [2,3] is on the left part up and down respectively.



The results show different RSD plot for changing the above conditions using the same number of peaks. The proposed approach is applicable to conditions in which only two spectral vectors are available, and from them information from more than two components is obtainable.

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Rapid Electrochemical Estimation of Antioxidant Potential of Eucalyptus *oleosa* extracts

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Abstract:

The present study describes a facile and rapid screening method based on cyclic voltammetry (CV) for estimation the antioxidant potential of leaf extracts of a plant (Eucalyptus *oleosa*) obtained from different amounts of plant (1, 3 and 5g) at various temperatures (25, 50 and 100 ºC). CV technique could be used as a simple and routine electrochemical method for screening antioxidant properties and estimation the antioxidant activity of foods and medicinal plants [1]. In this work, cyclic voltammetric measurements were carried out by a conventional three electrode system in a single compartment cell. The triple electrode system was composed of a glassy carbon working electrode (GCE), a saturated calomel reference electrode (SCE) and a platinum wire auxiliary electrode [2]. Before each measurement the working electrode was polished with alumina on a polishing cloth [3]. The plant extract solutions were prepared at different temperatures and using various amounts of plant materials in a 50 mL of distilled water. Then, the pH of solution was fixed at 7.0 by 0.2 M phosphate buffer as the supporting electrolyte. The cyclic voltammograms were recorded at several scan rates. Two main parameters i.e., the anodic peak current (Ipa) and the first oxidation potential (Epa) were obtained using rapid and simple cyclic voltammetry method without time consuming sample preparation step. The extracted samples resulted in different antioxidant capacities. The peak anodic current (Ipa) for the plant extract obtained by 1 g of plant material at the temperature of 25 ºC showed the highest height, which confirms the low oxidation potential of this sample and hence its high antioxidant capacity.

Key words: Cyclic voltammetry; Antioxidant; Plant extracts; Electrochemical; Eucalyptus *oleosa*.

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Rapid supramolecular solvent-based nonmagnetic particles for the determination of herbicides in water

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Supramolecular solvents have outstanding properties to be used at the microscale for analytical extractions [1,2]. Thus, they are made up of three-dimensional aggregates with regions of different polarity that offer a number of interactions for analyte solubilisation (i.e. ionic, hydrogen bond, _-cation, dipole–dipole, hydrophobic, etc.) that making them suitable for the extraction of organic compounds in a wide polarity range.

Supramolecular solvent (SUPRAS) combined with magnetic nanoparticles was applied for extraction and preconcentration of four herbicides (cyanazine, simazine, prometon and prometon) followed by HPLC-UV determination in water samples. A supramolecular solvent which consist of vesicles of decanoic acid in the nano- and microscale regimes was produced from the coacervation of decanoic acid aqueous vesicles. The created SUPRAS was dispersed in water sample containing triazines. After completion of extraction the dispersed SUPRAS was collected by using magnetic nanoparticles (MNPs). After elution of SUPRAS from MNPs surface 20 μL of it was injected into HPLC column for analysis. Several parameters affecting the extraction efficiency including pH, amount of decanoic acid and sorbent, sample temperature, stirring rate, salt addition, and extraction time were investigated and optimized. Under optimal conditions, preconcentration factors and relative recoveries of the studied compounds were obtained in the range of 120–140 and 91–100% respectively. The calibration curves were obtained in the range of 0.5–100 $\mu g\ L^{-1}$ with reasonable linearity (R^2 >0.99) and the limits of detection (LODs) ranged between 0.3 and 0.5 $\mu g\ L^{-1}$ (based on S/N = 3). The precision of the method, expressed as relative standard deviation (100 $\mu g\ L^{-1}$), was in the range of 2.9–5.8.

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Rapid synthesis of porous Cu-Sn/ Pt catalyst using hydrogen bubble dynamic template and their enhaced catalytic performance for methanol electrooxidation

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In recent years, numerous studies have focused on the preparation of porous materials due to their large surface area and, as a result, higher catalytic activity. A new green and promising template, gas bubble dynamic template, was proposed by which different shapes of nanostructured metals and alloys such as dendritic or porous structure have been successfully prepared from electrochemical deposition processes [1-3]. At first, the galvanostaticelectrodeposition method was employed for fabrication of porous copper-tin film. Electromagnetic stirring was posed to the system when a constant current was applied to the GCE. Then the deposited copper-tin was used as sacrificial template to prepare Ptporouse structures. The galvanic replacement was carried out in $PtCl_4$ solution for certain time. Then, the Cu-Sn/Pt porous structures were obtained. The as-prepared Cu-Sn/Pt/GCE exhibited high methanol oxidation peak current. However, more studies including optimization of experimental parameters and the characterization of the surface properties for this structure is under way. In this work, a self-supporting Cu-Sn/Ptthree metallic film with porous structure was electrodeposited at the surface of glassy carbon electrode (GCE) using a facile double-template fabrication process, including hydrogen bubble templating method and galvanic replacement reaction, and its performance investigated as a catalyst for methanol oxidation.

Keywords: Cu-Sn/Pt Structures; Bubble template; Galvanic replacement; methanol electrooxidation

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Reduce in chemical oxygen demand of industrial wastewater using electrochemical method

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Reuse of wastewater is suitable solution in water resources development strategy, which should be considered in the future. The refined wastewater is considered to be the new and proper source of water for industrial activities. In recent years, many efforts worldwide to reduce chemical oxygen demand (COD) using different methods of investigation have been addressed. One of the most plausible method is using of advanced oxidation by electrochemistry [1]. Due to development of various industries and environmental issues in Iran (pollutions of groundwater resources, surface and soil), new methods for studying of performance of that is necessary to be applied. This plan is according to the highly requested demand as well as unsuitable methods to reduce treated industrial wastewater with high amount of COD, reduction in water usage, a new complementary treatment has been suggested in this manuscript.

The main aims of the present study can be categorized as follow:

- The reuse of effluent for irrigation of plants, factories and industries,
- Solve environmental problems of various industries such as petrochemical output effluent, electroplating, dairy products, sugar and etc.,
- Increase in value of the generated effluent.

Table 1 shows the effect of different voltage at the same volume of electroplating wastewater (10000 liter) at the same time (5 hours).

Voltage (V)	2	5	10	15	20	25
Reduce of COD (%)	10±1.8	24±2.1	47±1.9	68±1.2	71±1.8	74±2

Keywords: wastewater, environmental problems, chemical oxygen demand, electrochemical method.

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Reduction of Chemical Oxigen Demand in South pars gas Complex waste waters using Co_3O_4/Graphene Oxide nanoadsorbent

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Chemical oxygen demand (COD) is a main parameter widely used to estimate the organic content of industrial wastewaters.Conventional treatments for these organic waste waters include biodegradation, acidification, electrochemical technologies, chemical precipitation, adsorption by activated carbon, ozonation and oxidation and so on. Graphene, a novel class of carbonbased nanomaterial, has attracted considerable research interests because of its extraordinary electronic, thermal and mechanical properties. This material has prompted tremendous theoretical and experimental efforts worldwide. Recently, magnetic materials are of considerable interests in material chemistry because of their unique physical properties and outstanding surface chemistry properties. Furthermore, an increasing number of studies have been concentrated on adsorption and separation using these materials, which is the so-called magnetic solid-phase extraction (MSPE). In this work, a Co_3O_4/Graphene Oxide nanoadsorbent was synthesized according the literature [1] and charachterized with TEM, FTIR and XRD and then used for COD reduction in industrial waste waters of South pars gas Complex. Adsorption equilibrium and kinetic data were determined for Co_3O_4/grapheneoxide nanocomposite and fitted to several adsorption isotherm and kinetics models, respectively. In general, Co_3O_4/ grapheneoxide nanocomposite can be utilized as an effective and facil adsorbent for the reduction of COD in refinery wastewaters.

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Removal of 2-nitro toluene from wastewater samples by using of activated carbon-magnetic nanocomposite

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ABSTRACT

Nitrotoluene compounds (NTCs) are highly toxic and coming to environment from production of pesticides, drugs, explosives, polymers, dyes, rubber chemicals, etc. [1]. Many techniques have been proposed for removal of NTCs in aqueous solutions [2]. The objectives of the present research are to introduce a simple, fast, high-efficient, cost-effective, and environment-friendly method for removal of NTCs from aqueous solutions. In current research, activated carbon-magnetic nanocomposite (AC-MNC) as adsorbent was prepared, and characterized by vibrating sample magnetometer (VSM), transmission electron microscopy (TEM) and thermo gravimetric analyzer (TGA). The adsorbent was used to removal of 2-nitrotoluene from aqueous solutions and gas chromatography (GC) technique was used for determination amounts of 2-nitrotoluene. Through an experimental design, the effects of different parameters such as pH value, shaking rate, amount of sorbent, contact time and the initial volume of solution were simultaneously studied on removal efficiency. According to the results, it was observed that pH variations do not remarkably affect the removal efficiency but efficiency was improved by other parameters. Optimum condition was achieved adsorbent amount (80 mg), contact time (12 min), stirring rate (400 rpm), and initial volume of solution (200 ml). The proposed method was applied removal of 2-nitrotoluen from industrial wastewater (ammunition and military industries) and drinking water samples

Key Words: 2-nitrotoluene, removal, activated carbon-magnetic nanocomposite.

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Removal of Alizarin Red and Purpurin from Aqueous Solutions Using $CoFe_2O_4$ Magnetic Nanoparticles

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Abstract

Many industries, such as dyestuffs, textile, paper, food, cosmetic, carpet and plastics, use dyes to color their products; as a result, these industries produce color-containing wastewater as an unavoidable by-product [1,2]. the presence of dyes in wastewaters is one of the major environmental problems as they are generally resistant to degradation by biological treatment methods. Therefore, wastewater contaminated with dye needs to be treated by physical and chemical means before discharging. The adsorption is one of the most applied techniques for dye removal [3]. This is likely due to its simplicity and high level of effectiveness.

In this research, the removal of two anthraquinone dyes, alizarin red and purpurin, by $CoFe_2O_4$ magnetic nanoparticles from textile effluent is described. The $CoFe_2O_4$ magnetic nanoparticles were synthesized and were used as adsorbent. Characterization of the obtained nanoparticles was achieved by FTIR, TEM and XRD. Batch adsorption experiments were performed to investigate the adsorption conditions and reusability of the nanoparticles. The influence of several parameters such as nanoparticle dosage, pH of the solution, dye concentration, contact time between reagents, temperature, and ionic strength was studied. Experimental results indicated that $CoFe_2O_4$ nanoparticles can remove more than 98% of alizarin and 94% of purpurin under the optimum experimental conditions of nanoparticles dosage of 15.0 mg, a pH of 4.0, and a contact time of 3.0 min, when the initial dyes concentrations in the range of 5.0-80.0 mg L^{-1} were used.

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Removal of As(III) from water samples using nickel- zinc- ferrite nano-composite

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Recently, application of nano-composite for the removal of pollutants has come up as an interesting area of research. The unique properties of nano-composite are providing unprecedented opportunities for the removal of metals in highly efficient and cost effective approaches and various nanoparticles and dendrimers have been exploited for this purpose [1, 2]. Nano-composite exhibit good adsorption efficiency especially due to higher surface area and greater active sites for interaction with metallic species. Magnetic nanoparticles as an efficient adsorbent with large specific surface area and small diffusion resistance have been recognized

In this work, $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nano-composite was synthesized and its removal ability for AS(III) was investigated. Physical characteristics of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ were studied using SEM and XRD measurements. The SEM results showed nanocomposite diameters of 27 nm. Prepared nano-composites were characterized by XRD measurements which indicated that the $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nano-composites were synthesized well. Nano-composites for the removal of ntcontamina arsenic from water samples were used. Influence of pH on the adsorption process was studied over the range 3.0–12.0, adjusted with 0.01 M NaOH or 0.01 M H_2SO_4. Effect of other operational parameters such as contact time and weight of nano-composites on the move of As(III) were investigated and optimum conditions were established. Batch studies were performed to address various experimental parameters for the removal of this ion.

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Removal of Brilliant Blue FCF dye from aqueous solution using of ROSA SP seed as a natural adsorbent: kinetic and thermodynamic studies of process

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ABSTRACT

The natural adsorbents have some benefit such as: nontoxic, clean, easy absorption and degradation in environment.[1]

Adsorbent prepared from ROSA SP (flower seed) as a natural, availability, inexpensiveness and the desired pores adsorbent. It has used to remove Brilliant Blue FCF dye successfully from several aqueous solutions in this study. SEM micrographs using a scanning electron microscope were studied. The surface morphology studies using SEM proves that, it contains move pores which lead to develop more adsorption sites. An FT-IR analysis for ROSA SP was performed.

Brilliant Blue FCF is classified as a low hazardous dye, but because of the height solvent it can cause to environmental and water pollution also in the industrial usage using with caution, skin contamination should be avoided [2].

The effect of various parameters such as pH, amount of adsorbent, size of adsorbent particles, and contact time on removal processing was investigated. Spectrophotometric technique was used for the measurement of concentration of dye before and after adsorption. To investigate the mechanism of adsorption, several kinetic models were tested including Lagergren, pseudosecond-order, particle diffusion, film diffusion, and Elovich models. Adsorption isothermal data could be interpreted by the Langmuir, Freundlich, Tempkin, and Dubinin_Rudushkevich (DR) isotherm models. The adsorption isotherm data will employ to calculate the thermodynamic parameters like ΔG, ΔH and ΔS [3].

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Removal of Cr(VI) from Aqueous Solution Using Ag_2O/Sawdust Nanocomposite

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Abstract

The presence of toxic heavy metals in water poses serious health and environmental hazards. Hence, significant research activities are aimed for analysis, minimization, containment and mitigation of such pollutions. Among the heavy metal ions, Cr(VI) has been found to be very toxic and cancerogenic. Chromium salts is widely used in many industrial applications, such as tanning, electroplating, pigmentation, catalysis, and wood preservatives [1]. Its two main oxidation states which are very stable under environmental conditions: Cr(III) and Cr(VI). Cr(VI) is highly toxic and dangerous than Cr(III) since it is highly soluble and mobile in water streams. Therefore its entering in the environment can severely endanger human health [2]. Thus, the removal of Cr(VI) seems to be very important issue in water and wastewater treatment.

This study describes the Cr(VI) removal in aqueous solution using Ag_2O/Sawdust nanocomposites, which were prepared by the precipitation method. The effects of several factors, including pH, contact time, initial concentration, adsorbent dosage, temperature, and ionic strength on sorption capacity were evaluated. The optimal pH value for the removal of the Cr(VI) by the introduced adsorbent was about 3. The adsorption processes were very rapid during the first 5 min and reached equilibrium within 20 min. Isotherm investigation was carried out using Freundlich and Langmuir models. Kinetics study was performed using pseudo first and pseudo-second-order models [3]. It was found that the adsorption data are best fitted with the Langmuir isotherm model and a pseudo-second-order kinetic model. Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD) were employed for characterization of the introduced adsorbent.

Keywords: Cr(VI), Adsorption, Ag_2O nanocomposite, Characterisation

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Removal of Cr(VI), As(V) and Hg(II) from aqueous solutions using functionalized mesoporous silica nanomaterials

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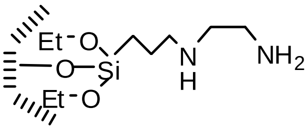
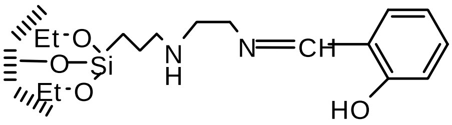
Toxic heavy metals such as Cr(VI), As(V) and Hg(II) in some effluents are considered persistent, non-biodegradable and bio-accumulative. Chromium is commonly found in the waste effluents from electroplating, metallurgical, tannery and textile industries [1]. Arsenic is one of the most toxic pollutants introduced into natural waters by geochemical reactions, industrial waste discharges and agricultural use of arsenic pesticides [2]. Mercury is one of the most dangerous and ubiquitous of pollutant, and its contamination is widespread and arises from a variety of natural and anthropogenic sources [3].

Adsorption of metal ions on various sorbents is popular due to its simplicity, low cost and interesting potential for overcoming the environmental problems. Among the known adsorbents, ordered mesostructured silica materials present high superficial areas, great pore volumes and uniform pore diameters because of their mesoscopic structure.

In this communication the preparation and application of two adsorbents based on SBA-15 mesoporous silica (A, B) for removal of Cr(VI), As(V) and Hg(II) ions from aqueous solutions are reported and Inductively coupled plasma optical emission spectrometry (ICP-OES) was utilized for measuring the concentrations of metals.



(A)



(B)

The adsorbents were characterized by means of transmission electron microscopy (TEM) and scanning electron microscopy (SEM). The effects of various parameters such as aqueous phase pH, initial metal concentration, temperature, time and the amount of adsorbent in the process were investigated. By using 0.02 g of the adsorbents in 20 ml of the water samples containing the studied metal ions (initial concentration of each 10 mg/l), under optimum conditions; pH=2.5, contact time=3 h, the sorption amounts of Cr (VI), As (V) and Hg (II) ions were 94%, 89% and 73% of Hg (II) by mesoporous A and at pH=3, contact time=15 min., the sorption amounts of Cr (VI), As (V) and Hg (II) ions were 96%, 77% and 54% of Hg (II) by sorbent B. The adsorbents were used successfully for removing the target ions from various real water samples. The analysis of the adsorption data showed that Langmuir isotherm can suitably describe the experimental data obtained by modified mesoporous silica materials.

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Removal of Cu(II) from aqueous solutions by modified nano-particles of clinoptilolite with cysteine

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Abstract

The purpose of this study is modification of nano-clinoptilolite (NCP) particles by use of Cysteine (Cys) to increasing removal of copper cations by the modified zeolite (NCP–Cys). The modified sorbent demonstrated affinity for soft and moderately soft heavy metal ions, such as Cu(II), probably as a result of the soft basic character of the thiol groups of Cysteine. Removal of copper from aqueous solution was done with both batch and column methods [1]. The removal capacity of raw micro and nano – clinoptilolite particles (MCP and NCP), NCP-Cys, MCP-Cys, modified Cu(II)-exchanged clinoptilolite (NCP-Cu-Cys and MCP-Cu-Cys) samples was compared. The sorption capacities of NCP–Cys showed that this modified form is a good sorbent for the removal of copper cations. The effects of analytical parameters such as pH, dose of Cys, concentration of copper solution, contact time and selectivity were studied and the optimal operation parameters were found as follows: pH_{PZC}: 4, $C_{Cu(II)}$: 500 mg/L, contact time: 360 min and Cys dosage: 0.01 M. The kinetics of copper removal process by the proposed sorbent obeys from second order kinetics, that explain the chemical sorption because of formation of complex between copper and cysteine.The equilibrium adsorption is described by the Langmuir model and maximum adsorption of 0.521 mmol Cu/g was obtained [2]. Langmuir equation indicates the monolayer sorption of Cu(II) by the proposed sorbent. The study of some interfering cations confirmed that the proposed adsorbent has good selectivity for copper cations. In column studies, the effects of various parameters such as amount of sorbent, flow rate of solution and concentration of copper and influences of other cations on copper removal were investigated.

Keywords: Nano-Clinoptilolite; Modification; Sorption; Cysteine; Heavy metal ions

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Removal of $Cu^{۲+}$, Ag^{+} and $Pb^{۲+}$ Ions by using modified ZnO nanoparticle with S, N-substituted Thiouracil Derivative

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Exposure to heavy metals, even at trace level, is believed to be a risk for human beings. Thus, effective removal undesirable metals from water and wastewater systems are still very important challeng for environmental study. The synthesis of adsorbents for the removal of toxic metals ions is a continuing research objective for control of environmental pollution. Recently, ZnO nanoparticles have been used directly and in modified form for extraction of some heavy metals [1].In this work, removal of heavy metals by means of modified ZnO nanoparticles was investigated. For this purpose, S, N -Substituted thiouracil derivative [Fig1] was used for modification of ZnO nanoparticle's surface and the modified adsorbent was used for removal of Cu^{+2}, Ag^{+} and Pb^{+2} ions. The ions concentration was measured by flame atomic adsorption spectroscopy (FAAS).

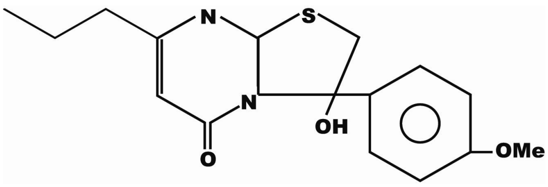

Fig 1. S, N -Substituted thiouracil derivative

The amount of adsorbent, extraction time and pH were optimized. Removal of Cu^{+2}, Ag^{+} and Pb^{+2} became possible after 30min in 25ml 0f 4mgL^{-1} solution of these three ions, and by using 35mg of modified adsorbent. Upraise of pH from 2 to 5 caused absorption value of all three ions to increase. The optimum pH for Cu^{+2}, Ag^{+} and $Pb^{+ ۲}$ removal were 5.The adsorption data was analyzed by Langmuir, Freundlich and Temkin isotherms. It was concluded that adsorption process follows Langmuir isotherm model for this three ions. The maximum adsorption capacity values of the modified ZnO nanoparticles (q_m calculated from Langmuir equation) were about 208µg, 114µg, 238µg of Cu^{+2}, Pb^{+2} and Ag^{+} ions per each mg of adsorbent, respectively.

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Removal of Diazinon Pesticide Using Amino–Silane Modified Magnetite Nanoparticles

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Diazinon is an organophosphorus insecticide with widespread agricultural and non-agricultural uses. Because of the great risk that it poses to human health and the environment, diazinon use has been reviewed [1]. Therefore the removal of pesticides is one of the major environment concerns in these days. In the other hand interest in studying nanostructure materials has grown significantly in recent years due to unique size and physical properties. Magnetic nanoparticles (MNPs) have attracted much research interest duo to many potential technological applications in different area, e.g., in catalysis as support, in targeted drug delivery, adsorption processes and environmental remediation. Also magnetic nanoparticles can be used as a novel and excellent adsorbents due to their unique advantages over traditional micro-sized adsorbents [2]. Application of magnetite nanoparticles in cleanup and enrichment of pesticides is a relatively new area of research [3].

In the present study, a co-precipitation method was used to prepare the magnetite nanoparticles (Fe_3O_4). The particles were subsequently coated with a dense silica layer and then the silica layered magnetic nanoparticles were functionalized with (3-aminopropyl) triethoxysilane (APS) to prepare the adsorbent, $Fe_3O_4@SiO_2\text{-}NH_2$. The magnetic adsorbent was characterized by FT-IR, TEM and SEM.

The prepared adsorbent ($Fe_3O_4@SiO_2\text{-}NH_2$) was used for removal of diazinon from aqueous solutions. Adsorption of diazinon on $Fe_3O_4@SiO_2\text{-}NH_2$ was investigated by UV spectrophotometry at 236 nm, through batch experiments. Various chemical parameters and equilibration times were considered to be optimized. A one at a time optimization method was used for the optimization of conditions such as adsorbent dosage, solution pH, contact time and initial concentration of diazinon. Furthermore the effects of solution temperature and water impurities on adsorption of diazinon onto $Fe_3O_4@SiO_2\text{-}NH_2$ were investigated. Then the data were evaluated for compliance with Langmuir, Freundlich and Temkin isotherm models. Results showed the maximum removal efficiency 84% under the optimized condition. Evaluations were also made for kinetic and thermodynamic of diazinon adsorption on $Fe_3O_4@SiO_2\text{-}NH_2$. Results showed the best fitness with the pseudo-second-order model ($R^2>0.99$) and equilibrium adsorption data could be better fitted with the Freundlich isotherm ($R^2>0.99$). Thermodynamic analysis also suggests the electrostatic interactions, as the main mechanism of adsorption.

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Removal of Direct Blue 131 from Aqueous Solution by Chitosan Modified Zeolite

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Zeolites are a class of porous materials that comprise inorganic oxide atoms covalently linked into a crystalline framework**.** The pore diameters of zeolites measure typically 5 A to 10 A and are consistently sized because of the crystalline formation. One specific family of zeolites comprises an alumino-silicate framework.

The natural zeolites have already found extensive applications to the environmental problems. The most of these applications are based upon their cation – exchanger properties. The adsorption results indicate that the natural zeolite has a limited adsorption capacity for reactive and anionic dyes, but is substantially improved upon modifying its surfaces with positively charged modifiers.

In this work, a new synthesized chitosan-modified zeolite employed as an adsorbent for removal of Direct Blue 131 (DB131) as an anionic dye from aqueous solution. Chitosan is a biopolymer that has many useful characteristics such as hydrophilicity, biocompatibility, biodegradability, and anti-bacterial properties. A batch system was applied to study the adsorption. The effect of experimental parameters upon dye adsorption was investigated and optimal conditions were obtained. These parameters are initial concentration of DR131, pH of solution, contact time, temperature and particle size, volume of solution, kind and amount of buffer, adsorbent mass, interfering ions, ionic strength. The points of zero charge (PZC) of zeolite before and after modifying with chitosan were calculated. The maximum adsorption capacity of modified zeolite at pH=2 and contact time of 75 minutes was 72.54 $mg.g^{-1}$. The equilibrium data were applied to Langmuir, Freundlich and Temkin equations. The adsorption isotherm was well fitted with Langmuir model. The kinetic of removal method was studied using the first and second order equations.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ۱۳۹۲

Removal of dyes (brilliant cresyl blue, di methylene blue, aniline blue, eosin methylene blue, comassise brilliant blue G250) using magnetite nanoparticles (Fe_3O_4) from aqueous solutions

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Abstrac

An adsorbent, silica-coated magnetite nanoparticles, was used for removal of cationic and anionic dyes brilliant cresyl blue (BCB), di methylene blue (DMB), aniline blue(AB), eosin methylene blue(EMB),andcomassise brilliant blue G250 (CBB) from water samples.Prepared nanoparticles were characterized by SEM, TEM, BET and XRD measurements. The prepared magnetic adsorbent can be well dispersed in the water and easily separated magnetically from the medium after loaded with adsorbate. The influences of parameters including initial pH, dosage of adsorbent and contact time have been investigated in order to find the optimum adsorption conditions. The optimum pH for removing of all the investigated cationic dyes from water solutions was found to be 9.0 and for anionic dyes 3.0. The experimental data were analyzed by the Langmuir adsorption model. The maximum predicted adsorption capacities for BCB, DMB, AB , EMB and CBB dyes were obtained as192.25,111.363, 358.33,131.49 , and 398.53 mg g^{-1}, respectively.Desorption process of the adsorbed cationic dyes was also investigated using acetonitrile as the solvent. It was notable that both the adsorption and desorption of dyes were quite fast probably due to the absence of internal diffusion resistance.

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Removal of heavy metal ions Cd^{2+}, Zn^{2+}, Pb^{2+}, Cu^{2+} and Co^{2+} from aqueous solution by thiosalicylhydrazide-modified magnetic nanoparticles

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Abstract: A novel magnetic nano-adsorbent has been synthesized by the covalent immobilization of poly (acrylic acid) (PAA) and a thiosalicylhydrazide (TSH) agent on the surface of Fe_3O_4 nanoparticles. The prepared magnetic nanoparticles (MNPs) were characterized by Scanning electron microscopy (SEM), Atomic force microscopy (AFM), FT-IR spectroscopy, X-ray diffraction and Vibration sample magnetometer (VSM). The SEM images showed that the diameters of the Fe_3O_4@PAA@TSH MNPs are about 10-35 nm. The ability of the Fe_3O_4@PAA@TSH MNPs for removing heavy metal ions (Pb^{2+}, Cd^{2+}, Cu^{2+}, Zn^{2+} and Co^{2+}) from aqueous solution was studied and the effects of the solution pH, contact time, metal ions concentration and background electrolytes on the adsorption characteristics of the modified MNPs were investigated. The results showed the conditions of solution pH=5.2, 100.0 mg.L^{-1} initial concentration of heavy metal ion , 1.0 g.L^{-1} adsorbent and time of 40 minutes dose the optimum conditions for this application. Finally, adsorption process was investigated with Langmuir and Freundlich isotherms. The experimental adsorption equilibrium data were fitted to Langmuir adsorption and the maximum adsorption capacities of Pb^{2+}, Cd^{2+}, Cu^{2+}, Zn^{2+} and Co^{2+} were found to be 188.7, 107.5, 76.9, 51.3 and 27.7mg.g^{-1} adsorbent, respectively. The adsorption was fairly rapid, because it doesn't take more 40 minutes to reach the equilibrium state and kinetic data were precisely fitted by the pseudo-second-order model. The assumption that the metal ions adsorption is due to complex formation between active sites of the adsorbent and the heavy metal ions was further confirmed by investigating the adsorption isotherms and kinetics. The desorption experiments under batch conditions revealed that the Fe_3O_4@PAA@TSH MNPs are reusable adsorbent for practical removal of the heavy metal ions. In addition, it was found that presence of amine and sulfur binding sites on the surface of the prepared MNPs not only can significantly improve the adsorption capacity of the adsorbent but also can notably reduce the interferences of the alkaline/earth metal ions. These improvements are most probably due to higher affinity of the sulfur and amine sites of the modified MNPs for complex formation with the heavy metal ions via soft-soft interactions. For testing the ability of the prepared nanoparticles for removal of heavy metal ions from industrial samples was evaluated using different industrial wastewaters from Moham industrial complex, Raad plating company and Iran Aircraft Manufacturing Industrial Company (HESA) and obtained optimum conditions is adsorbent concentration 1.0 g.L^{-1}, contact time 40 minutes, solution pH=5.2, solution volume 50.0 mL and stirrer speed 300 round per minutes that is applied to real samples. The obtained results showed that Fe_3O_4@PAA@TSH MNPs can be used for removal of heavy metal ions from industrial wastes.

Key Words: Magnetic nanoparticle; Poly acrylic acid; Thiosalicylhydrazide; Heavy metals.

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Removal of heavy metal ions Cd^{2+}, Zn^{2+}, Pb^{2+}, Cu^{2+} and Co^{2+} from aqueous solution by thiosalicylhydrazide-modified magnetic nanoparticles

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Abstract: A novel magnetic nano-adsorbent has been synthesized by the covalent immobilization of poly (acrylic acid) (PAA) and a thiosalicylhydrazide (TSH) agent on the surface of Fe_3O_4 nanoparticles. The prepared magnetic nanoparticles (MNPs) were characterized by Scanning electron microscopy (SEM), Atomic force microscopy (AFM), FT-IR spectroscopy, X-ray diffraction and Vibration sample magnetometer (VSM). The SEM images showed that the diameters of the Fe_3O_4@PAA@TSH MNPs are about 10-35 nm. The ability of the Fe_3O_4@PAA@TSH MNPs for removing heavy metal ions (Pb^{2+}, Cd^{2+}, Cu^{2+}, Zn^{2+} and Co^{2+}) from aqueous solution was studied and the effects of the solution pH, contact time, metal ions concentration and background electrolytes on the adsorption characteristics of the modified MNPs were investigated. The results showed the conditions of solution pH=5.2, 100.0 $mg.L^{-1}$ initial concentration of heavy metal ion , 1.0 $g.L^{-1}$ adsorbent and time of 40 minutes dose the optimum conditions for this application. Finally, adsorption process was investigated with Langmuir and Freundlich isotherms. The experimental adsorption equilibrium data were fitted to Langmuir adsorption and the maximum adsorption capacities of Pb^{2+}, Cd^{2+}, Cu^{2+}, Zn^{2+} and Co^{2+} were found to be 188.7, 107.5, 76.9, 51.3 and 27.7$mg.g^{-1}$ adsorbent, respectively. The adsorption was fairly rapid, because it doesn't take more 40 minutes to reach the equilibrium state and kinetic data were precisely fitted by the pseudo-second-order model. The assumption that the metal ions adsorption is due to complex formation between active sites of the adsorbent and the heavy metal ions was further confirmed by investigating the adsorption isotherms and kinetics. The desorption experiments under batch conditions revealed that the Fe_3O_4@PAA@TSH MNPs are reusable adsorbent for practical removal of the heavy metal ions. In addition, it was found that presence of amine and sulfur binding sites on the surface of the prepared MNPs not only can significantly improve the adsorption capacity of the adsorbent but also can notably reduce the interferences of the alkaline/earth metal ions. These improvements are most probably due to higher affinity of the sulfur and amine sites of the modified MNPs for complex formation with the heavy metal ions via soft-soft interactions. For testing the ability of the prepared nanoparticles for removal of heavy metal ions from industrial samples was evaluated using different industrial wastewaters from Moham industrial complex, Raad plating company and Iran Aircraft Manufacturing Industrial Company (HESA) and obtained optimum conditions is adsorbent concentration 1.0 $g.L^{-1}$, contact time 40 minutes, solution pH=5.2, solution volume 50.0 mL and stirrer speed 300 round per minutes that is applied to real samples. The obtained results showed that Fe_3O_4@PAA@TSH MNPs can be used for removal of heavy metal ions from industrial wastes.

Key Words: Magnetic nanoparticle; Poly acrylic acid; Thiosalicylhydrazide; Heavy metals.

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Removal of heavy metals from chemical wastewater by using magnetic nanoparticles

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With increasing industrial activities, wastewater from many industries such as tannery, chemical manufacturing, mining, battery manufacturing industries, etc., contains toxic heavy metals, which are not biodegradable and tend to accumulate in living organisms, causing various diseases and disorders[1]. Heavy metals are nowadays among the most important pollutants in source and treated water, and are becoming a severe public health problem, can be toxic to aquatic life and cause natural waters to be unsuitable as potable water sources [2]. In order to reduce heavy metal pollution problem, heavy metals in the environment removed by some processes such as oxidation–reduction processes, filtration, electrochemical treatment, evaporation, ion exchange or reverse osmosis, and adsorption [3]. In this work, effect of the Fe_3O_4 magnetic nanoparticles (MNP) as a cadmium (Cd) absorbent was studied. Beside this, pH of the solution, contact time, and absorbent amount as effective parameters were investigated, each one at a time. To this end, pH values were set at 2, 4, 6, 8, and 10; also, contact times were adjust at 10, 30, 50, 70, and 90 minutes. In addition, absorbent MNP amounts were selected on the three various amounts such as 0.1, 0.25, 0.5, and 1 gr. The remaining of the Cd into the solution was measured by atomic absorption system (AAS). Meanwhile, scanning electron microscopy (SEM) was used for investigation of the surface morphology of the Fe_3O_4 MNP before and after absorption process. To characterization of the crystallite structures X-ray diffraction (XRD) method was used. Also, for more information and understanding regarding the distribution of particles on the absorbent surface, dot-mapping method was used. Results show that, best ellemination was happened at the ph=8 and 10 miutes ass contact time. SEM images show that, surface morphologies of the absorbent after the elemination process was changed, so that, absorbent's surface mophology before elemination process was more intensity than after elemination process.

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Removal of heavy-metal ions from aqueous solution using electrospun acrylonitrile-maleic acid copolymer nanofibers.

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As industry expands, the contamination of water resources by heavy metals is a serious environmental problem worldwide. Environmental pollution by toxic metals occurs globally through military, industrial, and agricultural processes and waste disposal. Fuel and power industries generate 2.4 million tons of As, Co, Cr, Cu, Hg, Ni, Pb, Se, V, and Zn annually [1]. Presence of heavy metal ions in the environment has been great concern because of their toxic nature and other adverse effects on many life forms. For instance, excessive intake of copper results in an accumulation in the liver and it is also toxic to aquatic organisms even at very low concentrations [2]. Lead is a general metabolic poison and enzyme inhibitor [3]. It can cause mental retardation and further brain damage, especially in children.

In recent years, insoluble polymeric sorbents having different functional groups that can be complexed with metal ions have become well-known for the removal of metal cations from aqueous solutions [4]. The main advantages of such materials are easy loading and, in most cases, stripping of cations with simple chemicals, reusability, high efficiency, easily handling, and possible low cost. The adsorption of heavy metal ions by a polymeric sorbent depends on the macromolecular characteristics and specific surface area of sorbent. Electrospinning is a simple and versatile method for fabricating continuous fibers with diameters ranging from micrometers to several nanometers. High specific surface area with excellent adsorption capacity can be obtained by electrospinning.

acrylonitrile-maleic acid copolymer nanofibers with high acid group content prepared with different maleic acid ratios by a water-phase precipitation copolymerization process with $K_2S_2O_8$–Na_2SO_3 as initiator system and the resultant copolymers were used to fabricate electrospun fiber for the removal of Cu(II), Hg(II), Ni(II), Co(II) and Pb(II) ions from aqueous solutions. The copolymer was characterized by Fourier transform infrared spectroscopy, scanning electron microscopy, X-ray diffraction analysis, BET analysis, titration and swelling studies. The effects of pH, time, and initial metal ion concentration on the metal ion adsorption capacity were investigated. when pH is lower than 4.0, all sites of copolymer-carboxylic groups are protonated (-COOH), and metal-copolymer complexes can be hardly formed. At pH 6.0, the available complexing sites are enough to bind all the metal ions, and the metal rejection arrives at nearly 100%. Adsorption percentage for single metal ions were 85, 76, 93, 95, and 98% for Cu(II), Hg(II), Ni(II), Co(II) and Pb(II) respectively The adsorption isotherm models were applied on experimental data and it is shown that the Freundlich and Langmuir equations was the best model. It is stated that these electrospun copolymer can be regenerated efficiently (>95%) and used repeatedly.

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Removal of pollutant dyes using titania-alumina photocatalysts

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Titanium oxide is one of the most well known photocatalysts. It is non-toxic, accessible and low cost photocatalyst. But low surface area is a drawback of commercial TiO_2 products and developing new methods to increase the specific surface area values is attracting many interests. Spreading TiO_2 onto porous and stable materials like alumina is one these methods [1]. In this study, dissolved metallic aluminum and Degussa-P25 were used as sources for alumina and titania, respectively. The phase transformation of the metallic aluminum powder to alumina is observed upon special treatment. The prepared samples were characterized using X-ray diffraction (XRD) and surface area measuring (BET) methods. Methylene orange (MO) and methylene blue (MB) were used as probe to evaluate the photocatalytic activity of the prepared photocatalysts. The photocatalytic reaction was conducted in a photochemical reactor. In each experiment, 0.05 g of the catalyst was added to 50 mL of an aqueous solution that contained 25 mg/L MO or MB. Prior to illumination, the suspensions were continuously stirred in the dark for 30 min to ensure the adsorption/desorption equilibrium of the pollutants on the photocatalyst powders. At given time intervals, portions of the suspension was sampled, centrifuged, and filtered to remove the photocatalyst. The concentration of the pollutants was then analyzed using a UV–vis spectrophotometer. The measurements were done at absorption maxima at 464 and 665 nm for MO and MB, respectively. The titania/alumina exhibited the higher photocatalytic activity during the decomposition of the dyes in comparison with titania.

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Removal of reactive black 8 dye using zinc oxide nano particles

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A sorption system using nano zinc oxide, ZnO, particles as sorbent was investigated to remove reactive black 8 (RB8) dye from aqueous solutions. The system variables studied include the sorbent nanoparticles with the size of 50, I, and 10 nm, II. Dye solution containing a concentration of 10 to 150 mg L^{-1}. In all experiments, 25 ml containing dye solutions with 0.2 g of ZnO sorbent were treated separately. The concentration of RB8 determined, using UV absorption before and after exposure. The results revealed that the dye removal capacity of ZnO increased as the sorbent particle size decreased. Maximum saturated monolayer sorption efficiency of ZnO nanoparticles for the adsorption of RB8 dye was 36 and 97% for II and I, respectively. For a set of complexation with n central cores, equation 2 could be used.

$$\frac{A_{ad}-A_{\max}}{A_{\max}}[ZnO]=\frac{A_{ad}-A_{\max}}{A_{ad}}[RB8]\frac{1}{n}-\frac{K_d}{n} \qquad (1)$$

Where n is proportional to the number of sites on the surface area of the given ZnO particles, K_d is the dissociation equilibrium constant for the hypothetical complex in which the ZnO particle plays the role of central atom surrounding by RB8 molecules as legands, [ZnO] is the certain amount of ZnO particles suspended in water molecules. [RB8] are concentrations of the adsorbed RB8 molecules on the surfaces of ZnO particles. A_{ad} represents the absorbance value at the certain adsorbed RB8 dye and A_{max} represents the observance upon the saturation of all surfaces on the given ZnO particles. The saturated monolayer sorption capacity, C_{max}, can also be obtained. A plot of $\frac{A_{ad}-A_{\max}}{A_{\max}}[ZnO]$ versus $\frac{A_{ad}-A_{\max}}{A_{ad}}[RB8]$ should be a linear plot by a slope of 1/n and the vertical-intercept of $\frac{K_d}{n}$, which n and K_d can be obtained. The free energy of the sorption reaction (complexation process), considering the sorption equilibrium constant, K_a $(1/K_d)$, (ΔG^0) is given by the following equation:

$$\Delta G^0 = -RT\ln K_a \qquad (2)$$

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Removal of reactive blue19 from aqueous solutions by eggshell: Optimization of effective parameters using Taguchi method

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Abstract

Dyes contain carcinogenic materials which can cause serious hazards to aquatic life and the users of water. Textile industry is the main source of dye wastewater which results in environmental pollution. Many studies have been conducted to investigate the use of low cost adsorbent as an alternative technique for the adsorption of dye. The objective of this study is to determine the potential of eggshell powder as an adsorbent for reactive blue 19 removal and find out the best operating conditions for the adsorption of eggshell powder process at laboratory scale. The influences including pH,dosage of adsorbent,contact time,rate and ionic strength have been researched by taguchi experimental design in order to find the optimum adsorption conditions.[1]The adsorbent was analyzed by SEM and BET analysis. The experimental data were analyzed by the Langmuir,Freundlich and Temkin adsorption isotherm models.The results were obtained at 25^0C. Equilibrium data fitted very well with the Freundlich model.It was shown that the sorbent can remove more than 91% of the investigated dye.[2,3]

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Removal of reactive red 74 dye from textile industries waste using zinc oxide nanosorbent

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The growing population and industrial activity in the multi recent decades, makes a large amount of pollutants entering the water. Pollutants such as dyes can be found in the effluent of textile industry is mentioned. Due to the toxicity of these pollutants are removed from water resources is essential. In this study, removal of Reactive Red 74 dyes from aqueous solutions using zinc oxide nanoparticles were investigated. It has been used in the textile industry. Reactive red 74 was purchased from a cosmonaut chemical Co. Zinc oxide nanoparticles with an average particle size of 10 nm and a flower-shaped morphology were synthesized by chemical precipitation method and was used as adsorbent [1]. Impact of various factors such as the pH of solution, contact time, amount of adsorbent with a solution dye and adsorbent mass on the adsorption efficiency were optimized. pH = 3 time = 60 minutes, adsorbent=0.2 gr was considered as the optimum conditions. Maximum capacity was=28.8 mg/g. The results show that the pseudo-second-order reaction kinetics model has attracted and adsorption process obey the Langmuir isotherm. The results showed that zinc oxide nanoparticles as an adsorbent is capable for the removal of Reactive Red 74 dyes from aqueous solutions, such wastewater of textile industries. The survey found that the efficiency of dye removal with zinc oxide nanoparticle is much more than the micro scale zinc oxide particle as adsorbent.

Reference:

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Removal of reactive red 74 dye from textile industries waste using zinc oxide nanosorbent

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The growing population and industrial activity in the multi recent decades, makes a large amount of pollutants entering the water. Pollutants such as dyes can be found in the effluent of textile industry is mentioned. Due to the toxicity of these pollutants are removed from water resources is essential. In this study, removal of Reactive Red 74 dyes from aqueous solutions using zinc oxide nanoparticles were investigated. It has been used in the textile industry. Reactive red 74 was purchased from a cosmonaut chemical Co. Zinc oxide nanoparticles with an average particle size of 10 nm and a flower-shaped morphology were synthesized by chemical precipitation method and was used as adsorbent [1]. Impact of various factors such as the pH of solution, contact time, amount of adsorbent with a solution dye and adsorbent mass on the adsorption efficiency were optimized. pH = 3 time = 60 minutes, adsorbent=0.2 gr was considered as the optimum conditions. Maximum capacity was=28.8 mg/g. The results show that the pseudo-second-order reaction kinetics model has attracted and adsorption process obey the Langmuir isotherm. The results showed that zinc oxide nanoparticles as an adsorbent is capable for the removal of Reactive Red 74 dyes from aqueous solutions, such wastewater of textile industries. The survey found that the efficiency of dye removal with zinc oxide nanoparticle is much more than the micro scale zinc oxide particle as adsorbent.

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Removal of the Acid Violet 5 from contaminated water using a surfactant–modified natural zeolite

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Adsorption of anionic dye Acid Violet 5 (AV-5) on raw or Non-Modified Zeolite (NMZ) and Surfactant-Modified Zeolite (SMZ) adsorbents was investigated to explore the feasibility of removing AV-5 from aqueous solution using natural Clinoptilolite zeolite adsorbents. The SMZ was prepared by adsorbing the cationic surfactant hexadecyltrimethyl-ammonium (HDTMA) bromide on the external surface of the zeolite [1]. The NMZ and SMZ adsorbents were characterized with scanning electron microscopy (SEM) , energy dispersive X-ray spectroscopy (EDS) and X-ray fluorescence(XRF) for morphology, elemental composition. N_2-BET method was also applied to determine surface area and pore volume. Results revealed that the surface area and pore volume were reduced due to pore blocking caused by the surfactant molecules adsorbed on the zeolite [2]. The effects of different reaction parameters such as the initial AV-5 concentration, contact time, adsorbent dosage and pH on AV-5 adsorption were investigated using UV-Vis spectroscopy technique, all absorbance values were determined at maximum wavelength (λ_{max}= 530 nm). Results indicated that adsorption efficiency of AV-5 on SMZ is increased by contact time and adsorbent concentrations. However, adsorption efficiency decreases by increasing pH of the aqueous solution. The maximum adsorption capacity for AV-5 on SMZ was 76.92 mg g^{-1}. Empirical adsorption models of Langmuir and Freundlich were applied for the experimental data. Results showed that Langmuir isotherm was more consistence for this process (R^2= 0.97). The experimental data fitted very well with the pseudo-second-order kinetic model. It was found that AV-5 removal efficiency of the NMZ were significantly improved after modification with cationic surfactant (HDTMA-Br). The surfactant-modified zeolite appears to be a promising adsorbent for removing AV-5 from water.

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Removal Reactive Black 5 dye with prepared nano manganese dioxide in watermelon peel media

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Textile industries effluents contain dye and other organic toxic compounds which are hazardous to aquatic life. Study of the textile wastewater represents, it is the most polluting industries in accordance to the discharge volume and effluent composition [1]. Reactive dyes which are typically azo-based chromophores are extensively used in many areas of textile because of their applicable favorable characteristics such as bright color, water fast soluble and simple application techniques. The presence of reactive dyes is aquatic system can cause serious harmful effects on the aquatic life by increasing toxicity and chemical oxygen demand and by hindering photosynthetic activity [2].

Aim of this study was using prepared nano manganese dioxide, placing in watermelon peel as a new low- cost biosorbtion to remove RB5 from wastewaters. Nano MnO_2 was prepared from reduction of $KMnO_4$ with alcohol in the presence of cetyl trimethyl ammounium bromide in stainless steel autoclave at 116 °C for 24 h [3]. After cooling down to room temperature, the sample was washed, filtered and dried in vacuum at 80 °C for 2 h. The product was characterized by IR spectroscopy, X-ray powder diffraction patterns and scanning electron microscopy. In the second step collected and dried watermelon peel was mixed with nano MnO_2 (ratio 3:1 respectively) in ultrasonic hemogenizer. Nano MnO_2 and nano watermelone peels-MnO_2 were used as a photocatalyst to be compared to gather in order to remove Reactive Black 5 (RB5) in simulated wastewater.

Important factors in the removal of RB5 such as pH, contact time, initial dye concentration and photocatalyst value were studied by UV-Vis spectroscopy. In the best condition of removing the dye, Nano MnO_2 and nano watermelone peels-MnO_2 showed 98.81 and 84.81 % yield for removing the dye. In spite of decrease in the yield of removal by nano watermelone peels-MnO_2 but this result is very good for compound that one-third of it is MnO_2 and two-thirds of it is waste. So wastes of some fruits can be suitable for different applications.

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Resonance light scattering method for the determination of triamterene using gold nanoparticles as probe

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Triamterene is a potassium-sparing diuretic used in combination with thiazide diuretics for the treatment of hypertensionand edema and with caution in patients with prediabetes or diabetes mellitus as there may be a change in glucose control. The National Collegiate Athletic Association (NCAA) Drug made this drug for testing in 1991. Programs In the present work, resonance light scattering (RLS) method using gold nanoparticles have been applied for the determination of triamterene in human urine and serum samples. In RLS technique, the wavelength of the incident beam is close to the absorption band of the molecule, which leads to a significant enhancement in scattering signals. The determination of triamterene using gold nanoparticles is based on the binding of triamterene onto gold nanoparticles which results in the ligand-induced aggregation of gold nanoparticles. This, in turn, leads to the enhancement of the RLS signals of the gold nanoparticles. The effects of important experimental factors such as pH, concentration of gold nanoparticles and incubation time on RLS signal were investigated and optimized. At the optimum conditions linear range and limit of detection for triamterene were 5.0-60 µg L^{-1} and 1.27 µg L^{-1}, respectively. This method was applied to the determination of triamterene in human urine and serum samples with satisfactory results.

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Retention Behavior of Phenolic acids and Flavonoids in Low-Submicellar Liquid Chromatography

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Addition an anionic surfactant such as sodium dodecyl sulfate (SDS) to the hydro-organic mobile phase in reversed phase liquid chromatography (RPLC), at concentrations lower than critical micellar concentration (CMC) leads to extended modification of stationary phase with surfactant monomers while, a small amount of free surfactant monomer existed in the mobile phase. On the stationary phase, the alkyl chain of the adsorbed SDS monomer associated to the alkyl-chains bonded to the silica support, and the polar head groups oriented away from the surface [1]. At these conditions, hydrophilicity of stationary phase was increased, as a consequence the retention of non-charged hydrophobic solutes decreased as compared with conventional hydro-organic RPLC. This is the basis of a rather uncommon mode of RPLC which known as low-submicellar liquid chromatography (LSLC) [2]. In this study, retention behavior of two important groups of natural poly-phenolic compounds with wide different polarities i.e. phenolic acids and flavonoids (five phenolic acids galic, chlorogenic, caffeic, p-coumaric and ferulic acid and four flavonoids myricetin, morin, quercetin and kaempferol) was investigated in the LSLC and compared with conventional RPLC with hydro-organic mobile phases. In the hydro-organic mode methanol (35, 40, 45, 55% v/v) and acetonitrile (20, 25, 30, 40% v/v) were examined. In the LSLC nine mobile phase compositions covering three concentration levels of methanol (35, 45 and 55 %v/v) and SDS (0.001, 0.004 and 0.007 M) were investigated (all mobile phases were acidified by 0.2% of H_3PO_4). Retention behaviors of selected phenolic compounds were studied by construction of plots of logk´ (capacity factor) of solutes against SDS and methanol or acetonitrile concentration. In the absence (hydro-organic mobile phase) or constant SDS concentration, increasing in concentration of organic solvent leads to relatively same decrease in retention of both group of solutes. But, at the constant methanol content, increase in concentration of SDS leads to more decreases in retention of more hydrophobic solutes (flavonoids), while the retention of less hydrophobic solutes (phenolic acids) was not significantly affected by SDS concentration. This was behavior like organic solvent gradient in conventional RPLC, and referred as the "gradient effect" in LSLC under isocratic conditions. In order to achieve adequate retention models for reliable and quantitative description of the retention of target phenolic compounds in the LSLC, the retention data of each solute were fitted to 16 different hyperbolic and logarithmic retention models. The performance of the investigated models was measured using the R^2 and the mean relative prediction error (RE). Among the all investigated models, the hyperbolic retention model $1/k = c_0 + c_1\varphi + c_{11}\varphi^2 + c_2[S] + c_{12}\varphi[S]$ showed the best description capability for both phenolic acids and flavonoid (R^2: 0.9981, RE: 2.8%).

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Role of modified granular activated carbon by H_3PO_4 and HNO_3 from natural adsorbent for removal of Everzol Red 3bs from aqueous solutions

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Synthetic dyestuffs are used extensively in textile, paper, printing industries and dye houses. The effluents of these industries are highly coloured and the disposal of these wastes into receiving waters causes damage to the environment as they may significantly affect photosynthetic activity in aquatic life due to reduced light penetration. Colour removal has been the target of significant attention in the last few years, not only because of its toxicity but also to its visibility [1]. Even the presence of very low concentrations of dyes (less than 1ppm) in the effluent is highly visible and is considered undesirable [2,3].However, it has to be emphasized that the majority of synthetic dyes currently used are the highly water soluble azo-reactive dyes. Azo dyes are characterized by the existence of nitrogen double bonds (-N=N-) and the presence of bright color is due to these azo bonds and associated chromospheres. The present study aims to develop a procedure for Everzol Red 3bs removal. The study was based on using powdered activated carbon, and modified with nitric acid and phosphoric acid as chemical agents. The main parameters, such as effect of pH, effect of sorbent dosage, concentrations, and various contact times that influence on the sorption process have been investigated and optimized. The results showed that as the amount of the adsorbent increased, the percentage of Everzol Red 3bs removal increased accordingly. The optimum pH value for lead adsorption was determined to be 4.The maximum removal of Everzol Red 3bs was obtained at pH 4, with a removal level of 92% for granular activated carbon modified by H_3PO_4 and 96.03% for granular activated carbon modified by HNO_3 and an adsorbent dose of 0. 5 g and 10 mg/L initial Everzol Red 3bs concentration at room temperature. The experimental results were analyzed by using Langmuir, Freundlich, adsorption models. The correlation coefficients for the Langmuir better than did the Freundlich for granular activated carbon modified by H_3PO_4 and HNO_3The kinetic study of Everzol Red 3bs absorption on granular activated carbon was performed based on pseudo- first order and pseudo second order equations. The data indicate that the adsorption kinetics follow the pseudo- second order rate. The procedure was successfully applied for Everzol Red 3bs removal from aqueous solutions.

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Scanometric determination of magnesium with titan yellow agent

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Magnesium is an essential mineral for human nutrition mainly found in foods like cereals, nuts, cacao, meat, milk and vegetables. Magnesium has several important functions. It is involved in energy metabolism, acting as a metal activatoror co-factor for enzymes requiring adenosine triphosphate (ATP), in replication of DNA and in the synthesis of RNA and proteins; it appears to be essential for all phosphate transferring systems [1]. Its deficiency occurs, in general as complications of other diseases like alcoholism, diabetes, and kidney failure and in some post-operative periods.Magnesium deficiency can be treated by oral or parental administration of some magnesium salts (magnesium supplement tablets). Over supply in severe cases lead to coma and death [1].Several analytical methods, which are used, for magnesium analysis in various samples (matrices) include ion chromatography with a piezoelectric detector, inductively coupled plasma atomic emission spectrometry, atomic absorption spectrometry, ion selective electrode and UV-Vis spectrophotometry [2]. Some of these methods are expensive and non-portable analytical equipment and cannot be used sufficiently for routine analysis in small local laboratories.

In this study trace amounts of Mg was determine by Scanometry [3], as a new, simple, fast, sensitive and inexpensive method based on the formation of the Magnesium - titan yellow complex. The method is based on the reaction in solution phase, in Plexiglas® cells. The solution was scannedand finally the RGB (red, green and blue) color model was used in color monitor.Titan yellow is the reagent most frequently used for the rapid determination of magnesium. Operational parameters on the effective intensity (green parameter) such as the concentration of NaOH, titan yellow, hydroxylamin ion, starch and time, were investigated and optimized.Under the optimal conditions, the calibration curve was linear in the range of 0.1-30 µg per ml. In addition, the effects of foreign species, including cations and anions were investigated. The proposed method was successfully applied to the determination of the Mg in different real samples.

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Scanometry, new method for determination of the acidity constant of indicators: application for calculation of acidity constant of methyl red and comparison with spectrophotometric result

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The accurate determination of acidity constant values is often required in various chemical and biochemical areas. These are of vital importance in understanding the distribution, transport behavior, binding to receptors and mechanism of action of certain pharmaceutical preparation [1]. The acidity constants of organic reagents play a very fundamental role in many analytical procedures such as acid–base titration, solvent extraction and complex formation. Potentiometric, spectrophotometric, conductometric and spectroscopic are conventional methods for determination of acidity constants of organic compounds [2].

In this study, scanometry as a new, simple, fast and inexpensive method [3] was used for determination of the acidity constant, pK_a, of methyl red indicator, at first time. The method is based on solution phase, in Plexiglas® cells. The solution was scanned and finally the RGB (red, green and blue) color model was used in color monitor. The comparison between the current and traditional UV-Vis spectrophotometry methods was studied and the results revealed similar trends in both methods.

The HypSpec program was used for calculation of acidity constant with spectrophotometric data. The calculated pK_a values of the cited indicator by scanometry and spectrophotometric methods are 4.45 and 4.62 respectively. The distribution diagrams of species were plotted using the Hyss2009 program.

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Second-order calibration for determining fatty acids in pomegranate seed by vortex assisted-extraction-dispersive liquid-liquid microextraction and gas chromatography-mass spectrometry

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Getting useful chemical information from complex mixtures with a large number of chemical components has been a challenging task to chemists. Development and evolution of new chromatographic systems coupled to sophisticated detection systems for identification and quantification of chemical compounds in complex samples has become one of the most important developments in analytical chemistry in the recent years [1]. In spite of recent technological advances, current chromatographic analyses are faced with some fundamental challenges that can directly affect chromatographic results. For this purpose, multivariate resolution methods (i.e., multi-set and multi-way methods) have been proposed to overcome fundamental chromatographic challenges occurred during analysis of complex mixtures [2].

In the present contribution, the fatty acids (FAs) of pomegranate seed were extracted using vortex assisted-extraction-dispersive liquid-liquid microextraction (VAE-DLLME) and were analyzed using gas chromatography-mass spectrometry (GC-MS) under optimum conditions. Regarding the complexity of pomegranate extract and occurrence of fundamental GC-MS problems such as baseline/background contribution, elution time shifts, low S/N and peak overlap, second-order calibration based on multivariate curve resolution-alternating least squares (MCR-ALS) [3] was used to obtain pure qualitative and quantitative results. Herein, the extraction and separation of FAs were performed in less than 20 min by VAE-DLLME-GC-MS. Resolution, identification and quantification of target FAs in the standard mixture and real samples (pomegranate seed) were carried out successfully despite of the presence of some uncalibrated interferences in these samples. Three parameters of explained variance (R^2), lack of fit (LOF) and reverse match factor (RMF) were considered for evaluation of MCR-ALS results of calibration samples. The values of R^2, LOF (%) and RMF (%) were in the range of 94.06-99.47, 7.24-24.36 and 70.8-97.7, respectively. In addition, regression coefficients (R^2) and relative errors (REs, %) of calibration curves of different FAs were in the satisfactory range of 0.9934-0.9989 and 3.70-7.45, respectively. Application of the proposed strategy to pomegranate extract showed that linolenic acid, oleic acid, palmitic acid, stearic acid and 11-eicosenoic acid were respectively the main fatty acids of pomegranate seed with concentration 9098.0, 4873.0, 3147.0, 1960.0 and 1019.0 mg kg^{-1} and relative standard deviations (RSD, %) between 0.15-11.57. It is concluded that second-order calibration combined to VAE-DLLME-GC-MS is a fast and simple strategy to be used for qualitative and quantitative analysis of complex samples such as natural products.

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Selective preconcentration of trace amounts of copper by Cloud point extraction coupled flame atomic absorption spectrometry

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The cloud point extraction (CPE) procedure for pre-concentration of copper (II) ion by using a new schiff base ligand is developed. This method is based on the formation of Cu(II)-N,N′-bis(2-hydroxynaphthaldiminato)-1,8-diamino-3,6-dioxaoctane that extracted into the micellar phase (Triton X-114) at a temperature above the cloud point temperature (CPT). After phase separation, the surfactant rich phase was diluted by acidic ethanol and determined by flame atomic absorbtion spectrometry (FAAS)[1].

Results and discussion

To achieve the optimal condition for maximum extraction efficiencies for analytes, some analytical parameters including pH, ligand concentration, surfactant concentrations, incubation temperature, and other parameter were examined. The pH plays a unique role on metal-chelate formation and subsequent extraction. The effect of pH in the range of (2.0–12.0) adjusted with HCl and NaOH solution, in water samples was investigated. Result shown that the extraction recovery increase up to pH 6.5. Thus, the value of pH 7.0 was selected for the following experiments. The effect of concentration of ligand was investigated in the range of 0.1×10^{-5} to 10×10^{-5} $molL^{-1}$. The results shown that the extraction recovery increased by increasing the ligand concentration up to 4×10^{-5} $mol\ L^{-1}$ and then constant. A concentration of 1.0×10^{-4} $molL^{-1}$ of ligand was chosen for the subsequent experiments. The effect of non-ionic surfactant concentration was studied in the range of 0.01–0.25% (W/V). A considerable decrease in the absorbance signal is observed with increasing the surfactant amounts higher than 0.2% (W/V). Thus, a concentration of 0.15% (W/V) Triton X-114 was used for subsequent experiments. The cloud point temperature of Triton X-114 is near 25˚C. It was desirable to employ the shortest incubation time and the lowest possible equilibration temperature. Time and temperature was studied in the range 0.5-20 min. and 40-90 ˚C, respectively. The results showed that an equilibration time of 10 min. and 75˚C were adequate to achieve quantitative extraction. The effect of ionic strength was examined by studying the response for $NaNO_3$ concentration in the range (0-1) $molL^{-1}$. The results shows that the recovery of copper were constant to 0.2 $molL^{-1}$ $NaNO_3$, and then decrease. Based on these results, no salt was needed for further study. The proposed method was applied to the determination of Cu(II) in water samples with satisfactory results. Under the optimum conditions, Detection limit was 0.89 μgL^{-1} for 10 replicate determinations. The relative standard deviation (n=10, 200 μgL^{-1}) was ±2.65%. Also, the calibration graph was linear in the range of 3-800 $\mu gL^{-1}$with the correlation coefficient was 0.9993. Accordingly the enrichment factor was 100 that reached for only a 10 mL water sample. In this study, the use of micellar systems as a preconcentration and separation for Cu^{+2} offers various advantages including safety, low cost, preconcentrated Cu^{+2} with high recovery and very good extraction efficiency.

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Selective Separation and preconcentration of Cu(II) with novel modified Alumina and Its Determination By Flame Atomic Absorption Spectrometry

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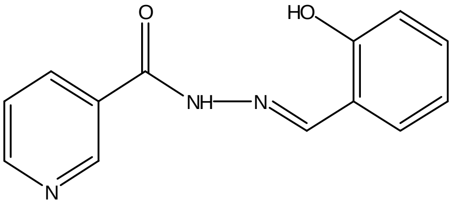
Abstract

Surfactant aggregates formed on solid surfaces, such as alumina or hydrated iron (III) oxide are called ad micells, whose interior tends to incorporate springly solube organic constituents [1]. Previously, we proposed a separation technique with admicellar sorbents for inorganic trace analysis into hydrophobic chelates with ligand and successfully incorporated in ad micelles[2]. The analyte ions were adsorbed quantitatively on adsorbent due to their complexation with new ligand (Scheme 1). First, 0.5g of SDS was added to a 40 mL suspension of 1.5g of purified alumina and 0.2 g ligand. Then the mixture was acidified to pH 2. After mixing, the supernatant solution was discarded and the remaining was used as modified solid phase sorbent in the extraction of Cu(II), Cd(II), Ag(I), Ni(II), Zn(II) and Pb(II) ions from aqueous solution. A nearly complete desorption was achieved for those metal ions in 8 ml of 4 mol L^{-1} nitric acid with the aid of ultrasonic irrdiation. The effects of main parameters such as pH, amount of alumina, amount of ligands, flow rate of sample solution, type and concentration of elution agent were examined and optimized. The percentage of extracted metal ion determined by atomic absorption spectroscopy and exhibits a good extractive affinity for Cu (II) of > %95.



Scheme 1. The structure of the ligand

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Selective Synthesis of Graphene Quantum Dots and Carbon Nanodots at Large Scale through the Electrochemical Exfoliation of Graphite Electrode thorough the Intercalation Phenomenon

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A facile electrochemical exfoliation method [1-3] has been introduced for selective, highly pure and large scale synthesis of luminescent graphene quantum dots (GQDs), carbon nanodots (CDs) and their hybrid. For this purpose, an amperometric detection system has been selected during applying an optimum direct current (DC) potential (+8.50 V) between a two-electrode system including stainless steel as cathode and graphite rod as anode under optimized condition i.e. pH 7.0, 0.5M and 1.0 M NaCl solution. The synthesized carbon nanomaterials has been characterized using analytical techniques such as Fourier-Transform Infrared (FT-IR), transmission electron microscopy (TEM), scanning electron microscopy (SEM), X-ray diffraction(XRD), particle size analyzer (PSA), XPS measurements, Raman spectroscopy, etc. According to the results, no nanomaterials are precipitated attime scales lower than ~15 s, due to the unsaturation of the electrolyte solution. According to the microscopic images, the CDs are then considered as the unit cell of GQDs. The color of the electrolyte solution is then changed to yellow during the formation of luminescence CDs. Coagulation of CDs leads to the formation of QCDs at 20-45 s time duration and ~1.0 M NaCl solution. According to the scanning electron microscopy (SEM), the average inter-layer thickness of GQDs is evaluated to ~60 nm. In this study, aging of the electrochemical reaction as well as enhancing the electrolyte concentration to ~1.0 M NaCl separates the GQD's sheets during the sonication process as well as intercalation of the NaCl that results to get hybrid (mixture) of CDs and GQDs at 300-600 s time duration and pure CDs at times higher than ~600 s.

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Selective transport of Cu(II) ion in presence of Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Pb(II) metal cations through a bulk liquid membrane using benzyl bis(thiosemicarbazone) as carrier

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Abstract

Among the separation techniques, ion transport is a selective, efficient and simple method. In the recent years, the liquid membrane has widely been used to study ion transport with a concentration gradient [1, 2].

In this work a dichloromethane membrane system incorporated with benzyl bis(thiosemicarbazone) **(LH_2)** as carrier is introduced for the selective transport of Cu(II) ion from an aqueous source solution containing Mn(II), Co(II), Ni(II), Cu(II), Zn(II) and Pb(II) metal cations to a receiving aqueous phase. The optimum transport conditions were established by studies the composition of receiving phase, time of transport, stirring rate, pH of source phase, presence of fatty acid in the membrane as a phase modifier, type and concentration of surfactant and concentration of mineral acid in receiving phase and carrier concentration. Two replicate experiments showed that 27% of the initial concentration of Cu (II) ion in the source aqueous phase (pH 5) is extracted into the receiving phase (HNO_3 3.0×10^{-1}M) through the bulk liquid membrane (**LH_2** 4.0×10^{-4}M in CH_2Cl_2) after 24 h. A kinetic model was also used to describe the Cu (II) ion transport behavior, assuming that it obeys the kinetic laws of two consecutive irreversible first-order processes. The possible mechanism of transport was also discussed. The method was applied for the recovery of Cu (II) ions from real samples.

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Selective transport of metal ions across polymer inclusion membranes (PIMs)

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Abstract

The use of polymer inclusion membranes (PIMs) for selective and efficient transport of metal ions is increasingly retaining attention [1-2]. We then prepared and characterized cellulose triacetate membranes derivatives as fixed carriers (2,2'-Dithiobis[benzothiazole])(L) and containing 2-nitrophenyl octyl ether (2-NPOE) as a plasticizer.We applied them for the investigation of facilitated transport of Zn^{2+}, Pb^{2+}, Co^{2+}, Ni^{2+},Cu^{2+} and Ag^{+} ions from aqueous nitrate source phase (10^{-4}M) and the receiving phase containing of buffered at pH=3. The polymer inclusion membranes (PIM) method were prepared by mixing a solution of cellulose triacetate (CTA) in dichloromethane with solution containing of a plasticizer (2-NPOE) in dichloromethane and L (3×10^{-3}M). The cell was enclosed by a water jacket and thermostated at 25 °C for 24 h. All transport runs were terminated after 24 h and atomic absorption spectroscopy was used to determine the amount of each of the metal cations.

We found that the facilitated transport of metal ions through our PIM proceeds through a carriermediated diffusion mechanism.

The results show that for method (PIM) the selectivity of L_1 for zinc(II) cation is higher than the other cations. These results may open news avenues in zinc removal from waste waters.

Keywords**:** PIM, Transport, Zn(II)**.**

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Sensitive spectrophotometric determination of Zinc in the presence of major interferences in highly complex matrix- electric arc furnace dust

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Electric Arc Furnace Dust (EAFD) is one of the most important by-products in steel-making industry. Over seven million tons toxic dust is produced per year and still more than 50% of the generated dust is lost by sending it to landfill. EAFD contains 5-30 % zinc and is considered as a secondary zinc resource[1-2]. Since the economic feasibility of zinc recovery process is depended to the zinc content, determination of zinc percent in EAFD is very important. On the other hand, dust contains more than 15 elements and tens of chemical complexes that cause problems in zinc determination. The aim of this study is to propose a simple and practical method to determine zinc percent in dust. For this purpose, we used EAFD of Esfarayen Steel Co. as initial zinc source and visible spectrophotometry as the determination method. Zincon (2-carboxy-20-hydroxy-50-sulfoformazylbenzene) was applied as the chromogenic reagent[3]. The best condition for zinc determination obtained at pH=9 using universal buffer. For solving matrix effect and removal of some spectral interfereces standard addition method and partial least squares calibration were applied. Results indicate that although Fe^{2+} ions don't react with zincon but seriously interfere in combination of Zn^{2+} ions with zincon. In order to eliminate negative effect of Fe^{2+}, iron oxide precipitation using ammonia solution was used. According to the results, the zinc content of used EAFD was 19.2 % which was consistent with atomic absorption results.

Keywords: Zinc Determination, Electric Arc Furnace Dust, Zincon, Spectrophotometry, Iron Interference

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

Sensitive turn-on fluorescence assay of silver ion based on the fluorescence resonance energy transfer between harmine and Au NPs

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Resonance energy transfer (RET) is a non-radiative process whereby an electronically excited ‘donor’ molecule transfers its excitation energy to an ‘acceptor’ molecule such that the excited state life time of the donor decreases [1]. Gold nanoparticles (Au NPs) have received great attention for potential biological analysis in recent years, because of their interesting size-dependent optical properties. Au NPs possess a broad absorption band with high extinction coefficient (about 3~5 orders of magnitude higher than that of common organic molecules) in the UV-vis region. Thi distinguished optical properties allow Au NPs to act as efficient acceptors for most fluorophores through both energy transfer and electron transfer processes [2].

In this work, we have explored the quenching of the fluorescence of harmine from the β-carboline family by Au NPs. Harmine and several related alkaloids occur in a number of plant species and contain the β-carboline group, i.e. they are pyrido(3,4-b)indoles and are expected to show strong fluorescence and emissions [3]. Considering a good overlap between the fluorescence spectrum of harmine emitting at 420 nm and the absorption spectrum of Au NPs the harmine-Au NPs pair has been chosen for a plausible energy transfer system in which harmine acts as the donor and Au NPs as the acceptor. The fluorescence of harmine molecules was severely quenched when they were attached to the surface of Au NPs by electrostatic attraction. The quenching constant and thermodynamic parameters for this interaction were determined. It was found that upon the addition of silver ions, harmine molecules are partly released from the Au NP surface. This is the basis for sensitive turn-on detection of silver ions. Under the optimum conditions, the enhanced fluorescence intensity displayed a linear relationship with the concentration of silver ion in the range of 9.0-470 μg L^{-1}, with a limit of detection of 5.0 μg L^{-1}. This method is simple, selective and relatively free of interference from co-existing substances and was applied to the determination of silver ion in some natural water samples.

[1] V.V. Mokashi, A.H. Gore, V. Sudarsan, M.C. Rath, S.H. Han, S.R. Patil, Evaluation of interparticle interaction between colloidal Ag nanoparticles coated with trisodium citrate and safranine by using FRET: spectroscopic and mechanistic approach, J. Photochem. Photobiol. B 113 (2012) 63.
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Separation and determination of antidepresant drugs in biological samples using ionic liquid – based ultrasound – assisted-nano particle microextraction method before High performance liquid chromatography

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Abstract:

In the present study, a sample preparation method by combination of ionic liquid and nanoparticle microextraction which assisted by ultrasonic (IL-USA-NP-ME) has been reported for preconcentration and extraction of Nortriptyline and Doxepin in biological samples prior to their high performance liquid chromatography (HPLC) [1-3]. The properties of AgOH nanoparticle is characterized by FT-IR, SEM and XRD. This nanoparticle posses great adsorptive ability for preconcentration of antidepresant drugs. Experimental parameters such as amount of nanoparticle, the pH and ionic strength of sample solution, the ultrasonic time (min), the ultrasonic temperature (℃), kind and volume of ionic liquid were optimized by central composite design (CCD). The calibration graphs were calculated for eight working aqueous standards spiked with two drugs at different concentrations. Under the optimal conditions, the response linear over concentration range of 0.005-3 $\mu g\ mL^{-1}$. Limit of detection is based on a signal to noise ratio of 3, calculated for five replicate runs and was between 0.001-0.0025 µg mL^{-1}. The obtained relative standard deviation (RSD%) was 3%, based on five replicates. Finally, the method was applied for the determination of drugs in real samples (urine and plasma). The relative recoveries at spiked level of 0.01 µg mL^{-1} were in the range of 89– 95%.

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Separation and Identification of Chemical Composition of the Volatile Oils from *Teucrium polium* Using the Head Space Solid Phase Microextraction Method Prior to Gas Chromatography-Mass Spectrometry

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Abstract:

Teucrium has been used since ancient time for a variety of medicinal purposes as anti bacterial, sedative, antipyretic and gastric lesions[1,2,3] . The Flora of Iran comprises 12 species of *Teucrium* of which 3 are endemic[4].The present report deals with the analysis and comparison of composition of the volatiles obtained by head space-solid phase microextraction (HS-SPME) procedures from the crushed dry *Teucrium polium*. The plant material was collected from south of Sabzevar, Khorasan Razavi Province, Iran[4]. The SPME headspace volatiles were collected using a fiber coated with polydimethylsiloxane (PDMS). The composition of the volatile essential oils was investigated by GC-FID and GC-MS. Kovat's retention indices were calculated using co-chromatographed standards hydrocarbons. The individual compounds were identify by MS and their identity was confirmed by comparing their retention indices relatives to C8 –C32 n-alkanes and by comparing their mass spectra and retention times with those of authentic samples or with data already available in the NIST library and literature Adams[5]. Eleven components, representing 99.26 % of the total, were identified in the oil from the flowers, of which the major components were □-pinene(57.99%),sabinene(18.44%), limonene (7.26 %), myrcene (7.2 %) [5]. A comparison of the chemical composition of volatiles of *Teucrium polium* from this study with previous studies on the same genus showed variation of the major components. These results imply that although the use of HS-SPME has the advantages of simplicity, solvent-free sampling and lowering the amount of sample needed, it can also affect the results of the essential oils analysis.

Key words: *Teucrium polium*; Head Space Solid Phase Microextraction; essential oil; □-pinene limonene, myrcene.

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Separation and Removal of dyeing pollutant from aqueous solutions using Ionic Liquid

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Abstract

Ionic liquids (ILs) are regarded as ‘‘designer solvents’’ because of this tunable nature, which increases their potential applications. Their most often cited attribute is their negligible vapor pressure even at elevated temperatures, which offers some advantages like product recovery and recycling. Replacing organic solvents currently used in different extraction processes with ILs is a ‘‘hot’’ research topic [1].

A simple, rapid and efficient method, ionic liquid based dispersive liquid–liquid extraction (IL-DLLME), has been applied to extract and remove Rhodamine B (as a model dyeing pollutant and commercial dyes) from aqueous solutions. In this methodology a binary solution, containing the extraction solvent (1-hexyl-3-methylimmidazolium bis(trifluormethylsulfonyl) imid) and a suitable disperser solvent, was rapidly injected into the water sample containing dye [2]. Therewith, a cloudy solution was formed, and most of the dye molecules were extracted into fine IL droplets and removed from aqueous phase. Afterwards, IL-phase was dissolved in 50μL of pure acetone and transferred to 50 μL quartz cylindrical micro-cell. The spectra of these complexes was collected by the charge coupled device (CCD)-linear array detector and the multi wavelength absorbance data were processed for spectrophotometry and the absorbance was measured at 556.0 nm against blank. Thus, this technique combines extraction and concentration of the analytes into one step and avoids using toxic chlorinated solvents. Under optimum conditions (pH 5. and a 0.25%w/v of salt concentration) and by applying about 90–400mg/10mL of IL with the proper amount of dispersant, quantitative extraction (>95%) of Rhodamine B could be achieved at initial concentrations in the range of 50–280mg L^{-1} of this dye. Experimental surveys were also accomplished for recovery of the IL by applying a reverse dispersive liquid–liquid extraction using acidic stripping solutions. The simplicity in regeneration of the IL and the dye recovery may make this IL-DLLE suitable for practical application for some types of biphasic industrial separations. IL-DLLE provides a high recovery and low toxicity because only small amounts of a ‘‘rather green extraction solvent’’ (IL) is used [3].

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Separation and Removal of dyeing pollutant from aqueous solutions using Ionic Liquid

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Separation enantiomers of Dextrodopa in aqueous media by using surface molecularly imprinted silica nanoparticles in electrokinetic chromatography

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Molecular imprinted polymer (MIP) techniques have been increasingly used in a variety of fields including chromatography, sample pretreatment, purification, sensors, drug delivery, and catalysts, etc. MIP is a specific artificial receptor that shows favored affinity to the template molecule. The cavities of the template are produced by carrying out polymerization of a reaction mixture followed by eliminating the template molecules by washing [1].

A new research based on surface molecularly imprinted technique is about for the synthesis of surface molecularly imprinted silica nanoparticles (MI-SiNPs) in aqueous media, using L-DOPA (L-3, 4-dihydroxyphenylalanine) as template molecule. The synthesized MI-SiNPs were characterized by transmission electron microscopy (TEM) and thermo gravimetric analysis (TGA) and the results showed MI-SiNPs were uniform in particle size (∼76 nm) and have core–shell structure with shell thickness of ∼5 nm. So MI-SiNPs have terminal carboxyl groups, they are good water-compatibility and good suspension stability in buffer rich of water. When MI-SiNPs were used as pseudostationary phases (PSPs) in electrokinetic chromatography (EKC) for the enantioseparation of DOPA, a resolution of 2.9 can be achieved in less than 12 min with symmetric peaks. These excellent separation features are mainly attributed to the fast mass transfer and good accessibility of the interaction sites locating at the surface of the MI-SiNPs. The effects of some important separation factors on the enantioseparation of DOPA such as pH and concentration of buffer solution, content of MI-SiNPs added, content of organic modifier in buffer solution, were studied, and an optimum separation condition of 30% (v/v) methanol in 20 mmol L^{-1} phosphate buffer (pH 7.0), with 0.50 mg mL^{-1} MI-SiNPs added was selected.

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Silver Nanoparticles-Polyaniline for Microextraction in Packed Syringe

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A rapid, convenient and reliable method based on microextraction in packed syringe (MEPS) in combination with high performance liquid chromatography (HPLC) assay for the determination of the loop diuretic furosemide (FUR) in urine was developed. A nanocomposite based on silver nanoparticles (Ag-NPs)/polyaniline (PANI) was synthesized and used as the packing material of MEPS. This nanocomposite was prepared conveniently using interfacial polymerization without the need for any templates or functional dopants [1]. The feasibility of the synthesized nanocomposites was examined by isolation of FUR from diluted urine sample. After extraction, the analyte was desorbed by 200 µL of methanol. After complete drying, the residue was dissolved in 30 µL of methanol and an aliquot of 25 µL was, finally, injected into the HPLC system. Important parameters influencing the extraction and desorption processes were optimized [2,3].The linearity was studied by preconcentration of 5 mL of diluted urine sample spiked with a standard solution of FUR inthe concentration range of 15–750 µg L^{-1}. The coefficient of determination was satisfactory (r^{2} >0.99). The relative standard deviations (RSD %) value under the optimized condition was found to be 8.8%. The limit of detection (LOD) and limit of quantification (LOQ) were 7 and 15 µg L^{-1}, respectively.

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Simple Chemiluminescence Determination of Ketotifen in Pharmaceuticals

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A new method using chemiluminescence (CL) detection has been developed for the, simple determination of ketotifen drug. The method is based on the catalytic effect of ketotifen on the CL reaction of tris(1,10 phenanthroline)ruthenium(II), $[Ru(phen)_3^{2+}]$, with Ce(IV) in sulfuric acid medium. The CL response was detected using a lab made chemiluminometer. Effects of chemical variables were investigated simultaneously using an experimental design and the response surface methodology (RSM). Under optimum conditions, the CL intensity was proportional to the concentration of the drug in solution over the range 0.34-34.00 $\mu g\ mL^{-1}$. The limit of detection (S/N=3) was 0.3 $\mu g\ mL^{-1}$. Effects of common ingredients in pharmaceuticals were investigated on the determination of ketotifen The relative standard deviation for determination of 10 replicates at a level of 3.4 $\mu g\ mL^{-1}$ of ketotifen was 6.0%. The minimum sampling rate was 70 samples per hour. The method was applied successfully to the determination of ketotifen in pharmaceutical formulations.

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دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢
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Simple three variable QSPR model for polychlorinated biphenyls:
Henry's law constant

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Abstract:

The exchange-rate of a chemical between air and water is of great importance in environmental science. Some physicochemical parameters such as vapor pressure and Henry's law constant are used to determine the distribution of a chemical between air and water [1]. Polychlorinated biphenyls are ubiquitous in the global environment because of their biological and chemical stability and their historical widespread use in the power-generation industry (Giesy and Kannan, 1998; King et al., 2000). Experimental determination of Henry's law constant is time-consuming and expensive. Quantitative structure property relationship (QSPR) methodology provides a promising method based on descriptors derived solely from the molecular structure to fit experimental data [2]. The aim of the present study was to construct a predictive QSPR model for interpretation of the relationship between molecular structures of PCB and their corresponding Henry's law constants. Multi linear regression (MLR) method was used for model construction. A data set of molecules including the Henry's constants of 209 PCB compounds that 41 of them were separated as test set. The developed three-parameter linear model which is utilizing the molecular descriptors: BEHv1, GGI2 and VRA2 was constructed based on training set chemicals. The proposed model was completely evaluated by statistical tests such as internal and external validation, leave-one-out cross validation and Y-randomization test. The statistical results of these tests were $R^2_{train} = 0.9681$, $R^2_{pred} = 0.9638$, $Q^2_{Loo} = 0.9658$ and $RMS_{train} = 0.168$ and RMS test = 0.170, which reveal high predictive ability of the proposed model.

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Simultaneous derivatization, extraction and preconcentration of some phenolic compounds by air-assisted liquid–liquid microextraction prior to their determination by GC-FID

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Nowadays, there is an increasing demand for green analytical methods for monitoring organic pollutants such as phenols. These compounds are typically found in domestic and industrial wastewaters, natural waters and potable water supplies [1]. Chlorophenols can be generated as a result of hydrolysis, oxidation, and biodegradation of chlorinated pesticides. They can also be formed during chlorination of drinking water. They are carcinogenic compounds and US Environmental Protection Agency (EPA) has regulated eleven phenolic compounds as priority pollutants, and especially, chlorophenols are known to be most toxic among them. Analysis of phenols in aqueous samples has been done with several analytical methods such as dispersive liquid–liquid microextraction combined with spectrophotometry, solid phase extraction-gas chromatography/mass spectroscopy, supercritical-fluid extraction-gas chromatography [2] and etc. In this work, a new microextraction, of method named air-assisted liquid-liquid microextraction (AALLME) [3] for extraction and preconcentration phenols in water and wastewater is described. Butylchloroformate and 1,1,1-trichlorethane were used as derivatizing agent and extraction solvent, respectively. Some important parameters which affect the extraction efficiency were completely studied. Under the optimum extraction conditions, the method showed wide linear ranges (1-500 µg L^{-1}). Enrichment factors were in the ranges of 568 to 1128, and limits of detection were 0.15-0.42 µg L^{-1}. Relative standard deviations for the extraction of 100 and 200 µg L^{-1} of each selected phenols were in the ranges of 1.7- 4.1 % and 1.27-4.05% for intra-day (n=6) precision. Finally phenols, contents of different samples such as tap water, river water, industrial waste water, and municipality wastewater were determined by the proposed AALLME-GC-FID method.

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Simultaneous detection of ascorbic acid and sudan I using a nanocomposite-based electrochemical sensor

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Abstract: Ascorbic acid is an necessary vitamin with a recommended of daily intake of about 70 mg. Continuing interest in the benefits of a well-balanced vitamin intake has resulted in the fortification of a lot of food products with a variety of vitamins. With insufficient uptake, the symptoms of scurvy appear [1]. On the other hand, Sudan I is a synthetic azo-colourant which has been commonly used as a colouring agent in chemical industries and an additive in foods in daily life, particularly in those containing chilli powders, ketchup, olive oil, because of their intense red–orange colour [2]. In this study, the electrooxidation of ascorbic acid, sudan I and their mixture has been studied using an ZnO/CNTs nanocomposite as a sensor and novel electroactive mediator modified carbon paste electrode using cyclic voltammetry, chronoamperometry, electrochemical impedance spectroscopy and square wave voltammetry methods. Square wave voltammetry of ascorbic acid at the modified electrode exhibited linear dynamic ranges in the concentration ranges of 0.02 to 400.0 µM. The detection limit (3σ) of 0.009 µM ascorbic acid was achieved. The modified electrode successfully resolves the overlapped voltammetric peaks of ascorbic acid and sudan I by ≈210 mV. The RSD% for 5 replicates determination of 0.5 and 20.0 µM of ascorbic acid were 1.2% and 1.0%, respectively, whereas for 20.0 and 30.0 µM sudan I, they were 1.9% and 1.5%, respectively. The modified electrode was used for the determination of these compounds in pharmaceutical and food samples.

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Simultaneous determination of adenine and guanine using Pt-Pd/porous silicon nanocomposite modified carbon nanotubes paste electrode

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An electrochemical sensor was developed for Simultaneous determination of guanine and adenine usingPt-Pd/porous silicon (PSi) nanocomposite as a modifier. The Pt-Pd/PSinanocomposite have been synthesized by chemical etching of silicon powder in a HF/HNO_3 solution [1]followed by electrodlessdeposition of Pt-Pd nanoparticles on the etched silicon.The resulting nanocomposite was characterized by X–ray diffraction, scanning electron microscopy, cyclic voltammetry and FT–IR spectroscopy. Incorporation of Pt-Pd/PSinanocompositein the carbon nanotube paste electrode significantly increases the oxidation peaks currents but reduces the peaks potential of guanine and adenine. The modified electrode was employed for detecting purine bases using differential pulse voltammetry.The effects of different parameters such as pH, electrode composition and potential scan rate were investigated.Using differential pulsevoltammetry, we could measure guanine and adenine in one mixture independently from each other by a potential difference of about 300 mV. Differential pulsevoltammetric peaks current of guanine and adenine increased linearly with their concentrations in the ranges of 0.1–14.0 µM, and 0.4–14.0 µM respectively. The detection limits of 50 nM, and 200 nMwere achieved for guanine and adenine, respectively. Finally, the proposed electrochemical sensor was employed to determine guanine and adenine in single-strand deoxyribonucleic acid (ssDNA) samples with satisfactory results.

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Simultaneous determination of Ascorbic acid, dopamine and uric acid with application of Silver nanoparticles-polypyrrole nanocomposite modified Glassy Carbon Electrode

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Ascorbic acid (AA), dopamine (DA) and uric acid (UA) are biologically important compounds in human metabolism. Metal nanoparticles exhibit excellent optical and electronic properties because of their high surface area to volume ratio and exhibit excellent catalytic activity than the respective bulk metals. Among other nanoparticles, silver nanoparticles have gained much interest due to its applications in catalysis, anticancer activity, optical sensor and as a potential antibacterial agent. Nanocomposites derived from silver nanoparticles dispersed in polypyrrole (PPy) are particularly important due to their excellent electrocatalytic properties, sensor and supercapacitors applications [1-3].

The electrochemical behavior and electrocatalytic activity of silver nanoparticles/PPy/GCE were characterized by cyclic voltammetry and electrochemical impedance spectroscopy. The morphology of electrodes was characterized by scanning electron microscopy. The modified electrode was successfully used to simultaneously determine AA, DA and UA. In pH 4.0 PBS, at a scan rate of 100 mV/s, the modified electrode gave three separated oxidation peaks at 110 mV, 350 mV and 440 mV for AA, DA and UA, respectively. The peak potential differences were 240 mV and 90 mV. The sensor showed excellent sensitivity, selectivity, and stability. The proposed method could be applied to the determination of DA, AA and UA in real samples with satisfactory results.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ۶ الی ۸ اسفند ماه ۱۳۹۲

Simultaneous determination of ascorbic acid, epinephrine and uric acidby poly(*p*-aminophenol)/graphenenanocomposite modified glassy carbon electrode

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Abstract

A nanocomposite of poly(*p*-aminophenol) /graphenemodifiedglassy carbon electrode (P(*p*AP)/Gr/GCE)was prepared byelectropolymerization techniquefor the simultaneous determination of ascorbic acid (AA), epinephrine (EP) anduric acid (UA). Surface morphology and electrochemical properties of the prepared nanocomposite modified electrode were investigated by scanning electron microscopy (SEM), cyclic voltammetry and electrochemical impedance spectroscopy techniques.The cyclic and differential pulse voltammetric methods were used to investigate the modified electrode for the electrocatalytic oxidation of AA, EP and UA in the aqueous solution. The separation of the oxidation peak potentials for EP–AA and EP–UA was about 150 mVand 200 mV, respectively.Under the optimum experiment conditions, the linear current responses were obtained in the ranges of 5–1100 µM, 0.05–350 µM and 0.05–400 µM for AA, EP and UA, respectively.Thedetection limitsof 0.9, 0.01 and 0.009µM(S/N = 3)were obtained forAA, EP and AA, respectively.The modified electrode was also successfully used for the simultaneous determination of EP and AA in real samples.

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Ulnjt9Sv

Simultaneous determination of caffeine and gallic acid in green tea using photoluminescence spectroscopy and chemometric methods

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Green tea is considered as a dietary source of antioxidant nutrients which acts upon human health. Green tea leaves that contain three main components in the form of simple hydroxy benzoic acids such as gallic acid, propyl gallate and xanthic bases (caffeine), have been reported to prevent or delay a number of many degenerative disease and act mainly upon the central nervous system and stimulating wakefulness[1]. Therefore, it is important to establish a simple and reliable analytical method for determination of these compounds in the presence of unexpected interferences in green tea sample. Multi-way models (e.g PARAFAC, APTLD), can be used to study such data, providing estimates of the spectra and concentration profiles of the underlying chemical analytes if the data are approximately trilinear. Excitation wavelengths between 240-335 nm with 5 nm intervals and emission wavelength between 250 to 496 nm with 0.5 nm intervals was selected for the samples. After performing parallel factor analysis, obtained results are 0.045 gr/gr gallic acid in green tea with R^2 =0.9335 and 0.317 gr/gr caffeine in green tea with R^2 =0.9695 and for alternating penalty trilinear decomposition, 0.054 gr/gr gallic acid in green tea with R^2 =0.9472 and the 0.332 gr/gr caffeine in green tea with R^2 =0.9841. In this research, a rapid, simple and selective method was used for the direct determination of caffeine and galic acid in green tea that works based on photoluminescence spectroscopy data. This method enabled us to handle the direct interfering effect in green tea matrix [2, 3].

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Simultaneous determination of captopril and acetaminophen at a modified carbon nanotubes paste electrode

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Captopril (CAP) with the chemical name of (2S)-1-[2S)-2-methyl-3-sulfanylpropanoyl] pyrrolidine-2-carboxylic acid, is an angiotensin-converting enzyme inhibitor and has been widely used as antihypertensive drug. CAP is used to treat high blood pressure heart failure. It decrease certain chemical that tighten the blood vessel, so blood flows more smoothly and the heart can pump blood more efficiently [1]. In this study, a carbon paste electrode modified with carbon nanotubes and with 7-(3,4-dihydroxyphenyl)-10,10-dimethyl-9,10,11,12-tetrahydrobenzo[c]acridin-8(7H)-one was used to prepare a novel electrochemical sensor. One of the very important and potent characteristics of the proposed electrode is its ability to lower the overpotential of CAP, and to increase the rate of electron transfer processes, which is believed to be due to the modification of the nanostructure of the electrode surface by using carbon nanotubes and hydroquinone derivative. Cyclic voltammetry was used to investigate the redox properties of this modified electrode at various scan rates. The apparent charge transfer rate constant, k_s, and transfer coefficient, α, were calculated. The objective of this novel electrode modification was to seek new electrochemical performances for detection of CAP. The peak potential of CAP oxidation at the surface of modified electrode shifts by about 490 mV toward negative values compared with that at the bare electrode. Also the peak crrent of CAP increased at modified carbon paste electrode. The values of electron transfer coefficients (α), catalytic rate constant (k), diffusion coefficient (D) and detection limit were calculated for hydroxylamine, using electrochemical approaches [2].

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Simultaneous determination of deltamethrin and permethrin in water samples using homogeneous liquid-liquid microextraction via flotation assistance and gas chromatography

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Abstract

Pyrethroids are non-systemic insecticides and acaricides which are used for the control of a wide range of pests in cereals, ornamentals, vegetables, cotton, and other crops. Their residues will represent a serious hazard to human health such as cancer, infertility, nerve disorders, immunological and respiratory diseases [1]. A new method [2] was developed for simultaneous determination of deltamethrin and permethrin in water samples with homogeneous liquid-liquid microextraction via flotation assistance (HLLME-FA) and gas chromatography-flame ionization detection (GC-FID). In this research, a special extraction cell was designed to facilitate collection of the low-density solvent extraction. The sample solution was added into the extraction cell, which contained appropriate mixture of n-hexane (as extraction solvent) and acetone (as homogeneous solvent). Using air flotation, extraction solvent was collected from the top of the solution. Under the optimum conditions, good linearity was observed in a range of 1.0 -200 μg L^{-1} with the correlation coefficient (r^2)> 0.9980 for both of analytes. The limits of detection (LODs) were 0.2 and 0.3 μg L^{-1} for deltamethrin and permethrin, respectively ($S/N=3$). The developed method was successfully applied to determine the two pesticides in three different water samples.

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Simultaneous determination of epinephrine and uric by new molecular self-assembled monolayers on gold electrode characterized by EIS, QCM, STM and CV techniques

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Abstract

In the present paper, we use a gold electrode modified by 2-(3, 4-dihydroxy phenyl) benzothiazole self-assembled monolayer (DHT-SAM) for the determination of epinephrine (EP) and uric acid (UA). Initially, DHT-SAM was characterized by cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), scanning tunneling microscopy (STM) and quartz crystal microbalance (QCM) techniques. CV was used to investigate the redox properties of the modified electrode at various scan rates. The apparent charge transfer rate constant, k_s and transfer coefficient (α) were calculated. Next, electro oxidation of epinephrine (EP) and uric acid (UA) on a gold electrode modified by a self-assembled monolayer of DHT mediated was investigated. At the optimum pH of 7.0, the oxidation of EP occurs at a potential about 200 mV less positive than that of an unmodified gold electrode. The values of transfer coefficients ($\alpha = 0.35$), catalytic rate constant ($k = 4.4 \times 10^4\ M^{-1}s^{-1}$) and diffusion coefficient ($D = 1.03 \times 10^{-6} cm^2s^{-1}$) were calculated for EP, using electrochemical approaches. Differential pulse voltammetry (DPV) exhibited a linear dynamic range over the concentration range of 0.5–400.0 µM and a detection limit (3s) of 0.11 µM for EP in pH= 7. Finally, simultaneous determination of EP and UA at the modified electrode was described and used for the determination of EP in EP ampoule.

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Simultaneous Determination of Glutathione and Cysteine Based on Fluorescence Resonance Energy Transfer between Gold Nanoparticles and FITC

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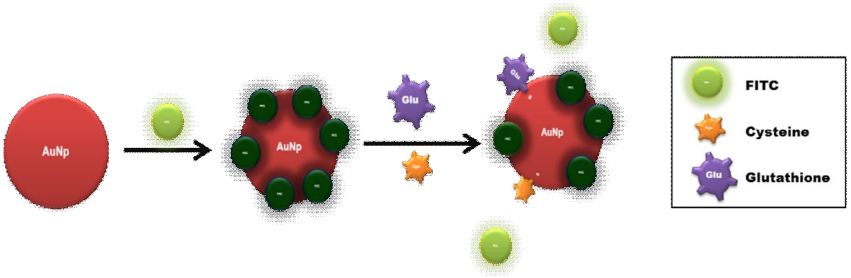
Turn-On Fluorescence detection is a selective and sensitive method for many analytical purposes. Fluorescence quenching is a commonly observed consequence when fluorophores and gold nanoparticles (Au NPs) are in same media. Here, we report a “turn-on” fluorescent nanoprobe for simultaneous determination of glutathione and cysteine using H-point standard addition method (HPSAM). This probe develops based on the fluorescence resonance energy transfer (FRET) between Au NPs and fluorescein isothiocyanate (FITC), in which FITC acts as the donor and Au NPs as the acceptor. FRET, electron transfer processes and inner filter effect (IFE) cause the fluorescence intensity of FITC strictly quenched as a result of noncovalently adsorbed on AuNPs surface [1, 2]. This probe operated on a basic principle that glutathione and cysteine have stronger affinity to Au NPs, so deposit on the surfaces of the Au NPs and induce the release of FITC molecules into the solution with different rates, and thus restored the florescence signal of FITC [2, 3]. We try to omit IFE signal in every part of our work using UV-Vis spectra and mathematical calculations. The H-point standard addition method (HPSAM) was applied to kinetic data for simultaneous determination of glutathione and cysteine. Under the optimum conditions (i.e. pH=8.5, C_{AuNPs}=0.6 nM), the fluorescence intensity of the released FITC displays a linear relationship in the range of 20 to 200 nM of Glutathione in the presence of cysteine. The proposed method was successfully applied to the determination of glutathione in the presence of different concentration of cysteine in several synthetic mixtures.



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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

Simultaneous determination of isoprenaline and acetaminophen at a TiO_2 nonastructured cabon paste electrode

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Isoprenaline (IP) is an important neurotransmitter used for the treatment of neural disorders such as Parkinson disease. The IP cardiovascular effects, which are usually compared to those of adrenaline and noradrenaline, showed that it can successfully relieve nervous tension from almost every kind of smooth musculature [1]. Application of nanomaterials in various fields of science and technology has been extensively developed due to the unique properties of these materials. TiO_2 nanoparticles are an attractive, biocompatible and environmentally benign material widely used in toothpaste and cosmetics. The major barriers involve the low solubility and poor stability of TiO_2 nanoparticles and the TiO_2 film on the electrodes [2]. In this study, a carbon paste electrode modified with TiO_2 nanoparticles and 7-(3,4-dihydroxyphenyl)-10,10-dimethyl-9,10,11,12-tetrahydrobenzo[c]acridin-8(7H)-one and was used for simultaneous determination of IP and acetaminophen. Cyclic voltammetry was used to investigate the redox properties of this modified electrode at various scan rates. The apparent charge transfer rate constant, k_s, and transfer coefficient, α, were calculated. The peak potential of IP oxidation at the surface of modified electrode shifts by about 340 mV toward negative values compared with that at the bare electrode. Also the peak current of IP increased at modified TiO_2 carbon paste electrode. The values of electron transfer coefficients (α), catalytic rate constant (k), diffusion coefficient (D) and detection limit were calculated for IP, using electrochemical approaches.

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simultaneous determination of levodopa and uric acid by chemical and electrochemical deposition of gold nanoparticles and reduced graphen at glassy carbon electrode for formation of self-assembled monolayers

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Abstract: A novel layer-by-layer assembly of graphene Sheets, gold nanoparticles and 2-(2, 3-dihydroxy phenyl) benzothiazole (DPB) modified glass carbon electrode (GC/GR/AuNPs/DPB) was successfully fabricated by chemical and electrochemical depositionnanoparticles of gold and reduced graphen at glassy carbon electrode and applied to simultaneous determination of levodopa (LV) and uric acid (UA).It was observed that electrochemical deposition nanoparticles of gold and reduced graphen have higher electrocatalyticactivity for the oxidation of LV and UA when compared with chemical deposition. Simultaneous determination of LV and UA on GC/GR/AuNPs/DPB was conducted with a differential pulse voltammetry technique, and two well defined and fully resolved anodic oxidation peaks were observed. This newelectrode due to large surface area and high electron transfer ability presented very large current response from electroactive substrates.

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Simultaneous determination of thiocyanate and oxalate in urine using a carbon ionic liquid electrode modified with TiO_2-Fe nanoparticles

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Abstract: A carbon ionic liquid electrode (CILE) [1] modified with TiO_2-Fe nanoparticle [2] was employed for simultaneous determination of thiocyanate and oxalate by differential puls voltammetry. The electrocatalytic oxidation of 1.00×10^{-3} mol L^{-1} thiocyanate and 1.00×10^{-3} mol L^{-1} oxalate was investigated with this electrode in 0.04 mol L^{-1} universal buffer (pH 7.0). For the preparation of the electrode, a desired weight of TiO_2-FeNPs was added to graphite powder and ionic liquid so that the weight ratio was 45:45:10, in order, and the electrode was then heated in an oven at 80 °C for 2 minutes. After optimizing the operational conditions, a linear range of 1.00×10^{-5} to 1.75×10^{-3} mol L^{-1} for thiocyanate with a detection limit of 6.38×10^{-6} mol L^{-1} and a linear range of 5.00×10^{-5} to 3.00×10^{-3} mol L^{-1} for oxalate with a detection limit of 2.31×10^{-5} mol L^{-1} were obtained. The electrode showed good reproducibilities for both thiocyanate and oxalate with relative standard deviations of 2.19% and 0.232%, respectively. The repeatabilities were 4.40% and 6.36%, in the same order. The fouling effect and possible interferences were also investigated. The anodic transfer coefficient was calculated as 0.448, 0.439, respectively, for thiocyanate and oxalate. The method was successfully applied for analysis of thiocyanate and oxalate spiked in urine samples and good recoveries were obtained.

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Simultaneous determination of uric acid and ascorbic acid at the surface of bare carbon paste electrode by differential pulse and chemometrics

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Ascorbic acid (AA), Vitamin C, an essential nutrient found mainly in fruits and vegetables and one of the most important cellular antioxidants. The major problem with simultaneous determination of UA and AA by electrochemical methods is the closeness of the oxidation potentials of these two compounds which results in an overlapped voltammetric response which makes their signal discrimination very difficult. In addition, AA oxidation at bare electrode is very often suffers from fouling effects [1].

Differential pulse voltammetry was applied for the simultaneous determination of mixture of uric acid and ascorbic acid at the surface of bare carbon paste electrode. The electrooxidation of these compounds at bare electrode is sluggish, and there is no suitable peak separation between them. It relies on oxidative reaction of uric acid and ascorbic acid at a carbon paste electrode in a phosphate-citrate buffer (pH=4). The peak voltammograms of UA and AA show high overlap, and non-linear characteristics. Thus, it is difficult to determine the compounds in a mixtures by using voltammetric method in a bare electrode.

In this approach, chemometrics strategies such as classical least squares (CLS), principal component regression (PCR) and partial least squares (PLS) regression have been applied to determine uric acid in the presence of ascorbic acid without electrode modification. The linear ranges for uric acid and ascorbic are 1–1000 μM and 5–1000 μM , respectively. The effects of the different variables on the sensitivity and selectivity are discussed.

Key word: Carbon paste electrode, Ascorbic Acid; Uric Acid, Partial least squares, Differential pulse voltammetry.

References:

[1] M. Amiri, A. Bezaatpour, Z. Pakdel, K. Nekoueian, *J Solid State Electrochem*. 16 (2012) 2187-2195.

Simultaneous determination of uric acid and ascorbic acid at the surface of bare carbon paste electrode by differential pulse and chemometrics

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Ascorbic acid (AA)**,** Vitamin C, an essential nutrient found mainly in fruits and vegetables and one of the most important cellular antioxidants. The major problem with simultaneous determination of UA and AA by electrochemical methods is the closeness of the oxidation potentials of these two compounds which results in an overlapped voltammetric response which makes their signal discrimination very difficult. In addition, AA oxidation at bare electrode is very often suffers from fouling effects [1].

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Simultaneous electrochemical determination of epinephrine, uric acid and tyrosine using carbon ceramic electrode modified by cobalt acetylacetonate complex

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Epinephrine (EP) is one of the most important neurotransmitters in mammalian central nervous systems, which exists in the nervous tissue and body fluids as large organic cations. This compound controls the nervous system in its performance for a series of biological reactions and nervous processes [1]. Determination of EP in the presence of UA, because of their coexistence in human fluids, is attractive to biological and analytical researchers.

Carbon ceramic electrode (CCE) has been utilized to a great extent for electrochemical sensors, whose electrode surfaces could be renewed by a simple polishing step. Beside graphite particles gold, glassy carbon and multiwalled carbon nanotube particles also have been used as conducting materials for ceramic composite electrodes [2].

In this study, a new modified CCE with acetylacetonate complex (Co-L3) for the simultaneous determination of epinephrine (EP), uric acid (UA) and tyrosine (Tyr) has been described. The electrocatalytic behavior of EP, UA and Tyr was studied in pH 1.0 chloroacetic acid buffer solution by cyclic voltammetry (CV) using the modified CCE. This modified electrode exhibits potent and persistent electroxidation behavior followed by well-separated oxidation peaks towards EP, UA and Tyr with increasing of the oxidation current. The catalytic peak current obtained, was linearly dependent on the EP, UA and Tyr concentrations in the range of 33-434μM and the detection limits for EP, UA and Tyr were 4.2, 5.0 and 4.5 μM, respectively. The analytical performance of this sensor has been evaluated for simultaneous detection of EP, UA and Tyr in human serum and urine samples.

Keyword: Modified carbon ceramic electrode, Eepinephrine, Uuric acid, Tyrosine, Simultaneous electrochemical determination

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Simultaneous electrochemical determination of hydroquinone and catechol at modified carbon paste electrode Based on Au@SiO$_2$ nanoparticle

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Catechol and hydroquinone are two dihydroxybenzene isomers, which are widely used in pesticides, medicines, antioxidant, dye, and photography chemicals. They are considered as environmental pollutants by the US Environmental Protection Agency (EPA) due to their high toxicity and low degradability in the ecological environment. These isomers are often coexisting inenvironmental samples. Therefore, it is necessary to develop simple and rapid analytical method for simultaneous determination of dihydroxybenzene isomers [1].

Recently the application of the carbon paste electrodes modified with nanostructured materials exhibit considerable improvements in electrochemical behavior of many important compounds. Chemically modified electrodes involving silica based materials have attracted much attention in the field of electroanalysis [2]. Silica-coated gold nanorods (GNRs@SiO2) are a kind of novel biocompatible nanocomposite, which has been widely used as the electrochemical sensors.

In this work carbon paste electrodes (CPE) modified with Au@SiO$_2$ nanoparticles were used for the sensitive simultaneous electrochemical determination hydroquinone (HQ) and catecol (CT). The electrode prepared by mixing graphite powder, paraffin oil and Au@SiO$_2$ nanoparticles. The electrochemical response characteristics of these modified electrodes toward HQ and CT were investigated by cyclic voltammetry and differential pulse voltammetry. Prior to establish the detection method experimental variables were optimized. Under the optimum experimental conditions the peak current was linearly dependent on the HQ and CT concentration in the range of 10×10^{-6} to 1×10^{-3} mol $L^{-}1$ and 30×10^{-6} to 1×10^{-3} for HQ and CT, respectively. Finally, the method was successfully utilized for determination of HQ and CT in a real samples.

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Simultaneous kinetic spectrophotometric determination of butylated hydroxytoluene and butylated hydroxyanisole using N-PLS regression

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Antioxidants play a significant role in retarding the lipid oxidation reactions in food products, since oils and fats, tend to encounter problems of oxidation and rancidity, which affect food quality and may endanger our health. To prevent lipid food peroxidation, manufacturers prefer to use synthetic phenolic antioxidants such as butylated hydroxytoluene (BHT), butylated hydroxyanisole (BHA) because of their chemical stability, low cost and availability [1]. Usually, the maximum limit of BHT, BHA should be no more than 200 mg/kg in vegetable oils, either single or in combination. Thus, determination of synthetic antioxidants in foods is necessary to ensure the fulfillment of the legal requirements and quality control procedures in the food industries. In this study, a spectrophotometric method for the simultaneous determination of two preservatives, BHA and BHT is described based on their different kinetic properties. These antioxidants react differentially with Fe(III), and the reduced product of which, Fe(II), will be complexed by chromogenic reagent 2,2-dipyridyl. The absorption spectra were measured in the wavelength range of 420–560 nm. Three-way data matrices were generated by acquisition of visible spectra (420–560 nm) as a function of time for mixtures with different concentrations of BHA and BHT. The concentrations of the calibration matrix ranged from 0.1 to 35.0 mgL^{-1} for BHT, and from 0.1 to 28.0 mgL^{-1} for BHA. The N-PLS modeling was used for regression and the relative errors of prediction (REP) for the validation set were 7.5%, 9.1% for BHA and BHT, respectively. The new method was successfully utilized to determine the amounts of BHA and BHT in real vegetable oils without the need for tedious pretreatments.

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20th Iranian Analytic Chemistry Conference
25–27 Feburay

Simultaneous Preconcentration Flotation-Separation and CVAA-FAE-FAA Spectrometry Determination of Hg(II), Al(III), Fe(III), Cd(II) and Ag(I) In Real Samples

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Flotation as a preconcentration method has unique advantages such as rapidity and excellent recoveries of investigated trace elements, simple and inexpensive equipments and ability to analyze a large volume of sample solution and to obtain a greater preconcentration factor than the conventional carrier precipitation technique [1]. Precipitates and ions in aqueous solutions can be floated to the solution surface by a rising stream of gas bubbles, with (or sometimes without) the aid of surfactants [2]. Multicharged anionic complexes, containing an analyte element, form sparingly soluble ion-association compounds with singly-charged cations of some basic dyes [3]. We have developed a procedure for the simultaneous determination of trace levels of Hg(II), Al(III), Fe(III), Cd(II) and Ag(I) in the real samples based on precipitate flotation with dye of bromocresol green (figure 1) as a reagent ion-association.

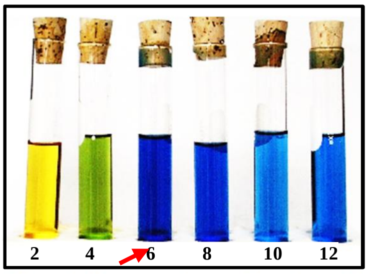
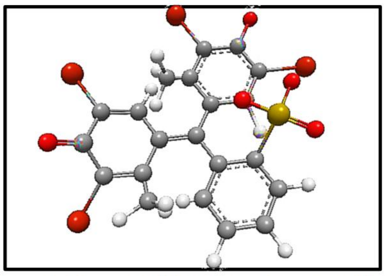
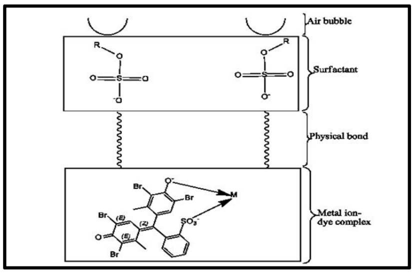


Figure 1. Bromocresol Green in various pH



Scheme 1. Structure of bromocresol green at pH=6



Scheme 2. Structure of the dye-metal ion-SDS system

In this research cold vapor atomic absorption (CVAA), flame emission atomic (FEA) and flame atomic absorption (FAA) spectrometry were used as an instrumental method for a quantitative and sensitive determination. The precipitates were floated with the aid of tiny air bubbles and supported by the stable foam layer of sodium lauryl sulfate (SDS) (Scheme 2) and influence pHs, type and amount of surfactant, metal ions concentration, ionic strength, the presence of foreign ions and air flow rates for better efficiency of flotation process were examined. Good results were obtained under the optimum conditions, according to which nearly 98% of investigated metal ions, at pH 6 and at room temperature (~25°C), in presence of KNO_3 0.04 mol L^{-1} as electrolyte and quantitatively eluted with 4 mL of 1M HNO_3 in methanol were recovered. The preconcentration factor of the method is 212.5, the detection limits (n=5) of Hg(II), Al(III), Fe(III), Cd(II) and Ag(I) ions are 0.014, 0.8, 1.73, 0.34 and 0.3 ng mL^{-1} and the RSD (n=5) 0.9-2%, respectively. Beer's law was obeyed over the concentration range 0.04-0.64 ng mL^{-1} for Hg(II) and 8-80 ng mL^{-1} for other investigated metal ions. This flotation technique is simple, rapid and also applicable to the determination of trace elements at low ppb levels.

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Simultaneous Removal of Anionic dyes in wastewater samples using Surfactant modified TiO_2 nanoparticles

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Abstract

The utilization of modified TiO_2 nanoparticles with a cationic surfactant (cetyltrimethylammonium bromide (CTAB)) as a novel and efficient adsorbent was successfully carried out to remove two anionic dyes from aqueous solutions in single and binary systems. Alizarin Red (AR) and Indigo Carmine (IC) were used as anionic dyes. The influence of various factors such as initial pH, initial concentration and sorbent mass on the dye adsorption was investigated [1]. In addition, statistical experimental design was utilized to optimize removal of dyes by CTAB-modified TiO_2 nanoparticles through a batch adsorption process. As a result, a regression model was derived using a response surface methodology (RSM) through performing the 416B model of hybrid design [2]. Model adequacy was checked by such diagnostic tests as analysis of variance (ANOVA), lack of fit test and residuals distribution. The proposed quadratic model resulted from the hybrid design approach fitted very well to the experimental data. Hybrid design was applied to find out a suitable model that led to optimum outcome conditions [3]. The optimal conditions for dye removal were pH =2, Sorbent dose = 0.05g, C_{AR} (Initial AR concentration) =232 mg/L and C_{IC} (Initial IC concentration) =298 mg/L. Then isotherms and kinetics of adsorption were also investigated.

Keywords: Indigo carmine, Alizarin red, CTAB-modified TiO_2 nanoparticles, Hybrid design.

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Simultaneous Spectrophotometric Determination of Lisinopril and Argenine Based on Charge-Transfer Complex Formation with 1,2-Naphthoquinone-4-sulfunate

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Lisinopril is a member of angiotensin-converting enzyme (ACE) inhibitor's family. ACE inhibitors have been shown to be very effective and universally well-tolerated for the long-term treatment of hypertension and congestive heart failure.

Arginine -the natural precursor of nitric oxide- with lisinopril is combined to complete imperfect effect of lisinopril in kidney disease with balancing endothelin/NO ratio. Accordingly, the simultaneous determination of Lisinopril and Argenine is important. In other words, this work can be a pattern for simultaneous determination of other aminoacids.

1,2-Naphthoquinone-4-sulphonate (NQS), reacts with both primary and secondary amines and amino acids, non-selectively [1]. Hence spectrum of reaction product of lisinopril with NQS is very similar to that of arginine. Linear ranges for lisinopril and arginine are between 10 to 500 µM.

Hence these amino acids cannot be determined in the mixture directly. However, since reaction kinetics of lisinopril and arginine with NQS are different, multivariate calibration methods are suitable and applicable. Due to nonlinear effects in the kinetics of mixtures, artificial neural network (ANN) – a method for non-linear multivariate calibration – was selected [2].

For the application of neural networks, the kinetic curves corresponding to 40 binary mixtures of the considered analytes were obtained. And randomly splitted into three set consisting of training (18 mixture), test (12 mixture) and validation (10 mixture) set.

Principal component analysis (PCA) was applied to the raw of data matrix previous to ANN in order to reduce the volume of data and network running time . The optimum number of scores as inputs and concentration compositions as targets were applied to ANN. The network was trained and its related parameters were optimized with training and test sets. The prediction ability of constructed network then was investigated by validation set.

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Simultaneous ultrasound assisted removal of sunset yellow and erythrosine by gold nanoparticles loaded on activated carbon: central composite design

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Abstract

The present study was focused on the simultaneous ultrasound assisted removal of sunset yellow and erythrosine dyes from aqueous solutions by gold nanoparticles loaded on activated carbon (Au-NP-AC). Synthetic dyes represent a relatively large group of organic chemicals that are discarded by industries. Because of their high organic nature and intense colors, the effluents discarded by the food industry lead to entrance of huge amount pollutant to water bodies [1,2]. Therefore, it becomes necessary to removal these materials from such industrial effluents to produce a safe and clean environment. In this study, gold nanoparticle was synthesized and characterized using different techniques such as FESEM, XRD and TEM. Adsorption experiments were performed in a cylindrical glass vessel by addition of adsorbent (0.035 g) in to 50 mL of SY and erythrosine solutions at known concentration (25 and 25 mg L^{-1}) and pH 3.5. The vessel was immersed in an ultrasonic bath for 4.0 min sonication time at the room temperature. After this time, solutions were analyzed for the final concentration of dyes by using a UV-Vis spectrophotometer set at a wave-length of 431 and 520 nm for SY and erythrosine, respectively. The effects of variables such as sonication time, pH, initial dyes concentrations and adsorbent dosage on removal percentage were studied and optimized by central composite design (CCD) combined with response surface methodology (RSM) and desirability function (DF). A good agreement between experimental and predicted data using optimal model in this study was observed. The small amount of proposed adsorbent (0.035 g) is applicable for simultaneous removal of sunset yellow and erythrosine (25 and 25 mg L^{-1}) in short time (4.0 min) with high percentage removals (>99 %). Fitting the experimental equilibrium data to various isotherm models such as Langmuir, Freundlich, Tempkin and Dubinin-Radushkevich models show the suitability and applicability of the Langmuir model. Kinetic models such as pseudo first and pseudo-second order, Elovich and intraparticle diffusion models applicability was tested for experimental data and the second-order equation control the kinetic of the adsorption process.

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Simultaneous UV-VIS spectrophotometric determination of dextromethorphan andPseudoephedrine in pharmaceutical preparations

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Background and Aims: Dextromethorphan and pseudoephedrine combination is used to treat pulmonary tract inflammations. There are a lot of combination of dextromethorphan pseudoephedrine in the pharmaceutical market that mostly contains 15 mg of dextromethorphan and 30 mg of pseudoephedrine in each 5ml of the product. On the basis of our knowledge there aren't any reports regarding the simultaneous determination of the two drugs in combination by spectrophotometric method, therefore development of a simple, fast and inexpensive spectrophotometric method can be very useful for quality control laboratories. The main aim of this study was to develop an spectrophotometric method for simultaneous determination of dextromethorphan and pseudoephedrine in pharmaceutical products especially oral solution dosage forms[1].

Methods: Standard solutions of dextromethorphan and pseudoephedrine in the concentration range of 10-60 mg/L and 5-30 mg/L in distilled water were prepared, respectively. Absorption spectra of the solutions were drawn in the wavelength range of 200-400nm. Absorption spectra of the mixed standard solution of dextromethorphan and pseudoephedrine were also drawn to find a wavelength for simultaneous determination of the two drugs without any further pretreatment or separation steps. Validation parameters like linearity range, precision, accuracy, LOD, LOQ, stability of the solutions and interferences were studied. The developed and validated method was used to determine the drugs in real samples of pseudoephedrine and dextromethorphan –p as combination product present in local market.

Results: Results depicted fourth derivation at 291 nm and 256.01 nm are optimum for the determination of dextromethorphan and pseudoephedrine, respectively. Results of intra-day and inter-day precision showed that RSD% for dextromethorphan was less than 0.48 and 2.24%, and for psudoephedrine was less than 1.31 and 2.63%, respectively

Conclusion:Results show that the developed methods are proper, simple, cheep, fast, and sensitive for the simultaneous determination of dextromethorphan and pseudoephedrine. The method can be used in routine analysis of the drugs in simple and combination products in quality control laboratories.

Keywords: dextromethorphan , Pseudoephedrine, UV-Vis spectrophotometry

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

Simultaneous Voltammetric Determination of Acetaminophen and Ascorbic Acid Using a Glassy Carbon Electrode Modified with Carbon Nanotube and a Schiff-Base Complex of Copper

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Carbon nanotubes (CNTs), as a novel form of carbon, have recently come to forefront of chemical research due to their interesting electronic, chemical and mechanical properties [1]. Ascorbic acid (AA) and Acetaminophen (AC) plays an important role in human health [2]. At traditional electrodes, AA is oxidized at potentials close to that of AC, resulting in an overlapping voltammetric response [3]. A mixture of multiwalled carbon nanotube (MWCNT) and N,N-bis(5-nitrosalicylidene)-2,2dimetyl-1,3-propan-diamin copper(II) complex is applied for the modification of Glassy Carbon electrode (GCE). The modified electrode is used as a sensitive voltammetric sensor for simultaneous determination of AA and AC. The electrode showed efficient electrocatalytic activity in lowering the anodic overpotentials and enhancement of the anodic currents. The effects of potential sweep rate and pH of the buffer solution on the response of the electrode, toward AC and AA, and the peak resolution is thoroughly investigated by cyclic and differential pulse voltammetry (CV and DPV). The best peak resolution for these compounds using the modified electrode is obtained in solutions with pH 4.0. Peak potential separation nearly 418 mV is resulted for two compounds. The modified electrode showed good sensitivity and selectivity. The high sensitivity and selectivity and very low detection limit (sub-micromolar) together with the very easy preparation and surface regeneration of the modified electrode and the reproducibility of the voltammetric response make the prepared modified system very useful in the construction of simple devices for the simultaneous determination of AA and AC in clinical and pharmaceutical preparations.

Keywords: Carbon Nanotube, Ascorbic acid, Acetaminophen, Cyclic Voltammetry, Differential Pulse Voltammetry

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Simultaneous Voltammetric Determination of Epinephrine and Acetaminophen using Modified Carbon Nano Composite of $ZnFe_2O_4/TiO_2$ Paste Electrode

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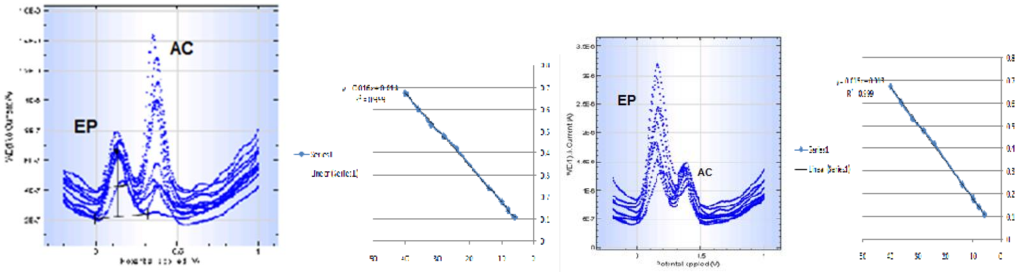
Abestract

Epinephrin(EP) is an important neurotransmitters in mammalian central nervous systems[1], and it exits in the nervous tissue and body fluid in the form of large organic cations.The change of its concentration may result in many diseases[2].Acetaminophen(AC) is a widely used analgesic and antipyretic medicine[3].AC cotains phenolic hydroxyl group, which is electrochemical active and can be oxidized.A carbon paste electrode(CPE) modified with $ZnFe_2O_4/TiO_2$, was prepared for determination of epinephrin(EP) in the presence of asetaminophen(AC). The electrochemical response characteristics of the modified electrode toward EP and AC were in vestigated by differential pulse voltammetry(DPV).Nano composite $ZnFe_2O_4/TiO_2$ in 0.1 M phosphate buffer solution(pH; 8) concentration of EP and AC mixed solutions of (5,10,20,25,30,44,46,52,62,80)µM EP in the presence of AC(30µM) and the second value is concentration of AC (6,8,10,14,24,28,32,36,40) µM in the presence of EP(10)µM.



Keyword: Epinephrin,Acetaminophene, Carbon Paste Electrode

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Solar Rechargeable Redox Flow Batteries

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Abstract:

Unlike the conventional conversion between electrical energy and chemical energy from fossil sources, solar energy is environmentally benign, abundant, and renewable. Solar cells are efficient devices that can convert solar energy to electrical energy, thanks to various photovoltaic technologies. Such solar-generated electrical energy can be stored in external rechargeable batteries [1].

In contrast, solar storable rechargeable batteries, in which the solar energy can be converted and stored directly, are highly desired for the utilization of solar energy. The practical application of suchdevices depends upon the discovery of inexpensive and abundantmaterials with an energy gap which matches the solar spectrumand which are capable of stable operation in solution over anextended period.

A redox flow battery (RFB) is a type of rechargeable battery that stores electrical energy, typically in two soluble redox couples contained in external electrolyte tanks sized in accordance with application requirements. Liquid electrolytes are pumped from storage tanks to flow-through electrodes where chemical energy is converted to electrical energy (discharge) or vice versa (charge) [2-4]. Vanadium redox flow batteries (VRBs) exploit the capability of vanadium to exist in solution infour different oxidation states and use this property to make a flowbattery that has only one active element in both anolyte and catholyte [5].If the energy conversion and storage from solar energy to electrical energy can be carried out in a redox flow cell, this would offer a new method for solar-generated electricity storage [6-8].

In this presentation, recent researches in the field of solar rechargeable battery are reviewed in general and feasibility of photoelectrocatalysis for utilizing light in charging the vanadium redox flow battery is covered in more details.

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Sol–gel/nanoclay composite as a solid-phase microextraction fiber coating for the determination of organophosphorus pesticides in water samples

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A novel SPME fiber coated with sol–gel silica/nanoclay composite was prepared. Clay/silica nanocomposites were prepared by a sol–gel technique involving hydrolysis reaction of alkoxysilanes and subsequent condensation reaction with hydroxyl groups of the nanoclay [1]. A method based on DI-SPME and gas chromatography corona discharge ion mobility spectrometry for the determination of OPPs in aqueous samples was developed. Commonly used fragile fused silica fiber was replaced with stainless steel wire, which made the fiber unbreakable. The new fiber possessed a homogeneous and porous surface; it also showed an excellent thermal and good stability. The Effect of different parameters influencing the extraction efficiency including extraction time and temperature, ionic strength, stirring rate, the amount of nanoclay, desorption time and desorption temperature were investigated and optimized. The SEM images revealed that the new fiber exhibited a rather porous and homogenous surface. The sol gel/nanoclay was characterized by FT-IR and the thermal stability of the fabricated fiber was investigated by TGA. Under the optimized conditions, the new coatings showed higher extraction efficiency and sensitivity for OPPs compared with Ppy/nanoclay and some commercial fibers (PDMS, PDMS/DVB and PA). Under the optimal conditions, LOD of the method was in the range of 0.003-0.012 ng mL^{-1} and LOQ was between 0.01 and 0.02 ng mL^{-1}, respectively. The calibration curves were linear in the concentration range from 0.01 to 2 ng mL^{-1} (r^2>0.993). RSD for intra- and inter-day precision for a single fiber were in the range of 2.8–4.9% and 4.5–7.7%, respectively. Fiber to fiber reproducibility was in the range of 7.0–10.2% (n = 3). Finally, the developed method was successfully applied for the extraction of selected OPPs from water samples. The relative recovery obtained for the spiked real water samples at 0.02, 0.2 and 2 ng mL^{-1} level were 87–98, 91–97 and 92-99%, respectively.

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Solid micro extraction technique for preconcentration of cadmium in real samples using cerium oxide nanoparticles and its determination by flame atomic absorption spectrometry

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Cadmium is a toxic elementwhich may lead to renaltoxicity, pancreatic cancer, or enhanced tumor growth [1].Environmental exposure to Cd often arises from sewage, pesticides, phosphate-based fertilizers, and the combustion of fuels [2].

For many years, flame atomic absorption spectrometry (FAAS) has been a standard analytical tool for determination of metals in various matrices owing to its wide application range, simplicity in operation and low cost of analysis. However, FAAS has a detectionlimit, which is not low enough to determine cadmium at tracelevels. Therefore, In order to achieve accurate, sensitive and reliable resultsat trace levels a preconcentration and separation step is needed prior to its determination by FAAS.

Solid phase extraction is one of the most commonly preconcentration techniques. Up to now, different adsorbents including nano-magnetic particles, ion imprinted polymers, carbon nanotubes were used for preconcentration of heavy metals [3].

In this work, cerium oxide nanoparticles as an efficient adsorbent for preconcentration of cadmium ion in real samples has been employed .The effects of the experimental conditions onthe extraction process were completely investigated and optimized.The calibration curve is linear inthe concentration range of 5–400 μgL^{-1} of Cd, The LOD was 5μgL^{-1} and RSD for 5 replicate analysis of 20 μgL^{-1} Cd was 4%.The enrichment factorbased on the slopes of calibration curves before and after the extraction was calculated to be 19. The effects of different interfering ions in the Cd determination was also investigated. The method was applied todetermineCd in environmental water and food samples.

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Solid phase extraction and determination of cisplatin by molecularly imprinted polymer (MIP) coupled with ETAAS

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Cisplatin, as the platinum compound cisdiammine dichloroplatinum(II) is generally known. This compound is an important chemotherapeutic agent for testicular cancer, including advanced forms of the disease. Long-term survival is experienced in more than 90% of cisplatin- treated patients with solid tumors of the testes [1]. Cisplatin can react with many structures in the cell, such as membranes, proteins and RNA, but its most important intracellular target is DNA [2].

In this work, a new molecularly imprinted polymer (MIP) has been prepared using Cisplatin as the template molecule. The polymer was synthesized in a non-covalent approach with acrylamid (AA) and allyl glycidil ether/ tannic acid (AGE/TAA) as the functional monomer, N,N methylene diacrylamid as the cross-linker in the methanol solution and 2,2'-azobis-isobutyronitril (AIBN) as the initiator. The polymerization was performed via a free radical. The synthesized MIP was identified by FT-IR, and scanning electron microscopy (SEM). The effect of various parameters like pH, sorption capacity of polymer, sorption time, selectivity of polymer and desorption solvent were examined. Electrothermal atomic absorption spectrometry (ETAAS) was used for measurement of the analyte. The prepared MIP adsorbent showed a capacity of 12.5 $\mu g\ g^{-1}$ for cisplatin at pH=5.7. A recovery of 99.25% and a relative standard deviation (RSD) of 4.5% (n=10) were obtained for the analyte. The detection limit of the method (3σ, n=20) was calculated to be 40 ngL^{-1} for the analyte. Finally, the proposed method was applied successfully for the extraction and determination of cisplatin in biological fluids (human blood, serum and urine).

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Solid phase extraction and preconcentration of omperazole from aquatic and urine samples using magnetice multi-wall carbon nanotube (MWCNT/Fe_3O_4) followed by spectrophotometric determination

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In this study, magnetic multi-wall carbon nanotubes (MWCNTs) were prepared by assembling magnetic nanoparticles on to the acid-treated multi-wall carbon nanotubes. The properties of magnetic MWCNT were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR) and transmission electron microscopy (TEM). Combination of solid phase extraction (SPE) and spectrophotometric methods was used for determination and preconcentration of omperazole in aquase and urine samples using MWCNT/Fe_3O_4 nanocomposite as a sorbent. This adsorbent was found to be advantageous over conventional solid phase extraction (SPE) in term of operational simplicity and low time consuming. Magnetic MWCNT,was easily separated from the aqueous solutions with the help of an external magnet ; so, no filtration or centrifugationwas necessary. After extraction and collection of magnetic MWCNT, the adsorbed analytes with acetonitrile were eluted and analyzed by spectrophotometry. The spectrophotometric method was based on charge transfer complexation reaction of omperazole with δ acceptor iodine had was measured at 360nm. Extraction parameters such as desorption solvent, sorbent amount and pH were optimized. The method had a linear calibration plot in the range from 0.025 to1.0 µgmL^{-1} with RSD of 2.3% at 0.5 µgmL^{-1} (n =11).The maximum adsorption capacities obtained from Langmuir model for omperazole on magnetice MWCNT nanocomposite was 175.43 mg g^{-1}. Finally, the established MSPE-Spectrophotometrice method was successfully applied to the determination of omperazole from urine samples. Morever, excellent average recovery values have been obtioned (98.9%).

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Solid phase extraction for determination of trace amount Cu(II) by Single walled carbon nanotubes (SWCNTs)in water samples and determination it by FAAS

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Abstract

Copper on the other hand occurs as CuS, CuS_2 , $CuFeS_2$, $CuSO_4$.$5H_2O$ and other forms. More than 75% of copper production is used in electrical industries. [1]It is also used in pigments, metallic blends and household. Hence determination of lead and copper in industry and environment are both very important.[2] A preconcentration step is advisable in trace analysis. Lead and copper heve been so far determined by various methods such as spectrophotometry[3]. A novel, simple method has been developed for the preconcentration of Cu(II) based on the adsorption of its Schiff's Naphtol-2-(Pyridylazo-2)-1 (PAN) complex on a Single walled carbon nanotubes (SWCNTs) onto C_{18} cartridge . The influence of acidity, eluting agents, stability of the cartridge, sample volume and interfering ions has been investigated in detail. The adsorbed complex could be eluted using HNO_3 4M and the concentration of Cu(II) was determined by flame atomic adsorption spectrometry (FAAS). A detection limit of 0.10 µgL^{-1} could be achieved and the developed procedure was successfully applied for the determination of Cu(II) in spiked water samples . The preconcentration factor attainable for quantitative recovery (>97%) of Cu(II) was 125 for a 1000mL sample volume. The mentioned method was successfully applied on the determination of Cu(II) in different water samples. In this method, relative standard deviation (RSD) is %1.25.

Key Words: **Keywords:** Copper(II), Preconcentration, Solid phase extraction, Single walled carbon nanotubes (SWCNTs). Flame atomic adsorption spectrometry (FAAS).

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Solid phase extraction of Ag with syringe technique from water samples by modified magnetic nanoparticles

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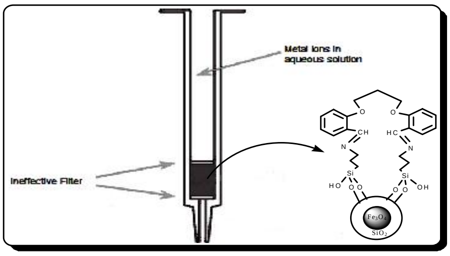
Abstract

Solid phase extraction (SPE) is one of the most commonly used sample pretreatment techniques due to its simplicity, high enrichment factor and low consumption of organic solvent. The surface modification of inorganic nanoparticles such as silica, titanium dioxide and magnetic nanoparticles is gaining increasing attention in the field of nanocomposites [1]. The greatest advantage of these nanoparticles is a large active surface area for a given mass of particles by high selectivity, which causes them to be more reactive to some other component such as metal ions [2]. In this work, a sensitive and simple magnetic solid phase extraction (Scheme 1) procedure was presented for the preconcentration of Ag ions in water samples. Some glass wool placed on the bottom of 5 mL syringe and filled with appropriate amount of modified magnetic nanoparticles and used as a SPE sorbent. An optimal amount of aqueous solution of metal ions containing Cu(II), Cd(II), Ag(I), Ni(II), Zn(II) and Pb(II) was passed through the column and the adsorbed metals were quantitatively elution using 8 ml of 4 mol L^{-1} hydrochloric acid. The percentage of extracted metal ions was determined by flame atomic absorption spectroscopy. The effect of some parameters, such as pH, flow rate, adsorbent amount and recovery was evaluated. At the optimized conditions the SPE device exhibits a good extractive affinity for Ag (I) with enrichment factor of 250 and recovery of >95%.



Scheme 1. The structure of the nanoparticles

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Solid phase extraction of thorium(IV) ions using C18-silica disks modified by oxime ligand

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Thorium is the most abundant radioactive element in nature and has widespread application in variable areas [1]. Thorium not only has chemical toxicity like other heavy metals do, but also has radioactivity which can cause a large scale permanent damage of bone, kidney and liver [2]. direct analysis of thorium is still a barrier because of the very trace content and the presence of complex matrix [3]. Therefore, preconcentration and selective separation of thorium from natural samples is very important and need much more attention. 3-tert-butyl-2-hydroxy-5-methyl benzaldehyde oxime (**HL**) are synthesized and characterized by conventional spectroscopic methods. Hydroxyoxime (**HL**) is used as a modifier of octadecyl silica disks for preconcentration of trace amounts of thorium(IV) ions, followed by hydrochloric acid elution and spectorophotometric determination using Arsenazo(III). The effect of parameters influencing the extraction efficiency, i.e. pH of the sample solutions, amount of the oxime, type and volume of stripping reagent, sample and eluent flow rates were evaluated. Under optimum experimental conditions, the capacity of the membrane disks modified by 5 mg of the ligand was found to be 410.2 (±1.3) µg of thorium. The detection limit and the concentration factor of the presented method are 2.32 µg/L and 200 respectively. The method was applied to the extraction, recovery and detection of thorium in different synthetic and a water samples.

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Solid phase extraction of trace amount of Gold using $NiFe_2O_4$ magnetic nanoparticles and its determination by flame atomic absorption spectrometry

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Abstract

Gold is an expensive metal which occurs on the earth in very low natural contents [1, 2]. The determination of gold is difficult for the high concentration of interfering matrix components and the low concentration of gold in most real samples. In order to resolve the two important problems that are the economical impact of losing gold and environmental concern, it is crucial to develop a simple, rapid and efficient method for the separation and preconcentration of gold prior to determination. Nowadays, SPE method is one of the most effective preconcentration methods because of its advantages such as ease of use, high preconcentration factor and flexibility to choose the solid phase for optimum results [3]. In this work, a simple and fast solid-phase extraction procedure has been developed for preconcentration and determination of traces amount of gold from aqueous solutions by $NiFe_2O_4$ magnetic nanoparticles (15<nm) sorbent, prior to flame atomic absorption spectrometry (FAAS) determination. The effect of different variables such as pH of solution, contact time, adsorbent amount, adsorption capacity, volume and type of eluent, and interference of some ions on the recoveries of the gold ions were investigated. Under optimal conditions, the adsorption capacity was found to be 34.6 mg g^{-1}. The sorption of Au (III) ions was quantitative in the pH range of 3.0–7.0 and quantitative desorption was achieved using 5 mL of 0.1 mol L^{-1} $Na_2S_2O_3$ solution. The preconcentration factor was 80. Detection limit based on the $3S_b$ criterion was 1.31 µg L^{-1} and relative standard deviation (R.S.D) for six replicate measurements was found to be 1.57%. The established method has been successfully applied to analyze real samples, and satisfactory results were obtained.

Keywords: Gold, Separation, Preconcentration, $NiFe_2O_4$, Magnetic nanoparticles, Flame atomic absorption spectrometry

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Solid phase extraction of trace amount of Gold using $NiFe_2O_4$ magnetic nanoparticles and its determination by flame atomic absorption spectrometry

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Abstract

Gold is an expensive metal which occurs on the earth in very low natural contents [1, 2]. The determination of gold is difficult for the high concentration of interfering matrix components and the low concentration of gold in most real samples. In order to resolve the two important problems that are the economical impact of losing gold and environmental concern, it is crucial to develop a simple, rapid and efficient method for the separation and preconcentration of gold prior to determination. Nowadays, SPE method is one of the most effective preconcentration methods because of its advantages such as ease of use, high preconcentration factor and flexibility to choose the solid phase for optimum results [3]. In this work, a simple and fast solid-phase extraction procedure has been developed for preconcentration and determination of traces amount of gold from aqueous solutions by $NiFe_2O_4$ magnetic nanoparticles (15<nm) sorbent, prior to flame atomic absorption spectrometry (FAAS) determination. The effect of different variables such as pH of solution, contact time, adsorbent amount, adsorption capacity, volume and type of eluent, and interference of some ions on the recoveries of the gold ions were investigated. Under optimal conditions, the adsorption capacity was found to be 34.6 mg g^{-1}. The sorption of Au (III) ions was quantitative in the pH range of 3.0–7.0 and quantitative desorption was achieved using 5 mL of 0.1 mol L^{-1} $Na_2S_2O_3$ solution. The preconcentration factor was 80. Detection limit based on the $3S_b$ criterion was 1.31 μg L^{-1} and relative standard deviation (R.S.D) for six replicate measurements was found to be 1.57%. The established method has been successfully applied to analyze real samples, and satisfactory results were obtained.

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Solid phase extraction of trace amounts of Fe (III) ion using 3-(trimethoxysilyl)-1-propanethiol and gold coated magnetic nanoparticles modified by 2-amino-5-mercapto-1,3,4-thiadiazole and determination with atomic absorption spectrometry

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Many research groups have explored the application of several nano sized SPE adsorbent such as nanoparticles (NPs) and nanotubes [1]. In resent years, magnetic nanoparticles have been studied because of their potential applications as magnetic carriers for varius biomedical uses such as cell and DNA sepration, bio-sepration and preconcentration of various anions and cations. This technique allowed the determination of trace amounts of heavy metal ions in environmental and biological samples [2-3]. A new magnetic solid sorbent waz synthesized using magnetic nanoparticles (MNPs) coated by 3-(trimethoxysilyl)-1-propanethiol (TMSPT) and gold and modified with 2-amino-5-mercapto-1,3,4-thiadizole. The sorbent was characterized by Fourier transform infrared spectroscopy (FT-IR) and used for solid phase extraction of Fe^{3+} ions. These magnetic nanoparticles can be easily dispersed in aqueous samples and retrived by the application of external magnetic field via a piece of permanent magnet. Fe^{3+}ions were measured by atomic absorption spectrometry. The optimal extraction/preconcentration conditions including pH of sample solution, desorption conditions (type, concentration, and volume of the eluent), adsorption and desorption time, sample volume, and coexisting ions were studied. Under optimal conditions, the detection limits of 3.2 ng ml^{-1} were obtained with a relative standard devation (RSD%) of 4.5% for Fe^{3+} ions.Finally, the method was successfully applied to extraction and determination of Fe^{3+} ion in Ferrous sulphate tablet.

keyword: Solid phase extraction, magnetic nanoparticles, Fe (III),2-amino-5-mercapto-1,3,4-thiadiazole

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Solid phase extraction of trace amounts of Fe (III) ion using 3-(trimethoxysilyl)-1-propanethiol and gold coated magnetic nanoparticles modified by 2-amino-5-mercapto-1,3,4-thiadiazole and determination with atomic absorption spectrometry

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Solid Phase Extraction of Trace Amounts of Rhodamine B Using Silica-Coated Magnetic Nanoparticles Modified with soybean oil

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Rhodamine B is used as a color additive in cosmetic, food, pharmaceutical, and colorant in textiles and plastic industries. The carcinogenicity and toxicity of rhodamine B towards human and animals have been experimentally proved [1]. Numerous methods have been developed for the determination of rhodamine B in cosmetics and food samples. Ultraviolet–visible spectrometry is an important instrument at this area due to its simplicity and lower cost than other instruments. The main limitations in ultraviolet–visible spectrometric determination of rhodamine dye is lower level of analyte then the quantitation limits of ultraviolet –visible spectrometry. The preconcentration methods, such as liquid extraction, cloud point extraction and solid phase extraction are used to solve this problem [3].

In this study, a novel magnetic sorbent material prepared by modifying silica-coated magnetic nanoparticles (Fe_3O_4/SiO_2) with soybean oil for extraction and determination of rhodamine B from various samples was developed. The magnetic composite was characterized by X-ray diffraction (XRD), thermo gravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FT-IR). The analytical conditions including pH of solutions, contact time, adsorbent dosage, and ionic strength for the quantitative recoveries of rhodamine B was investigated by applying central composite design. The design involved 30 experiments with six replicate at center point, which were performed in random order. Finally the proposed method was successfully applied in determining of rhodamine B in matches tips and hand washing liquid soap samples.

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Solid phase extraction using MWCNT/Fe_3O_4 nanocomposite modified by coupling agent APTES for preconcentration and determination of cadmium and lead in real samples

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In this study Multiwall carbon nanotube (MWCNT)/iron oxide magnetic nanocomposites were prepared and functionalized with using a coupling agent (3-Aminopropyl triethoxysilane) (APTES) .and were characterized by scan electron microscopy (SEM), X-ray diffraction (XRD), Fourier transform infrared specteroscopy (FT-IR) and Transmission electron microscopy (TEM) for estimate the extent of the MWCNT chemical modification. The results indicated that the APTES/MWCNTs magnetic composites were synthesized. The modified adsorbent used as a solid phase extractor for preconcentration Cd^{2+} and Pb^{2+} ions in a batch system coupled to flame atomic absorption spectrometry (FAAS), was evaluated. First the metal ions were adsorbed on modified magnetite nanotube.The adsorbed ions were desorbed in a small volume of desorption solvent and determined spectrometrically. The effects of adsorption time, solution pH, extraction time and amount of volume for obtained optimal conditions were investigated at room temperature. Calibration curve in the range of 0.21-250 and 1.1- 750 ng mL^{-1} for volume 200.0 mL was linear for cadmium and lead, respectively, with the correlation coefficient 0.9975 and 0.9965 were achieved. The detection limit of method for metal ions cadmium and lead were 0.37 and 0.08 ng mL^{-1}, respectively, and enrichment factor for this method was founded 100.0.Relative standard deviation of three separate determinations by the method was found to be 3.1% and 3.6 % for metal ions cadmium and lead, respectively . In The application of Langmuir and Freundlich isotherms are applied to fit the adsorption data of cadmium and lead. The Langmuir model fits the adsorption isotherm data of Cd and Pb better than the Freundlich model. The maximum adsorption capacities obtained from Langmuir model for Pb and Cd on functionalized nanocomposite were 142.85 and 60.44 mg g^{-1} at 25 ◦C, respectively. Application of the developed method for the determination of lead and cadmium in real samples gave satisfactory results.

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Solid-phase extraction combined with dispersive liquid-liquid microextraction for the determination of uranium in different water samples

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Uranium find extensive application as nuclear fuel in power plants and their sources are soil, rocks, plants, sand and water. Uranium is known to cause acute toxicological effects for human and their compounds are potential occupational carcinogens [1]. A novel approach for the determination of uranium in different water samples is presented. Analyte was extracted from the matrix and transferred to a small volume of a high density, water insoluble solvent using solid-phase extraction (SPE) followed by dispersive liquid-liquid microextraction (DLLME) [2,3]. Variables affecting the performance of both steps were thoroughly investigated and their effects on the efficiency of the whole sample preparation process are discussed. Under optimized conditions, 100 mL of water sample with pH adjustment at 6 and containing suitable amount of 1-(2-pyridylazo)-2-naphthol (PAN) as chelating agent were first concentrated using a C_{18} cartridge and then target compound were eluted with 1.5 mL of methanol. This extract was mixed with 40 μL of chlorobenzene as an extraction solvent and the blend added to 5 mL of ultra pure water. After centrifugation, the settled organic phase was evaporated and then analyzed by inductively coupled plasma – optical emission spectrometry (ICP-OES). The calibration graph was linear in the range of 0.5-500 μg L^{-1} for the analyte and the limit of detection (LOD) was 0.1 μg L^{-1}. The relative standard deviation (RSD) was below 7%. The optimized procedure was successfully applied to the extraction and determination of uranium in different water samples.

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Solid-phase microextraction based on cetyltrimethylammonium bromide-coated magnetic nanoparticles for spectrofluorimetric determination of dexamethasone from biological samples

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Abstract:

A simple, rapid, and sensitive method based on the combination of magnetic solid phase extraction (MSPE) and spectrofluorimetric determination was developed for extraction and preconcentration of dexamethasone (DEX) form biological samples using cetyltrimethylammonium bromide (CTAB) coated Fe_3O_4 nanoparticles (NPs) as a sorbent. Mixed hemimicelles formed on the surface of MNPs by CTAB showed great adsorptive tendency toward the analytes[1,2]. The experimental parameters affecting the extraction efficiency such as type and volume of desorption solvent, pH value, sorption and desorption time, amount of sorbent were investigated and optimized. Under optimized conditions the calibration curve for dexamethasone determination showed a good linearity in the range of 30.0 – 600 ng mL^{-1} with a R^2 of 0.998. The relative standard deviation (RSD) for the determination of 200 ng mL^{-1} DEX was 1.2% (n=5), and the detection limit (S/N=3) was 7.4 ng mL^{-1} in the original solution. Finally, the applicability of the proposed method was evaluated by extraction and determination of the DEX in urine and plasma samples. The average recovery of spiked samples was 99.2 %. The results indicated that CTAB-MNPs method has excellent clean up and high preconcentration factor and can be served as a simple and sensitive method for monitoring of DEX in the urine and plasma samples.

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Solid-phase microextraction of phthalate esters from aqueous media by electrochemically deposited Graphene oxide/polypyrrole composite fiber

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Abstract

Phthalate esters have been widely used as plasticizers in various products such as disposable cups**.** Phthalates are a group of endocrine-disrupting chemicals found in PVC or #3 plastic. Phthalate exposure has been linked to early puberty in girls and a risk factor for later-life breast cancer [1]. In this work, a new fiber based on electrochemical polymerization of pyrrole with graphene oxide and SDS as a dopant was prepared on platinum wire for solid-phase microextraction (SPME) of phthalate esters, including di-n-butyl phthalate (DBP), di-isobutyl phthalate (DIBP), butyl benzyl phthalate (BBP) and dioctyl phthalate (DOP) in aqueous samples. The feasibility of direct-immersion (DI) SPME for the determination of phthalate esters was studied. The effects of the four parameters including injection temperature, ionic strength, extraction temperature, and extraction time were investigated in DI-SPME. Liner range of DBP and DOP were .1-178 µg/l, for DIBP was .2-178 µg/l and for BBP was 1-356 µg/l. Relative standard deviations were between10-19. The results showed that the procedure is feasible for determination of very low amounts of phthalate esters in various aqueous media.

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Solid-State Thermal decomposition of $[Cu(NH_2C_2H_5)_4](NO_3)_2$: a New and Simple Route to CuO Nanopowder

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Copper oxide (CuO), as an important oxide of transitional metal with a narrow band gap (Eg = 1.2 eV), has been extensively studied because of its importance for fundamental investigations and practical applications, such as gas sensors, magnetic storage media, varistors, solar energy transformation, lithium ion electrode materials, semiconductors, and catalysis [1,2].

In this paper, a simple low-temperature method is reported for the preparation of pure phase CuO nanopowder. In the first step, $[Cu(NH_2C_2H_5)_4](NO_3)_2$ precursor was prepared from the copper (II) nitrate source, and then the precursor was decomposed to produce CuO nanopowder. The copper oxide powder was obtained by calcinations of $[Cu(NH_2C_2H_5)_4](NO_3)_2$ precursor at 400 ^{o}C for 1 h in air without any additive or template. No impurities were detected by XRD. The results indicates that a single phase of CuO could be obtained by the decomposition $[Cu(NH_2C_2H_5)_4](NO_3)_2$ precursor at 400 °C.

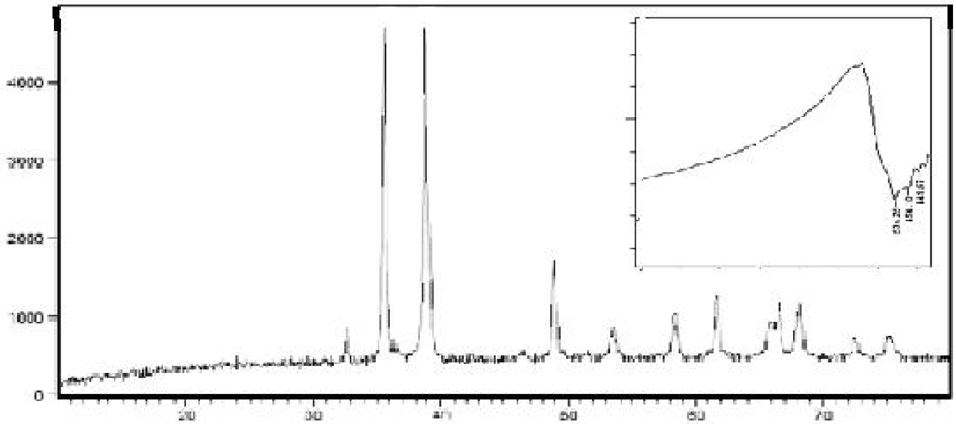

Fig.1.FT-IR spectrum and pattern XRD of the CuO nanoparticles

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Solvent influence upon complex formation between 1,10-diaza-18-crown-6 with the Ce^{3+} cation in some pure and binary mixed non-aqueous solvents

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The complexation reaction of the Ce^{3+} cation with the macrocyclic ligand, **1,10-**diaza-18-crown-6 (DA18C6) was studied in acetonitrile-methanol (AN-MeOH), acetonitrile-methylacetate (AN-MeOAc), acetonitrile-ethylacetate (AN-EtOAc) and ethanol-dimethylformamide (EtOH-DMF) binary solvent systems at different temperatures using conductometric method. The conductance data show the stoichiometry of the complex in all pure solvent systems, is 1:1 [M:L] . The stability constant of the resulting 1:1 complex was obtained using a GENPLOT computer program [1,2]. The stability constant of $(DA18C6.Ce)^{3+}$ complex in various neat solvents at 25°C and 35°C decreases in order: $MeOH > EtOH > DMF > AN$ and $MeOH > DMF > EtOH > AN$, respectively and in the case of binary solvent solutions at 25°C it was found to be: $AN - MeOAc > AN - MeOH > EtOH - DMF$. A none-linear behavior was observed for changes of $\log K_f$ of $(DA18C6.Ce)^{3+}$ complex versus the composition of the binary mixed solvents. In some of the binary solvent systems, it seems that a 2:1 [M_2L] and also 2:2 [M_2L_2] and 2:3 [M_2L_3] complexes are formed in solutions. The values of thermodynamic parameters ($\Delta H^\circ_c, \Delta S^\circ_c$) for formation of $(DA18C6.Ce)^{3+}$ complex were obtained from temperature dependence of the stability constant using van't Hoff plots[3]. The results show that in all cases, the $(DA18C6.Ce)^{3+}$ complex is entropy stabilized but from the enthalpy viewpoint, depending on the nature of the medium, the complexation process is enthalpy stabilized or destabilized. The result obtained in this study, show that the order of stability, the thermodynamics and even the mechanism of the complexation processes may change with the nature and composition of the solvent systems.

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Soxhlet extraction and GC-MS analysis of essential oil of Pistacia Atlantica Kurdica nuts and optimization of process using factorial design of experiments

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Abstract

The Anacardiaceae are a family of flowering plants with about 400 species of mainly tropical trees and shrubs bearing fruits. Among this family Anacardium occidentale and Pistacia vera are most known, the trees that are cultivated for their nuts (Cashew and Pistachio) [1]. Pistacia Atlantica Kurdica (p. a. Kurdica) is one of the subspecies of Pistacia atlantica. P. a. kurdica is widely spread around the Zagros Mountains and particularly in Western and Northern of Iran, in other words in the Kurdish regions of Iran specially in Kurdistan province. The essential oils of some of the species of the genus Pistacia have been characterized [2]. However, the subspecies of P. a. kurdica essential oil of nuts have not been characterized in region of Kurdistan province of Iran.

In this study soxhlet extraction technique is used for the essential oil extraction of p. a. Kurdica nuts. Soxhlet extractions were performed at three extraction time (7h, 11h and 13h) by using two solvents (n-hexane and methanol). A general full factorial design with two factors (extraction time and types of extractor solvent) was implemented. The Soxhlet extraction method presented a good yield of components in extract. The study shows that n-hexane extracted almost half times more than methanol. The highest yield obtained with n-hexane was 31.9%; while the highest yield obtained by methanol was 20.45%. For both of solvents, the extracted mass yield percent increased with increasing the extraction time from 7h to 11h and then it decreased from 11h to 13h. The chemical components of extracted essential oil were separated on silica gel column (n-hexane) and analyzed with GC-MS method.

Keywords: Soxhlet extraction, Pistacia atlantica Kurdica, Essential oil

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Spectrofluorimetric determination and preconcentration of trace amounts of gold with Rhodamine B and compare with spectrophotometry method

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A sensitive fluorimetric method was proposed for determination of gold based on the formation of a violet complex with Rhodamine B in hydrochloric acid and was compared with spectrophotometry method. The $AuCl_4^-$ ion reacts with the reagent to form an association complex which can be extracted into toluene. Fluorescence quenching of rhodamine B is measured in the absorption solution at an excitation wavelength of 565 nm and an emission wavelength of 580 nm.Different parameters affecting the process of complexation and extraction of gold such as ionic strength, time of extraction, concentration of acid and complex agent and etc. were optimized. The technique could be free of interferences using EDTA and TEA.

Under optimized conditions, the dynamic range was obtained between 0.2 to 6ppb with detection limit and RSD of 0.08 ppb and 4.8% respectively by using spectrofluorimetry and the dynamic range for spectrophotometry method was obtained between 0.1 to 1.4ppm with detection limit of 5ppb and RSD of 2.8%. The complex was found to have a stoichiometry of 1:1. The spectrofluorimetry method is generally used because of their specificity, selectivity and low detection limits. The proposed method has been applied satisfactorily to determine gold in tap water and sea water samples. The recovery of the spiked amounts was obtained 86% and 88% which demonstrates that the proposed method exhibits a good reliability.

Spectrofluorimetric determination and preconcentration of trace amounts of gold with Rhodamine B and comparing with spectrophotometry method

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Fluorescence is the most common and useful type of photoluminescence in analytical chemistry. Its capacity of detection is approximately one order of magnitude greater than that of molecular absorption spectroscopy [1] and its selectivity is also clearly greater than that of other spectroscopic methods, so it is applied for the analysis of concentrations in the ng/mL range[2]. A sensitive fluorimetric method was proposed for determination of gold based on the formation of a violet complex with Rhodamine B in hydrochloric acid and was compared with spectrophotometry method. The $AuCl_4^-$ ion reacts with the reagent to form an association complex which can be extracted into toluene. Different parameters affecting the process of complexation and extraction of gold such as ionic strength, time of extraction, concentration of acid and complex agent and etc. were optimized. The technique could be free of interferences using EDTA and TEA. Under optimized conditions, the dynamic range was obtained between 0.2 to 6 ppb with detection limit of 0.08 ppb and RSD of 4.8% respectively by using spectrofluorimetry and the dynamic range for spectrophotometry method was obtained between 0.1 to 1.4ppm with detection limit of 5ppb and RSD of 2.8%. The complex was found to have a stoichiometry of 1:1. The proposed method has been applied satisfactorily to determine gold in tap water and sea water samples. The recovery of the spiked amounts was obtained 86% and 88% which demonstrates that the proposed method exhibits a good reliability.

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Spectrofluorimetric determination and preconcentration of trace amounts of gold with Rhodamine B and comparing with spectrophotometry method

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Spectrofluorimetric determination of fluoride in toothpaste based on fluoresce quenching of 2-[(E)-(2-Hydroxy-4-methoxybenzylidene) amino]-4, 5, 6, 7-tetrahydro-1-benzothiophene-3-carboxamide as new fluorescer

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Modern toothpastes are designed to do more than just clean teeth and reduce decay. Hence they contain many additives, either for health or cosmetic reasons, including components to prevent tooth decay, colorants, flavors and preservatives. Fluoride is an important component of toothpaste as it protects the tooth by making the enamel harder in toothpastes. The fluoride usually in the form of free fluoride salts of sodium fluoride (NaF) is used. A part from NaF, the main sources of fluoride in toothpastes are sodium monofluorophosphate (MFP), aminefluoride and SnF_2. For determine the amount of fluoride ion can be used different methods. Spectrophotometry, gas chromatography,hplc and potentiometric including methods cited.

In this work we used a new fluorimetric method for determination of fluoride ion based on quenching effect of fluoride ion on fluorescence of 2-[(E)-(2-Hydroxy-4-methoxybenzylidene) amino]-4, 5, 6, 7-tetrahydro-1-benzothiophene-3-carboxamide. Because previous methods were less selectivity and sensitivity, in this work we used a new sensitive fluorimetric method. Merident brand toothpaste was used an analytical sample for determination of fluoride ion. This method is capable for measuring a wide range of fluoride concentrations of fluoride ion. In this method, the ionic strength was kept constant (I=0.001 M KNO_3). The fluorescence intensity is measured with the excitation and emission wavelengths 271 and 300 nm, respectively. A linear calibration graph was obtained from 0.9 to 8.2 ppm. The relative standard deviation was very good (1.06% at. 4.5 ppm F^-) and the detection limit was acquired 0.04 ppm. Also interference of some anions and cations were investigated. The proposed method was successfully applied for the determination of fluoride samples.

Keywords: toothpaste, fluoride, Spectrofluorimetric

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Spectrophotometric and Spectrofluorometric studies of safranin and cysteine interactions with gold nanoparticles

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Abstract

Gold nanoparticles (GNPs) have drawn worldwide attention for the fabrication of smart sensing devices meant for detection of threat agents. The benefits offered by GNPs in sensory research are fivefold; a) GNPs possess excellent compatibility with almost all types of chemically and biologically molecules, b) high surface area to volume ratios, c) GNPs serve as an excellent scaffold to immobilize large quantities of organic or biomolecules, thus increasing the chances of their interaction with a target analyte, d) mostly negatively charged, e) can be tailored to electrostatically interact with positively charged biomolecules [1-3].

The interaction between cysteine and safranin with citrate capped gold nanoparticles was studied. The measurements performed using resonance light scattering spectroscopy and absorbance spectroscopy. The size and surface characteristics of safranin-capped gold nanoparticles in the solution containing cysteine was also investigated by DLS and TEM techniques. The average diameters of synthesized Au nanoparticles were 19.0 nm and their surface potential were -56.3 mV. Interaction of the GNPs and safranin or cysteine leads to increase of their sizes and surface potential.

The influence of parameters such as pH, ionic strength of the solution and safranin concentration was studied on the spectrophotometric and spectroflourimetric characteristics of GNPs. This system was used for determining of cysteine. Under the optimum conditions there was two LDR range (9.0×10^{-8} – 1.0×10^{-6} and 5.0×10^{-6} – 9.0×10^{-5} M) for cysteine determination with RSD less than 5% and also detection limit of 3.7×10^{-8} M cysteine was obtained.

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Spectrophotometric determination of carbendazim in soil and water samples using dispersive liquid-liquid microextraction followed by microvolume UV–vis spectrophotometry

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Carbendazim [methyl 2-benzimidazolecarbamate (MBC)] is widely used systemic fungicides for control of a broad range of fungi affecting fruits, nuts, vegetables, turf, and field crops. Because of its extensive use and suspected carcinogenic activity, monitoring and determination of this fungicide in environmental water, soil, and various crops is required [1, 2]. Various analytical methods have been reported for the determination of carbendazim in different crop matrices and environmental samples, but its preconcentration followed by spectrophotometric determination has not been report yet. Dispersive liquid-liquid microextraction (DLLME) is a miniaturized form of liquid–liquid extraction (LLE) that employs microliter volumes of extraction solvent. This technique is based on a ternary component solvent system similar to homogeneous LLE and cloud point extraction. The advantages of the DLLME method are simplicity of operation, rapidity, low cost, high recovery and enrichment factors. This method has been applied for the determination of trace organic pollutants and metal ions in the environmental samples, and also coupled with different detection techniques, such as Uv-vis spectrophotometery [3].

A novel, rapid and simple method for the sensitive determination of trace amounts of fungicide carbendazim was developed by combination of dispersive liquid–liquid microextraction (DLLME) and microvolume UV–vis spectrophotometry. The method is based on the reduction of Fe(III) to Fe(II) by carbendazim, its reaction with potassium Ferricynide to form a bluish green product, and its extraction into CCL_4 using DLLME technique. In this method, there is no need for additional dispersive solvent because methyltrioctylammonium chloride, which is used to form ion pair with bluish green product, also acts as a disperser. Under the optimum conditions, the calibration graph was linear over the range of 5 to 600 ng mL^{-1} of carbendazim with limit of detection of 2.1 ng mL^{-1}. Relative standard deviation for eight replicate determinations of carbendazim at 50 and 300 ng mL^{-1} concentration levels were calculated to be 3.9 % and 1.0 %, respectively. The proposed method was successfully applied to determination of carbendazim in soil and water samples.

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Spectrophotometric Determination of Imidacloprid Insecticide in Potato samples by Rank Annihilation Factor Analysis (RAFA) Method and Compared with HPLC

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The use rank annihilation factor analysis (RAFA) for spectrophotometric studies of pesticide residue is proposed. Imidacloprid is a neonicotinoid insecticide which derives from the nicotine moiety market due to: (i) a broad spectrum of efficacy; (ii) systemic and translaminar action; (iii) pronounced residual activity and a unique mode of action; imidacloprid has become the largest insecticide treatment in recent years [1]. These compounds are used extensively for the control of agricultural pests by spraying and are also widely used in seed dressings and soil additions. So mesearment of its residue in agricultural products is important. Rank Annihilation Factor Analysis (RAFA) is a powerful chemometrics method based on rank analysis for two dimensional data. This method use hard and soft modeling methods for solving problems in chemical systems. In this work, UV-Vis spectrometric data from pH-metric titration of Imidacloprid in potato samples makes X_U matrix. X_U is unknown matrix and X_s in known matrix from standard solution of Imidacloprid. This method need only one standard solution, the RAFA is based on the principle that the rank of two-way bilinear matrix of pure compounds is one. When concentration of imidaclopride acts as an optimizing object, and simply combined with the pure spectrum of imidaclopride, the rank of original data matrix can be reduced by one by annihilating the information of the real concentration from the original data matrix. K is a coefficient that can be reduced X_U rank one unit. This coefficient is equal to the concentration of imidacloprid. Rank of X_U obtains use of Principle Component Analysis (PCA), $1+n_i$ and rank of X_s is 1. $X_k=X_U-kX_S$ [2]. The residual standard deviation (R.S.D.) of the residual matrix after bilinearization of the background matrix is regarded as the evaluation function. The performance of the method has been evaluated by using synthetic data.
This method iteratively done until obtains k that can reduce the rank of X_U one unit. All calculations are performed in MATLAB 6.0 (Math Works, Cochituate Place, MA).
However, the proposed method can also be applied for pesticide residue measurement in different matrixes. So it can be developed in many laboratory equipped with UV-Vis spectrometery instead of HPLC/UV instrument.

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Spectrophotometric determination of Sudan IV in industrial and environmental water samples after separation and preconcentration by modified magnetic nanoparticles

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Synthetic dyes used in various industries, including food, textile, and petroleum, are potential pollutants for food and environmental samples, in particular natural water samples. Sudan dyes are a class of azo dyes. They have been illegally used as additives in food products to maintain their red–orange color and thus stimulate sales of these products in the market. Sudan dyes are thought to be carcinogenic yet have been determined as a contaminant in chili powder [1]. Many methods have been reported for determining Sudan dyes in environmental water samples. However, the low concentrations of Sudan dyes increase the difficulty in determining them directly in environmental water samples. So a good sample preparation technique with high extraction efficiencies, low consumption of organic solvents and short time is usually necessary prior to instrumental analysis [2].

In presented work, a solid phase separation-enrichment procedure for an azo dye that is Sudan IV in real samples including industrial and natural water samples has been established for use prior to its spectrophotometric determination. The method is based on the adsorption of Sudan IV on a modified magnetic nanoparticles and its elution sorbent with 5 mL of ethanol. Sudan IV in the eluent was determined by UV–visible spectrophotometry at 516 nm. Sodium stearate was used for modification of magnetic nanoparticles. The effect of analytical conditions, such as pH, amount of sorbent, amount of sodium stearate, type and volume of eluent and sample volume was examined. The influences of matrix components on Sudan IV recoveries were studied. Under the optimum conditions, the preconcentration factor was 50. The detection limit was 3.0×10^{-7} mol L^{-1} and the relative standard deviation was 2.7 %. The results indicated that the novel method could be applied successfully for the determination of Sudan IV in industrial and environmental water samples.

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Spectrophotometric determination of Zinc using a novel optochemical sensor in real samples

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Abstract

Optodes represent a group of integrated analytical systems with rapid growth for measurement of heavy metals due to their easy fabrication, good sensitivity and selectivity and low cost. The sensing phase consists of reagent dyes immobilized in organic or inorganic matrices [1,2]. Zinc, as the second most abundant transition metal in the body and containing its highest concentrations in the brain, plays diverse roles in many vital biological processes as structural and catalytic cofactors, neural signal transmitters or modulators, and regulators of gene expression and apoptosis [3]. A highly selective and low cost optode was developed for determination of trace concentrations of zinc in water. In this method N-cetylpyridinium chloride was applied in order to convert Bromopyrogallol red to a hydrophobe ion-pair (BPR-CPC) that was subsequently immobilized on a triacetyl cellulose membrane. At pH 8, the proposed sensor displays a calibration curve over a concentration range of 40.0 to 566.0 µM with a response time of less than 10 min, detection limit of 3.2 µM, high stability (2 months life time) and high reproducibility (RSD ≤ 2.5%). The sensor shows a good selectivity towards zinc ion compared to the considered cations including alkali, alkaline earth and transition ions.

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Spectrophotometric investigation of complex formation between two new synthetic hydroxy thioxanthone derivatives and some transition and heavy metal ions in non-aqueous solution

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Thioxanthone and derivatives are an important class of molecules which are a common heterocyclic scaffold in biologically active and medicinally significant compounds. They are the core structure of a wide variety of naturally occurring and synthetic compounds that exhibit a number of interesting pharmacological activities [1] such as: extraordinary anti-tumor, anti-parasitic, anti-cancer activity, anti-hypertensive, anti-oxidative, anti-thrombotic and cytotoxic effects. Moreover, they are potential anti-cancer drugs, even some thioxanthones containing plant extract are directly used in traditional medicine. Thermodynamic studies of complexation reactions of thioxanthones with transition and heavy metal ions not only result in important information on the thermodynamics of complexation reaction, but also lead to a better understanding of the high selectivity of these ligands towards different metal cations. In spite of the subject importance, there is only few thermodynamic information about their complexation with metal ions [2].

In this investigation the complexation behaviour of 3-(2-(6-methylbenzo[d]thiazol-2-yl)-2-hydroxy-*9H*-thioxanthen-9-one and (E)-3-(2-(4-chlorophenyl)diazenyl)-2-hydroxy-*9H*-thioxanthen-9-one, as new synthesis hydroxy thioxanthones with some transition and heavy metal cations such as Co^{2+}, Ni^{2+}, Cu^{2+}, La^{3+}, Zn^{2+}, Cd^{2+}, UO_2^{2+}, Pb^{2+}, Ga^{3+}, Y^{3+} and Th^{4+} in N,N-Dimethyl formamid (DMF) at 25 °C and 0.05 M tetra ethyl ammonium perchlorate (TEAP) were studied by spectrophotometric method. The overall and stepwise stability constants of complexes have been evaluated using the Hypspec program. The observed types of formation complexes between thioxanthones and the cited metal ions ML, ML_2 or/and M_2L. The corresponding distribution diagrams were depicted by Hyss2009 program as a new version of the older one [3]. The effect of ligand structure on the stability of complexes was investigated.

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Spectrophotometric method for acidity constants determination of few dyes Using HypSpec Program

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Different methods are available for the determination of dissociation constants, such as potentiometry, spectrophotometry, conductometric and solubility methods [1]. The potentiometric titration and spectrometric method are commonly used and widely accepted techniques. Poor solubility of the compounds hampers traditional potentiometric methods. Spectrophotometric methods can be applied to the compounds having solubility even 1 μM. However, this method is limited to molecules having chromophores at ionization center, which shows spectral dissimilarity at protonated and deprotonated form [2]. Therefore spectrophotometric method will be more useful in this case and applicable to the compounds whose concentrations or extinction coefficients are not determined.

The most widely used programs and algorithms for the determination of acidity constants from spectrophotometric data are KINFIT, SQUAD, SPECFIT, DATAN and HYPSPEC**.** HypSpec can process UV/visible, infrared, Raman, luminescence and fluorescence data, subject to the single requirement that the spectral intensity of each chemical species should be proportional to the concentration of that species in solution.

In the present work UV-Vis spectrometry was used to obtain our spectral data and we then used commercially available HypSpec software that allowed us to perform spectral subtraction followed by spectral analysis to determine the protonation constants. The dissociation constants of some dyes including Carmine, Victoria Blue R and Giemsa in aqueous solution were determined by means of HypSpec program as a new version of PHab program [3]. Also the distribution diagrams were depicted by Hyss2009. The obtained results show the good agreement with those from literature.

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Spectrophotometric method for the determination of trimethoprim in pure form and biological samples by formation charg – transfer complex with iodine

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Trimethoprim (5 – (3, 4, 5 – trimethoxybenzyl) pyrimidine – 2, 4 – diamine) is a synthetic (man –made) antibiotic that interferes with the production of tetrahydrofolic acid, a chemical that is necessary in order for bacteria and human cells to produce proteins. Trimethoprim inhibits production of tetrahydrofolic acid by inhibiting the enzyme responsible for making tetrahydrofolic acid from dihydrofolic acid. Trimethoprim inhibits the bacterial enzyme more than the corresponding human enzyme [1]. Trimethoprim is active against a wide range of Gram-positive and Gram-negative aerobic bacteria, is readily absorbed by the oral route and is widely distributed in body fluids and tissue [2]. It is also effective as an antimalarial agent [3]. In the present study, a simple and sensitive spectrophotometric method has been developed for the assay of trimethoprim in pure form and human blood serum. The method is based on the formation of charg–transfer complex between the basic nitrogen of trimethoprim and iodine. This reaction was done at room temperature and the formed complex was measured at 360 nm. Reaction conditions such as solvent, reagent concentration and reaction time have been optimized to obtain higher sensitivity and longer stability of the complex. Beer's law was obeyed in concentration range of 15 – 70 µg mL^{-1} with correlation coefficient ≥ 0.9945. The molar absorbtivity was found to be 1.56×10^{4} L mol^{-1} cm^{-1} mL^{-1} and limit of detection (LOD) was obtained. The described method can be readily utilized for the analysis of trimethoprim in tablet and syrup.

Key Word**:** Trimethoprim, Spectrophotometry, Charg–transfer complex.

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Spectrophotometric study of the stability constant of complexation of two new synthetic Thioxanthone with metal ions

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Synthetic derivatives of thioxanthone compounds are of importance in wide ranging fields as plant science, biochemistry, medicinal chemistry and pharmacology. Thioxanthone derivatives have been studied extensively owing to their medicinal properties such as antihistaminic, antiparasitic, neuroleptic, antitumor, antimalarial, antiinflammatory, antiplatelet activities and anticancer, antifungal effects; they exhibit efficient antibiotic activity against some bacteria[1]. So many Thermodynamic studies of complexation interactions between analytical reagents and different metal ions were investigated but the interaction of thioxanthones as good analytical regents in luminescence, electrochemical and spectroscopic fields [2] with transition and heavy metal less attention has been. In this study the complexation behavior of 4-hydroxy-1-methyl-3-((4-nitrophenyl)diazenyl)-9H-thioxanthen-9-one and 3-((4-chlorophenyl)diazenyl)-4-hydroxy-1-methyl-9H-thioxanthen-9-one, as new synthesis thioxanthones with some transition and heavy metal cations such as Hg^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Cd^{2+}, UO_2^{2+}, Pb^{2+}, In^{3+}, Ga^{3+}, La^{3+}, Y^{3+} and Th^{4+} in N,N-Dimethylformamid (DMF) at 25 °C and 0.05 M tetra ethylammonium perchlorate (TEAP) were studied by spectrophotometric method. The overall and stepwise stability constants of complexes have been evaluated using the Hypspec program. The observed types of formation complexes between thioxanthones and the cited metal ions ML, ML_2 or/and M_2L. The corresponding distribution diagrams were depicted by Hyss2009 program as a new version of the older one [3]. The effect of ligand structure on the stability of complexes was investigated.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ۶ الی ۸ اسفند ماه ۱۳۹۲

Spectroscopic, docking and molecular dynamics simulation studies on the interaction of two Schiff base complexes with Human Serum Albumin

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Abstract

Schiff bases and their metal complexes are important class of compounds in medicinal and pharmaceutical field. They show biological applications including antibacterial, antifungal, antiviral and antitumor activity [1, 2]. Since Schiff bases are potential anticancer drugs, it was of interest to obtain insight in to their modes of binding with the most abundant plasma carrier protein, Human Serum Albumin (HSA), using spectroscopic techniques and molecular modeling.

HSA has long been the center of attention of pharmaceutical industry due to its ability to bind a diverse range of metabolites and drugs [3].

This paper presents spectroscopic, molecular docking and molecular dynamics studies on the interaction of two Schiff base complexes with HSA protein. It was shown that the fluorescence of HSA has been quenched due to interacting with Schiff base complexes and the absorbance of Schiff base complexes in the presence of HSA has been changed. So, on the basis of spectroscopic studies both Schiff base complexes bind strongly to HSA. Molecular docking of two Schiff base complexes revealed the main differences of binding modes of these complexes and the critical amino acid residues which are located in the subdomain IB of HSA. Furthermore, the molecular dynamics simulations of these Schiff base complexes with HSA were also performed. The results represent the stability of both complexes according to their RMSDs during the simulations. Molecular dynamics results suggested that both Schiff base complexes can interact with HSA, without affecting the secondary structure of HSA but probably with a slight compactness of its tertiary structure. All approaches employed in this study indicated that Compound II has a higher binding affinity to HSA than the Compound I. To sum up all these experimental and molecular modeling results clarified that Schiff base complexes bind to HSA effectively, which could be a useful guideline for Schiff-base drug design.

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Square wave stripping voltammetric determination of captopril in pharmaceutical samples at the glassy carbon-mercury film electrode

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For many years mercury electrode was the transducer of choice in stripping voltammetry of trace chemical species owing to their high sensitivity, reproducibility, and renewability. However, because of the toxicity of mercury in common polarography, alternative electrodes and design are highly desired.

Electrodes consisting of thin metal films are commonly employed in stripping analysis since their elaboration is simple and regeneration of their surface is possible [1-3].

Mercury film electrodes have been the most widely employed metal film electrodes in stripping analysis. The application of thin mercury film electrodes (TMFEs), in situ or ex situ produced on glassy carbon (GC) have been increased.

In this study, in situ mercury film electrode production has shown extremely useful for highly sensitive adsorptive stripping voltammetric measurements of captopril at micromolar level. Operational parameters have been optimized and the stripping voltammetric performance has been investigated using square wave scans. The adsorptive stripping response is linear over the range of 2×10^{-6} M – 14×10^{-5} M of captopril, with a detection limit of 1.2×10^{-7} M. The modified electrode can be properly used for determination of captopril in two tablet samples with satisfactory results.

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Square-Wave Voltammetric Determination of Azathioprine Using a Graphene-Chitosan Modified Glassy Carbon Electrode

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Azathioprine (Aza), 6-(1-methyl-4-nitroimidazole-5-ylthio)-purine, is primarily an immunosuppressive agent, used mainly in all transplantation procedures [1]. In this study, an effective electrochemical method with nanomolar detection limit based on the GR–CS modified electrode was described for the determination of Aza. This paper proposed a Simple, rapid, green and one-step electrodeposition approach to synthesis of graphene–chitosan (GR–CS) composite onto glassy carbon electrode (GCE) under controlled potential [2]. Compared to the redox peaks at the bare electrode, the value of peak-to- peak potential separation (ΔEp) becomes smaller and both of the peaks get sharped. In addition, the current response for Aza at the bare electrode was obviously lower than that at the modified electrode. Based on the results the sensitivity of the GR–CS sensor towards Aza was greatly improved due to the excellent electric conductivity and the large surface area of GR. the maximum current response of Aza was approached at pH 7. The linear range is wide from 99.0 nM to 27 μM, with a detection limit of 47 nM. The prepared modified electrode was successfully applied for voltammetric determination of trace amounts of Aza in a pharmaceutical preparation and recovery of Aza added to blood serum samples.

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Stripping Voltammetric Detection of Thorium on the Oxine Modified carbon Paste Electrode

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Thorium is a fuel of atomic energy and their sources are soil, rocks, plants, sand and water [1, 2]. In this work, a novel approach for the determination of Thorium in different water samples is presented. The polarographic wave of Th(IV) is often masked by the large reduction wave of hydrogen ion because of the extremely negative reduction potential of Th(IV) in aqueous solution. As a result, Thorium cannot be determined by direct polarography or conventional stripping voltammetry. Thus, higher sensitivity can be achieved by square wave anodic stripping voltammetry of the thorium complex with oxine at a carbon paste electrode (CPE). Voltammograms were performed with a three electrode system consisting of a modified CPE as the working electrode, an Ag/AgCl reference electrode, a platinum wire auxiliary electrode. pH adjusted at 1 and a scan rate of 5 mV/s, and also cathodic voltammetric peak corresponding to the reduction of oxine in the complex is occurred at -1.1V (vs. Ag/AgCl). The peak current of the complex was linearly dependent upon the concentration of Th(IV) over the range of 2.0×10^{-9}-2.2×10^{-8} M, the limit of detection (LOD) 0.2 M, accumulation time 180 s, and accumulation potential was at -0.8 V. The method has been successfully applied to the determination of trace Th(IV) in different water samples.

Keyword: Thorium; Oxine complex; Carbon paste electrode; stripping voltammetry.

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Study effect of Platinum loading and Nafion content on hydrogen peroxide reduction reaction in catalyst layer of PEM fuel cell by cyclic voltammetry technique

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The catalyst layer of polymer electrolyte membrane fuel cells are consist of a powdered catalyst to catalyze the ORR electrochemical reaction at the electrode surface and Nafion to establish the protonic connection. For optimal performance of the catalyst layer, the complete interaction between the powder catalyst, Nafion and reactant is necessary. Hydrogen peroxide is responsible to be as the oxidizing agent in this work [1].

In order to have optimal amounts of platinum loading and Nafion content and to speed up the reduction of hydrogen peroxide, we prepared several inks with different platinum loading (0.1, 0.2, 0.3, 0.4 $mg.cm^{-2}$) and different Nafion content (10, 20, 30, 40 wt% catalyst). Then each Ink was placed on glassy carbon electrode and was entered to three electrode system as the working electrode accompanied with a reference electrode (Ag/AgCl), an auxiliary electrode (Graphite rod) and 0.1 M perchloric acid as electrolyte solution.

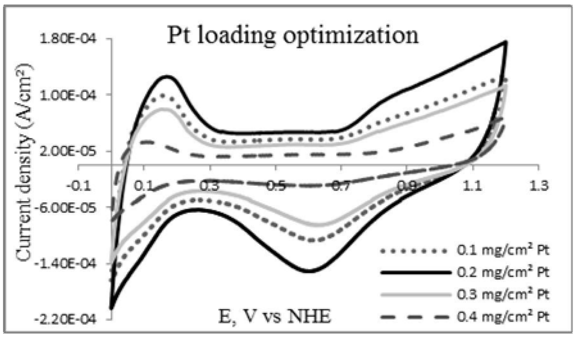

Fig.1-Cyclic voltammetry curves for different inks that made with 30 %wt catalyst Nafion content,scan rate of 50 mV/S

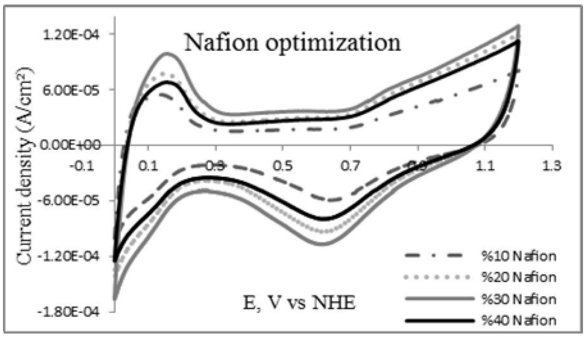

Fig.2-Cyclic voltammetry curves for different inks that made with 0.1 $mg.cm^{-2}$ Pt loading, scan rate of 50 mV/S

EAS (m^2_{Pt}/gPt)	Electrode (Pt Loading) ($mg.cm^{-2}$)
38.54×10^{-6}	0. 1
39.46×10^{-6}	0. 2
9.25×10^{-6}	0. 3
2.56×10^{-6}	0. 4

Table 1- Active surface area for different inks that made with 30 %wt catalyst Nafion content,scan rate of 50 mV/S

EAS (m^2_{Pt}/gPt)	Electrode Nafion content ($mg.cm^{-2}$)
27.43×10^{-6}	10 %wt catalyst
35.82×10^{-6}	20 %wt catalyst
38.54×10^{-6}	30 %wt catalyst
30.65×10^{-6}	40 %wt catalyst

Table 2- Active surface area for different inks that made with 0.1 $mg.cm^{-2}$ Pt loading, scan rate of 50 mV/S

The results show increasing Nafion content until 30%wt of the catalyst, increases the proton transfer and improved cell function and subsequent cell performance will be reduced, because the catalyst particles is insulated by nafion and there is no possibility of moving electrons. Moreover, increasing the platinum loading to 0.2 $mg.cm^{-2}$ improves cell performance and increases electrochemical active surface area. Higher values of this amount affords to decrement of its performance, Because the catalyst layer thickness increases and therefore mass transfer is reduced.

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Study of effective parameters on response of graphite electrode modified by ionic liquid in potentiometric measurement of Cu (II)

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Copper is the third most used metal in the world. It is one of the most widely distributed elements in the environment of industrialized countries. Copper (II) ions are soluble in water, where they function at low concentration as bacteriostatic substances, fungicides, and wood preservatives. The Cu (II) ion at lower concentrations is an essential trace nutrient to all higher plant and animal life. A number of methods are used for copper ion determination at low concentration level. Potentiometric measurements with copper ion-selective electrode allow directly the determination of free ion concentration in aqueous samples. Ion-selective electrodes provide analytical procedures for such situations as they are fast, convenient, require minimum sample pretreatment, and may also be suitable for online analysis. Further, ion selective electrodes possess wide dynamic ranges, simplicity of design, lower cost.

In this work, the graphite electrode was coated with an ionic liquid as a modifier with electrochemical method. The ionic liquid 1,3-disulfonic acid imidazolium hydrogen sulfate used as the electrolyte in electrochemical synthesis [1]. This electrode can be used as a sensitive potentiometric sensor for measurement of concentration of copper nitrate solutions. The effect of different parameters such as methods of electrosynthesis, pH, temperature, interferences on the electrode response was studied. The results showed that the prepared electrode shows a linear Nernstian response over the range of 1.0×10^{-6} to 1.0×10^{-1}M with a slope of 29.5±1.0 mV per decade change in concentration. A detection limit of 8.0×10^{-7} M was obtained. The sensor was found to have a short response time about 20s. The best method of electrosynthesis of this sensor was found in controlled potential coulometry (CPC). The optimum pH working range of the electrode was found to be 3.0 –7.0 [2].

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Study of electropolymerization of 4-nitroaniline and its application in preconcentration of heavy metals from natural water by cloud-point extraction

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Global, environmental and social problems such as water pollutions have received great attention. Heavy metals are important environment pollutants threatening the health of human populations. The toxic effect of heavy metals on human health has been investigated intensively [1,2]. Therefore, control of heavy metals special in waters is always a main task in analytical chemistry. The low concentration of heavy metals and matrix effects make it difficult to analysis trace metals in water and environment samples.

In this work, cloud-point extraction method as a benign method of separation, using non ionic surfactant (TX-114) is being applied for extraction of heavy metals before analysis [3]. This investigation involved electrochemical synthesis of a new and selective chelating agent by electropolymerization of 4-nitroaniline in acetate medium. The prepared polymer was characterized by DSC, CV, FT-IR, UV-VIS spectrometry and a suitable chemical structure was suggested. The analytical procedure is based on complexation and reaction of prepared polymer with $Cd^{2+}$and cloud-point extraction the complex. After phase separation by centrifuging and cooling in refrigerator, the surfactant rich phase containing complex was first dissolve in 1ml in 50:50 water-methanol mixture containing 1% acid nitric then subjected to FAAS. All parameters like concentration of surfactant, pH, concentration of prepared polymer and salts were optimized. In present of foreign ions no significant interferences were observed. Under optimum conditions limits of detection (LOD) and quantification (LOQ) were 0.7 and 2.5ngmL^{-1} the mean, preconcentration factor, standard error and RSD for five replicate of 10 ngmL^{-1}of Cd^{2+} were conculated to be 9.78 ngmL^{-1}, 29.3, 2.2% and 3.36% respectively.

Keywords: electropolymerization, 4-nitroaniline, cloud-point extraction.

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Study of gold nanoparticle size effect on direct colometric determination of cyanide

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Cyanide (CN^-), as a highly toxic lethal venom, is of more importance in analytical chemistry. Thus, developing a rapid, highly sensitive, and selective detection of CN^- is extremely essential for human life safety. In this study, we report a simple method to CN^- quantification based on gold nanoparticle (Au NPs, synthesized with citrate reduction) Surface Plasmon Resonance (SPR). CN^- has much effective modifying ability on Au NPs surfaces through $Au(CN)_2^-$ complex formation [1]. Thus, etching of Au NPs might be happen in the presence of CN^- that leads to a significant reduction in NPs SPR which is proportional to CN^- concentration. On the other hand, NPs size can have a remarkable effect on this phenomenon. Based on this effect, CN^- etching ability was tested on different sizes of Au NPs and it was showed that Au NPs with size of 13 nm in diameter more influenced compared with other sizes. The diminution effect of CN^- on these NPs is linearly altered over the concentration ranges of 0.5–14 µmol L^{-1}. The limit of detection was 0.15 µmol L^{-1}, and other common anions nearly have no notable influence. The method was successfully applied to the determination of CN^- in water samples.

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Study of NH_4^+ cation complexation with 1,13-bis(8-quinolyl)-1,4,7,10,13-pentaoxatridecane (kryptofix5) in some mixed non-aqueous solvents

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Abstract:

Kryptofix5 is a quinoline-terminated podand which was investigated by Vogtle and Weber in 1979 whose early work involved synthesis of podands,which are able to form stable crystalline compounds with a wide variety of alkali metal salts as well as transition metal cations [1].Podands are open-chain hosts,whose specific cation selectivity and other properties make them useful to compare to crown ethers [2,3]. In the present work , the complexation reaction of 1,13-bis(8-quinolyl)-,4,7,10,13-pentaoxatridecane (Kryptofix5) with NH_4^+ cation was studied in some binary solvent solutions of methanol-acetonitrile (MeOH-AN) , methanol-ethylactate (MeOH-EtOAc) , methanol -1,2-dichloroethane (MeOH-DCE) and methanol-methylactate (MeOH-MeOAc), at different temperatures by conductometry method. The stability constant of the resulting 1:1 complex at each temperature was determined using a computer fitting conductance-mole ratio data.Anon-linear relationship was observed for changes of $\log K_f$ of $(Kryptofix5.NH_4)^+$ complex versus the composition of the binary mixed solvents.The results show that the stability of $(Kryptofix5.NH_4)^+$ complex changes with the composition of the binary solvent solutions. The corresponding standard thermodynamic parameters($\Delta H_c^\circ$and ΔS_c°) were obtained from temperature dependence of the stability constant. The results show that the values of standard enthalpy(ΔH_c°) and standard entropy(ΔS_c°) change with the nature of the non-aqueous solvents.

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Study of Noncovalent Ineraction of macromolecules With Gold Nanoparticles By Thermal Lens Spectroscopy

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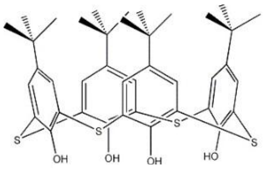
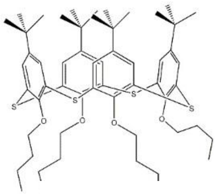
Analytical Instrumentation and Spectroscopy Laboratory

Photothermal spectroscopy is a group of high sensitivity spectroscopy techniques used to measure optical absorption and thermal characteristics of a sample. The basis of photothermal spectroscopy is the change in thermal state of the sample resulting from the absorption of radiation [1]. Plasmonic nanoparticles (NP) are the best known converters of optical energy into heat due to their unique mechanism of plasmon resonance. In this work we used of a single beam thermal lens apparatus under diod laser excitation (532 nm). In order to investigate the interaction of gold nanoparticles with macromolecules that have electronegative groups, we used thiacalix[4]aren derived (1)[1] and thiacalix[4]aren derived (2)[2], the structure of this macrocycles shows in fig (1) and fig (2) respectively.



Fig(1)



Fig(2)

After synthesis of gold nanoparticles, the solutions with different concentration of thiacalix[4]aren deriveds (50 ppm, 100 ppm, 300 ppm, 600 ppm) and same concentration of gold nanoparticles(80 ppb) was prepared and thermal lens signals for them were obtained. Overally Termal lens signals with increasing concentration of thiacalix[4]aren derived(1) was significantly increased, But the solutions that Includeded different cocentration of thiacalix[4]aren derived (2) were showed less increase in gold nanoparticles thermal lens signals than previous solutions. It means thiacalix[4]aren derived (1) more prevent from quick dispersion of heat and thus more enhance thermal lens signals of gold nanoparticles than thiacalix[4]aren derived (2). This is due to the fact that gold nanoparticles have stronger noncovalent ineraction with thiacalix[4]aren derived (1) than thiacalix[4]aren derived (2) [2].

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[1] 15,11,17,23 Tetra tertio butyl- 25,26,27,28 Tetra hydroxyl- Tetra thiacalix[*4*] arene.

[2] 5, 11, 17, 23- Tetra tertio butyl-25,26,27,28-Tetra butoxy-2,8,14,20-Tetra thia calix[*4*] arene.

Subject:

Study of photocatalytic degradation of methyl blue in presence of nanocatalysts of NiO, NiS, Ni_2O_3 and Ni_2S_3 and nanocomposits of $NiO_{0.5}S_{0.5}$ and $Ni_2O_{1.5}S_{1.5}$

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Abstract

Textile industries produce large volume of colored dye effluents which are toxic and non-biodegradable. These dyes create severe environmental pollution problems by releasing toxic and potential carcinogenic substances into the aqueous phase.Various chemical and physical processes such as precipitation, adsorption, air stripping, flocculation, reverse osmosis and ultra-filtration can be used for color removal from textile effluents. However these techniques are non-destructive, since they only transfer the non-biodegradable matter into sludge, giving rise to new type of pollution, which needs further treatment [1]. Photocatalytic degradation has proven to be a promising technology for the removal of various organic pollutants in wastewater for its many attractive advantages, including the environmental friendly feature, relatively low cost and little energy consumption [2].

In this research, nano catalysts of NiO, NiS, Ni_2O_3, Ni_2S_3, $NiO_{0.5}S_{0.5}$ and $Ni_2O_{1.5}S_{1.5}$ were synthesized by controlled precipitation method. The prepared nanoparticles were characterized by FT-IR spectrophotometry, X-ray diffraction patterns, transmission electron microscopy and energy dispersive x-ray analysis. The photodegradation of methyl blue was studied at presence of prepared photocatalsts and under UV irradiation. a degradation efficiency > 91 % was obtained at optimized conditions of pH 7, 0.5 g/L of photocatalyst dosage and 20 mg/L of day. The photodegradation reaction was shown a psedue first-order kinetic. The photoreactivity of prepared nanocatalysts was seen as : $Ni_2O_{1.5}S_{1.5} > NiO_{0.5}S_{0.5} > NiS > NiO > Ni_2O_3 > Ni_2S_3$.

The reuseability of as photocatalyst $Ni_2O_{1.5}S_{1.5}$ was investigated in four-cycles of degradation reaction with degradation efficiency of 90−80 %.

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[1] S. Kansal, M. Singh,"Studies on photodegradation of two commercial dyes in aqueous phase using different photocatalysts", J. Hazard. Mater. 141 (2007) 581−590.

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Subject:

Study of photocatalytic degradation of methyl blue in presence of nanocatalysts of NiO, NiS, Ni_2O_3 and Ni_2S_3 and nanocomposits of $NiO_{0.5}S_{0.5}$ and $Ni_2O_{1.5}S_{1.5}$

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Abstract

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Study of self aggregation processes of some organic dyes using chemometric methods

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The self-aggregation processes are the important processes which are interest in chemistry. Some organic dyes can aggregate to form dimmers, trimmer and higher aggregation and show new spectrophotometric properties. So the UV-Vis absorption spectrophotometric method has been used to study the aggregation processes and extract some thermodynamic parameters such as equilibrium constant, enthalpy and entropy [1-3]. But the main problem of UV-Vis absorption data is spectra overlapping. In such cases, recording multiwavelength data and its analyzing using chemometrics method is very good solution. In this work, two chemometrics methods, traditional fitting of parameters and target transform fitting were used to find parameters. Our investigation on simulated data showed that the first method has a main disadvantage which is that in the presence of interfering species such can lead to incorrect results. But the second method resolved such data successfully.

Self- aggregation process of dye can be studied at two different methods. For this purpose, some samples with different concentration of dye were prepared at defined constant temperatures, 298 K and the absorption spectra were recorded. This process was repeated for three other temperatures, 303, 308 and 313K. From the analysis of obtained data equilibrium constant of dimization and trimmerization process were obtained. After determination of equilibrium constant at different temperatures, thermodynamic parameters can be realized using van’t hoff equation. The disadvantages of this method are the long time and high consumption of material. To overcome to these disadvantages of the method, temperature changing in the constant concentration and recording spectrum in each temperature can be used. In this method, a solution by known concentration at which equilibrium is established between the species was prepared and at different temperatures the absorption spectra of the solutions were recorded. Thus the size of obtained data matrix was number of temperatures × number of wavelengths. The analysis of these data gave ΔH^0 and ΔS^0, directly. The proposed methods were used to calculation of equilibrium constant, ΔH^0 and ΔS^0 of self-aggregation processes of methylene blue and crystal violet.

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study of ternary system containing o-phenylenediamine, dipicolinic acid and Cu2+ ion by potentiometric titration method using Hyperquad program

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Polymerised o-phenylenediamine (OPD) is used for various applications such as for immobilization of bioactive materials, electrocatalysis, corrosion inhibition, and as electrochromic materials [1]. Among dicarboxylic acids, dipicolinic acid (dipic) as a very important carboxylate derivative, has attracted much interest in coordination and solution chemistry [2]. In the recent years, solution study about a large number of proton transfer compounds including dipic and some amino bases and their complexes with metal ion have been reported [2], but so far the interaction between dipic and OPD, in 1:2 ratio and its metal complexes has not reported.

In this study, the protonation constants of dipic and OPD, in all of the probability protonated forms, the equilibrium constants for the dipic-OPD (1:2) proton transfer system and the stoichiometry and stability constant of the complexation of this system with Cu^{2+} ion in aqueous solution were investigated by potentiometric pH titration method. In order to evaluate the stoichiometry and stability of Cu^{2+} complexes with the dipic-OPD proton transfer system in aqueous solution, the equilibrium potentiometric pH titration profiles of dipic, OPD and their 1:2 mixture were obtained in the absence and presence of Cu^{2+} ion.

The potentiometric pH titration curves of dipic, OPD and their corresponding 1:2 mixture in the presence of Cu^{2+} ion were fitted by Hyperquad 2008 program as a new version of the older one [3]. The corresponding distribution diagrams were depicted using Hyss 2009.

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Study of the electrochemical behavior of $LaFe_{0.2}Ni_{0.2}O_3$ nanoparticles for the methanol oxidation in DMFCs

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Abstract

Direct methanol fuel cells (DMFCs) are considered due to their high efficiency and low pollution. One of the problems is usage the noble metals with high price and poisoning as catalysts in DMFCs [1]. Perovskites due to thermal stability and unique crystalline structure are the useful catalyst [2]. We followed different approaches for the development of an effective and low-cost catalyst for DMFCs. Nanocrystalline ($LaNi_{0.2}Fe_{0.2}O_3$) powder was prepared by co-precipitation method. It was characterized by X-ray diffraction (XRD) and scanning electron microscope (SEM). The nanocatalyst activity for methanol oxidation was tested with a glassy carbon (GC) electrode by cyclic voltammetry, chronoamperometry and anodic Tafel polarization techniques. The results showed that it has a suitable catalytic activity toward methanol oxidation. The CV curves on the GC/$LaNi_{0.2}Fe_{0.2}O_3$ electrode in 0.8 M methanol and 1M NaOH at various sweeping rates in the range of 10-300 mV s^{-1} were recorded. The linear relationship between the square root of the scan rate and the peak current density estimate the control of the methanol oxidation via the mass transport of the methanol (R^2=0.99) [3]. The polarization resistance (R_p) is a criterion for charge exchange on the surface catalyst. The R_p and j_0 was obtained 77.43Ω and 8.94 mA cm^{-2}, respectively. It shows the larger exchange current density lead to the faster reaction by mentioned catalysis.

Keywords: $LaFe_{0.2}Ni_{0.2}O_3$ perovskite

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Studying the Adsorption Process of Riboflavin on Silver-Deposited Fe_3O_4 Nanoparticles

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There is an over-expression of riboflavin receptors on the cell membrane of breast and prostate cancer cells [1]. These receptors allow riboflavin to enter the cell and assist in the formation of the redox cofactors that are necessary for basic functions of the cell. So riboflavin receptors can be used as a biomarker to direct the drug to the cancer cells only. In this study, the adsorption of riboflavin onto silver-deposited Fe_3O_4 nanoparticles (Ag/Fe_3O_4) is described. The Ag/Fe_3O_4 magnetic nanoparticles were synthesized [2] and were used as adsorbent. Characterization of the obtained nanoparticles was achieved by FTIR, TEM and XRD. Batch adsorption experiments were performed to investigate the adsorption conditions. The influence of several parameters such as nanoparticles dosage, pH of the solution, riboflavin concentration, contact time between reagents, temperature, ionic strength and presence of halide anions was studied. Experimental results indicated that Ag/Fe_3O_4 nanoparticles can adsorbed more than 90% of riboflavin under the optimum experimental conditions of nanoparticles dosage of 4.0 mg, a pH of 6.0±0.2, and a contact time of 2.0 minutes, when initial riboflavin concentrations of 0.02 mmol L^{-1} was used. Orientation of riboflavin molecule toward Ag/Fe_3O_4 surface was proposed to be perpendicular and dissolution of the silver layer from Ag/Fe_3O_4 nanoparticles was seen in the presence of halide anions. Adsorption isotherm modeling revealed that the Dubinin-Radushkevich model could better describe the adsorption of riboflavin onto Ag/Fe_3O_4 nanoparticles as compared to Freundlich model. Also, it was concluded that the adsorption process is physical in nature. Kinetic data was appropriately fitted to the pseudo-second-order adsorption rate model.

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Sub-nanomolar detection of zinc on the ion-imprinted polymer modified glassy carbon electrode

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There is an increasing demand for sensors able to give a stable response, in short time, but in the same time sensitive, selective, with high precision to use and inexpensive. Monitoring trace heavy metals like zinc in industrial wastewater is important for human health and the environment [1,2].

In this work, A simple, low cost, easy, rapid, sensitive square wave anodic stripping voltammetry (ASV) has been developed for determination of ultra trace levels of zinc using the glassy carbon electrode (GCE) modified by Zn(II) ion-imprinted polymers. Zinc ion-imprinted polymers were synthesized by thermal polymerization and zinc ions were moved by leaching the IIP by HCl and utilized for the electrode modification. The potential was scanned between -0.3 to 0.4 and variable affecting parameters such as pH, deposition duration time, deposition potential and stirring speed were optimized. The resulting calibration curve exhibited a linear response over a concentration range of 5×10^{-10} to 1×10^{-8} with correlation coefficient 0.9989. The detection limit was 0.5nM. Finally, the method was successfully applied to the determination of Zinc in the biological samples and human hair.

Keywords: Ion imprinted polymer(IIP), Anodic stripping voltammetry(ASV), Glassy carbon electrode(GCE)

References

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Successive projections algorithm applied to spectrophotometric data for the simultaneous determination of methyl paraben and phenol in cosmetic products

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Parabens are a group of effective preservatives widely used for the control of microbial organisms in a wide variety of cosmetic products [1]. It has been shown that phenol (also used in different cosmetics) is strong skin sensitizer and allergen and parabens possess estrogenic activity [2]. In order to ensure compliance to existing or future regulation, reliable methodologies to identify and quantify preservatives are required. In this study, a novel variable selection strategy for multiple lineal regression (MLR), the successive projections algorithm (SPA), was applied to spectrophotometric data (200–350 nm) for the simultaneous determination of methyl paraben and phenol in cosmetic products. This selection method uses simple operations in a vector space to minimize variable collinearity and has become an interesting variable selection strategy for multivariate calibration. For this purpose, variable selection by the SPA is used in order to obtain simple MLR models based on a small subset of wavelengths. In this work, twelve and ten wavelengths for phenol and methyl paraben, respectively, were selected to construct calibration models in order to solve successfully the serious spectral overlapping in samples containing these analytes. The concentrations of the calibration matrix ranged from 0.1 to 25.0 mgL^{-1} for phenol, and from 0.1 to 20.0 mgL^{-1} for methyl paraben. The relative errors of prediction (REP) for the validation set were 2.5%, 3.1% for phenol and methyl paraben, respectively. Cosmetic products were also analyzed and a recovery study was carried out to verify the accuracy of the proposed method with satisfactory results (94-105%). The proposed method is a simple and efficient spectrophotometric method to determine phenol and methyl paraben directly and simultaneously in cosmetic products without any derivatization or complex separation techniques.

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Sulfated β-cyclodextrin doped polypyrrole film deposited on pencil graphite as an ion selective electrode for determination of Phenobarbital

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Conducting polymers (electroactive conjugated polymers, ECPs) have emerged as one of the most promising transducers for chemical sensors. This is related to the unique electrical, electrochemical and optical properties of conjugated polymers that can be used to convert chemical information into electrical or optical signals in the solid state [1]. In the present research, a conducting polypyrrole (ppy) matrix doped with the sulfated β-cyclodextrins (sβ-CD) was electrosynthesized on a pencil graphite electrodeand used as a potentiometric sensor for the detection of phenobarbital. The sβ-CD was immobilized within the ppy backbone during the growth of the polymer at the surface of pencil graphite electrode by means of cyclic voltammetry. The effect of polymerization condition and on the characteristics of potential response of the sensor was investigated. The concentration of pyrrole and sβ-CD, the electrode type, the number of voltammetric cycles and pH were optimized. Under the optimized conditions, the proposed sensor works satisfactory in the concentration range of 10^{-3} to 10^{-1} mol L^{-1} of phenobarbital with a sub-Nernstian slope (~ 40 mV decade^{-1} activity) and a fast response time of 5 s. The developed potentiometric sensor has been applied for the potentiometric determination of phenobarbital in wastewater and pharmaceutical formulation samples.

Keywords: Phenobarbital, sulfated β-cyclodextrin, polypyrrole, ion selective electrode.

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Supramolecular chemistry approach to the design of a highly selective fluorescence chemosensor for identification of mercury ions

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Abstract:

Exposure to even very low levels of toxic heavy metals such as lead, mercury and cadmium is known to cause neurological, reproductive, cardiovascular, and developmental disorders, which are more serious problems for children particularly. Accordingly, great efforts have been devoted to the development of fluorescent and colorimetric sensors, which can selectively detect these metal ions [1]. In this study, a new fluorescent probe 1, 5-(4-(N-octyl-N-phenylamino)benzylidene)-dihydro-2 thioxopyrimidine-4,6(1H,5H)-dione, as an electron donor–acceptor supramolecular compound, was designed and synthesized for a highly selective colorimetric identification of mercury ions [2]. The free probe 1 has a relatively strong fluorescence emission band at 376 nm in methanol solution. Adding Hg2+, as a fluorescent quencher to probe 1, leads to emission turn-off. Moreover, the maximum absorbance intensity of probe 1 was decreased with about 37 nm red-shift (from 493 to 530 nm), accompanied with the absorbance color change from orange to violet, which was attributed to the reaction of the thiol groups in probe 1 with mercury ions to form a stable complex. The detection limit for mercuric ions using fluorescence spectroscopy was estimated to be ≈ 2.4 ×10^{-9} μM. Similar spectral behavior upon binding probe 1 with other metal ions such as Al3+, Cu2+, Ag+, Pb2+, Zn2+, Cd2+, Ni2+, Fe3+, Co2+ and Mn2+ has also been reported. The interference from these metal ions can be neglected. Therefore, these results clearly validate that probe 1 can be exploited as a selective colorimetric visual chemosensor for Hg2+ in non aqueous solutions.

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Supramolecular solvent-based microextraction of Loratadine and its determination using high-performance liquid chromatography

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Loratadine is a long acting and non-sedating tricycle antihistamine, used for relief of allergic seasonal symptom and skin rash [1]. Supramolecular solvents have a unique array of physicochemical properties that render them very attractive to replace organic solvents in analytical extractions [2]. Optimization of Supramolecular solvent-based microextraction of Loratadine and its determination of with high-performance liquid chromatography were investigated. A factorial design (FD) and a central composite design (CCD) were applied to evaluate the Supramolecular solvent-based microextraction procedure. The effect of four parameters on extraction efficiency was investigated. The factors studied were decanoic acid concentration, THF percentage (v/v), pH and extraction time. According to half factorial design results, the effective parameters were decanoic acid concentration, THF percentage (v/v) and pH. Next, a central composite design was applied to obtain optimal condition. The optimized conditions were obtained at 110 mg of decanoic acid, 2.62 mL of THF and pH=3. The enrichment factor of Loratadine was 150. The linearity was 5–250 ng mL^{-1} and the extraction recovery was > 92 %. RSD for intra and inter day of extraction of Loratadine were 4.2% and 7.8%, respectively for five measurements. The developed method was successfully applied for the determination of Loratadine in urine and water samples.

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Surface modification of magnetic nanoparticles by LTA zeolite and their using for removal of Pb^{2+}ions from aqueous solutions

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Zeolites are crystalline alumino silicates which have open framework structures of three-dimension tetrahedral units. They can be holding great potential for application as ion exchangers (water-softening agent) and affinity toward heavy metals due to their high specific surface area[1]. Pore diameters of Zeolite A is small at 4.2Å and it has a void volume fraction of 0.47, with a Si/Al ratio of 1.0 and decomposes at 700ºC[2]. One of the disadvantages using of zeolite is difficulty of cumulation among time-consuming, so for solved this problem and accelerated cumulation we used magnetic nanoparticles. In recent years, magnetic nanoparticles(MNs) attracted a great deal of attentions. In this work crystaline zeolite was prepared on the surface of MNs without changing the properties of zeolite and were to provide suit conditions for removal ions from aqueous solutions. The zeolite was synthesized via a sol-gel process and hydrothermal technique and magnetic zeolite A was synthesized by adding Fe_3O_4 in the precursor of zeolite. Magnetic zeolite A was characterized with scanning electron microscopy (SEM), XRD and FT-IR spectroscop. Magnetic zeolite A was used for removal of heavy metal (Pb^{2+} ions) from aqueous solutions. Finally collected samples were analyzed with atomic absorption spectroscopy (AAS). Based on the results, adsorption capacities were obtained more than 94% at less than 15 minutes at room temperature for different concentrations. Showed that this adsorbent can be used as a good candidate for fast and efficient removal of Pb^{2+} ions from aqueous solutions. This condition were obtain without time consuming steps. A comparison study showed that the magnetic zeolites had good capacity for lead removed as well as non-magnetic zeolites.

Keywords: Magnetic nanoparticles, Heavy metal, surface modification, zeolite A

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Surfactant assisted dispersive liquid phase microextraction followed by ion chromatography for determination of chloroacetic acids in aqueous samples

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Abstract

Chloroacetic acids (CAAs) are certain by-products from chlorination disinfection process of drinking and portable waters. Potential health risks to humans from long term exposure to particular CAAs has led to increased efforts to monitor and reduce their concentration in drinking waters [1]. Due to the fact that CAAs exist as anions in drinking waters, ion chromatography (IC) is an obvious choice for their separation and detection. Because of trace levels of CAAs in drinking water by most current methods, including IC, it is necessary to employ a preconcentration step [2]. Surfactant assisted dispersive liquid phase microextraction (SADLPME) is a powerful preconcentration technique and has been shown many advantages such as very high extraction efficiency, low cost and low matrix effects [3]. In this research SADLPME was used for extraction and preconcentration of mono-, di- and trichloroacetic acids from aqueous matrix before IC analysis. Ion exchange chromatography and ion exclusion chromatography as two different modes of IC were examined for separation of CCAs. Ion exchange chromatography showed better peak shapes and good resolution for target analytes. Affecting parameters such as type of surfactant, kind of extraction solvent, ultrasonication effect and extraction time were investigated and optimized for extraction procedure. In this research, cationic types of surfactants were utilized because CCAs are in anionic forms. Aliquat 336 (as an organic solvent soluble surfactant) and cetyltrimethyl ammonium bromide (as a water soluble surfactant) were applied for this investigation. Aliquat 336 revealed better results. Under the optimum conditions, analytical parameters such as linearity, precision and limit of detection were evaluated. Limit of detections were between 0.31 and 1.2 μg L^{-1}and relative standard deviations were less than 9.1% under the optimized condition. The calibration curves were obtained in the range of 1–1000 μg L^{-1}with a good linearity (r^2> 0.9942). Finally the method was successfully applied for the determination of CAAs in tap, pool and river waters.

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Surfactant assisted solid-phase extraction of benzodiazepines from water samples using silica supported Fe_3O_4 magnetic nanoparticles with high-performance liquid chromatographic analysis

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Abstract

In this work, a simple and effective analytical method for solid phase extraction and pre - concentration of five benzodiazepines in water samples was developed by using silica-coated (Fe_3O_4/SiO_2core/shell) magnetite nanoparticle modified by surfactants. The five kinds of benzodiazepines were selected as the model analytes including diazepam, oxazepam, clonazepam, alperazolam and midazolam to validate the extraction facility of Fe_3O_4/SiO_2 microspheres. Fe_3O_4 nanoparticles were prepared by the chemical co-precipitation method of $Fe^{+3}$and Fe^{+2} as reaction substrates and NH_3–H_2O as precipitant [1].Then, the surface of Fe_3O_4 was coated with silica by hydrolysis of tetraethylorthosilicate as the silica source using a sol–gel procedure[2]. The properties of the Fe_3O_4/SiO_2 nanocomposite were characterized by VSM and FTIR. Compared with pure magnetic particles, silica coatings have high stability when pH value is sufficiently low and would protect the Fe_3O_4 core from oxidation in acidic conditions[3]. In order to increase their adsorptive affinity towards organic compounds, cetyltrimethylammonium bromide (CTAB) were added, which was adsorbed on the surface of the Fe_3O_4/SiO_2 nanoparticles. The variables involved in SPE were studied and optimized. The optimum conditions were: mass of sorbent, 50 mg; donor phase volume, 30 mL; pH, 10; and amount of surfactant, 20 mg. After magnetic separation of adsorbent, the adsorbent was eluted with 0.50 mL methanol prior to its analysis by high-performance liquid chromatography. Under optimum conditions, the calibration curves weres linear in the range of 0.10 –10 $\mu g.mL^{-1}$. Detection limits obtained for diazepam , oxazepam , clonazepam , alperazolam and midazolam were 0.0186, 0.0162, 0.097 , 0.032 and 0.024 $\mu g.mL^{-1}$, respectively.

Keywords: Solid-phase extraction(SPE), Nanocomposite Fe_3O_4/SiO_2 , Sol–gel, Benzodiazepines drugs, Water samples.

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Survey and determination of mycotoxins in wheat from Iran by LC/Fluorescence using immunoaffinity column Cleanup

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Wheat is the one of most important grains consumed in the world. Food contamination with toxigenic moulds has attracted increasing attention over the last three decades. Most grain, such as wheat, maize, bean and rice, can be infested by filamentous and microscopic fungi. Some genera can produce toxic secondary metabolites, namely mycotoxins, which impact on food safety [1]. AFs (B_1, B_2, G_1 and G_2) are mycotoxins produced by the fungi Aspergillus flavus and A. parasiticus. These mycotoxins are hepatotoxic and carcinogenic in humans. A. flavus produces AFB_1 and B_2, while A. parasiticus gives rise to AFB_1, B_2, G_1 and G_2 [2]. In this study a liquid chromatographic method was developed for the simultaneous determination of aflatoxins (AFs) (B_1, B_2, G_1, G_2), ochratoxin A (OTA) and zearalenone (ZEA) toxins in wheat. HPLC analyses were performed on a Sykam (Eresing, Germany) HPLC system equipped with an S2100 pump, an S7131 reagent organizer, an S4011 column thermo controller and a RF-10Axl fluorescence detector. For aflatoxin and zearalenone analysis, Genesis RP C18 analytical column (150 × 4.6 mm, 5µm) and for ochratoxin analysis, Chromolith® performance RP18 analytical column (100 × 4.6 mm) was used. The excitation wavelength for determination of AFs, OTA and ZEA was 365, 333 and 275 nm and the emission wavelength was 445, 443 and 450 nm, respectively. For aflatoxin, ochratoxin and zearalenone, mobile phase consisted of a solution of water: methanol:acetonitrile (60:20:20), water:acetonitrile:acetic acid (99:99:1) and methanol:acetonitrile:water (8:46:46), respectively. Clarity software was used for data management. For determination of AFs, OTA and ZEA concentration a UV-Visible spectrum of AFs, OTA and ZEA stock solution against solvent used for solution in reference cell was obtained using a Shimadzu UV-1700 Pharma spec. (Tokyo, Japan). Spectrophotometer equipped with a standard 10 mm path length spectrophotometer cell. For this study, a total of 231 wheat samples were analyzed. Mycotoxins were extracted and purified from the samples using immunoaffinity (IAC) columns. 2.67% and 2.21% of examined wheat samples contained AFB_1 and OTA. The method was based on the extraction of AFs, OTA and ZEA finely ground wheat sample with 80% methanol and 60% acetonitrile solution, respectively. 2.60%, and 2.16% of examined wheat samples contained AFB_1 and OTA. LOD was 0.27, 0.04, 0.23, 0.04, 1.18 and 21.56 for AFB_1, AFB_2, AFG_1, AFG_2, OTA and ZEA, respectively. Considering these results, these special commodities should not be a health concern for these collectives; however, special attention should be focused on imported foods distributed in special retail shops.

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Survey of the interaction between oleuropein and DNA using voltammetry, fluorescence titration and thermal denaturation studies

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The study of interactions between small molecules and DNA is of importance because these interactions are the basis of carcinogenic and therapeutic properties of many carcinogenic species, antitumor and antivirus drugs [1]. A large percentage of chemotherapeutic anticancer drugs are compounds that interact with DNA directly or prevent the proper relaxation of DNA (through the inhibition of topoisomerases) [2]. Oleuropein, the major active compound in olive leaf, is well known for its benefits for human health. Since the anti-cancer activity of oleuropein has been reported [3], we studied the interaction between Ole and double-stranded DNA employing fluorescence titration, cyclic voltammetry and thermal denaturation studies. The fluorescence intensity of Ole showed a gradual decrease with the increase of DNA. The fluorescence titration resulted in a binding constant and number of binding sites of 8.61×10^3 L/mol and 1.05, respectively. Additionally, both anodic and cathodic peak currents of Ole decreased in the presence of DNA. The peak potential of the oxidation wave shifted to a more positive value whereas that of the reduction wave shifted to a more negative value. According to these observations, it seems that the decreases of peak currents of Ole after an addition of DNA are caused by the intercalation of Ole to the bulky, slowly diffusing DNA, which results in a considerable decrease in the apparent diffusion coefficient. In DNA denaturation, also called DNA melting, duplex DNA was thermally denatured into single-strand components in the absence and presence of Ole and at the same time, the absorbance of the DNA at 260 nm was monitored at various temperatures. The value of T_m was obtained from the plot of $A_{25\,°C}/A$ versus temperature and was found to increase by 5°C upon binding to Ole, thereby confirming the intercalative mode of binding.

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Survey on activated carbon produced from oak wood for removal of trihalomethanes precursors from water

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Abstract

Experiments were carried out to remove trihalomethane precursors by adsorption technique using activated carbon prepared from oak wood (ACOW) as a novel adsorbent. The prepared adsorbent was characterized by BET surface area measurement and SEM analysis. A series of batch experiments were performed to investigate the effect of experimental conditions (e.g. pH value, initial Total Organic Carbon (TOC) concentrations, contact time and adsorbent dose) on the removal of TOC and UV254 from water [1, 2]. The TOC adsorption efficiency and UV254 removal remains almost constant within the pH range of around 3-8. At the optimal adsorption condition of ACOW for TOC and UV254 elimination (pH = 6.5, contact time 120 min) about 53% and 62% removal efficiency was obtained for TOC and UV254 removal using adsorbent dose of 5 g/100 mL at initial TOC concentration of 1 mg/L. Fitting the experimental equilibrium data to various isotherm models such as Langmuir, Freundlich, Tempkin and Dubinin- Radushkevich models showed that the adsorption data of the ACOW at well with the Freundlich isotherm equations this indicates that THM precursor uptake was mainly directed by a heterogeneous physical adsorption [3]. The kinetic data were best fitted to pseudo-second-order and intraparticle diffusion kinetic models. These results demonstrate that the ACOW are easily prepared, enabling promising applications for the removal of TOC and UV254 from water, therefore it can be an attractive option for THM precursor removal from water.

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Synthesis and antibacterial activity of CuO-montmorillonite nancomposite

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Cuo-montmorillonite (CuO-MMT) nanocomposite was synthesized by thermal decomposition methods[1]. Diffuse reflectance spectroscopy (DRS), X-ray diffraction analysis (XRD), Fourier transform infrared spectroscopy (FT-IR) and transmission electron microscopy (TEM) were used to characterize the product. The resultant particles are nearly spherical and particle size is in the range of 3-5 nm and the support size is obtained more than 100 nm by XRD and TEM. The XRD patterns confirmed the formation of CuO nanoparticles and implied that CuO nanoparticles can exist in micropore of MMT. The FT-IR and DRS results showed that low amounts of Cu^{2+} ions have located at inner layers of MMT. The antibacterial activity of CuO-MMT nanocomposite was tested against *Eschericia coli* [2]. The results indicated that MMT had no antibacterial activity and the MIC value for the matrix was 100 ng/ml. But the MIC values obtained for CuO-MMT and CuO nanoparticles were 0.1 ,10 ng/ml respectively and they showed efficient bacterial effect. The diffuse reflectance spectra shows that the value of band gap enegy for CuO-MMT nanocomposite (2.7 eV) is more than CuO nanoparticles (1.2 eV) so in additional to antibacterial effect, this nanocomposite can be studied as photocatalyst under visible light.

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Synthesis and application of amine-functionalized magnetic sorbent for efficient enrichment of parabens from different matrices

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An Amino-functionalized magnetic mesoporous nanoparticle (Fe_3O_4@MCM-41-NH_2) was synthesized and applied to the extraction and determination of three parabens, methyl-paraben, propyl-paraben and butyl-paraben, by in situ derivatization solid-phase extraction method. Due to the hydrogen bonding and hydrophilic interactions, the functionalized magnetic nanoparticles displayed excellent extraction performance for parabens. This approach improved both the extraction efficiency and sensitivity in a simple way since the derivatisation reaction occurs at the same time as the extraction procedure. Acetylated derivatives were selectively determined using gas chromatography. Parameters affecting SPE procedure including amount of sorbent, type of eluting solvent, extraction and desorption times, and ionic strength were studied and optimized through a one at a time method. The performance of the method was studied in terms of accuracy, linearity, precision and limits of detection. Limits of detections were < 0.05 ng mL^{-1} and linearity was in the range of 2-250 ng mL^{-1}. Finally, the applicability of the proposed method is demonstrated for several real samples including tap water, sea water, Shampoos and moistening creams. Quantitative recoveries (≥82%) and satisfactory precision (RSD≤12%) are obtained. Since no matrix effects are observed, quantification was readily carried out by external calibration with ultrapure water standards.

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Synthesis and application of amino-functionalized Fe_3O_4-graphene nanocomposite as an adsorbent for extraction and determination of Cu (II)

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Abstract:

In small quantities, certain heavy metals are nutritionally essential for a healthy life, while at higher levels of concentration their presence causes problems due to increased toxicity. For this reason, the development of reliable methods for removal and determination of heavy mrtals has been a very active area of research [1,2]. In this work, a solid phase extraction procedure was presented for separation and preconcentration of trace levels of Cu (II) ion using amino functionalized Fe_3O_4 graphene nanocomposite. Experimental parameters such as pH, type of eluent solvent, sample volume, temperature, amount of nanocomposite and ultrasonic time were studied in detail. 50 mL copper ion(10 μg L^{-1}) was add to 0.005g nanocomposite at pH6.0 then the mixtures in the flasks were ultrasonicated for 10 minutes to guarantee a good dispersion of nanocomposite and then assemble the nanocomposite by magnet and copper ion desorbed from nanocomposite with 0.5mL HCl 0.1M and then 0.5mL sample directly injected into the flame atomic absorption spectroscopy. The effect of potentially interfering ions on the recovery of Cu (II) was also examined. Under optimized conditions, the method provided a enhancement factor of 95.08, had a linear range from 1.0-100 ng mL^{-1} of Cu (II), a detection limit of 0.9 ng mL^{-1} and a relative standard deviation 1.23 % at 10 ng mL^{-1} of Cu (II). The method was validated by the extraction and determination of Cu (II) in eggplant, red lentil, and mushroom samples.

Keywords: Flame atomic absorption spectrometry (FAAS); Separation and preconcentration ; copper ;Eggplant; Red lentil; Mushroom

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Synthesis and application of Graphene-based magnetic nanocomposite solid-phase extraction for preconcentration and determination of lead and cadmium in milk, yoghurt and water samples

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Graphene (G) as a novel class of nanotube shows fast adsorption equilibrium and analyte elution. Because the large delocalized electron system of G can form a strong stacking interaction with the benzene ring [1], it could be considered as an adsorbent for adsorption of benzenoid compounds. Liu et al suggested G as a sorbent for solid phase extraction(SPE). A new procedure for SPE, based on magnetic or magnetically modified adsorbents, called magnetic solid-phase extraction (MSPE), was developed [2], which demonstrated superior adsorption efficiency and a rapid separation from different matrices by an external magnetic field [3].In this study, a graphene-based magnetic nanocomposite (G-Fe_3O_4) was synthesized and developed as the adsorbent for solid-phase extraction of Pb(II) and Cd(II) in milk, yoghurt and water samples prior to their analysis by flame atomic absorption spectrometry (FAAS). The structure of the resulting products was confirmed by Fourier transform infrared (FT-IR) spectra, X-ray diffraction (XRD) spectrometry. Effects of various parameters on the preconcentration of ions were studied. To obtain the adsorption capacity, both the Langmuir and Freundlich adsorption isotherm models were used. The calibration graph was in the range of 0.5-90 µg L^{-1} for Cd and 1.5-200 µg L^{-1} for Pb with a detection limit of 0.16 and 0.50 µg L^{-1} for Cd(II) and Pb(II), respectively. The relative standard deviations (RSDs, n=10) of 50 µg L^{-1} of Pb and Cd were 3.30 and 2.13 %, respectively. The maximum sorption capacity of the nanocomposite was found to be 16.94 and 13.69 mg g^{-1} for Pb(II) and Cd(II), respectively. The results indicated that the present method can be reliably used for determination of Pb(II) and Cd(II), with good recoveries in the range of 95 and 105.5% for real samples.

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Synthesis and application of manganese- zinc sulfide nanoparticles loaded on modified activated carbon for the extraction and preconcentration of heavy metals

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In the part of the project, ZnS-Mn nanoparticle loaded on activated carbon has been synthesized and characterized by different techniques including XRD and SEM. After modification by a new Schiff' base ligand (SMPhEA) it was applied as a solid phase extractant for simultaneous determination of trace amounts of Cu (II), Pb (II) and Fe(III) by flame atomic absorption spectrophotometry. Experimental conditions for effective adsorption of trace levels of Cu (II), Pb (II) and Fe(III) were optimized by using one at the time method. The optimum pH value for the separation of metal ions on the new sorbent was 5, and the adsorbed metal ions could be completely eluted with 2.0 mL 2.0 mol L^{-1} HNO_3 solution. Common coexisting ions did not interfere with the extraction and determination of target metal ions. The detection limits of the method were found to be 2.21, 0.97 and 2.34 μgL^{-1} for Cu (II), Pb (II) and Fe(III), respectively. The relative standard deviation (RSD) of the method was lower than 4.0% (n = 8). The enrichment factor was 375, 500, 250 for Cu (II), Pb (II) and Fe(III), respectively. The method was successfully applied for the extraction and determination of trace Cu (II), Pb (II) and Fe(III) in various real water and food samples with satisfactory results. Adsorption isotherms of heavy metal ions on adsorbents were determined and correlated with common isotherm equations such as Langmuir, Freundlich and Tempkin models. These studies were showed that the Langmuir isotherm model was fitted well with adsorption data for Cu (II) and Pb (II). The maximum adsorption capacity for Cu(II) and Pb (II) was 13.75 mg g^{-1} and 11.76 mg g^{-1} at 298 K, pH = 5 according to the Langmuir isotherm. The adsorption kinetics data were best fitted with the pseudo-second-order type for Cu (II) and Pb (II). .Finally thermodynamic parameters are determined at four different temperatures and it has been found that the adsorption process for Cu (II) and Pb (II) on the adsorbent is endothermic. because Fe(III) tends to hydrolyze in pH=5.5 and high concentrations, study adsorption isotherms for Fe(III) failed to do.

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Synthesis and application of novel Al-Mg layered double hydroxid with biodegradable counter ions for removal of cyanide ions from industrial wastes

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Abstract

The groundwater is an important component of our nation’s fresh water resources. In many parts of the nation, ground water serves as the only reliable source of drinking and irrigation water. Unfortunately, this vital resource is vulnerable to contamination, and ground water contaminant problems are being reported throughout the country. groundwater quality can be adversely affected or degraded as a result of human activities that introduce contaminants into the environment. groundwater quality may be adversely impacted by a variety of potential contaminant sources. Industrial and some commercial developments present the greatest risk. Groundwater contamination can result from deliberate waste disposal, and accidental leaks or spills of hazardous chemicals during transport, storage or use.

Cyanide (CN^-) is one of the most hazardous materials that can be released into groundwater from industrial water.

Cyanide can be removed from aqueous solutions using different but expensive methods. In this study, Mg/Al layered double hydroxides (LDHs)was used to remove Cyanide. Removing the cyanide from the aqueous solutions with the Mg/Al layered double hydroxides (LDHs)was studied via considering the effects of absorbent dose, initial cyanide concentration, contact time and pH in batch phase.

LDHs have the general structure of $[M_{1-x}^{2+} M_x^{3+} (OH)_2](A^{n-})_{x/n}.mH_2O$, Prepared from divalent and trivalent metal salts in basic solution and specific temprature and pH condition and they have many variety industrial applications.

In this research novel type of the (LDHs) containing amina acids were prepared and their characteristics were studied by XRD, IR, AAS, FE-SEM, AFM and TEM techniques . It was found that prepared (LDHs) have hydrotalcite-like structure.[1]

It was found that removal efficiency is higher at neutral and acidic pH. Due to their regeneration characteristics. Mg/Al layered double hydroxides (LDHs) can be used as a convenient option to reduce the level of Cyanide from the industrial waste water and specifically electroplating industries, where the problem of Cyanide in sewage is ubiquitous.[2]

The maximum adsorption capacity of the used LDH for CN^- was 37.5(±2) mg/g.

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Abstract

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Synthesis and Application of Palladium nanoparticles loaded on modified activated carbon for extraction and preconcentration of heavy metal ions with Application of response surface methodology for optimization of variables affecting the extraction efficiency

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In this project, palladium nanoparticles loaded on activated carbon (Pd-NP-AC) has been synthesized and characterized by different techniques including XRD and SEM. Then Pd-NP-AC was modified by a new Schiff' base ligand (N-(3-Bromo salisilidin)-4-nitro-1,2-phenilyne diamine) (BSNM) and was applied as a new solid phase extractant for determination of Fe and Cu ions in different water and waste water and food samples by flame atomic absorption spectrophotometry (FAAS).The significant of variables affecting the extraction efficiency were determinated by using a placket–burman design. Then the significant variables optimized by using a Box-Behnken design. The optimum experimental conditions obtained from this statistical design were included: pH=5.5; amount of chelating agent (ligand), 0.0084g and amount of adsorbent 0.1066 g. The adsorbed metals ions on adsorbent were eluted using 3 mol L^{-1} nitric acid. At the optimum conditions, analytical performance of the method was evaluated based on limits of detection, the relative standard deviation (RSD%) and preconcentration factor was evaluated and the results were 3.1 µg L^{-1} and 1.6 µgL^{-1}, 2.6 and 3.2, and 250 for Fe(III) and Cu(II), respectively. Also, the effect of matrix ions on the recoveries of the analytes was investigated. The method has been successfully applied for the extraction and determination of trace Fe(III) and Cu(II) in real samples with satisfactory results. Langmuir, Freundlich and Dubinin–Radushkevich (D–R) models were employed to study adsorption isotherms for Cu(II). The Cu(II) adsorption followed pseudo-second-order kinetics, and the maximum Cu(II) adsorption capacity was 9.57 mg g^{-1} at 298 K, pH = 5.5 according to the Langmuir isotherm. The values of thermodynamic parameters such as enthalpy change (ΔH°), entropy change (ΔS°) and free energy change (ΔG°) were evaluated for the adsorption of the Cu ion and it has been found that the adsorption process for Cu (II) on the adsorbent is endothermic. because Fe(III) tends to hydrolyze in pH=5.5 and high concentrations, study adsorption isotherms for Fe(III) failed to do.

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Synthesis and application of zinc oxide nanoparticles (n-ZnO) for removal of lead and palladium ions from aqueous solutions

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In recent years, application of nanoparticles with size range of 1–100 nm has received significant attention due to their novel properties and has come up as an area of extensive research [1]. Nano particles differ in characteristics from those of bulk materials. Due to numerous applications, studies on the synthesis and properties of nanoparticles have received significant attention in the past several years. Among of nanoparticles, ZnO nanoparticles (n-ZnO) have received more attention. ZnO nano particles have wide spread applications as biosensors, gas sensors, solar cells, ceramics, nanogenerators, photo detectors, catalysts, active fillers for rubber and plastic, UV absorbers in cosmetics and anti-virus agent in coating, pigments, optical materials, cosmetics, and in treatment of water and wastewater [2] also.

In the present work, ZnO nanoparticles have been synthesized and applied for removal of metal ions Pb and Pd from aqueous solutions. Adsorption experiments showed, ZnO nanoparticles has high good adsorption for these ions. Optimal experimental conditions including pH, adsorbent dosage, temperature, salt concentration and contact time have been established. Lead ions are retained on the sorbent (n-ZnO) in the pH=5.7 and palladium ions in the pH=6.8. The maximum equilibrium capacities of ZnO nanoparticles from Langmuir models were 142.85 and 333.33 mg g^{-1} for Pb(II) and Pd(II), respectively. Using the equilibrium concentration constants obtained at different temperatures, various thermodynamic parameters, such as ΔG^0, ΔH^0 and ΔS^0 have been calculated. ΔH^0s were 57.73 and -32.15 kJ mol^{-1} for palladium and lead, respectively. The thermodynamic studies indicated that the adsorption was spontaneous and exothermic for palladium adsorption and endothermic for lead.

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Synthesis and characterization of a biopolymer coated on glassy carbon electrode for electrochemical sensing

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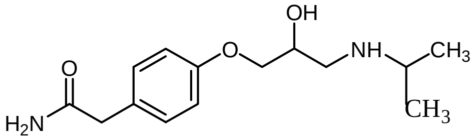
Polydopamine(PDA), a mussel-inspired adhesive catecholamine, is a biopolymer with excellent biocompatibility and low cytotoxicity, whichmakes it a versatile platform for bioapplication [1].Polydopamine coatings have recently attracted considerable interest for a variety of applications including biomedicine. Apart from numerous applications that benefited from this PDA coating reaching from biosensing to drug delivery, a renewed interest in catechols and their derived compounds has been sparked [2].

Hypertension is a growing disease of medical concern. Tremendous increase in the use of antihypertensive medications such as β-blockers points toward an increasing number of hypertension cases in last decade. Atenolol (I) is one of the most widely used -blockers. It is a hydrophilic β_1-receptor blocking agent, which is of immense therapeutic use in the treatment of various cardiovascular disorders, such as angina pectoris, cardiac arrhythmia and hypertension [3].



(I)

In this report, a facile and general approach was developed to prepare uniform layer of PDA at the surface of glassy carbon electrode by using electrochemical method. The thin film was characterized using scanning electron microscopy (SEM) and electrochemical techniques. The glassy carbon modified by PDA has been applied for adsorption and electrochemical sensing of atenolol. The adsorption parameters such as pH, adsorption time and agitation rate were optimized. In optimum condition, the various concentrations of atenolol were adsorbed at the surface of modified electrode. Differential pulse voltammetry has been used for quantitative determination of atenolol. The effects of voltammetric pH and scan rate have been studied for proposing the mechanism of sensing.

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Synthesis and characterization of chitosan/titana nanocomposite for removal of tartrazine from food samples: Optimization and modeling using a response surface methodology (RSM) based on the central composite design

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Metal oxide nanoparticles are currently the focus of considerable research due to their wide application in electric, optical, mechanical, magnetic and chemical field [1]. Because of their large surface area to volume ratio, the properties of the nanoparticles are different from its bulk material. In this report, chitosan immobilized titanium dioxide nanohybrid was prepared by chemical precipitation method. Fourier transform infrared spectroscopy and X-ray diffraction confirmed the formation of nanohybrid. Transmission electron microscopy analysis showed the immobilization of chitosan on the surface of TiO_2 nanoparticles. The nanohybrid was also characterized by thermogravimetric analysis (TGA) and zeta potential. Response surface methodology (RSM) was employed to investigate the effect of different operating parameters on the uptake of tartrazine using chitosan/titana nanocomposite. A two level three factor (2^3) factorial central composite design (CCD) was used for the optimization of the process parameters and to evaluate the effects and interactions of process variables: initial solution pH (2.5.0–6.0), adsorbent dosage (0.03-0.07 g), and initial concentration (15-35 µ g L^{-1}) were studied. The optimum removal efficiency of the nanoadsorbent for tartrazine adsorption was found as 99.0%. A good agreement between experimental and predicted data was achieved that efficiency of this model for prediction of real optimum point. Optimized values of adsorbent nanoparticle dosage and pH for tartrazine adsorption were found as 0.07 g L^{-1}, and 3, respectively. In order to investigate the mechanism of tartrazine removal, various adsorption isotherms such as Langmuir, Freundlich, Temkin were fitted. The experimental data revealed that the Langmuir isotherm gave a more satisfactory fit for tartrazine removal and the monolayer adsorption capacity was found to be 27 mg/g. The adsorption process was rapid and obeyed pseudo-second-order kinetics. The results showed this nanocomposite can be used for the efficient removal of tartrazine from different food and drinking samples.

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Synthesis and characterization of ion imprinted polymers for selective solid phase extraction of Ni (II) ions from aqueous samples

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Analysis and extraction of samples with complex matrix needs special attention in order to avoid matrix interferences beside preconcentration of the analytes and converting them into a more suitable form for the instrumental detection. The best solution for the complex sample analysis is the selective extraction of the interesting analytes using sorbents with tailor-made recognition sites such as Ion imprinted polymers (IIPs). IIPs can be synthesized by copolymerzation of a complex of a template and a functional monomer with a cross-linking agent in the environment of a porogenic solvent to mold template complementary binding sites [1,2]. After leaching the template ions, a rigid three dimensional network remains with cavity complemented to the target compounds. MIPs showed many advantages such as high selectivity, easy preparation and low cost that makes them suitable candidate as a sorbent for the sample preparation of very complex samples [3].

In this work, a new ion imprinted polymer was synthesized and applied for the selective extraction of Ni (II) ions from aqueous solutions. At the first step N-(2-Hydroxyphenyl)acrylamide was synthesized as a monomer. Next, the prepared monomer was reacted with Ni (II) ions to form a complex and finally it was used for polymerization with ethylene glycol dimethacrylate and AIBN as cross linker and initiator, respectively. Ni (II) ions were leached from prepared IIPs using EDTA (0.2 M) reagent. Prepared IIPs particles were packed in a cartridge for the selective solid phase extraction of Ni (II) ions. Important parameters effective in SPE of Ni (II) ions was studied and optimized. Optimum condition includes: extracting 20 mL Ni (II) solution at room temperature with 30 mg of IIPs with mesh size of 100-200 at pH of 6.9 and loading flow rate of 0.4 mL min^{-1}, following desorption using 10 mL of EDTA (0.2 M). Breakthrough volume for the prepared sorbent was not occurred until 200 mL of sample. Synthesized IIPs showed high selectivity among the interfering ions such as Cd (II), Co (II) and Zn (II) ions in which extraction recovery of Ni (II) was around 90% while for other ions it was less than 20%. Under the optimized method, LOD of the method was 20 ppb with linearity in the range of 0.1- 100 mg L^{-1}.

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Synthesis and characterization of magnetic metal-organic framework (MOF) as a novel sorbent, and its optimization by experimental design methodology for determination of palladium in environmental samples

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This paper describe sthesynthesis and application of novel magnetic metal-organic frame work (MOF) [(Fe3O4–Pyridine)/Cu3(BTC)2] for preconcentration of Pd(II) and its determination by flame atomic absorption spectrometry (FAAS)[1]. ABox–Behnken design was used to find the optimum conditions for the preconcentration procedure through response surface methodology. Three variables including amount of magnetic MOF, extraction time, and pH of extraction were selected as factors for adsorption step, and in desorption step, four parameters including type, volume, and concentration of eluent, and desorption time were selected in the optimization study. These values were 30mg, 6min, 6.9, K2SO4+NaOH, 6mL, 9.5 (w/v%)+0.01 molL^{-1}, 15.5min, for amount of MOF, extraction time, pH of extraction, type, volume, and concentration of the eluent, and desorption time, respectively. The preconcentration factor(PF), relative standard deviation (RSD), limit of detection (LOD), and adsorption capacity of the method were found to be 208, 2.1%, 0.37ngmL^{-1}, and 105.1 mgg^{-1}, respectively. It was found that the magnetic MOF has more capacity compared to Fe3O4–Py. Finally, the magnetic MOF was successfully applied for rapid extraction of trace amounts of Pd(II) ions in fish, sediment, soil, and water samples.

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Synthesis and characterization of magnetic nanoparticles functionalized with sulfamic acid groups as a recyclable catalyst for organic transformation

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Mounting interest is recently expressed for surface functionalization of nanocatalysts in which the nanoscale architecture of active centers plays a dominant role in determining their efficiency and selectivity [1-2]. Surface functionalized iron oxide magnetic nanoparticles (MNPs) are a kind of novel functional materials, which have been widely used in biotechnology and catalysis [3-4]. Good biocompatibility and biodegradability as well as basic magnetic characteristics could be denoted for functional organic materials grafted to MNPs. Magnetic nanocatalysts can easily be separated and recycled from the products by an external magnet. Moreover, their catalytic performance is enhanced, for the available surface area of the nonporous MNPs is external and the internal diffusion is practically avoided. The silane agents such as 3-aminopropyltriethyloxysilane (APTES) and mercaptopropyltriethoxysilane (MPTES) are often considered as potential candidates for modifying the surface of MNPs directly. Such surface modification enhances the biocompatibility and provides rather high density surface functional endgroups which allow for connecting to other metals, polymers or biomolecules [5].

In this report, a novel organic–inorganic hybrid heterogeneous catalyst (SA-MNPs) was synthesized by anchoring chlorosulfuric acid on amino-modified magnetic Fe_3O_4 nanoparticles. Characterization of the functionalized magnetic nanoparticles by XRD, FT-IR, TGA, and TEM demonstrated the successful grafting of sulfamic acid into the amino-functionalized Fe_3O_4 nanoparticles. The many organic transformation reactions over the SA-MNPs catalyst resulted in high to excellent yields, under solvent-free conditions at room temperature. The heterogeneous catalyst could be recovered easily and reused many times without significant loss of its catalytic activity.

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Synthesis and Characterization of Modified Fe_3O_4 Magnetic Nanoparticles with ZrO_2 and its Application in Electrochemical Determination of Cr (III) and Cr (VI)

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Abstract

Magnetic nanoparticles are of great interest for researchers from a wide range of disciplines, including magnetic fluids, catalysis, biotechnology/biomedicine, magnetic resonance imaging, data storage, and analytical applications [1-2]. Recently, successful application of such magnetic nanoparticles in the mentioned areas is highly dependent on the appropriate surface engineering to ensure achieving desirable interactions with analyte. To this end, magnetite nanocrystals have been coated with different materials like hydrophilic polymers or inorganic shells [2-3]. Herein, modified Fe_3O_4 magnetic nanoparticles (NPs) by zirconium oxide (ZrO_2) were prepared (Fe_3O_4/ZrO_2). Fe_3O_4/ZrO_2 NPs with different magnetization was synthesized by changing weight ratio of Fe_3O_4 to ZrO_2 as (2:2), (2:3), (2:5) and (2:7). Prepared Fe_3O_4/ZrO_2 NPs was characterized with XRD, SEM, EDXA, TEM, FTIR and VSM techniques. In addition, interaction of Fe_3O_4/ZrO_2 NPs with Cr (III) and (VI) ions was investigated by construction of a modified magnetic carbon paste electrode in the presence of $Fe(CN)_6]^{3-/4-}$ and PBQ/H_2Q, as a probes, via electrochemical methods. Electrochemical studies showed, on can detect and determine each form of the chromium ions in the presence of another by controlling the pH and choosing appropriate redox probe via cyclic voltammetry (CV) or electrochemical impedance spectroscopy (EIS) methods. Our experimental results showed Cr (III) can be pre-concentrate from 10^{-9} to 10^{-3} M in pH=3.5 and determined in the presence of $Fe(CN)_6]^{3-/4-}$ as a redox probe and also, Cr (VI) can be determined after pre-concentration in pH = 4.0 from 10^{-9} to 10^{-3} M in the presence of PBQ/H_2Q as a redox probe, by using CV and EIS techniques. Observed results will be presented and discussed.

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Synthesis and characterization of Nano-crystalline Zeolites: investigation its performance in vegetable oils refinement process

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Nanozeolites with the size of less than 200nm have received much of interest recently. Crystalline structure of zeolites with tridimensional network of well-defined micropores (pore diameter less than 15 A) brings both advantage and disadvantage. This feature provides zeolite with a consistent adsorption behavior toward guest molecules. Free fatty acids in vegetable oils resulted from the breaking of the triglyceride ester bonds, are normally removed during the refining process in the industry. Adsorption has been proved to be an excellent way to treat fatty acid effluents, offering significant advantages such as the cheapest, easy availability, easy operation and efficiency, comparing to many conventional methods especially from the economical and environmental point of view. Thus, the adsorbent properties of zeolites, clays, fibers, activated carbon, membranes, chitosan, and ion exchange resin were studied [1]. A common approach is to modify the general method of synthesis of zeolites, which is carried out in an aqueous phase. The principle of the synthesis is derived from the classic nucleation and crystallization theory. First a 250 mL polypropylene bottle containing a magnetic stir bar was washed with water under ultrasonication. Then appropriate amounts of H2O, 25 wt% aqueous tetramethylammonium hydroxide solution, and aluminum isopropoxide were added, in that order, and stirred vigorously until the solution became clear. Tetramethylammonium bromide was then added to the clear solution and stirred until completely dissolved. Aqueous 30 wt% colloidal silica was then added to complete the solution, and the bottle was sealed tightly. After each batch was sealed, it was aged for 3 days at room temperature with vigorous stirring, and was then heated with stirring in oil bath for a period of at least six days. All samples were washed by three repetitions of centrifugation, then decanting, and redispersion in H2O with ultrasonication before analyses were performed. Washed colloidal suspensions of nanocrystals were dried overnight in aven at 80C°. The prepared samples were characterized using X-ray diffraction (XRD) and surface area measuring (BET) methods. Nano-crystalline Zeolites is an effective and inexpensive adsorbent and has been widely used in various oil extraction industries to purify vegetable oil.

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Synthesis and characterization of nanomagnetite particles with various functionalities for biological and medical applications

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Abstract

Magnetic nanoparticles (MNPs) due to their high specific area and adsorption capacity are especial adsorbent, for this reason MNPs were used in biological and medical research [1-2]. Among these magnetic nanoparticles, magnetite (Fe_3O_4) nanoparticles were studied for their magnetic properties and easy separation. Magnetic nanoparticles were synthesized by co-precipitation method. Ferric chloride and ferrous chloride were dissolved in mL deionized water, which was then stirred and heated to 85^0C. The solution was bubbled with nitrogen .Next ammonia solution was added slowly. After synthesis, the precipitates were collected, washed with ethanol and DI water and dried. Then nanoparticles were modified; next the polymer with special groups was grafted to MNP, and at the end modified. Modified nanoparticles could adsorb drug via ionic bond. Fourier transform infrared spectroscopy (FT-IR), and elemental analysis (CHN) and Thermo gravimetric analysis (TGA) and Transmission Electron Microscope (TEM) were done for characterizing of adsorbent that showed the modification of MNPs was correctly carried out. The ability of drug adsorption of these nanoparticles was measured with UV-Vis spectroscopy. Many factors such as pH, temperature, contact time and adsorbent capacity were studied. In addition, mechanisms of drug delivery were analyzed. According to results, adsorbent release drug slowly and during the first 30 hours, adsorbent releases the drug approximately 50%. In addition, in temperature of 37^0C was observed the maximum drug delivery.

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Synthesis and characterization of Ni-Geopolymer nanocomposite and its application for nitro reduction

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Geopolymers can be regarded as amorphous analogues of zeolitic materials (three dimensional network of SiO_4 and AlO_4 tetrahedra with negative charge of the network balanced by extra-framework ations, variability in chemical composition, ion exchange properties, presence of rings forming cationic sites for bare cations), but possessing some advantages as network formation at ambient or low temperatures, mesoporosity and low cost preparation. It is shown that the geopolymer network enables incorporation of transition metal ions as active centres for catalytic reactions. These findings open a potential for synthesis of new types of robust catalysts for heterogeneously catalysed reactions. The synthesis yields an amorphous or semi-crystalline monolithic material with three-dimensional network of AlO_4 and SiO_4 tetrahedral connected by oxygen corners exhibiting high strength and resistance to high temperatures, chemicals, etc. [1-2].

Recently, new type of redox heterogeneous catalysts based on geopolymer aluminosilicate materials, was developed and their catalytic properties have been demonstrated on industrially important reactions.

The reduction of aromatic nitro compounds to corresponding anilines is one of the most important transformations in synthetic organic chemistry as the anilines obtained are commercially important starting materials and intermediates for a number of valuable compounds such as pharmaceuticals, agrochemicals and dyes. Among the methods used for reducing the aromatic nitro compounds, catalytic transfer hydrogenation is the best one being simple, eco-friendly with safe operation [3].

This work concerns the preparation and characterization of Geopolymer catalyst containing nickel nanoparticles prepared based on metakaollin. This novel nanocomposite was effectively employed as a novel heterogeneous catalyst for reduction of nitro compounds in the presence of $NaBH_4$ as a reducing agent. The reaction was carried out in room temperature and an aqueous medium. The catalyst showed good activity for the reduction of a number of nitro compounds yielding the respective amine compounds in excellent yield (~98%) at room temperature along with good reusability.

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Synthesis and characterization of novel tetra cyclopyrrole ether cation and its application for nano-composite carbon paste electrode selective to nitrate ion

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Novel tetra cyclopyrrole ether cation ($C4PE^{2+}$) was synthesized and characterized by different spectroscopy methods. Our recently studies showed a selective interaction of $C4PE^{2+}$ with nitrate anions. C4PE cation was used as a recognition element in the form of a composite with carbon paste as an electrode ($C4PE.(NO_3)_2$) for the potentiometry sensing of nitrate in water samples. Nitrate ions take part in several important environmental transformations involving nitrogen. This anion is widely used in explosives, fertilizers, drugs and many industrial products and hence their effluents may contain appreciable concentrations of nitrates and related ions. The large amounts of this species may be toxic and cause eutrophication. Hence, in recent decades a great interest has been applied to development of new methods based on removal and determination of nitrate in water and waste water samples [1]. Chemically modified carbon paste electrodes (CMCPEs) are prepared by addition of a suitable modifier to carbon paste. The operation mechanism of such CMCPEs depends on the properties of the modifier materials used to import selectivity and sensitivity towards the target [2]. In recent years, pyrrole-containing entities have emerged as among the most versatile and useful of all known anion-binding agents. They have seen application in areas as diverse as anion sensing and transport and have allowed for the stabilization of supramolecular structures with species as simple as fluoride anion and complex as DNA [3]. In this study a nitrate-selective carbon paste electrode based on C4PE was proposed. The electrode has a linear response to nitrate with a detection limit of 1×10^{-5} M and Nernstian compliance (57.5 ± 0.6 mV/decade) between pH 3.6 and 9.6 with a fast response time less than 10 s. The selectivity coefficient values indicate high selectivity for nitrate ions over various anions ($NO_3 > NO_2^{-} > Cl^{-} > Br^{-} > I^{-} > F^{-} > CH_3COO^{-} > SO_4^{2-} > IO_3^{-} > ClO_4^{-}$). The electrodes were used over a period of 60 days with good reproducibility. The analytical usefulness of the proposed electrode has been evaluated by its application in the determination of nitrate ions in mineral and drinking water samples.

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Synthesis and Characterization of Pt-Cu/porous silicon nanocomposite as a novel electrode material for Amperometric determination of Hydrazine

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Pt-Cu/Porous Silicon nanocomposite powder was prepared by chemical etching of silicon (Si) powder in a HF/HNO_3 solution [1], followed by electrodless plating of Pt-Cu alloy on the etched Porous silicon (PSi) powder in a solution containing $CuSO_4$and K_2PtCl_6as a metal precursors. The resulting nanocomposite was characterized by X–ray diffraction, scanning electron microscopy, cyclic voltammetryand FT–IR spectroscopy. The Pt-Cu alloy nanoparticles supported on PSi shows the good chemical and electrochemical stability as well as electrocatalytic effect on hydrazine oxidation, hencethe Pt-Cu/PSinanocomposite was used to modify carbon ionic liquid electrode to fabricate hydrazine sensor. TheelectrocatalyticactivityofPt-Cu/PSinanocomposite was investigated for hydrazine oxidation in alkaline and neutral solutions using cyclic voltammetryandchronoamperometry. The novel developedsensordisplayed a fast amperometricresponsetime of less than 4 s, long time stability, good signal reproducibility and a linear range from 0.3 to 2100 μmolL^{-1} of hydrazine with a detection limit of 100 nmolL^{-1}. The sensor exhibitedno interference from common interferences such as Ca^{2+}, Mg^{2+}, Ba^{2+}, Cd^{2+}, Pb^{2+}, NO_3^-, SO_4^{2-}, $C_2O_4^{2-}$. The good analytical performance and easy preparation method made this novel electrode material promising for the development of effective hydrazine sensor.

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Synthesis and characterization of three new nano-colored toners

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Abstract

In this paper, to improve the image quality of colored printed document[1], three novel cyan, magenta and yellow nano toners were synthesized by emulsion polymerization techniques [2] of styrene/Methyl methacrylate with Phthalocynanine Blue –Beta (P.B. 15:2), Irgalite red FBL (P.R. 48:4) and Benzidine Yellow G (P.Y. 12), respectively. The yields of these new products were excellent and they have been fully characterized by FT-IR, SEM, Differential Scanning Calorimetry (DSC) as well as the color of the image, light fastness, and water fastness and abrasion fastness tests. The spectroscopic and SEM results indicate that the the particle sizes of the pigment were converted about 96 nm.

Color assessment of synthesized toner with nano pigment shows that cyan hue has higher amount of color differences. It can be concluded that the cyan nano pigment in comparison with magenta and yellow ones is more sensitive to lower particle size. Magenta and yellow Shades of the synthesized toner are more color stable in the light than cyan. The cyan hue of conventional toner has the most color difference caused by light too. In water fastness test, cyan and magenta hues of synthesized toner with nano pigment shows more color difference compared with yellow hue. But in the conventional toner, a maximum difference in color shades is shown in cyan hue. Results of abrasion tests showed that the cyan synthesized toner with nano pigment has a maximum color change. Conventional toner compared to synthesized toner has lower values of color differences. Due to the effect of particle size on stability, higher color difference between shades of synthesized toner with nano pigment and conventional toner can be related to the smaller particles of synthesized toner. The amount of binder used in toner formulation employed also affect the abrasion fastness of toner. In general, according to the results of the light, water and abrasion fastness tests, the pigments used in this project can be used in the production of toner.

Keywords: Nanopigment, Colored-Toner, Emulsion Polymerization, ball mill, DSC,FT-IR, styrene/Methyl methacrylate

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Synthesis and electrochemical properties of a sulfur-multi walled carbon nanotubes composite cathode for lithium-sulfur batteries

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Abstract

Among lithium secondary batteries, a lithium-sulfur battery has attracted much interest due to its high theoretical specific capacity of 1672 mAh g^{-1} and theoretical energy density of 2600 Wh kg^{-1}. However, development of lithium-sulfur batteries has been encountered with some serious problems including low active material utilization and poor cycle life property, restricting their practical applications. These problems are caused by electrical insulating nature of sulfur [1], which leads to poor electrochemical accessibility and the low utilization of sulfur in the electrode. Moreover, polysulfides, which form during the first discharge step, are generally soluble in conventional organic solvent based electrolyte, which cause the rapid irreversible loss of sulfur active materials over repeated cycles [2]. To successfully operate the lithium-sulfur battery, the sulfur cathode material must be well combined with a conductive additive and a strong adsorbent agent [3]. In this study, a sulfur-multi walled carbon nanotubes composite (S/MWCNTs) was prepared using a two-step procedure of liquid-phase infiltration in CS_2 and heating process at 155 °C for 24 h as a cathode material for lithium-sulfur batteries. MWCNTs were used due to their good conductivity, unique structure and mechanical properties. The prepared composite material was characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and thermogravimetric analysis (TGA). From the results, it is found that sulfur was distributed homogeneously on the surface and into the pores of MWCNTs. The electrochemical performances of bare sulfur and composite cathodes were also investigated by cyclic voltammetry, electrochemical impedance spectroscopy and galvanostatic charge–discharge tests. The MWCNTs in the composite structure act as a adsorbent agent to trap polysulfides, leading to the improvement of the discharge capacity, the cycle performance, and the columbic efficiency of the composite cathode in lithium-sulfur batteries. A reversible capacity of 680 mAh g^{-1} was maintained after 50 cycles for the composite cathode, which is much higher than that of the bare sulfur cathode (131 mAh g^{-1}).

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Synthesis and evaluation of a magnetic molecularly imprinted polymer for solid phase extraction of diclofenac from urine Samples

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Abstract

Molecularly imprinted polymers (MIPs) show a strong chemical affinity and recognition to the target compounds in complicated matrix, due to the shape recognition, hydrogen bonding, and hydrophobic interaction [1]. Due to the simple preparation and flexibility of MIPs, molecularly imprinted technique has been widely used in sample preparation techniques especially in SPE and SPME, greatly improving the extraction selectivity for trace target compounds from complicated matrix [2]. Combining the molecularly imprinted technique with magnetic polymer beads to produce magnetic MIP (mag-MIP) beads can achieve many advantages during sample preparation such as the higher selectivity, larger extraction capacity and prevention of cross-contamination [3]. In this study, a mag-MIP has been prepared by precipitation polymerization method using Fe_3O_4 as magnetic bead, methacrylic acid (MAA) as functional monomer, trimethylolpropane triacrylate (TRIM) as a cross-linking and tetrahydrofuran (THF) and toluene as solvent, for solid phase extraction of diclofenac and its determining by Uv-Vis spectrophothometeric method. The influences of the sample pH and type of solvent on sorption and desorption processes was optimized. Water and methanol/acetic acid were distinguished as proper solvents for sorption and desorption of diclofenac, respectively. The calibration curve results show linearity in the range of 0.220-5 mgL^{-1} and detection limit of 0.066 mgL^{-1} (3s, n = 6). The results revealed the obtained mag-MIP exhibits high affinity for diclofenac and excellent discrimination between imprinted polymer and non-imprinted polymer (NIP) observed. Under optimum conditions, the prepared mag-MIP was successfully applied for extraction and determination of diclofenac in human urine.

Keywords: Magnetic molecularly imprinted polymer; solid-phase extraction; diclofenac.

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Synthesis and modification of CdS quantum dots and their applications for cancer detedtion

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Quantum dots (QDs) are luminescent semiconductor nanoparticles with several unique properties, such as tunable emission wavelengths ranging from visible to infrared, with a wide range of optical and electronic absorption [1]. Surface coating property is an important factor in determining QDs uptake into mammalian cells [2]. Water soluble and bio-functionalized semiconductor nanoparticles have an important concern and have been increasingly utilized in biosensors and bioimaging. In this work cadmium sulfide quantum dots modified with mercapto acetic acid (MAA) were synthesized in a colloidal solution. The cadmium sulfide nanoparticles were about 10 nm in size. Then amino_terminated poly ethylene glycol (PEG) was conjugated to the surface of modified QDs in order to reduce cytotoxicity as well as to enhance cell compatibility. The anticancer drug was conjugated with QDs and was used for in vitro cancer imaging.

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Synthesis and modification of nano β zeolite by ethylenediamine and monoethanol amine for adsorption of Cd^{+2} and Pb

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Abstract

Natural zeolites, known for their excellent sorption properties towards metal cations, are widely used for the purification of wastewaters. Toxic heavy metals such as cadmium, lead, chromium, arsenic, and selenium are constantly released into the environment. And are dangerous environmental pollutants due to their toxicity and strong tendency to concentrate in environment and in food chains [1]. In this study β zeolite (BEA) with high aluminum content (BEA, Si/Al = 7.4) was synthesized by hydrothermal method and to enhance the zeolites sorption capacity, it was modified by ethylenediamine (EDA) and monoethanol amine (MEA) through immobilization of amines on the zeolite surface by reflux method [2]. The treated β zeolite was examined for adsorption of Cd^{+2} and Pb^{2+} under different experimental condition including shaking time, pH and initial concentration was studied and optimized. At the optimized condition (pH= 4.0, shaking time =150 min, initial concentration=3000 $mg.L^{-1}$), 2544 $mg.L^{-1}$ of Cd^{+2} and (pH= 4.5, shaking time =120 min, initial concentration=2500 $mg.L^{-1}$), 2040 $mg.L^{1}$ of Pb^{2+} were adsorbed. It was revealed that modification of β zeolites with EDA and MEA does enhance the adsorption capacity of zeolite for Cd^{+2} and Pb^{2+} [3]. In this research, beta zeolite was firstly synthesized and characterized by X-ray diffraction and TG-DTG techniques [4]. After modification, it was used for adsorption of Cd^{2+} and Pb^{2+} from aqueous solutions. The effect of different parameters including pH, contact time, concentration on the adsorption was investigated and optimized.

Keywords: β-zeolite; Amine modified; Adsorption

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Synthesis and Structure Studies of 4-Phenyl-3-Thiosemicarbazide Modified Magnetic Nanoparticles and Application in the Removal of Heavy Metal Ions

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Abstract: Heavy metals are major pollutants in the environment due to their bioaccumulation in living cells and their non-biodegradable nature. The objectives of this research were synthesis the magnetite nanoparticles (Fe_3O_4) and determining their efficiency in the removal of heavy metal ions from aqueous solutions. In this study, magnetic nano-adsorbent has been synthesized by the covalent immobilization of poly (acrylic acid) (PAA) and 4-phenyl-3-thiosemicarbazide (TSC) as a complexing agent on the surface of Fe_3O_4 nanoparticles. Scanning electron microscopy (SEM), atomic force microscopy (AFM), FT-IR spectroscopy, X-ray diffraction and vibration sample magnetometer (VSM) were used for characterization of the synthesized magnetite nanoparticles. Atomic absorption spectroscopy (AAS) was used for determination of heavy metal ions in the solution before and after treatment with adsorbent. The ability of the Fe_3O_4@PAA@TSC MNPs for removing heavy metal ions Pb^{2+}, Cd^{2+}, Co^{2+}, Cu^{2+} and Zn^{2+} from aqueous solution was studied and the effects of pH, contact time, metal ion concentration and background electrolytes on heavy metal ions removal efficiency were investigated in batch system. SEM results showed that the diameter of the particles is in the range of 40-60 nm. Results also showed that the optimum pH value for adsorption is 5. The kinetic data were properly fitted by the pseudo-second-order model. The assumption that the metal ions adsorption is due to complex formation between active sites of the adsorbent and the heavy metal ions was further confirmed by investigating the adsorption isotherms and kinetics. The equilibrium time of 45 min and 0.050 g used of adsorbent for the volume 50.0 mL of wastewater solution. Finally, adsorption process was investigated with Langmuir and Freundlich isotherms. The experimental adsorption equilibrium data were fitted to Langmuir adsorption and the maximum adsorption capacities of Pb^{2+}, Cd^{2+}, Co^{2+}, Cu^{2+} and Zn^{2+} were found to be 181.1, 125.0, 86.2, 67.1 and 57.8 mg g^{-1}, respectively. The adsorption capacity increased and the adsorption efficiency decreased with increasing the concentration of heavy metal ions and reducing the amount of adsorbent. The desorption experiments under batch conditions revealed that the Fe_3O_4@PAA@TSC MNPs are reusable adsorbent for practical removal of the heavy metal ions. The ability of the prepared nanoparticles for removal of heavy metal ions from industrial samples was evaluated by using different industrial wastewaters from Moham industrial complex, Raad plating company and Iran Aircraft Manufacturing Industrial Company (HESA) and obtained optimum conditions is adsorbent concentration 1.0 g L^{-1}, contact time 45 minutes, solution pH=5.0, solution volume 50.0 mL and stirrer speed 300 round per minutes that is applied to real samples..

Key Words: Heavy Metal; Poly Acrylic Acid; 4-Phenyl-3-Thiosemicarbazide; Magnetite; Fe_3O_4.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ١٣٩٢

Synthesis and treatment of carbon nanostructures by HNO_3 for removal of fluoride from aqueous solutions

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In this study, chemical vapor deposition (CVD) method was used for the synthesis of carbon nanostructure (CNS) films on macroporous kaolin substrates using cyclohexanol and ferrocene, respectively as carbon source and catalyst. 0.01g of CNS was deposited on the surface of each disc. The structure of the CNS films was studied by SEM method (Fig 1)[1]. To enhance the adsorption behavior of discs, chemical surface treatment was carried out with 13M nitric acid at 110°C for 6 h. FT-IR spectra of the treated sample showed that the hydroxyl and carboxyl groups were produced on the carbon nanotube surface. The surface functional groups were determined according to the Boehm titration [2]. The fluoride adsorption capacity of carbon nanostructures depended on the pH of the solution. The point of zero charge (pH_{PZC}) of the treated sample was measured by acid–base titration and the value of 3.035 was obtained [3]. The amount of fluoride adsorbed on the carbon nanostructures was determined by ion-selective electrode. Under optimized conditions, the maximum adsorption capacity was obtained as 277.21 meq/g (Fig 2). The experimental results indicated that the synthesized carbon nanostructure was a promising candidate for fluoride removal from aqueous solutions.

Key words: chemical vapor deposition, carbon nanostructures, Boehm titration, Fluoride, HNO_3

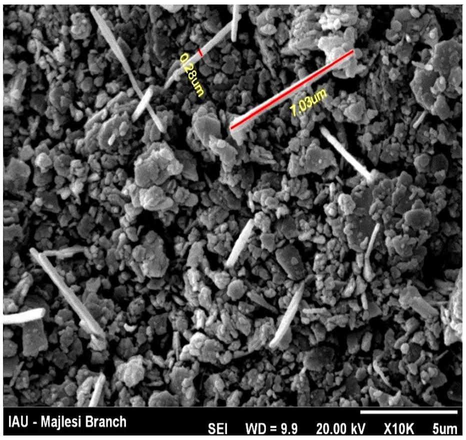

Fig 1. SEM image of carbon nano-structure.

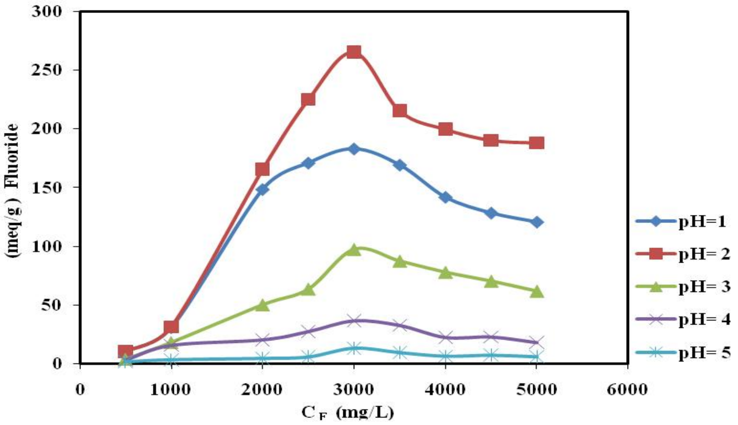

Fig 2. Adsorption isotherms of treated carbon nanostructures at 25°C.

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Synthesis magnetic Fe_3O_4/CS/PVA nanofibrous membrane and it application as a new sorbent for removal of Arsenic

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Arsenic toxicity which has become a major concern worldwide, therefore it is needed to develop the technology with improved materials and systems with high efficiency to overcome this problem [1]. Different sorbents have been synthesized and used for removal of arsenic in different samples [2]. The aim of this study is developing of a simple and efficient method for removal of arsenic from drinking water based on synthesis of magnetic fibrous materials. Ultrafine Fe_3O_4 powder synthesis by co-precipitation method and the particle size of Fe_3O_4 nanoparticles is characterized by X-ray diffraction, and scanning electron microscope to study the particle sizes, compositions, morphology and structure. After preparing of Fe_3O_4 powder, magnetic Fe_3O_4 /chitosan (CS)/poly vinyl alcohol (PVA) nanofibrous membrane was achieved by electrospinning with average fiber diameters in nano size scale. The influences of polymer concentration, applied voltage and Fe_3O_4 nanoparticles loading on the fabrication of nanofibrous membrane were optimized. Then, different parameters that may be affected on the adsorption of total arsenic (III and V) by Fe_3O_4/CS/PVA sorbent were optimized. Under optimum conditions, about 98% of arsenic adsorption was achieved at pH of 6.7-8.3 from the initial concentration containing 100.0 ng mL^{-1}. The Langmuir and Freundlich adsorption isotherms were investigated for a range of arsenic initial concentrations of 10.0-200.0 ng mL^{-1} under the same conditions. Moreover, it suggests that the Langmuir isotherm is more adequate than the Freundlich isotherm in simulating the adsorption isotherm of arsenic.

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Synthesis of a new ionic imprented polymer for the extraction of mercury from water samples

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Abstrac:

Contaminate water recourses by heavy metal is a serious worldwide environmental problem. Many metals such as mercury, cadmium, chromium and lead are known to be significantly toxic [1].

Molecular imprinting is a unique technique used for preparing polymers with synthetic recognition sites having a predetermined selectivity for analytes of interest [2]. Ionic imprinted polymers (IIPs) are an important branch of MIPs and are similar to MIPs, but they recognize inorganic ions after imprinting, especially metal ions [3]. The ionic imprinted polymer (IIP) is a kind of novel adsorbent, which can be used in the preconcentration of the templet ion of a trace amount and in the separation of the templet ion from other coexisting species or complex matrix, owing to the specific interaction of ligand with the templet ion.

The ionic imprinted polymer (IIP) of mercury ion as the template was synthesized by the formationof binary complexes of mercury (II) acetate with methacrylic acid and 1,3_dimethylbarbituric acid as functional monomers followed by therma copolymerization with ethylene glycol dimethacrylate as cross-linking monomer in the presence of 2,20 azobisisobutyronitrile as initiator and dimethyl sulfoxide as porogenic solvent. 50 mmol L-1 HCl solution was used to leach out mercury ions from the IIP.

The proposed IIP-based ISE was used to analyze mercury in spiked water samples.It can be seen that the recoveries of water samples vary from 96% to 104%, indicating that the present IIP-based sensor is can be used for reliable determination of mercury in water samples.

Key word: Ion imprinted polymer, Mercury(II) ion, potentiometric sensor,

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Abstrac:

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Synthesis of Ag/AgCl/montmorillonite nanocomposite and its application in degradation of methylene blue dye

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plasmonic photocatalytic nanostructured system was investigated on silver chloride/silver nanoparticles under visible light. Ag/AgCl nanoparticles were readily prepared using dispersing agent and light irradiation. The synthesis of Ag/AgCl nanoparticles in montmorillonite has been realized by chemical synthesis between montmorillonit, silver nitrate and hydrochloricacid [1]. The nanocomposite was characterized by X-ray diffraction analysis (XRD), diffuse reflectance spectroscopy (DRS), transmission electron microscopy (TEM) and fourier transform infrared (FT-IR). The XRD results demonstrated that silver nanoparticles were introduce on the surface of silver chloride nanoparticles and Ag/AgCl nanoparticles were inserted in the interlayers of montmorillonite and replacing the interlayered cations and water molecules [2]. DR spectra show surface resonance Plasmon for Ag nanoparticles. Photocatalytic activity of Ag/AgCl/montmorillonite nanocomposite was studied for degradation of Methylene Blue (MB) under visible light [3]. Several parameters were examined, catalyst amount (0.003-0.1 g L^{-1}), pH (2-10) and initial concentration of MB (0.32-16 mgL^{-1}). The extent of degradation was estimated from the residual concentration by spectrophotometrical. The support size was obtained in the range of more than 100 nm by TEM. In the same way, the average size of Ag/AgCl nanoparticles was obtained about 8 nm.

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Synthesis of Alumina, Silica, and Bentonite Aerogels using super critical technology and feasibility study of using them in Bioethanol conversion Reaction

By:

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Abstract

Aerogels are one of the highly applicable materials, having very high surface area. Beside of high surface area, these materials have some attractive specification such as very low density. Friability, flexibility and dimensional stability are some of important parameters of mechanical properties of Aerogels [1]. With respect to these specifications, Aerogels have many industrial application purposes [2]. Aerogel are produced via sol-gel process and one-step of special drying, in addition [3]. In this thesis, three types of Alumina, Silica and Bentonite Aerogels have been produced by using of super critical technology. Produced Aerogels structures have been studied by FT-IR method. Surface area of these Aerogels has been examined via BET method. BET test results showed that the highest surface area is belongs to corresponding Alumina Aerogel. Surface area of this aerogel was obtained at 277.211m^2/gr. As one of their application, these Aerogels have been deployed for catalyst basis for bioethanol conversion reaction. Among the produced catalysts, the Nickel-loaded catalyst which consists of 8% W/W Nickel on Alumina Aerogels has been showed the best performance. Qualitative measurements showed that major products of reaction are 1-Butanol, Diethyl ether and 1-Hexanol. Obtained yields for 1-Butanol, Diethyl ether and 1-Hexanol was 25.4±0.23%, 8±0.78% and 5.5±0.11% respectively for this catalyst. Studies showed that the optimum temperature and pressure was 250ºC and 141 bars, respectively.

Keywords: Aerogel Silica, Super-Critical State, 1-Butanol

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Synthesis of Alumina/Ni-Co Oxides, As a Catalyst For Ethanol and the Mixture of Ethanol and Ammonia Conversion to Valuable Products

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Nowadays due to high consumption rate of fossil fuels, demands for alternative sources are crucial. One of the solutions is the of biodiesels such as Dimethyl ether (DME), Diethyl ether (DEE) & Butanol. During this reaction using Ammonia as a feed in addition to the primary alcohol source can direct the products to some other substances such as acetonitrile, and Triethyl amine (TEA) [1-3].With respect to expensiveness of valuable metal salts such a Mo, Zr, Pd and etc., interests on producing catalysts with lower price salts such as Nickel and Cobalt which show the same properties, have been raised. Using supports such as Alumina, calcium carbonate and etc. not only increase the surface area, but also can modify the catalyst activity. Based on these facts, during this project, Nickel and Cobalt nitrate salts were loaded on the alumina as a support [4]. Amorphous high surface area Al_2O_3/Ni-Co was prepared using sol-gel method. The characteristics were investigated via XRD, SEM, BET, DSE & FT-IR. The result showed the high porosity in the structure of the synthesized catalyst. Through this process two different works were carried out. In the first part just Ethanol and in the second part Ethanol and gaseous Ammonia were reacted on the catalyst. Data of these reactions showed the presence of DEE in the first procedure and TEA, Acetonitrile, N-ethylacetamide and DEE in the second one. Identification of these products were done, using GC-MS & FT-IR. For the quantitative analysis, GC-FID technique. Results showed the yield of 15% for DEE and 8%, 13%, 19% for N-ethylacetamide, DEE and acetonitrile, respectively.

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Synthesis of an ion imprinted chelating resin for trace enrichment of lead(II) in environmental samples

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A new lead(II) ion-imprinted polymer (IIP) material has been prepared based on 2-(3,4-dihydroxyphenyl)-3,5,7-trihydroxy-4H-chromen-4-one (quercetin) as a chelating agent by using the radical bulk polymerization method [1]. The entrapped lead(II) ion was removed from the polymer using 2 M HNO_3. The prepared material was then characterized using IR spectroscopy and thermal analysis. The rebinding behavior of the lead(II) ion by the IIP was studied in the batch and column approaches. Concentration of template ion in the resulting solutions was determined by inductively coupled plasma atomic emission spectrometry (ICP-AES). The influence of pH on the rebinding of lead(II) ions was investigated in the range from 1.5 to 8.0 with an optimum value of 6.0. The result of batch kinetic tests revealed that uptake of lead(II) ions from solution is fairly rapid. The experimental adsorption data were successfully described by the Langmuir–Freundlich model. The maximum adsorption capacity of the imprinted polymer was calculated as 63.2 mg/g. Selectivity of the prepared polymer toward template ion was investigated in the presence of strong competitors such as Cd(II), Cu(II), Hg(II), and Zn(II). Finally, analytical performance of a packed micro-column containing 250 mg of the synthesized IIP was evaluated for FAAS determination of lead(II) in environmental samples.

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Synthesis of CNT/TiO$_2$-CoO-SiO$_2$-Ag composite as anode material for Lithium-ion battery

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As an allotrope of graphite, carbon nanotubes (CNTs) have been approved to be a good anode material for lithium batteries due to their unique structure (one-dimensional cylindrical tubuleof graphite sheet), high conductivity , low density, high rigidity and high tensile strength MWCNTs can have reversible capacities anywhere from 300 to 600mAh/g [1]. Ag addition increases the electronic conductivity of the composites and allows convenient transfer of Li-ion in the composite structure [2]. The electrochemical results show that the TiO$_2$ nanoparticles in the composite restrain the formation of surface film and make a contribution to the overall reversible capacity. Silicon thin-film shows very high first-cycle coulombic efficiency (95%), excellent cyclic performance and high reversible capacity. Among all of the transition-metal oxides studied, cobalt oxides showed a high theoretic reversible specific capacity[3].

In this work Multiwall Carbon nanotubes (MWCNTs) loaded with TiO2/CoO-SiO2-Ag nanoparticles (CNT/TiO2-CoO-Si-Ag) are a composite showing promise as an anode material in lithium-ion batteries. Here we prepare (MWCNT/TiO2-CoO-Si-Ag) via sol gel method. EPD (electrophoretic deposition) method used for deposition nanoparticles on foil copper as anode. The, structure, morphology and electrochemical performance of the composite were investigated by SEM, FT-IR, X-ray diffraction, and a variety of electrochemical Techniques such as cyclic woltammetry (CV) and Chorono charge and discharge method. The results show that the TiO$_2$-CoO-SiO$_2$-Ag nanoparticles were uniformly deposited on the surface of CNTs with sizes of 25-40 nm. The MWCNT/TiO$_2$-CoO-SiO$_2$-Ag anode materials showed superior cycling stability and a high reversible capacity of 180 mAh/g after 75 cycles.

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Synthesis of Fe-Cu/TiO_2 nanostructure and its use in construction of a sensitive and selective sensore for metformin determination

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Abstract

A carbon paste electrode modified with Fe-Cu/$TiO_2$was prepared and used for low level determination of metformin (MET) using square wave adsorptive stripping voltammetry (SWAdSV).The Fe-Cu/TiO_2nanoparticlewassynthesized by a modified sol-gel method. The surface structure and compositionof nanoparticles were characterized by scanning electron microscopy (SEM), X-ray powder diffraction analysis(XRD) and N_2physisorption. Also, electrochemical properties of the prepared nanocomposite modified electrode were investigated by cyclic voltammetry and electrochemical impedance spectroscopy (EIS) techniques.Under optimized conditions, the modified electrode exhibited a linear response over the concentration range of 15 nMto 75μMMET, with a detection limit of 3nM. The proposed sensor exhibited a high sensitivity, good selectivity and was successfully applied for MET determination in real samples such human urineandpharmaceutical formulations.In comparison to other METelectrochemical sensors that have been reported,the proposed sensor has the lowest limit of detection [1-3].The RSD value was found to be 2.1%, which indicated that the Fe-Cu/TiO_2-CPE has good repeatability.Furthermore, the stability of the proposed modified electrode was checked by recording the SWAdSV of 10μMof MET every other day over a month. The maximum deviation was found to be 3.8%.

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Synthesis of FePt/CNTs nanocomposite and a novel ionic liquid and application of them in preparation of a high sensitive electrochemical sensor in food samples

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Abstract: Sudan I is a synthetic azo-colourant which has been commonly used as a colouring agent in chemical industries and an additive in foods in daily life, particularly in those containing chilli powders, ketchup, olive oil, because of their intense red–orange colour. In 1975, Sudan I was classified as Group 3 carcinogen by the International Agency for Research on Cancer (IARC). Most phenol derivatives are known as harmful compounds [1]. In particular, some of them (e.g. bisphenol-A) are regarded as endocrine disruptors that might cause abnormal feminizing responses of animals. Natural estrogens including 17ß-estradiol can also be classified as phenol derivatives. Some of those estrogenic compounds are suggested to exhibit synergistic effects with other estrogenic compounds. Thus, detection and determination of phenol derivatives is very important. In this study, a novel ionic liquid modified FePt/CNTs carbon paste electrode was used as a powerful sensor for simultaneous determination of sudan I and bisphenol A for the first time. This modified electrode exhibited a potent and persistent electron mediating behavior followed by well separated oxidation peaks of sudan I and bisphenol A.

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Synthesis of graphene decorated with silver nanoparticles composite and its application for electrochemical determination of paracetamol

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Abstract:

Over the last few years, research has been directed towards the synthesis and application of metal nanoparticles for their unique properties compared to the bulk metal.[1] Among the different metal particles, silver nanoparticles (Ag-NPs) have received considerable attention because of their unusual properties and potential applications in diverse fields.[1]

Graphene, as the fundamental 2D carbon structure with exceptionally high crystal and electronic quality, has emerged as a rapidly rising star in the field of material science.[2] Electron transfer properties of graphene will be discussed, involving its unusual electronic structure, extraordinary electronic properties and fascinating electron transport [2]. Graphene-based nanomaterials are in the forefront of chemical research.[3]

Paracetamol or acetaminophen (AP) is generally an antipyretic and analgesic drug that is used for the relief of fever and headaches. This substance is commonly used for relieving mild to moderate pain associated with headache, backache, arthritis and postoperative pain, and is used universally for reducing fevers of bacterial or viral origin.

The current study a solution-based chemical approach has been used to prepare silver nanoparticles–graphene (Ag–G) hybrids through sequential reduction of graphene oxidation and silver ions and characterized with IR, XRD and TEM methods. This study reports oxidation of acetaminophen at modified carbon paste electrode (CPE) with silver nanoparticle–graphene (Ag–G) hybrids. Differential pulse voltammetry (DPV) studies show that the CPE/Ag-G electrode has a good electrochemical behavior of acetaminophen (AP) in aqueous buffer solutions, compared to the bare CPE and CPE/graphene. Excellent analytical features are achieved, including high sensitivity, low detection limit and satisfactory dynamic range, by differential pulse voltammetry (DPV). The response current is linear with the AP concentration in the range of 0.025 – 1.0mM with the linear regression equation $y = 2\times10^{-6} C_{AP} - 5\times10^{-6}$ ($R^2 = 0.986$) and LOD is 4.45×10^{-7} M. The proposed methods obtain satisfactory results in detection of acetaminophen in two commercial tablets.

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Synthesis of graphene-alumina nanocomposite adsorbent in separation and preconcentration of lead ion

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Abstract:

Graphene with its exceptional properties has been widely used in the preparation of next generation functional nanocomposites[1]. We have modified graphene nanoparticles With aluminium oxide(alumina) and used these for selective solid-phase extraction of trace amounts of lead metal ions. Heavy metal ions are toxic pollutants which exist in waste watersand are concerning for industries and environmental organizations all over the world[2]. The nanoparticles were characterized by FT-IR , XRD and SEM. Extraction time, amount of adsorbent ,pH value, type, volume, concentration of eluent and elution time were found to be critical factors for uptake. Quantitative extraction of trace amounts of Pb(II) from solutions was accomplished at an optimal pH value of 7 within 10 min. The metal ions were eluted from the sorbent with hydrochloric acid 1M.The ions were then determined by FAAS. The resulting calibration curve is linear in the concentration range of 8–1200 μgL^{-1} and limit of detection (LOD) was 4 $ngmL^{-1}$, while the relative standard deviation (RSD) was 1.44% (at 100μg L^{-1}Pb level and n=8). The enrichment factor is 22.65 (for 50 mL samples),and the adsorbent can be used for at least 5 cycles of preconcentration and elution. The adsorbent was successfully applied to the rapid extraction of trace quantities of Pb (II) ion in tea, rice, tomato , kohl and water samples.

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Synthesis of molecularly imprinted polymer as a sorbent for solid phase extraction of warfarin

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Warfarin, as a vitamin K antagonist, is the most commonly prescribed anticoagulant drug for the prophylaxis and treatment of venous and arterial thromboembolic disorders [1,2]. The aim of this work was to develop a method for the clean-up of warfarin from biological sample employing a new molecularly imprinted polymer (MIP) as a selective sorbent for solid phase extraction (SPE). This molecularly imprinted polymers (MIPs) was synthesized using warfarin as a template, pyrrole as a functional monomer, VTEOS as a cross-linker. The molar ratio of 1:4:20 (template: functional monomer: cross- linker) showed the best results. Non-imprinted polymers (NIPs) were prepared and treated with the same method, but in the absence of Warfarin. The prepared polymer was characterized by Fourier transmission infrared spectrometry (FT-IR), Scanning electron microscopy (SEM). Effective parameters on warfarin retention for example, type and volume of elution solvent = 3 mL of methanol, pH of sample solution = 3, maximum loading capacity = 16 mg g^{-1} and breakthrough volume = 750 mL and selectivity were studied. The extraction recovery was > 99 %. RSD for intra and inter day of extraction of warfarin were 3.8% and 7.1%, respectively for five measurements. The developed method was successfully applied for the determination of warfarin in biological samples.

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Synthesis of nano fiber based onmolecularly imprinted polymeric coating for solid phase microextraction of bisphenol A combined with electrochemical determination

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Abstract:

Solid-phase microextraction (SPME) is widely used in analytical laboratories for the analysis of organic compounds. Selectivity is an important challenge of solid phase microextraction fibers. Low selectivity of SPME fibers was resolved by using high selective analytical devices such as HPLC and GC. However, these methods are expensive and time consuming. So, in recent years molecularly imprinted polymers (MIP) because of their high selectivity to a special molecule, high mechanical and chemical stability, ease of preparation and low cost have gained great attention as coating of solid-phase microextraction fibers [1,2].

In the present work, a simple approach to preparation of MIP coating for microextraction of bisphenol A (BPA), is electro-polymerization; whichallows for the generation of a rigid and uniform MIP film with good adherence on the stainless steel of any shape and size. Moreover, the thickness and density of the film are adjustable in this process by controlling conditions.This high selective coating can couple with electrochemical methods, that arecheaper, faster and their simpler operationthan chromatographic methods to determine of BPA [3]. The effect of the extraction parameters including pH, sampling temperature, salt concentration and extraction time on the extraction efficiency have been studied.The calibration graph showed linear response range between10and100 ng l^{-1} and detection limit for BPA was 4 ng l^{-1}.Finally the preparedfiber was used for determination of BPA in thermal papers as real samples.

Keywords: Solid phase microextraction, Molecularly imprinted polymer, Electropolymerization, Bisphenol A.

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Synthesis of nano-composite alumina-chitosan-acetic acid and its application for the removal of indigo carmine dye

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Dyes are highly visible material that even a small amount of them which are released into the environment may cause the appearance of color, which attract the critical attention of public and local authorities [1]. The indigo carmine is considered as a highly toxic indigoid class of dye. Contact with this dye can cause skin and eye irritations. It can also cause permanent injury to cornea and conjunctiva [2]. Adsorption is an important technique for the removal of colors and any other pollutant from industrial wastewaters.

In this research, a new nano-composite of alumina-chitosan-acetic acid was synthesized. In order to synthesize the new nano-composite, firstly, activated alumina was mixed with chitosan in acetic acid solution. Then, in the presence of triton x-100, the polyanion of poly sufide was added to the alumina-coated chitosan at room temperature. Finally, synthesized nano-composite was added to chloroacetic acid at pH 8.0 for 12 h and then, for protonation of nano-composit it was placed in concentrated hydrochloric acid at room temperature. The protonated nano-composite was centrifuged, washed with distilled water to neutral pH and dried at 60° C. The synthesized nano- composite was characterized by XRD, TEM, and FTIR spectra and used as an adsorbent for removal of indigo carmine dye from aqueous solutions. Nano-composite of alumina-chitosan with chloro acetic acid shows exceptional adsorption capability for indigo carmine. 0.05 g of protonated nano-composite was suspended in 50 mL solution containing 100 mg/L indigo carmine dye and stirred. At successive time intervals, the suspension was centrifuged and absorption of supernatant solution was recorded with spectrophotometer in maximum wavelength of dye (λmax =620 nm). The dye concentration was measured spectrophotometrically by using a calibration curve. Effect of different variables such as pH, volume and type of acid, electrolyte, amount of adsorbent, stirring time and volume of solution were investigated. The interferences of the various species accompanying indigo carmine of 50 μg mL^{-1} were tested under the optimum conditions. The sorption data was then correlated with Langmuir and Freunlich isotherms and the results showed that the data are fitted better with Langmuir isotherm and maximum capacity of the adsorbent was 83.3 mg g^{-1}. The kinetics of indigo carmine adsorption on nano-composite was also studied by fitting the data to first order and pseudo second order kinetics hypothesis. It was observed that the removal of indigo carmine by the adsorbent undergoes via pseudo second order.

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Synthesis of phenyl-modified nanosilica particles and its application as a sorbent in solid phase extraction of polychlorinated biphenyls from aquatic media

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The main goal of this research was to develop a solid phase extraction method based on using nanosilica particles modified by phenyl group for the extraction of polychlorinated biphenyl compounds (PCBs) from aqueous media. Firstly nanosilica particles were synthesized based on sol-gel method and subsequently it was modified using phenyl group [1,2]. The entire sorbent was characterized by studying FT-IR spectra and SEM images. In addition, TGA curve showed 11.5% presence of phenyl group in this structure. The potential of entire nanoparticles in solid phase extraction (SPE) of seven PCBs including PCB28, PCB31, PCB52, PCB101, PCB 138, PCB153, and PCB180 was studied. All determinations were carried out using Gas chromatography- electron capture detector (GC-ECD) system. The parameters influencing solid phase extraction including extraction time, salt amount along with desorption solvent and desorption time were optimized. Under optimized condition, validation of method was performed. Calibration curves showed linearity in the range of 1-100 ng/mL for PCB31, PCB101, PCB 138, PCB153, PCB180 and 5-100 ng/mL for PCB28, PCB52, respectively. These ranges were obtained along with good correlation coefficients in the range of 0.993 to 0.998. As expected highly accessible surface area and π-π interaction make modified nanoparticles a promising candidate to use as a sorbent.

Keywords: Phenyl-modified nanosilica particles, Solid phase extraction, Polychlorinated biphenyls, Gas chromatography- electron capture detector.

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Synthesis, characterization and application of pure and doped ZnS quantum dots as green nanophotocatalysts for the degradation of pollutant dye

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Abstract

Quantum dots (QDs) are zero-dimensional semiconductors that may be used in a wide array of fields. QDs are nanometer-scale semiconductor crystals composed of groups II–VI or III–V elements, and are defined as particles with physical dimensions smaller than the exciton Bohr radius [1]. Among QDs, zinc sulfide (ZnS) is particularly suitable for use as a host material for a very large variety of dopant, due to the effects of dopants on the optical properties of QD semiconductors. However, due to the unique and novel properties of these nanocrystals, QDs and their molecular conjugates are becoming increasingly important for a wide range of applications in the biotechnology, physics, analytical chemistry, agriculture, chemical engineering and medicine. When a photon of light hits such a semiconductor, some of their electrons are excited into higher energy states. When they return to their ground state, a photon of a frequency characteristic of that material is emitted [2]. The heterogeneous photocatalysis using UV-radiation QDs is an interesting method for the treatment of water polluted with the organic substances [3].

In this study, ZnS QDs, as a pure and doped with transition metal were prepared for photodecolorization of methyl red as a model dye. The synthesis of QDs was carried out using a chemical precipitation method in aqueous solution, in the presence of 2-mercaptoethanol as a capping agent. The X-ray diffraction patterns indicated that the doped nanoparticles are crystalline, with cubic zinc blend structure. The effects of dopant content, pH, nanophotocatalyst amount, irradiation time, and initial dye concentration on the removal efficiency of dye were studied. Results showed that the QDs presented high dye decolorization efficiency, and doping with transition metal promoted the dye removal.

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Synthesis, characterization and photocatalytic activity of TiO_2 modified with ammonium hexafluorosilicate

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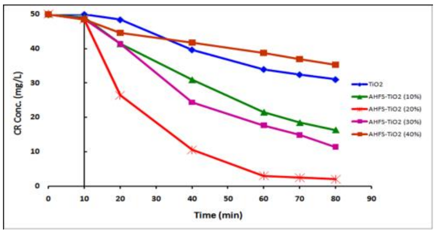
The photocatalytic efficiency of pure TiO_2 is rather low because of the fast recombination of photogenerated electrons and holes. Doping of certain elements or species into TiO_2 can greatly enhance its photocatalytic activity or make the catalyst active under visible light [1,2]. In this study TiO_2 nanoparticles with anatase crystalline phase were treated with $(NH_4)_2SiF_6$ as a structural modifier. Varying amounts of ammonium hexafluorosilicate (AHFS) were used **(**nominal weight of 10%–40%). The photocatalysts were characterized by X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectrophotometer, scanning electron microscopy (SEM), BET surface area measurement, energy dispersive X-ray analysis (EDX) and UV–vis diffuse reflectance spectroscoy (DRS). XRD analysis indicates that the samples possesses anatase crystalline lattice. FTIR shows two new bands at 1104 and 953 cm^{-1}, corresponding to the characteristic stretching vibrations of Si–O–Si and Si–O–Ti, respectively. SEM and BET analysis shows decreas of the particle size and increases the specific surface areas of TiO_2 (102 m^2 g^{-1}), respectively. EDX analysis of the nanoparticles shows that the samples are composed of elements Si, Ti and O with various percents. The DRS spectra of the samples indicates that there is a blue shift in UV–vis spectrum of AHFS-modified TiO_2 which may be due to the quantization of band structure for titania, which is often observed when the particle size is less than several nanometers. The photocatalytic activity of AHFS-modified TiO_2 nanoparticles was evaluated by the photocatalytic degradation of congo red (CR) solution under UV light irradiation Fig. 1 indicates that the AHFS-modified-TiO_2 nanoparticles prepared using a 20% of the modifier display the highest photocatalytic activity for the degradation of CR.



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Temperature-assisted dispersive liquid-liquid microextraction-gas chromatography-flame ionization detection:A simple method for the assessment of organophosphorous pesticides residues in river waters and fruit juice samples

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It is well known that pesticides are one of the most important compounds being used in agriculture. These compounds have caused a great harm to humans and the environment.In recent years, organophosphoruspesticides (OPPs) have been enormously used and are progressively replacing organochloridepesticides (e.g. DDT and aldrin) due to their low environmental persistence. However, according to the clinical reports, OPPs present a high acute toxicity that can cause a serious risk for the balance of aquatic systems and even danger to human health through the contamination andresidua of farm products [1].Due to adverse effect of pesticides on both human health and the environment, their monitoring is necessary. Several analytical methods were developed for the determination of OPPs, such as gas chromatography, high performance liquid chromatography and capillary electrophoresis. Several sample preparation methods have been used for the determination of pesticides residues such as liquid–liquid extraction, solid-phase extraction, solid-phase microextraction, liquid-phase microextraction, dispersive liquid–liquid microextraction(DLLME) and stir bar sorptive extraction[2,3]. In this studytemperature-assisted dispersive liquid-liquid microextraction(TA-DLLME) followed by GC-FID has been proposed for the determination of OPPs in food and environmental samples.TA-DLLME method is based on injection of the mixture of extraction and disperser solvents into an aquesed sample solution at an elevated temperature.After cooling of this solution, a cloudy state containing tiny droplets of extraction solvent was formed.The movement of extraction solvent droplets through the solution during the cooling process leads to an efficient transition of analytes from the aqueous phase into the extraction solvent.Figures of merit of the proposed method such as high enrichment factor (2171-5698), extraction recoveries nearly 50%, and low detection limits (0.47-1.00ng mL^{-1}) were satisfactory for determination of the selected OPPs pesticides.

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Temperature-assisted dispersive liquid-liquid microextraction-gas chromatography-flame ionization detection: A simple method for the assessment of phthalate esters residues in packed water, cola and physiological injection solution

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Phthalate esters (PAEs) are used primarily as plasticizers in polymeric materials, such as polyvinylchloride or polyvinylidenchloride copolymers, increasing their flexibility only through weak secondary molecular interactions with polymer chains . Being not covalently bound to the vinyl polymer matrix, their migration from plastic products to contact materials may occur through extraction (leaching) or evaporation processes [1]. Worldwide production of PAEs and their frequent application in different products of everyday use has resulted in their widespread presence in all parts of the environment, and in water especially. Certain phthalate esters and/or their metabolites are suspected human cancer-causing agents and endocrine disruptors [2]. Several analytical methods were developed for the determination of PAEs such as gas chromatography and high performance liquid chromatography. Several sample preparation methods have been used for the determination of PAEs residues such as liquid–liquid extraction, solid-phase extraction, solid-phase microextraction, liquid-phase microextraction, dispersive liquid–liquid microextraction (DLLME). In this study temperature-assisted dispersive liquid-liquid microextraction(TA-DLLME) followed by GC-FID has been proposed for the determination of PAEs in food and environmental samples. TA-DLLME method is based on injection of the mixture of extraction and disperser solvents into an aqueous sample solution at an elevated temperature. After cooling of this solution, a cloudy state containing tiny droplets of extraction solvent was formed. The movement of extraction solvent droplets through the solution during the cooling process leads to an efficient transition of analytes from the aqueous phase into the extraction solvent. Figures of merit of the proposed method such as high enrichment factor (352-3427), extraction recoveries nearly 50%, and low detection limits (0.27-0.91 ng mL^{-1}) were satisfactory for determination of the selected PAEs. Finally the proposed method was successfully applied to the determination of analyts in cola, packed water and physiological injection solution.

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Tetracycline loaded on Graphene oxide via ester bond formation

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Abstract

Drug delivery system as a new treatment approach to destruct cancer has emerged as an alternative of currently used cancer therapies. Tetracycline (TC) is one of the strong general cancer and antibiotic medicines nevertheless it has many side effects for many organs[1]. TC could be functionalized onto graphene oxide nanoparticles (GO) with strong chemical ester bond could be an appropriate interface for loading and releasing in vitro and in vivo cancer therapies and infectious diseases. In this work, GO reduces with pulsed laser irradiation after 10 min. The reduce graphene oxide (rGO) in order to functionalized by carboxylic group with the mixture of H_2SO_4/HNO_3 [2]and it was Acylation by $SOCl_2$ [3]. The Acyl chloride GO prepared to reaction with TC phenolic ring with ethanol and slightly few drops of ammonia. The phenolic ring of TC linkage with the carbonyl of GO. The chemistry effect of reduce graphene- tetracycline (rGO-TC) in each step of reactions characterized by UV-Vis and Furrier Transform Inferred (FT-IR) spectroscopy. Both UV-Vis and FT-IR are approved the ester bond between TC and GO. Transform Electron Microscopy (TEM) images used to estimate the thickness of each sheet is ~0.8 nm which is consistent with the typical thickness of single-layer GO. The particle size histogram also demonstrated the size of GO nano particles on 2 nm is more probable for piercing the cell membrane to caring the special medicine.

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The investigation of Cerium-binding to Human Serum Albumin by spectroscopic method

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Understanding the interaction between Cerium ion and human serum albumin (HSA) is of major pharmaceutical and clinical importance [1].The objective of this study was to access the conformational changes of HSA due to its binding to Ce^{3+} ion by Uv-Visible absorbance and Fluorescence spectroscopy. Solutions for experiments were prepared from stock solutions of protein and salt ($CeCl_3$) prepared in phosphate buffer (pH7.4). The concentration of protein and salt were 20 µM and 0.1mM, respectively. On all experiments, to a stock protein solution, different volumes of Copper solution (0.1mM) were added. All fluorescence spectra and intensities were recorded using a PERKIN Elmer Bio 100 spectrofluorimeter equipped with a xenon lamp source and 1cm quartz cells, excitation and emission wavelengths were 295 and 334nm, respectively and excitation and emission bandwidths were both 5nm. The UV absorption intensity of HSA decreased regularly with the addition of cerium ion concentration and the slight blue shift was observed in the wavelength with increasing Ce^{3+} ion concentration. The change of fluorescence intensity of HSA is related to the tryptophan residue when the small molecules are added to HSA. The fluorescence intensity of HSA increased regularly and the slight red shift was observed in the emission wavelength with increasing Ce^{3+} ion concentration, indicating that the HSA + Ce^{3+} complex was formed. Under simulative physiological conditions by Stern-Volmer equation, fluorescence data revealed that the binding constant (K_b) are $4.78\times10^{6}\,/\,M$ at 298K. The number of binding sites (n) is almost constant around 1.0 .Thermodynamic parameter $\Delta G = 27.46$ $kJ.mol^{-1}$ was calculated based on the binding constant .Instability of HSA structure is a bad side effect of using Ce^{3+} compounds as anti emeting drug [2].

Keywords: Human Serum Albumin, Fluorescence spectroscopy, Cerium (III) chloride, UV spectroscopy

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The construction of electrochemical biosensor based on Mg-Al-graphene LDH and CNT modified carbon paste electrode (CPE) for determination of furosemide

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Abstract

The surface modification of electrode in biosensors (sensors) is important and can improve selectivity and sensitivity in measurement. In recent years, anionic clays (LDHs) have been widely used for the fabrication of sensors due to their high ion-exchange capacity, good adsorption property, special structural feature, unusual intercalation property, good catalytic activity, high storage stability and chemical stability.

Furosemide used to treat excessive fluid accumulation (edema) caused by congestive heart failure, liver failure, renal disease, Kmadrary induced renal failure and antihypertensive drugs to control hypertension. Drug’s Side effects include increased urination, thirst, muscle cramps, weakness and constipation, dehydration, fever, rapid weight loss, increased blood urea and gout, temporary increase in blood cholesterol and triglyceride concentrations and so on. Therefore, concentration of this drug is extremely important.

The electrochemical behavior of furosemide was investigated on Mg-Al-graphene LDH and CNT modified CPE by cyclic voltammetery (CV). The CV of Furosemide on the modified electrode exhibited two well-defined irreversible anodic peaks in 0.1 M phosphate buffer solution.[1] The experimental parameters such as composition of modifier, pH, scan rate, applied potential and time were optimized. . Optimum values were 7,3%Mg/Al/Graphen-5%CNT,0.2V,60s respectively.

The results show that the peak current density of furosemide on Mg-Al-graphene LDH and CNT modified CPE is higher than the electrode without Mg-Al-graphene LDH.

According to scan rate, probable oxidation mechanism was proposed. In addition, the signal dependence on concentration confirmed.

The purpose of the present work is to construct a stable, sensitive and selective analytical method to determine furosemide in a simple, fast and inexpensive way.

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The effect of silica nanoparticles on solubility of sucrose in ternary system water-n-butanol -sucrose

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Ebrahim Nemati Lay, *Department of Chemical Engineering, University of Kashan*

Liquid-liquid extraction is a unit operation frequently employed in the pharmaceutical industry, as in many others, for recovery and purification of a desired ingredient from the solution in which it was prepared[1]. In recent years, nanofluids as new engineering materials that have high potential for use in industrial processes has been introduced. Finite studies have been done for the effect of these substances on the hydrodynamics and mass transfer in liquid-liquid systems. In this research work, the effect of silica nanoparticles on solubility of sucrose in ternary system water - n-Butanol- sucrose has been studied. n-butanol as a solvent and the deionized water including constant concentration hydrophilic silica nanoparticles as the continuous phase were selected. Silica nanoparticles with the size range of 10-15 nm was used to prepare nanofluid. In order to prepare nanofluids, 0.004 weight percents silica nanoparticles were dispersed in dionized water using ultrasonic system[2]. The results showed that adding nanoparticles to similar systems, solubility of sucrose in both extract and raffinate phases has increased. With increasing sucrose from 1,2,4 to 12.5 g in ternary system, percent of solubility in raffinate and extract phases increased by 4.3 - 7.21% and 40.6 -16.5%, respectively.

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The effect of temperature on the electrochemical oxidation of L-DOPA in aqueous solution

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Catecholamine drugs are used to treat hypertension,bronchial asthma, and organic heart disease and are used incardiac surgery and myocardial infarction [1−3]. 3,4-Dihydroxyphenylalanine(L-DOPA) is known as an important catecholamineandis considered to be the most effective medicine available for Parkinson's disease [1-3]. The thermodynamic parameters of L-DOPA electrooxidation reaction are dependent on the surrounding medium. In this work, we report the effect of temperature on the electrochemical oxidation of L-DOPA in aqueous solution. Based on the relation between the formal potential of the redox couple, $E^{\circ\prime}$, and pH, ($E^{\circ\prime} = E^{\circ} - 2.303mRT/2F$ pH) the standard redox potential of L-DOPA at different temperature can be obtained from the interception of the corresponding equation at pH 0. Finally, using temperature dependency of redox potential (Fig. 1), the thermodynamic parameters of enthalpy, entropy, and Gibbs free energy for the L-DOPA electrochemical reaction have been investigated in a range of temperature experimentally.

Ab initio calculations have been also used in order to calculate two proton-two-electron potential of L-DOPA in aqueous solution. Calculations have been completed using method of G3MP2/B3LYP for the gas-phase and solvation model of CPCM for the aqueous calculations. The results are in good agreement with the experimental values obtained by cyclic voltammetry.

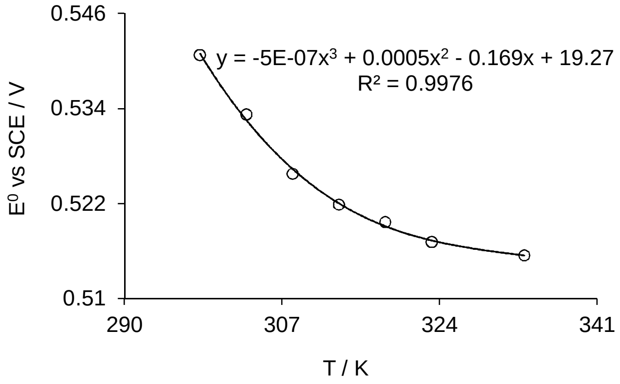

Fig. 1.The effect of temperature on the redox potential of L-DOPA.

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The effect of TiO_2 nanoparticles on the performance of DNA biosensors by methylen blue

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Abstract:

A key issue with any DNA biosensor is how to enhance the DNA immobilization amount. In order to enhance the immobilization amount of DNA and optimize the DNA interaction efficiency, different kinds of strategies have been developed. Including which, nanomaterials have been paid a great deal of attention in electrode surface modification for DNA biosensor fabrication. In this work, Multiwall carbon nanotubes (MWCNTs) and Titaniume dioxide nano particles (TiO_2NPs) were used in the fabrication of DNA biosensor . One of the problems of DNA biosensors fabrication is immobilization DNA on the surface of nanomaterials. It can be immobilized on to different carbon alotrop(such as CNT, graphit,…) via electrostatic interactions. In this work we use functionalized MWCNT(F-MWCNT) and TiO_2 NPs. However, the results are unsatisfactory because negatively charged F-MWCNTs repulse the negatively charged DNA[1]. In order to, we used positively charged polyelectrolytes as dispersant of MWCNTs and TiO_2NPs. At first, we compared two positively charged polyelectrolyte, poly dialylldimethylammonium chloride (PDDA) and chitosan, for DNA immobilization on the surface of MWCNTs and TiO_2NPs. In other study, to recognize the effect of TiO_2 NPs effect in DNA immobilization on the electrode surface, a comparision was done between MWCNTs and TiO_2NPs. We used a pencil graphite electrode (PGE) and differential pulse voltammetry technique .To construct DNA biosensor, different variables such as immobilization time of nanomaterials and polyelectrolyte on PGE, amount of MWCNTs and TiO_2NPs , incubation time of ds-DNA(double stranded DNA), and its concentration were optimized. In order to investigate efficiency of the prepared DNA biosensor methylene blue was chosen as an electroactive probe [2]. After interaction of ds-DNA with methylen blue by following the oxidation signals of guanine and adenine detectioin limits of PGE/PDDA-MWCNTs/dsDNA and PGE/PDDA-MWCNTs-TiO_2NPs/dsDNA were obtained 0.085 and 0.012 µM, respectively.

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The effects of Ge-doped on HOMO-LUMO orbital of armchair BNNTs

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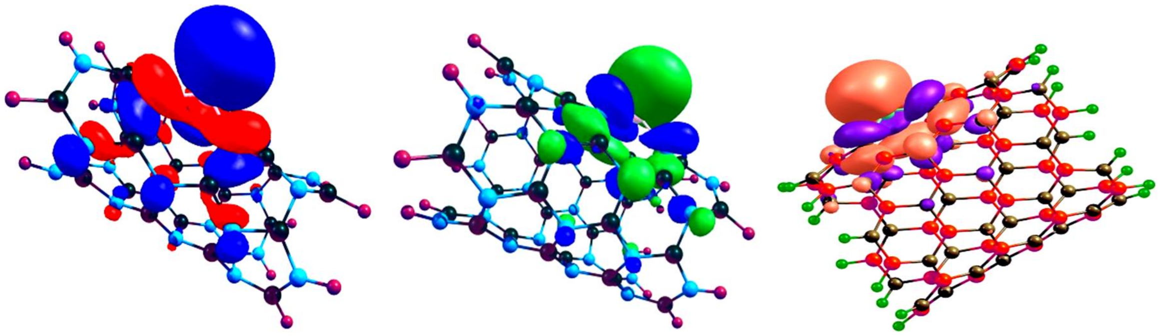
Ever since after discovery and synthies of Boron nitride nanotubes, The structural and electronic properties of this nanotube have also been studied through both experimental and theoretical [1-3] works.The purpose of this work is investigation the effect of Ge doped on the HOMO and LUMO orbitals of the (3,3) , (4,4) and (5,5) armchair BNNTs. For this meanes at first time all sturures optimized at the B3LYP/(6-31G(d)) level of DFT theory. Then the HOMO and LUMO orbitals are calculated and the quantum molecular descriptors: electronic chemical potential (μ), global hardness (η), electrophilicity index (ω), energy gap (E_{gap}), global softness (S), and electrongativity (χ) of the nanotubes are calculated. Comparison of results reveals that the charge density of HOMO and LUMO are localized near the Ge-doped and the energy gap between HOMO-LUMOand global hardness is decrease.



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The green synthesis of silver nanoparticles using seed extract of oak (*Quercus infectoria*) and anticancer effects

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Determination of quercetin in different parts of *Quercus infectoria* (gal and bark of oak tree) due to its anti cancer, leukemia, hepatocelular, carcinoma and estrogen reception and breast cancer is shown in many studied [1]. In this work, silver nanoparticles were synthesized by using seed of hull. The anticancer properties on MCF-7 cell line were determined. Different parameters such as concentration of seed of hull extract, concentration of $AgNO_3$, temperature and pH of solution were studied. UV–VIS spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images shows the nanoparticle formation in the medium. FT-IR analyses confirmed the presence of extract compounds as capping and stabilizing agent on the surface of nanoparticles. It was found that using 1mM solution of $AgNO_3$, 40 gr/L extract, pH=9 and T=45 ºC silver nanoparticles are formed in the size of 20 nm. Antioxidant capacity of synthesized nanoparticles was investigated by using DPPH method. Based on the results, for silver nanoparticles the IC_{50} = 0.03 µg/mL whereas for BHT method 50.63 µg/mL was determined. In vitro test via MTT test on MCF-7 cell line was done by MCF-7 and IC_{50}= 0.04 µg/mL was obtained.

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The green synthesis of silver nanoparticles using seed extract of oak (*Quercus infectoria*) and anticancer effects

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Determination of quercetin in different parts of *Quercus infectoria* (gal and bark of oak tree) due to its anti cancer, leukemia, hepatocelular, carcinoma and estrogen reception and breast cancer is shown in many studied [1-3]. In this work, silver nanoparticles were synthesized by using seed of hull. The anticancer properties on MCF-7 cell line were determined. Different parameters such as concentration of seed of hull extract, concentration of $AgNO_3$, temperature and pH of solution were studied. UV–VIS spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images shows the nanoparticle formation in the medium. FT-IR analyses confirmed the presence of extract compounds as capping and stabilizing agent on the surface of nanoparticles. It was found that using 1mM solution of $AgNO_3$, 40 gr/L extract, pH=9 and T=45 ^oC silver nanoparticles are formed in the size of 20 nm. Antioxidant capacity of synthesized nanoparticles was investigated by using DPPH method. Based on the results, for silver nanoparticles the IC_{50} = 0.03 µg/mL whereas for BHT method 50.63 µg/mL was determined. In vitro test via MTT test on MCF-7 cell line was done by MCF-7 and IC_{50}= 0.04 µg/mL was obtained.

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The green synthesis of silver nanoparticles using seed extract of oak (*Quercus infectoria*) and anticancer effects

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The importance of intra- and inter-group separation of actinides and lanthanides is well known. In view of the similarities in chemical properties of lanthanides, separation of these ions requests sophisticated methodologies [1]. Zeolites are valuable inorganic crystalline materials, which are porous having wide variety of industrial applications due to their unique properties including molecular sieving, ion-exchange and catalytic behavior. Considerable attention has been paid on utilizing zeolitic nano-particles because their advantages over conventional micron sized materials. Studies have been shown that the particle size and morphology of the zeolitic materials play an important role in their properties and efficiencies as catalysts, adsorbents for separation scopes [2]. Therefore, in this study we report on the application of natural clinoptilolite as sorbent for removing Th^{4+} and Eu^{3+} ions from aqueous solutions. The adsorption efficiencies of the sorbent were found to be pH dependent. The maximum adsorption of thorium and europium ions was obtained at pH 4.5 (85% for Th^{4+} and 50.0% for Eu^{3+}). However, the studied zeolite was shown to be a adsorbent with lower adsorption percentage towards europium ions. In order to improve the adsorptive properties of clinoptilolite, modification of the zeolite's surface by a Schiff base bis(2-hydroxybenzaldehyde)-1,3-propylendiamine has been done. The results show a significant increase in the adsorption values for Th^{4+} and Eu^{3+} ions beyond the pH values of 4.5 and 6, respectively. The effect of various parameters such as pH, contact time, metal concentration and amount of adsorbent on the process was investigated. A quantitative removal of the studied metal ions (100% for Th^{4+} and 96.6% for Eu^{3+}) was achieved by 0.1 g of the modified adsorbent disperse in 20 ml of the sample containing the metal ions (initial ion concentration 20 mgl^{-1}), after 60 min. Kinetics of the adsorption process were studied by considering a pseudo second-order model. This model predicts chemisorption as the adsorption mechanism of the process. Langmuir, Freundlich and Temkin models were tested for describing the equilibrium data. It was found that each of the metal ions adsorption was well described with Langmuir model.

The indirect electrochemical detection and quantification of DNA through its co-adsorption with Arylene-N,N'-bis(dimethoxyphosphorothioate) diamide using a glassy carbon electrode modified with multiwalled carbon nanotube

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Abstract

The voltammetric responses arising from the co-adsorption of Arylene-N,N'-bis(dimethoxyphosphorothioate) diamide (ADPPD) and DNA at multiwalled carbon nanotube modified glassy carbon electrode (MWCNT/GCE) are reported. Surface morphology and electrochemical properties of the adsorbed of DNA at modified electrode were investigated by scanning electron microscopy (SEM), cyclic voltammetry and electrochemical impedance spectroscopy techniques [1, 2]. The electrochemical behaviour of these two species shows that the adsorbed species are non-interacting and furthe this result highlights the two species occupy similar sites on the modified electrode surface. The results of voltammetric studies show an inverse linear relationship between the surface concentrations of the two species, such that it is possible to indirectly measure the quantity of adsorbed DNA to the electrode through the voltammetric signal of the co-adsorbed Arylene-N,N'-bis(dimethoxyphosphorothioate) diamide. Through use of this methodology it is possible to obtain a limit of detection of the DNA solution phase concentration of 1.4 µM [3].

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The role of FeO semiconductor incorporated on clinoptilolite nanoparticles in photodegradation of cotrimaxazole in a pharmaceutical aqueous solution sample

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Abstract: Over the past decades, considerable attention with respect to environmental problems has been devoted to the development of photocatalytic systems. The photocatalytic degradation of cotrimaxazole antimicrobial which has widely used in human and veterinary medicine, by using FeO-NCP as heterogeneous catalyst was investigated under Hg lamp in aqueous solution. Nanoparticles of clinoptilolite were prepared by mechanical ball-mill method and FeO-NCP was prepared during ion exchange and calcinations processes. The proposed catalyst was used in photodegradation of cotrimaxazole aqueous solution. For this goal, suspensions containing a definite amount of cotrimaxazole and catalyst were irradiated and at regular time intervals, the absorbance of the irradiated and un-irradiated solutions was recorded to calculate the degradation extent [1]. All samples were characterized by XRD, FT-IR, TEM, methods. The degradation results were confirmed by COD and HPLC. The optimum values for experimental parameters were 0.05 g L^{-1} of photocatalyst, 50 time diluted of the original prepared solution (one capsule/100 mL), and pH 3. The kinetics of the process obeyed first order kinetics. Using nano-clinoptilolite particles significantly increased the photocatalytic activity of FeO with respect to micronized one. The proposed catalyst retains above 60% of its initial activity after two successive using [2].

Keywords: Nanoclinoptilolite, Iron(II) oxide, Photocatalyst, Photodegradation

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The study solvent and surfactants effect on the spectrofluorimetric properties of (R)-5-(1-(4-(5-chloro-3-fluoropyridin-2-yloxy) phenoxy) ethyl)-1, 3, 4-oxadiazol-2(3h)-one as new herbicide

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Synthesis of the derivatives 1,3,4 –oxadiazol with a high biological ability to facilitate different chemical reaction expanded and research on their chemical properties as well as the biological behavior because of their antibacterial ,anti-fungal, analgesics, anti-inflammatory, antiviral, anticancer, anti blood pressure and etc has been accelerated in the past two decades[1]. One of the synthesized oxadiazol derivatives is (R)-5-(1-(4-(5-chloro-3-fluoropyridin-2-yloxy)phenoxy)ethyl)-1,3,4-oxadiazol-2(3h)-one which the herbicide characteristic of this compound has been surveyed intensively[2]. The importance of herbicide and especially herbicide residual on environmental and food samples determination with new and sensitive method are very critical. For determination of negligible amount of herbicides may be used the surfactants that usually used to slow down surface tension of the herbicide [3]. Therefore the study on the effect of surfactant and solvent on spectroscopic properties of herbicide for analytical propose is important.

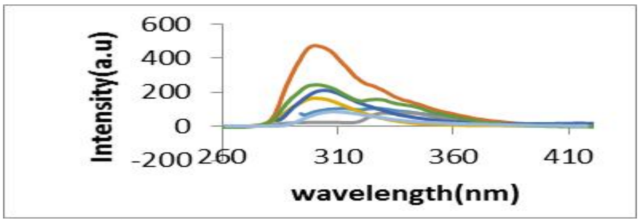

Fig. 1.influence of Triton X-100 (10^{-5}M) on fluorescence intensity of this herbicide (10^{-6}M) in—ETOH, —MEOH,—DMSO, — Dioxan, — DMF, — THF, — Acetonitril.

In this study spectroscopic properties of new herbicide in the prescence of solvents and varying surfactant were investigated.Herbicide compound (R)-5-(1-(4-(5-chloro-3-fluoropyridin-2-yloxy)phenoxy)ethyl)-1,3,4-oxadiazol-2(3h)-one as new herbicide in the presence of very low surfactants concentration have intense fluorescence. Therefore spectrofluorimertic method that is highly sensitive was used for this purpose. Ionic surfactants such as sodium dodecyl sulfate (SDS), hexadecyl trimethyl ammonium bromide (CTAB) and non-ionic surfactants such as Triton X-100, Tween 80 and Brij 35 were used and influenced the fluorescence intensity of this herbicide in any solvents. Experiments show that this herbicide has the highest affinity for lesser amount of Triton X-100. Fig. 1. Shows influence of Triton X-100 (10^{-5}M) on fluorescence intensity of this herbicide. Also determination probability of this herbicide was investigated and have good sufficiency indexes in competence with other herbicide determination methods.

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The use of gold nanoparticles for determination of morphine by colorimetric and resonance light scattering methods

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Morphine (MP), a group of narcotic pain relieve drugs, is frequently used to relieve severe pain for patients, especially for those who undergo a surgical procedure. However, when overdosed or abused, MP is toxic and can cause disruption in the central nervous system. Therefore, to prevent overdose-induced toxication, it is necessary to sensitively determine the concentrations of MP in patient's blood or urine. In the present work, a simple and sensitive method was developed for detection and determination of morphine hydrochloride in urine samples using gold nanoparticles as resonance light scattering (RLS) and colorimetric probes. The gold nanoparticles synthesized by sodium citrate reduction method have a negative charge layer on their surfaces because of self-assembling of citrate anions on their surface. The binding of morphine cations to gold nanoparticles results in the aggregation of them, which leads to the enhancement of the RLS signal of the nanoparticles. In UV-Vis spectrum, the addition of trace amount of morphine to gold nanoparticles solution decreases nanoparticles' plasmon absorption band at 520 nm and a new pick appears at 623 nm. A linear increase in absorbance at 623 nm observed as more concentrations of morphine were added. The aggregates were characterized transmission electron microscopy. The effects of important experimental factors such as pH, concentration of gold nanoparticles and incubation time on RLS and absorption signals were investigated and optimized. For both method, the linear range was 0.03-0.3 μgmL^{-1} and detection limit was 12 ng mL^{-1}. The methods were applied to the determination of morphine in urine samples.

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The use of non-modified magnetic nanoparticles for fast and efficient separation and preconcentration of trace amounts of cadmium (II) ions from food and water samples

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Recently, adsorption using magnetic nano-sized adsorbents has attracted significant attention due to its specific characteristics [1, 2]. It adopts magnetic particles as sorbents, which endow some unique features in extraction. For instance, after extraction, the magnetic particles can be readily isolated from sample matrix with a magnet. Compared with isolation of conventional sorbents by filtration or centrifugation, magnetic isolation is obviously much more convenient, economic and efficient. In addition, in magnetic solid phase extraction (MSPE), the sorbents are universally dispersed into sample solution to achieve extraction. In such a dispersive mode, the contact area between the sorbents and the analytes is large enough to ensure a fast mass transfer, which is beneficial to guarantee high extraction efficiency of this method. Moreover, MSPE is suitable for direct analysis of samples containing particles or microorganisms, which widely exist in environmental or biological matrices and may arouse blockage and lead to extraction failure on conventional SPE cartridges. Magnetic nano-adsorbents have the advantages of both magnetic separation techniques and nano-sized materials, which can be easily recovered or manipulated with an external magnetic field. All of these merits render MSPE a promising technique for sample preparation. In this study, a new method for the separation and preconcentration of trace amounts of cadmium in food and water samples by magnetic solid phase extraction (MSPE) with Fe_3O_4 magnetic nanoparticles (Fe_3O_4-MNPs) and its determination by flame atomic absorption spectrometry has been developed. For this purpose, Fe_3O_4-MNPs were synthesized via chemical precipitation method. The extraction of cadmium ions from the aqueous solution was performed with dithizone (DTZ) as the chelating agent. The various parameters affecting the extraction and preconcentration of cadmium were investigated and optimized. In this method, the analyte ions were quantitatively adsorbed on Fe_3O_4-MNPs and, then, Fe_3O_4-MNPs were easily separated from the aqueous solution by applying an external magnetic field. After extraction and collection of Fe_3O_4-MNPs, the analyte ions were eluted using 1.0 mol L^{-1} HNO_3 in methanol. Under the optimal conditions, the calibration curve showed an excellent linearity in the concentration range of 0.3–24.0 µg L^{-1} and the limit of detection was 0.1 µg L^{-1} of cadmium. The relative standard deviation was 2.6 % at the 10.0 µg L^{-1} level (n = 10) and the preconcentration factor was found to be 66.7. The developed method was successfully applied to the determination of cadmium in food (spinach, lettuce and radish) and water samples (tap, river, spring, mineral and sea water). The results show that, magnetic nanoparticles can be used as a cheap and efficient adsorbent for the extraction and preconcentration of cadmium from real samples.

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Thermal Lens Microscopy of Samarium- Phenanthroline Anticancer Complex

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1, 10-Phenanthroline (PL) is the parent of an important class of chelating agents that has excellent properties; the planar nature of 1,l0-phenanthroline is its ability to participate as either an intercalating or groove-binding species with DNA and RNA and the rigid PL molecule demonstrated distinct effects on in vitro cultured cells [1]. Therefore determination of this complex and PL singly, in biological samples is very important. We used photothermal spectrometry for determination of trace amount of Samarium complex and PL in a microchip. Photothermal spectrometry is a class of highly sensitive methods based on measurement of amount of thermal energy released by a sample subsequently to light absorption and nonradiative relaxation of the exited species. When a sample is irradiated by a laser beam, local heating near the beam axis produces a transverse temperature gradient which subsequently induces the formation of a refractive index gradient. Most generally, the refractive index is lowered by the temperature rise so that the refractive index gradient is negative and behaves like a diverging lens. Practically, one laser is used to create photothermal effect and a second laser is used to monitor the thermal lens [2]. Thermo optical spectroscopy is among the methods that have already proven their expedience in combining with chemical microchip, as well as for other microscale measurements. Thermal lens microscopy showed much potential when dealing with ultratrace on-chip determination of various substances and the analysis of biological materials [3].

Thermal lens spectrometry of Samarium complex in a fabricated microchip was measured. After optimization of chopper frequency, coaxiality of two laser beams and sample distance from objective lens for synthesized complex and PL, low concentration of them (about 12.5 ppm) in microliter scale (between 7-10 µl) were measured.

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Thermogravimetric analysis coupled with chemometrics: a reliable approach for pharmaceutical quality control

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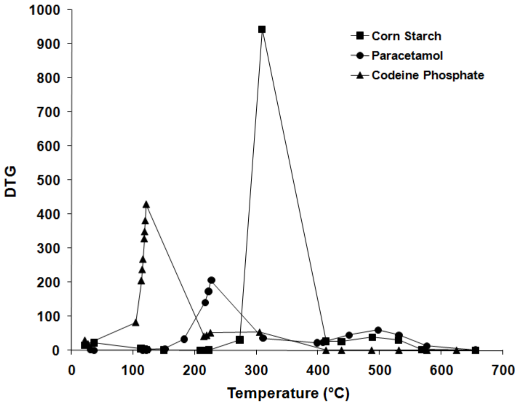
There are several methods based on official pharmacopoeias [1], spectrophotometry [2] and chromatography [3] for determination of paracetamol and codeine, whether as a single component or in combination with other chemicals. Although some of these methods are officially well known, most are relatively complicated in terms of required time and equipment. Thermo analytical techniques, such as thermogravimetry (TG), are quick techniques not requiring any sample pretreatment. A quantitative analytical approach has been proposed for simultaneous determination of paracetamol and codeine phosphate in pharmaceutical tablet preparation by thermogravimetric analysis (TGA) utilizing chemometric data processing techniques. Partial least squares regression (PLS) and successive projections algorithm (SPA) coupled PLS (SPA-PLS) were used for chemometric processing of data and the number of latent variables were optimized. The analytical performance of the proposed method was evaluated based on root mean squares errors (RMSE). Comparing the utilized chemometric methods, SPA-PLS has superiority toward PLS due to the variable selection in calibration, providing more reliability.



Selected variables after successive projection algorithm processing

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Thioacetamide modified copper electrode Application to electrocatalytic oxidation of hydrazine

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Abstract

Hydrazine and its derivatives are commonly known fuels in explosives, antioxidants, rocket propellants, blowing agents for plastic, photographic chemicals, corrosion inhibitor, insecticides, and plant and growth regulators [1]. Because this substance and its derivatives are applied in such a variety of different fields and has been recognized as a carcinogenic and hepatotoxic agent, its detection has attracted considerable analytical interest [2].

In the present study copper electrode modified with thioacetamide used for electrocatalytic oxidation of hydrazine in 0.1 M NaOH. Modified electrode showed excellent catalytic activity for hydrazine oxidation. With this modified electrode, the oxidation potential of hydrazine was shifted toward less positive value, presenting a peak current much higher than those observed on a bare copper electrode. The kinetic parameters such as the electron transfer coefficient (α_a), and the catalytic rate constant (K_{cat}) for the oxidation of Hydrazine were determined .The diffusion coefficient of hydrazine in the solution was also calculated ($4.08\times10^{-6} cm^2 s^{-1}$) by chronoamperometry. The experimental result showed that the catalytic oxidation peak current of hydrazine is linear dependent on the concentration of hydrazine in the range of 330 -5950µM with CV method.

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Thiosulphate ion-selective electrode (ISE) membranes studied by potentiometry and electrochemical impedance spectroscopy(EIS)

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Abstract:

In the body, thiosulfate converts small amounts of cyanide ion into harmless products and plays a role in the biosynthesis of cysteine.Thiosulfate is a reducing agent, and was widely used in photography, in the paper-making industry and is also useful in the extraction of silver from silver ore, in leather manufacture, and as a mordant in the textile industry.

In this work, a new ion selective electrode thiosulfate anion was prepared. Response surface methodology (RSM) in conjunction with central composite design (CCD) for modeling and optimization of the influence of some process variables (PVC (F_1), ionophore (F_2), additive (F_3) and plasticizer (F_4) amounts), on the performance of PVC membrane Thiosulfate ion-selective electrode is discussed. Potentiometry with ISEs is one of the most successful methods for detecting ionic species in complex samples.The wide use of ISEs in routine chemical and biochemical analysis has been accompanied by a search for ionophores that can chemically recognize specific ions and offer new or improved selectivities for different ions.Construction and application of ISEs as potentiometric sensors offer advantages such as simplicity, speed, fast response, low cost, and wide linear range. These characteristics have inevitably led to construction of sensors for ionic species[1].

Electrochemical impedance spectroscopy (EIS) is another technique used for evaluating interactions within the membrane [2]. In this technique, using an alternating small magnitude signal creates a perturbation at a steady state in the system [3].

The result of $S_2O_3^{-2}$ ion potentiometric analysis showed, the prepared electrode at the optimal amounts of PVC (0.0328 g), ionophore (0.005 g), additive (0.002 g) and plasticizer (0.0625 g) has a Nernstian slope 29.5 mV in the concentration range of 1.0×10^{-6} M to 1.0×10^{-1} M over pH range of 4.0-10.0.

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Thiosulphate ion-selective electrode (ISE) membranes studied by potentiometry and electrochemical impedance spectroscopy(EIS)

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Abstract:

In the body, thiosulfate converts small amounts of cyanide ion into harmless products and plays a role in the biosynthesis of cysteine.Thiosulfate is a reducing agent, and was widely used in photography, in the paper-making industry and is also useful in the extraction of silver from silver ore, in leather manufacture, and as a mordant in the textile industry.

Inthiswork, a newionselectiveelectrodethiosulfateanionwas prepared. Response surface methodology (RSM) in conjunction with central composite design (CCD) for modeling and optimization of the influence of some process variables (PVC (F_1), ionophore (F_2), additive (F_3) and plasticizer (F_4) amounts), on the performance of PVC membrane Thiosulfate ion-selective electrode is discussed.Potentiometry with ISEs is one of the most successful methods for detecting ionic species in complex samples.The wide use of ISEs in routine chemical and biochemical analysis has been accompanied by a search for ionophores that can chemically recognize specific ions and offer new or improved selectivities for different ions.Construction and application of ISEs as potentiometric sensors offer advantages such as simplicity, speed, fast response, low cost, and wide linear range. These characteristics have inevitably led to construction of sensors for ionic species[1].

Electrochemical impedance spectroscopy (EIS) is another technique used for evaluating interactions within the membrane [2]. In this technique, using an alternating small magnitude signal creates a perturbation at a steady state in the system [3].

The result of$S_2O_3^{-2}$ ion potentiometric analysis showed, the prepared electrode at the optimal amounts of PVC (0.0328 g), ionophore (0.005 g), additive (0.002 g) and plasticizer (0.0625 g) has a Nernstian slope 29.5 mVin the concentration range of 1.0×10^{-6} M to 1.0×10^{-1} M over pH range of 4.0-10.0.

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TiO_2 size reduction in presence of CNT and enhancement of photocatalytic performance in removal of azo dyes

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Abstract

photocatalytic degradation by semiconductors is a new, effective, rapid and destructive technique for water and wastewater treatment. TiO_2 is considered to be one of the best candidate photocatalysts [1]. On the other hand, carbon nanotube–inorganic hybrid materials are a new class of functional materials with exceptional properties. The main objectives of this paper are synthesis of TiO_2–CNT (carbon nanotube) hybrid, and the effect of CNT on synthesis and photocatalytic performance of TiO_2. TiO_2-CNT hybrids were prepared via the sol–gel process using pristine MWCNT and functionalized MWCNT as templates. Also, TiO_2 was prepared in the same method. The synthesized materials were characterized by XRD and FE-SEM. XRD results revealed that the crystalline structure of pure TiO_2 and TiO_2 coated on the surface of MWNTs were anatase[2]. The anatase crystallite size for characteristic peak was obtained equal to 20.7, 10.8 and 8.3 for pure TiO_2, TiO_2–pristine CNT and TiO_2-functionalized CNT, respectively. FE-SEM confirmed the presence of a layer of TiO_2 coating MWNTs with thickness in the range of 3-45 nanometer in both hybrids. In order to investigate the photocatalytic activity, photodegradation experiments were carried out using TiO_2 and TiO_2-functionalized CNT as photocatalysts and azo dye (acid blue 92) aqueous solution as model for organic contamination. With initial dye concentration 20 ppm, the photocatalyst dosage 80 ppm and pH=7, 35% and 48.3% decolourization was observed for TiO_2 and TiO_2-CNT hybrid, respectively. The effect of initial dye concentration was also examined employing TiO_2-functinalized CNT hybrid as photocatalyst. This effect was obtained with different initial concentrations (10–25 ppm). The best result for decolourization was obtained in initial dye concentration 15 ppm, and was equal to 81.9% after 120 min. The semi-log plot of dye concentration versus time was linear, suggesting first order reaction. First order reaction rate constant was calculated equal to 1.4×10^{-2} min^{-1} ($R^2=0.985$) at best performance of decolourization.

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TiO_2-nanoparticles modified with cetyl trimethyl ammonium bromide as solid sorbent for preconcentration of parathion prior to determination by gas chromatography

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TiO_2-nanaoparticles due to their unique properties have attracted high attention. They have been widely used as photo-catalyst for removal/ photo-degradation of organic pollutants [1, 2]. They have also been used for separation/ preconcentration of metal ions after modification by complex forming reagents [3]. Solution pH influences the surface charge of TiO_2; at pH values higher than pH_{pzc} (point of zero charge), the cationic surfactant can form admicelles on the negatively charged TiO_2. Parathion an organo phosphorous insecticide with agricultural applications is toxic towards human being and can cause dysfunction of central nervous system. Its interaction with oxidase enzyme has carcinogenic effect. Thus its determination is of great importance for pollution control. In the present work the performance of TiO_2-surface modified with cetyl trimethyl ammonium bromide (CTAB, cationic surfactant) and packed in a mini-column was investigated for solid phase extraction/ preconcentration of parathion prior to determination by gas chromatography.

Quantitative extraction of parathion was obtained at pH 7.0-9.0. Parathion was then eluted from column using 0.2 mL toluene and determined by gas chromatography. The variables affecting the efficiency, including solution pH, amount of modified TiO_2, and sample and eluent flow rates were optimized as 8.0, 6.0 mg modified with 73 µg CTAB, 3 ml min^{-1} and 0.2 ml min^{-1}, respectively. The adsorption capacity of modified-TiO_2 was found to be 35.6 mg g^{-1}. The detection limit (3σ) was found to be 0.079 µg L^{-1} for parathion. The proposed method was successfully applied for water samples.

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Title: Development of models QSPR for the prediction of thermal stability of explosion nitroaromatics

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Abstract: Explosive properties extensively in safety procedures, maintenance and improvement of the family better use of explosives, because of relevance to the structure and the ability to obtain explosives in the laboratory are very important. In order to get the best models causing the relationship between structure-property of explosives, was a extensive study on the molecular descriptors to determine the important properties of explosives, such as thermal stability. The amount of energy released by colorimetric analysis on the selection criteria for the classification of chemical explosives as the regulatory process . In this study, a set of 22 nitroaromatic derived from QSAR models developed using multiple linear regression analysis (MLRA) was studied and to measure the thermal stability of nitroaromatics particularly nitroben derivatives compounds of nitroaromatics with an increase in temperature of 4 ° C per minute crucible equipped with high pressure and heat gain from the analysis were used . According to the method used, the values of R^2, R^2_{cv} , F and s^2 respectively; 0.989, 0.984, 397 and 605.60 predicted and were obtained. The best model for each of the properties listed were selected. Then by the stability and robustness models (s, n, F, R^2) and cross validation (LMO, LOO) were evaluated. With regard to the external test set correlation coefficient R^2 which is equal to 0.989 the model is a satisfactory model.

Keywords: QSPR, MLRA, prediction, nitroaromatics , thermal stability, explosive.

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Title: Dispersive Liquid- Liquid Microextraction and derivatization for determination of Chloroacetic Acids in drinking water by GC/ECD

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Dispersive liquid- liquid microextraction is a simple and fast microextraction technique that have major advantage like used the negligible volume of extraction solvent, rapid achieving of a state equilibrium, low cost and high enrichment factors [1,2].

DLLME is based on the rapid injection of an suitable mixture of extraction solvent and disperser solvent into sample solution, the formation of a cloudy solution and then centrifugation and finally collecting extraction solvent [3].

Extraction solvent must in have the ability to extract the target analyte, have low solubility in aqueos phase and in traditional DLLME have density higher than water however since the number of organic solvent that have this feature are low and most of them are toxic so that we can use of solvent with a density lower than water[3].

In this study DLLME is used for extraction of chloroacetic acids (MCAA, DCAA, TCAA) with octanol as extraction solvent and acetonitrile as disperser solvent. One of major advantage of this work is fast extraction (0 min) and derivatization (2 min). A new device was designed for removing extraction solvent from solution. Under the optimized condition the proposed method yielded detection limits very low for DCAA and TCAA with ranges from 0.1- 0.02µg/l and relative standard deviation were below 2.8%.this method has been applied to the analysis of real water samples and good spiked recovery over the range 90-115% were obtained.

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Title: Photosensitizer Activity Prediction through Validation of Quantitative Structure Activity Relationship (QSAR) by multiple linear regression analysis (MLRA) Model.

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Abstract: A relatively new technique for the treatment of cancer is photodynamic therapy (PDT). In the PDT method that the content of certain compounds such as photosensitizers, oxygen and light are used for generating of so reactive singlet oxygen. Reactive singlet oxygen oxygen directly kills the tumor cells through damaging blood vessels and interfering with the immune system destroys. Through a quantitative structure-activity relationship (QSAR), and develop a model can change the structural characteristics of photosensitizers for resulting a few side effects of PDT and more activity. In this study, a set of 36 porphyrin derived from QSAR models developed using multiple linear regression analysis (MLRA) was studied. The models have respectively 24, 12 and 20 compounds in the training, testing and external testing set. According to the method used, the values of R^2, R^2_{cv} and R^2_{pre} respectively; 0.87, 0.71 and 0.70 predicted and were obtained. The theoretical half maximal inhibitory concentration $(IC_{50})_{cal.}$ values and five important biological activities respectively ranging from 0.54 to 8.78 micrometers and 0.602 to 0.918 (a number of biological activities always is between 0 and 1) were predicted. With regard to the external test set correlation coefficient R^2_{ext} which is equal to 0.52 the model is a satisfactory model.

Keywords: QSAR, PDT, IC_{50}, MLRA, photosensitizers, porphyrin, biological activities.

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Tosyl surface carbon nanoparticles at the surface of glassy carbon electrode: Simultaneous determination of ascorbic acid and uric acid

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Abstract

UA and AA commonly coexist in biological fluids of humans, mainly in serum, blood and urine. The major problem with simultaneous determination of UA and AA by electrochemical methods is the closeness of the oxidation potentials of these two compounds which results in an overlapped voltammetric response which makes their signal discrimination very difficult [1]. A sensitive and selective electrochemical sensor was fabricated via the drop-casting of sulfonate carbon nanoparticles (4-18nm) suspension onto a glass carbon electode (GCE).we have applied glassy carbon electrode modified by using Sulfonatecarbon nanoparticles for simultaneous determination of ascorbic acid and uric acid in different concentration. On the other hand, using MGCE, two well-defined anodic waves with a considerable enhancement in the peak current and a remarkably peak potential separation near 315 mV is obtained.The effects of pH and scan rate were studied.The prepared modified electrode exhibits a very good resolution between the voltammetric peaks of ascorbic acid and uric acid in acetate buffer pH 2 nearly 452 mV. A linear range of 1×10^{-3} to 1×10^{-8} and and 10^{-4} to 5×10^{-7} M from with detection limit of 10^{-5} M and 2×10^{-7} was obtained for ascorbic acid and uric aicd using differential pulse voltammetry, respectively.

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Towards pure electrospun polyaniline nanofibers by using of polyvinyl alcohol as template

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Electrospinning is an interesting approach for obtaining polymer nanofibers. These fibers have large aspect ratios with diameters ranging from 10 nm to 10μm and hence have received a great deal of attention by the scientific community for their vast potential applications. Polyaniline (PANI) is a well-know and widely studied conducting polymer that has poor processability by electrospinning due to its poor solubility in common solvents. However, it is possible to spun polyaniline nanofibers another polymer is added to the major polymeric solutions [1]. PANI in its basic form is non-conductive but it can become conductive in acidic media and when it is electrospun at the presence of an insulator copolymer the fibers/wires conductivity is decreased and this can be resolved by removing the side polymer. In this work a method for the preparation of highly pure PANI fibers was successfully implemented by electrospinning. Polyvinyl alcohol (PVA) insulating polymer was found to be a suitable medium to be used as a facilitating constituent to prepare PANI-PVA nanofibers. Eventually, the PVA constituent was removed from the polymeric network in order to prepare a pure PANI nanofibers mat. The use of a single solvent system with small preparation time for polymer solution blending, no need to use a dopant such as camphor sulfonic acid and also access to higher PANI ratio in respect to PVA are among major achievements [2,3].

Firstly, PANI powder was synthesized chemically by oxidative polymerization of aniline in an aqueous HCl solution. Aniline (0.1 M) was dissolved in 1 M aqueous solution of HCl below 5 °C and an aqueous solution of ammonium peroxydisulphate (0.1 M) was added to the above solution over a period of 30 minutes with vigorous stirring. The mixture was stirred continuously for 2 h. The precipitate was collected by filtration and then washed repeatedly with distilled water. In second stage, the blend polymer solution prepared by dissolving 120 mg of PVA and 60 mg of PANI then was stirred in 2 mL trifluoroacetic acid (TFA) for about 4 hour to get homogenized. After that, 1.5 mL of this solution was withdrawn by a 2.5 mL syringe which was eventually transferred to a syringe pump. A piece of aluminum foil (10 × 10 cm) was employed as a collector electrode. The collector and the polymer containing syringe needle were connected to the high voltage power supply terminals. The distance between the needle and the collector was set at 10 cm. A voltage of 15 kV was applied for the nanofibers production while a flow rate of 4.0 μL min^{-1} was set for the syringe pump to deliver the polymer solution. In final stage the prepared PVA/PANI electrospun nanofibers was located in doubly distilled water at 90°C to remove PVA template and access to highly pure PANI nanofibers.

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Two dimentional gaussian moving windows avereging for denoising of Gas Chromatography-Ion Mobility Spectrometry Row Data

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Ion Mobility Spectrometry (IMS) provides analytical information in a simple, rapid, and inexpensive manner [1]. This novel gas sensor become the dominant commercial technology in the field detection of trace amounts of organic compounds in different industries including forensics, military, security, and counterterrorism, industrial and environmental monitoring, biological, biomolecular researches, and medical applications, the last and perhaps the most interesting one is in both manned and robotic space explorations [2]. IMS cannot be work properly for identifying matrix compounds. To overcome this problem a gas chromatograph (GC) can be used to introduce individual components of mixture into an IMS and as a result attaining the high sensitivity of IMS along with high selectivity of GC [3].

The output signal of the hyphenated GC-IMS method is an extraordinary small, time-dependent current produced by mobile ions in atmospheric pressure. To exploit the qualitative and quantitative information hidden in this sensitive noisy signal various signal processing methods have been nominated for spectrum filtration and improving signal-to-noise ratio (SNR). In the present paper an attempt is made to investigate various spectrum filtration methods for removing noise from GC-IMS signal. The GC-IMS instrument used in this research was designed and constructed in Engineering Research Center of Isfahan, Detector group. Different methods for improving SNR are compared with each other and the efficiency of them will be mentioned. These methods are including Signal Averaging (SA) and Window Averaging (WA), Two dimentional gaussian moving windows avereging (2D GWA). The results of implementing these methods on raw data collected by data acquisition system will be presented and the consequent SNRs will be compared.

Keywords: *Gas Chromatography, Ion mobility Spectrometry, Signal-to-Noise ratio, Two dimentional gaussian moving windows averaging, Fourier transform*

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Two-dimensional asymmetric least squares for comprehensive baseline correction in comprehensive two-dimensional gas chromatography

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In recent years, comprehensive two-dimensional gas chromatography (GC×GC) has been accepted as a powerful technique with high resolving power (peak capacity) and high sensitivity for the analysis of complex mixtures, such as petrochemical, biological, food and environmental samples [1]. In gas chromatography (GC), the chromatographic peaks which correspond to chemical constituents in the sample, rise above a background level in the output. The baseline/background variations (e.g., noise, offset, drift and wander) are mostly due to small changes in experimental conditions and sample matrix [2]. Baseline/background contribution, particularly associated to noisy signals, may cause problems, especially for identification and quantification, since they change both, shapes and retention times of chromatographic peaks.

In the present contribution, a two-dimensional asymmetric least squares (2D-AsLS) algorithm is developed for comprehensive baseline correction in GC×GC. The algorithm is termed comprehensive because it functions to correct the entire chromatogram in both dimensions and it preserves the separation information in both dimensions. In this method, a smoother was combined with asymmetric weighting of deviations from the (smooth) trend to get an effective baseline estimator in both chromatographic dimensions [3]. This new method is fast, even for large datasets, and there is no need to prior information about peak shapes or baseline (polynomial) by the method. Also, the flexibility and the position of the baseline can be easily tuned with two parameters of asymmetry (p) and smoothness (λ). This 2D baseline correction algorithm was successfully applied to total ion current (TIC) chromatographic profiles obtained from GC×GC-TOFMS analysis of standard mixtures of polycyclic aromatic hydrocarbons (PAHs) and aromatic fractions of oil samples (i.e., twenty samples). In addition, GC×GC-FID data obtained from the analysis of essential oils were used to evaluate the performance of this new method. The three datasets were collected using slightly different temperature or pressure programs and different matrices to engender significant baseline drift in the raw data and then demonstrate subsequent corrections of that drifts upon comprehensive 2D baseline correction of the datasets. Finally, the performance of 2D-AsLS was compared with previously developed method of baseline correction in GC×GC based on subtraction of minimum value in each modulation cycle which showed an improvement. It is concluded that the newly developed algorithm of 2D-AsLS has potential to use as an efficient method for comprehensive baseline correction in GC×GC. Although the algorithm is demonstrated for baseline correction in GC×GC data, but it can also use to correct baseline in all forms of 2D separations, such as LC×LC, LC×CE, CE×CE, and LC×GC.

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Ultrasonic assisted Solid phase extraction pre-concentration and high performance liquid chromatographic determination of imidachloprid and thiacloprid using N660

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Neonicotinoids, such as imidacloprid (IMCP) and thiacloprid (THCP), are an important class of pesticides against a variety of insects [1]. Unlike traditional pesticides such as organophosphates or carbamates, most neonicotinoid pesticides contain a chloronicotinyl moiety, which has high agonistic affinity for nicotinic acetylcholine receptors found in many insects [2]. Despite of their specific toxicity for insects and high effectiveness, neonicotinoid residues are still harmful to human beings. Therefore, a suitable method for trace analysis of neonicotinoid residues is needed. Liquid chromatography has been widely employed for pesticides determination, in spite of the fact that the sample preparation usually requires laborious and time consuming steps. This study introduces a novel method of ultrasonic assisted solid phase extraction (SPE), pre-concentration and reversed-phase high performance liquid chromatographic determination of IMCP and THCP from an aqueous medium by N660 (Sterling V) (carbon black particles) as an adsorbent. Results demonstrated that N660 is quite efficient for extraction and pre-concentration of traces of IMCP and THCP at room temperature. The pre-concentrated pesticides were analyzed by HPLC on a Nucleodur (250 mm × 4.6 mm) column with water- acetonitrile- 0.1 M H_3PO_4 (pH 3), 50:45:5, as mobile phase (1 mL min^{-1}) in isocratic mode. A diode array UV detector was used at 270 nm for IMCP and then switched to 240 nm for determination of THCP (after 4.2 min from the injection). These tests investigated the effects of parameters such as pH, buffer and its volume, electrolyte concentration, composition and volume of the desorbing solvent, accepted tolerable volume, amount of adsorbent, ultrasonic radiation time and evidence of some co-existing ions on preconcentration and determination of IMCP and THCP. The method showed good linearity for determination of these compounds in the range of 1-5000 ng mL^{-1} with regression coefficients better than 0.9981. Evaluations for limits of detections (LODs) were 0.8 and 0.6 ng mL^{-1} for IMCP and THCP respectively. Relative standard deviations (RSDs) for 200 ng mL^{-1} and 1000 ng mL^{-1} were below 4.5% and 4.1%, respectively. Different samples, including tap water, Karoon River Water, soil, plant leaves and vegetables were used as real samples and IMCP and THCP in the samples and spiked samples were pre-concentrated and determined successfully.

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Ultrasonic synthesis of TGA-capped CdSe quantum dots and its application in Fluorometric determination of 4-nitrophenol

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Abstract

Luminescent thioglycolic acid (TGA) capped CdSe quantum dots (Qdots) have been produced at few minutes by using a simple and fast methodology in an aqueous medium by using ultrasound irradiation to accelerate the process of selenium reduction. The use of ultrasound allows the morphology to be better controlled and the surfaces defects of Qdots to be reduced. The structural and optical properties were characterized by TEM, XRD, absorption and fluorescence spectroscopy. The produced Qdots are in a strong quantum confinement regime and have only one fluorescence band. Moreover, the Qdots seem to be monodispersed, which is in accordance with the fluorescence results.

4-NP is one of the nitrophenols in the U.S. Environmental Protection Agency List of Priority Pollutants [1]. 4-NP is a hazardous substance which can cause a high environmental impact due to its toxicity and persistence. The origin of contamination comes from manufacturing, chemical industry and agricultural practices [2]. Consequently, qualitative and quantitative detection of nitrophenols is a matter of concern for environmental control. The detection of nitrophenols is usually accomplished by chromatographic techniques and different electrochemical methods such as polarography, cyclic voltammetry (CV), adsorptive stripping and differential pulse voltammetry (DPV) [3].

In this study, a simple and sensitive method for 4-NP determination was established based on the decrement of the fluorescence intensity of TGA-capped CdSe quantum dots in aqueous solution. Meanwhile, the fluorescence characteristics and the optimal conditions were investigated in detail. Under the optimized conditions, the linear range of QDs fluorescence intensity versus the concentration of 4-NP was 0.005–100 mg L^{-1} (with selected twelve concentrations and five replications), with the correlation coefficient (R^2) of 0.995 and a limit of detection of 0.00224 mg L^{-1} for the target molecule. There was no interference to coexisting foreign substances including other nitrophenols and some substituted phenols.

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Ultrasound assisted microextraction-nano material solid phase dispersion for extraction and determination of thymol and carvacrol followed by high performance liquid chromatography: experimental design methodology

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Abstract

In the present study, for the first time, a new extraction method based on ultrasound assisted microextraction nano material solid phase dispersion (UAME-NMSPD) was developed to preconcentration of thymol and carvacrol in pharmaceutical samples prior to their determination using high performance liquid chromatography/UV detection (HPLC-UV). Thymol and carvacrol are the two major components naturally found in oil of thyme [1]. Due to unique role of these two phenolic components as active constituents most of the pharmaceutical dosage forms are standardized according to their thymol or carvacrol contents [2]. Due to complicated matrices of real sample containing these compounds an efficient sample preparation for their separation and preconcentration is requirement. This method is based on dispersion of nanoparticles into aqueous media by ultrasonic device for adsorption of target analyte, and then adsorbed analyte is eluted and determined by HPLC-UV. The properties of nickel sulfide nanomaterial loaded on activated carbon (NiS-AC) were characterized by XRD, FESEM and UV-Vis analysis. Before the optimization, the type of extraction solvent was selected. Influence of the variables (sonication time, amount of adsorbent, ionic strength (NaCl %), pH and volume of extraction solvent) were investigated and optimized by central composite design (CCD) combined with response surface methodology (RSM) using the desirability function (DF). Under the optimum conditions (10 min ultrasonic time, pH 3, 0.011 g of adsorbent and 600 µL extraction solvent) the method has linear response over 0.005-4.0 µg mL^{-1} (r^2>0.993) with limit of detection 1.5 µg L^{-1} and the relative standard deviations (RSDs) less than 4.93% (n=3). At this optimum value of variables, the separation was achieved in less than 10 min using a C_{18} column and an isocratic binary mobile phase acetonitrile: water (60:40, v/v) with flow rate of 1.0 mL min^{-1}. In this method chlorine solvents was eliminated and sample preparation time as well as the consumption of the toxic organic solvent was lowered and lead to enhance in the sensitivity of the method.

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Ultrasound-assisted emulsification-microextraction combined with gas chromatography flame ionization detection for determination of hexanal and heptanal

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Aldehydes such as hexanal and heptanal (are formed by free-radical induced reactions with cellular lipids.) are generated by human body. These compounds have been much investigated, since elevated level of them is considered as the biomarker of different diseases [1-3].Ultrasound-assisted emulsification microextraction (USAEME) coupled with gas chromatography-flame ionization detection (GC-FID) was developed for extraction and determination of hexanal and heptanal in oil sample. Methanol (200 µL) was rapidly injected into oil sample. The mixture was immersed into an ultrasonic bath for water-in-oil (W/O) emulsion formation. After centrifugation, two distinct layers were formed. Several factors influencing the extraction efficiency such as the nature and volume of organic solvent, extraction time and centrifugation time were investigated and optimized. Under optimized experimental conditions, the calibration curves for hexanal and heptanal were linear in the range of 0.1-200 mg L^{-1}. The limits of detection were 0.03 mg L^{-1} for hexanal and 0.06 mg L^{-1} for heptanal, respectively. The relative recoveries of oil sample ranged from 96.2% to 101.0% and relative standard deviations (RSDs) values were less than 4.0%. Analysis of variance (ANOVA) was used to show effect of temperatures and times of formation of hexanal and heptanal in edible cooking oil sample.

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Ultrasound-assisted extraction of carotenoids from citrus peels using ostrich oil as a green solvent

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ABSTRACT

In this work a simple and efficient method for extraction of carotenoids from citrus peels was invented. Ultrasound-assisted extractions show significant effect on the extraction rate by increasing the mass transfer rate. Ostrich oil is one of the most valuable foundations in many branches of industry, cosmetics and nutrition. It appears to have anti-inflammatory properties and can be used as a green solvent in many studies to eliminate petroleum-based solvents. Due to the ability of carotenoids to absorb light, UV-Visible technique was used in this study as an easy-to-use and inexpensive method. The highest yield of carotenoids (27.42 mg/l) in λ_{max} =330 nm was obtained and the optimal extraction conditions were ultrasonic intensity of 16.5 W.cm^{-2}, temperature of 45 ^{o}C and sonication time of 30 min.

Keywords: Ultrasound-assisted extraction, Carotenoids, Citrus peels, Ostrich oil

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Ultrasound-assisted microextraction based on task-specific ionic liquid for the preconcentration of Ag(I) ions

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An ultrasound-assisted microextraction using task-specific ionic liquid was proposed for the extraction of metal ions prior to electro-thermal atomic absorption spectroscopy determination. Task-specific ionic liquid (TSIL) concept is introduced in extraction methods in order to synthesize ILs with properties needed to extract metal ions [1]. Partitioning of metal ions into an IL extracting phase in liquid–liquid systems is negligible owing to the tendency of the metal cations to remain hydrated in the aqueous phase. Thus, the use of an extractant molecule that forms complexes directly with metal ions to increase their hydrophobicity is necessary [2]. A TSIL acts as the hydrophobic solvent and the chelating agent at the same time reducing the chance for IL loss during the aqueous phase [3]. The TSIL used in the present work, was an imidazolium-based IL with a thiol-functionalized anion and a thiourea-functionalized cathion. In this method, Ag(I) ions were extracted into the fine droplets of the TSIL after chelate formation with the function groups. The fine droplets were made and dispersed as a cloud in the aqueous sample with the help of ultrasonic waves. After centrifuging step, Ag(I) determination was carried out in the IL-rich phase with electro-thermal atomic absorption spectroscopy. Several variable factors affecting the extraction and complex formation, such as pH, TSIL amount, sonication time, centrifugation time, salt addition were optimized. The optimized procedure was applied to the extraction and determination of ultra-trace amounts of silver ions in real samples. The obtained results showed that the proposed method is rapid, simple, sensitive, selective, low cost, volatile organic solvent-free, and efficient for the separation of trace amounts of Ag(I) ions.

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Ultrasound-assisted solvent extraction followed by dispersive liquid–liquid microextraction based on the solidification of floating organic drop for determination of organophosphorous pesticides in summer fruits

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Pesticides are essential in modern agricultural practices but, due to their biocide activity and potential risk to consumers, the control of the presence of pesticide residues in foods and vegetables is a growing source of concern for the general population [1]. Organophosphorus pesticides (OPPs) are a class of pesticides that generally act as cholinesterase inhibitors and are used as insecticides in a wide variety of crops [2]. Summer fruits are used almost daily in several countries, raw or processed as a canned product or juice.

This paper describes a rapid, specific and sensitive method for the quantitative analysis of OPPs in summer fruits by means of ultrasound-assisted solvent extraction (UASE) followed by dispersive liquid-liquid microextraction (DLLME) [3] combined with high performance liquid chromatography-ultraviolet detection (HPLC-UV). OPPs in fruits samples were extracted in to acetone, assisted by ultrasonic bath. Extracted OPPs were preconcentrated with 1-undecanol using DLLME-SFO technique and determined by HPLC-UV. Under the optimum conditions, the extraction recoveries were high and ranged between 44-61%. The linear range was wide (5-800 µg/Kg, four orders of magnitude) and limit of detections were low and were between 1-4 µg/Kg for most of the analytes. The relative recoveries of OPPs from fruit samples by spiking OPPs at the different concentration levels were 88-110%.

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Use of GC-MS Combined with Resolution Methods to Characterize and to Compare the Essential oil components of Green and Bleached Cardamom

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Gas chromatography-mass spectrometry combined with iterative and non-iterative resolution methods was used to characterize and to compare the essential oil components of green cardamom and bleached cardamom.

Green cardamom and Bleached cardamom essential oil components were extracted by microwave-assisted hydrodistillationand analyzed using gas chromatography-mass spectrometry. Thirty six and fifty five components were identified by direct similarity searches for green and bleached cardamom, respectively. These numbers were extended to fifty five and seventy one components, respectively with the help of multivariate curve resolution methods. Morphological score and subspace comparison were used for chemical rank determination of GC-MS data. Multivariate curve resolution-alternative least squares as an iterative method was used for resolving the overlapped and embedded peaks [1-2].

Comparison of the results of green and bleached cardamom showed that their volatile components are different from chemical components and relative percentages points of view. Major constituents in green cardamom are 1,8-cineol (47.18%) , alpha-terpinyl acetate (14.33%) , linalool (6.28%) , terpineol (4.94%) l-4-terpineol (2.48%) and in bleached cardamom are 1,8-cineol (34.12%) ,alpha-terpineol (26.91%) , alpha-terpinyl acetate (21.04%), linalool (6.39%) and l-4-terpineol (1.89%). In spite of different cultivation conditions, there are 33components which are common between the two types of cardamom.

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Use of Graphene/polyaniline nanocomposite electrosynthesized for determination of catechol by DPP

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Abstract

An electrochemical sensor based on graphene–polyaniline (GR–PANI) nanocomposite for determination of catechol is presented. The electrochemical behavior of catechol at the GR–PANI composite film modified composite graphite electrode (CG) was investigated by Differential Pulse Polarography (DPP). This electrochemical sensor shows a favorable analytical performance for catechol detection with a detection limit of 6.2×10−8M And high sensitivity604.5 μmM−1. Moreover, catechol and hydroquinone can be detected simultaneously without interference of each other in a large dynamic range.

Keywords: Differential Pulse Polarography (DPP) , catechol , polyaniline

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Abstract

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Keywords:Differential Pulse Polarography (DPP) , catechol , polyaniline

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Use of modified multi wall carbon nano tube with dithiooxamide for separation and determination of trace amounts of nickel ions in food and natural water samples

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Abstract

A new solid-phase extraction sorbent was used for the separation/preconcentration of Ni (II) ions prior to their determination by flame atomic absorption spectrometry (FAAS). Extraction experiments were carried out using batch system. It was prepared by immobilization of dithiooxamide on multi wall carbon nano tube (MWCNT). Nano sorbent was synthesized by the reaction of dithiooxamide with functionalized multi walled carbon nanotube. Initially, the surface of the multi-walled carbon nanotube oxidized by a mixture of nitric and sulfuric acid and then functionalized using thionyl chloride. The ligand has been attached to the multi-walled carbon nanotubes in a somewhat shorter time and lower temperature than previous reported methods [1-2].The properties of sorbent were characterized by scanning electron microscope (SEM) and FT-IR. A microsample introduction system was employed for the nebulization micro-volume of diluted solution into FAAS. Some effective parameters on extraction and complex formation, such as pH, concentration and volume of the chelating agent, adsorbent dosage, eluent concentration and volume, extraction time, salt effect and sample volume have been optimized. The calibration graph was linear range between 50-900 ng mL^{-1} with detection limit of 18.9 ng mL^{-1}. Relative standard deviation (RSD) for Ni ions were 1.6%. The proposed method has been applied to the determination of nickel ions at ng mL^{-1} levels in real samples sush as tomatoes, potato, wheat flour, red beans, oat, tap water, river water and sea water with satisfactory results.

Keywords: Solid phase extraction, Preconcentration, Nickel, Separation.

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Using CuO/ nanoclinoptilolite in photodegradation of 2, 6- dimethyl phenol

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Abstract

Advance oxidation processes, based on the irradiation of a semiconductor and generation electron-hole pairs, produce powerful hydroxyl radical can destroy organic pollutants to smaller fragment and finally to water and carbon dioxide [1-3]. In this work, we studied the degradation of 2,6- dimethyl phenol (2,6-DMP) in heterogeneous phase with CuO-nanoclinoptilolite particles. First, the pretreated micro-sized clinoptilolite was changed to nano particles by mechanical method. The general procedure for doping of Cu^{2+} in nano-clinoptilolite described as follows: 2 g of nano-clinoptilolite was added to the 50 mL copper(II) nitrate 0.5 M and was shaken for 24 h in the polyethylene bottle at room temperature. The solid product was filtered and washed with double-distilled water. Then it was dried at room temperature. Finally, the ion-exchanged zeolite was calcined at 450 °C in a furnace for 12 h. The obtained samples were referred to as CuO/nanoclinoptilolite (CuO/NCP). The proposed catalyst was used in photodegradation of 2,6- DMP aqueous solution under UV irradiation. The parent nano zeolite and the photocatalyst were characterized by XRD, FT-IR, DRS and TEM methods. The degradation process was monitored during the experimental runs through UV/Vis absorption as well as COD concentration determination and HPLC. In a typical photodegradation experiment, suspensions containing a definite amount of the pollutant and catalyst were irradiated and at regular time intervals, the absorbance of the irradiated and un-irradiated solutions was recorded to calculate the degradation extent. The effect of different variables on the performance of photodegradation was investigated by examining the catalyst dosage, CuO loaded extent, pH and initial concentration of tetracycline solution. The optimal conditions for each parameters was obtained as: 0.25 g L^{-1} of CuO/NCP, 30 ppm of 2,6–dimethylphenol, pH of 5.5 during 360 min. The CuO particles out of zeolite framework did not show significant degradation efficiency. The degradation process obeyed first–order kinetics.

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Using of nano liquid chromatography for evaluating of prepared porous silica monolithic capillary column

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Stationary beds based on porous monolithc materials for nano liquid chromatography (nano-LC) have been successfully fabricated by the sol-gel process [1, 2]. In this work silica monolithic capillary columns are prepared by silica alkoxide in an acidic condition utilizing a chelating agent and cross linker. Two kinds of columns have been prepared: a hybrid structure from a mixture of tetra methoxysilane (TMOS) and methyl trimethoxysilane (MTMS) and a common silica structure from TMOS only. Morphology of these capillary monolithic columns characterized with electron microscopes. Based on the image analysis of scanning electron microscopy (SEM) and transmission electron microscopy (TEM), macropores and mesopores are calculated 2 µm and 12 nm, respectively. Infrared spectrum of prepared silica monolithic is shown stretching O-H band at 3444 cm^{-1} due to free silanol terminations in the polysilicate reticulates and Si-O-Si and Si-O-H stretching vibrations at 1062 and 1058 cm^{-1}, respectively. Mercury intrusion-extrusion and nitrogen sorption were also used to determined pore size distribution and atomic force microscopy (AFM) used to determined specific surface area. These columns finally were assembled to liquid chromatography system and the major chromatographic performance characteristics (i.e., efficiency, resolution and permeability) of them were investigated.

Kinetic plots and van deemter plots for hydrophilic separation of ß-blockers such carvadilol and iso propanolol with the monolithic silica column are calculated, at flow rate 1 µL min^{-1} to 30 µL min $^{-1}$. The mobile phase consist of acetonitrile and acetate buffer (pH= 4.6, 50:50 v/v at λ= 242 nm). The retention time of carvadilol and propanolol was 6.9 min and 7.8 min in the flow rate 5 µL min $^{-1}$, respectively. The amino acids like tryptophan, phenylalanine and tyrosine were also analyzed with these columns. The influences of different mobile phase, different acid percentage, and different flow rate on separation of them were investigated.

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Using Orthogonal Array Design Optimization For Measurement Of Metal Cations By Cloud Point Extraction Method

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In this study, using 4-(2-pyridylazo)-resorcinol (PAR) as ligand, and in the presence of Triton X-114, preconcentration and determination of ions Cu (II), Ni (II), Fe (III) and Zn (II) were performed assisting flame atomic absorption(FAA)[1] and cloud point extraction[2]. Using one-at-a-time and orthogonal array design (OAD)[3] approaches, experimental conditions were optimized to obtain maximum extraction efficiency of metal ions. Effects of factors such as pH, ligand concentration, Triton X-114 concentration and temperature on the extraction efficiency of metals were investigated. The results from the two optimization procedures were compared. The interaction parameters were determined separately using orthogonal array design. The results show that overally the best levels for pH and temperature were respectively 6 and 30 °C. The optimum ligand concentration in case of Fe(III) ion was half of the other ions. The proper surfactant concentration was higher for Ni(II) ion, compared to other ions. Effects of speed and time of centrifugation and dilution factors on the extraction yields were evaluated and optimized. At the optimal conditions for ions Cu(II) , Zn (II), Ni (II) and Fe (III), the linear dynamic range of calibration model were in the range 50-200 ,50-200 ,50-300 and 50-500 μg L-1. limit of detections for metal ions were between 33 to 47 μg L-1 . From n=5 RSD% for ions were between 1.3 to 2.7. Finally, the proposed method was applied for determination of metal ions in tap water and blood samples in the optimized conditions. The obtained recoveries were between 96 to 102 percent.

key words**:** Flame atomic absorption(FAA), orthogonal array design(OAD), cloud point extraction(CPE).

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Using silver nanoparticles and functionalized carbon nanotubes for fabrication of a new screen printed biosensor to determination of cholesterol

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Abstract

Cholesterol has recently attracted much interest because its level in blood is an important parameter in the diagnosis and prevention of disease. Normally, the concentration of cholesterol in human serum is in the range 1.3–2.6 mg mL-1. There are two forms of cholesterol in blood: the free form (cholesterol) and the esterified form (cholesterol ester) [1-3].

For preparation of screen printed Biosensor, first silver conductive ink was sprayed onto a polyethylene terephthalate (PET) substrate and then the composite of acetone-extracted propolis (AEP) and functionalized single walled carbon nanotubes (f-SWCNT) , which acts as a second layer, coated on the working electrode and the auxiliary one. Finally, the cholesterol oxidase on the surface of the working electrode was immobilized. The f-SWCNT were then used to immobilize the enzyme ChOx covalently on the working electrode by coupling between the carboxyl (–COOH) groups of the single walled carbon nanotubes layer and the amino (–NH2) groups of the enzyme. The principle of measureing cholesterol is based on the detection of reduction signal of hydrogen peroxide produced in enzymatic reactions.The detection of cholesterol can be done in one step, a drop of sample was dropped onto biosensor and the reduction signal was obtained at an applied potential of −800mV (vs. pseudo Ag/AgCl).

The sensing device displays a linear response over the range of 0.5 – 5 mM (R^2= 0.998) for cholesterol. The detection limits and the sensitivity of the biosensor are 155μM and 6.9 nAμM−1.Cholesterol oxidase enzyme catalyzes reaction of free cholesterol only, so this biosensor for free cholesterol measurements in samples with different concentration were made. In this work the application of electrodeposited functionalized single-wall carbon nanotubes (f-SWCNT) and silver nanoparticles as an immobilization matrix for the construction of amperometric chlostrol screen printed biosensor is described.

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Visual, facile and green monitoring of hemoglobin using curcumin nanoparticles

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Hemoglobin (Hgb) is the protein component of the red blood cells that it’s responsible for the carry of oxygen between the respiratory organs such as lungs or gills to the rest of the body. The Hgb molecule has a quaternary structure in which the comprised of four polypeptide chains that each of chain bounds to a heme group. Meanwhile, each heme group contains an iron atom chelated to a protoporphyrin ring [1]. The determination of the Hgb concentration is greatly utilized in blood tests and to the appraisal of the physiological condition because diagnosis of many diseases such as anemia, thalassemia, heart disease, leukemia, sickle cell disease and excessive loss of blood requires a measurement of Hgb [2,3]. Due to the importance of accurate determination of Hgb in blood cells, till now numerous attempts have been made for determination of Hgb. The present context relates to an efficient, simple and visual measurement system for quantitative sensing of blood hemoglobin concentration using curcumin nanoparticle (CURN) as a safe, non-toxic and green nanoparticle. The Hgb monitoring is based on this phenomenon that at the presence of Hgb, the absorption intensity of CURN was decreased. Thus, in this study the analytical signal for the measurement of blood Hgb concentration was defined as the difference in absorption intensity of the CURN in the absence and presence of the Hgb. The method was optimized to enhance the selectivity and sensitivity of the method for conditions such as pH of the sample solution, buffer concentration, required amount of CURN, equilibrium time and tolerance amount of various probable co-existing chemicals. Under optimum conditions, the extent of differences in absorption intensity of the CURN was linearly proportional to the concentration of Hgb in two ranges of 1-40 µg mL^{-1} and 150-1200 µg mL^{-1} with an experimental detection limit of 0.1 µg mL^{-1}. The relative standard deviation for ten replicate measurements of 20 µg mL^{-1} and 600 µg mL^{-1} of Hgb were 2.8% and 3.1%, respectively. Eventually, this assay was successfully utilized for the measurement of Hgb in human blood with satisfactory results.

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Voltammetric determination of acetaminophone using a modified screen printed electrode by graphene

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Screen-printed technology consists of layer-by-layer depositions of ink upon a solid substrate, through the use of a screen or mesh, defining the geometry of the sensor. This technology has advantages of design flexibility, process automation, good reproducibility, a wide choice of materials. Screen-printed electrodes (SPEs) avoid some of the common problems of classical solid electrodes, such as memory effects and tedious cleaning processes. Moreover, the extensive range of forms of modification of SPEs opens a great field of applications for these electrodes. These modifications include for example the immobilization of nanoparticle and nanocomposite for the development of selective and sensitive biosensors [1].

Acetaminophen (ACE) is a widely used analgesic and antipyretic medicine. However, overdose of acetaminophen may be toxic and results in severe side effects. Therefore, the detection of acetaminophen is quite important. Acetaminophen contains phenolic hydroxyl group, which is electrochemical active and can be oxidized. Thus, various materials including carbon nanotube, nano-TiO_2 and etc were used to prepare electrochemical sensors for the detection of acetaminophen. However, electrochemical detection of acetaminophen using GNP has not been reported [2].

In this work, graphene nanoparticle (GNP) was synthesized by electrochemical method and characterized by transmission electron microscopy (TEM). GNP used as a modifier for preparation of modified SPEs and used as a sensor for determination of ACE. The electrochemical behavior of proposed SPEs has been studied in phosphate buffer solution with pH 3.0 with cyclic voltammetry. The catalytic peak current obtained, was linearly dependent on the ACE concentrations in the range of 1 – 500 μM. The detection limits for ACE were 0.2 μM. Finally, this sensor has been used for the determination of ACE in ACE tablet sample.

Keywords: Graphene nanoparticle, Screen printed electrode, Acetaminophen (ACE), Electrochemical determination.

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Voltammetric determination of aminopyrene using MWCNT-SDS-β-CD-nafion composite film modified electrode

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Nowadays, increasing attention is paid to the increase of health risks associated with exposure of human population to harmful substances contained in car emissions. Polycyclic aromatic hydrocarbons (PAHs) and nitrated polycyclic aromatic hydrocarbons (NPAHs), belong to widespread chemical carcinogens [1]. Aminopyrene as one of the representatives of the amino-PAHs is well-known genotoxic and carcinogens substances. Therefore, it is very important to monitor it in living and working environment.

Some polarographic and/or voltammetric methods have been used for the dtermination of trace aminopyrene due to an easy electrochemical oxidation of amino group on aromatic ring [2].

The special electronic properties of carbon nano tubes (CNTs), suggest that they can promote electron transfer reaction when used as the electrode modifier in electrochemical methods. Cyclodextrins (CDs, cyclic oligopyranose oligomers) are widely studied as examples of host molecular receptors. Sodium dodecyl sulfate (SDS) is an anionic surfactant.

In this study, a modified MWCNT-SDS-β-CD-nafion glassy carbon electrode is proposed to determine aminopyrene at trace levels by means of the differential pulse voltammetry method. The ratio of MWCNTs-SDS-β-CD-nafion was optimized and identified to be used in the modification of glassy carbon electrode. After adsorption of aminopyrene on MWCNT-SDS-β-CD-nafion glassy carbon electrode at 0.2 V for 150 sec, it was produced well defined oxidation peak in Robinson buffer. The effect of some other species was investigated on the determination of aminopyrene. It was found that the modified electrode has good sensitivity and selectivity. The oxidation mechanism was also studied.

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Voltammetric Determination of Cetirizine using a Carbon Paste Electrode Modified with Copper Hexacyanoferrate

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Cetirizine dihydrochloride is a non-sedative histamine H1-receptor antagonist used for the treatment of seasonal rhinitis and chronic urtiacaria or pruritsis of allergic origin [1]. A number of analytical methods have been used to quantify cetirizine in pharmaceutical formulations and biological fluids including gas chromatography, high performance liquid chromatography and capillary electrophoresis [2-3]. Although the results obtained from chromatographic methods are reliable with respect to selectivity, their separations are time consuming and they need expensive instruments. To overcome these defects, electrochemical methods are widely used because of their properties, such as selectivity, simplicity, and reproducibility.

In this study, a sensitive electrochemical method for the determination of cetirizine was developed using carbon paste electrode modified with copper hexacyanoferrate. Under the optimized pH of 7.0, the modified electrode showed high electrocatalytic activity toward cetirizine oxidation; the overpotential for the oxidation of cetirizine was decreased by more than 200 mV and the corresponding peak current increased significantly. The procedure was successfully applied to the determination of cetirizine in pharmaceutical and urine samples. The copper hexacyanoferrate modified carbon paste electrode gave comparable results to those results obtained using a high performance liquid chromatography method.

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Voltammetric determination of clonazepam at the glassy carbon electrode modified with nanocomposite of silver nanoparticles/multi walled carbon nanotubes

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Due to the exclusive properties of AgNPs such as high effective surface area and efficient electron transfer[1] with inherent properties of CNTs[2], an AgNPs/CNT/GCE was prepared for a sensitive determination of clonazepam CZP. The obtained composite was characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) analyses and electrochemical technique. Cyclic voltammetry (CV) and differential pulse voltammetry (DPV) respectively were used for electrochemical studies and measurements. It is successfully shown that AgNPs can be synthesized on the CNT without any additional reducing reagent. The results illustrated that the presence of the modifier film on the surface of the electrode significantly affect the sensitivity of the electrochemical responses towards CZP.The electrochemical behavior which has related to the reduction of the nitro group (NO_2) of CZP to hydroxylamine group (NHOH) [3]. High sensitivity and low detection limit of the AgNPs/MWCNTs/GCE are capable to the determination of trace amounts of CZP in pharmaceutical and scientific measures by means of differential pulse voltammetery. Experiments confirmed that the reduction peak currents increased linearly with CZP concentration in the range of 5.0×10^{-8}–2.5×10^{-6} mol L^{-1} with a detection limit of 6.0×10^{-9} mol L^{-1}. The modified electrode was effectively applied for determination of CZP in pharmaceutical measurements.

Keywords: Nanocomposite, Silver nanoparticle, Multiwalled carbon nanotubes, clonazepam

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Voltammetric Determination of Epinephrine at Poly (Glutamine-3-Substitued Thiophene) ModifiedGold Electrode

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A new poly (glutamine3-substituted thiophene) chemically modified gold electrode is proposed for investigation and determination of epinephrine (Ep) by cyclic voltammetry (CV) and differential pulse voltammetry (DPV) techniques. The functionalized monomerused for polymerization was synthesized through the Ullmann reaction [1,2]. The gold electrode was modified with electropolymerized films via its oxidative polymerization from an acetonitrile solution by galvanostatic and cyclic voltammetry methods. The oxidation and reduction peak potentials of epinephrine on the modified electrode recorded in a phosphate buffer solution (pH = 6.0) were 0.35V and -0.21V vs Ag/AgCl respectively. The peak position as well as sensitivity was affected by the potential window used for the determination. In addition the effects of pH and the type of electrolyte on the linearity range and detection limit were investigated. The high sensitivity for epinephrine was found to be due to electrocatalytic effect of amino acid on the oxidation reaction of epinephrine.The differential pulse voltammetric (DPV) peak current was linearly dependent on epinephrine concentration over the range 1.0×10^{-6}- 3.0×10^{-5}mol L^{-1}. The detection limit (3δ) was 3.25×10^{-6}molL^{-1}. The modified electrode has been used for determination of epinephrine in pharmaceutical formulation, with satisfactory results.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان، ٦ الی ٨ اسفند ماه ١٣٩٢

Voltammetric Determination of Gabapentin with a Ni-Catechol Complex Modified Multiwalled Carbon-Nanotubes Carbon Ceramic Electrode

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Abstract:

Gabapentin (GBP), 1-(aminomethyl) cyclohexane acetic acid, is an antiepileptic drug which is also effective in the prevention of frequent migraine headaches, neuropathic pain, and nystagmus [1, 2]. Today GBP is widely accepted clinically So Sensitive and reliable assays required to measure its blood concentrations at both high and low levels for furthering studies of its efficacy, does-related side effects and action mechanism. A modified carbon ceramic electrode (CCE) was used for the determination of GBP. A carbon ceramic electrode (CCE) consists of dispersed carbon based material embedded in a silicate matrix. This electrode was developed in early 1990s [3]. It has been fabricated by electrodepositing Ni (II)–catechol complex on the surface of MWCNT–CCE in alkaline solution (Ni (II)–CA/MWCNT–CCE). A quasi-reversible redox pair was observed at pH 13 (0.1 M NaOH solution). In the presence of GBP, both cathodic and anodic currents were enhanced. GBP could be oxided in less positive potentials (0.45V) than other electrodes. A linear calibration curve was obtained for GBP in the concentration rang 1.25×10^{-6} M - 6.3×10^{-5} M with a detection limit of 0.5 μM. The electrode showed excellent response stability over a period of 2 months and was successfully applied in the analysis of tablets of GBP.

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بیستمین کنفرانس شیمی تجزیه ایران
دانشگاه صنعتی اصفهان ، ٦ الی ٨ اسفند ماه ۱۳۹۲

Voltammetric determination of hydrochlorothiazide using a carbon paste electrode modified with graphene nano-sheets

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Hydrochlorothiazide has gained attention because it is a benzothiazide diuretic that acts directly on the kidney by increasing the excretion of sodium chloride and water and, to a lesser extent, that of potassium ions. hydrochlorothiazide is an antihypertensive substance and improves the action of other hypotensive substances, allowing a decrease in the dose of those below the level where these substances present secondary effects [1].

The subtle electronic properties of graphene suggest that it has the ability to promote electron transfer when used as the electrode material, which provides a new way to design novel electrochemical sensors and biosensors. Hence, the modified graphene electrodes have been successfully applied to study or determine some biological and organic molecules [2].

In this study, a carbon paste electrode modified with graphen nano-sheets and mediator was used to prepare a novel electrochemical sensor for the detection of hydrochlorothiazide. The response of catalytic current with hydrochlorothiazide concentration showed a linear relation. This new sensor was used for determination of hydrochlorothiazide in some real samples.

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Voltammetric Investigation of Oxidation of Desipramine in Aqueous Solution

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Desipramine (DES) are active ingredients of psychiatric drugs widely used in the treatment of depressive disorders[1]. In this work the electro-oxidation of desipramine has been studied in aqueous medium by means of cyclic voltammetry and controlled-potential coulometry methods. In order to understand the whole electrochemical mechanism involved in the reaction of DES, the cyclic voltammetry process was recorded in two repetitive cycles. In the first cycle, only two pseudo-reversible peak (O1) at +0.92 V and (R1) at +0.67V obtained which is due to the electro-oxidation of DES. The electrochemical oxidation of DES occurs at the nitrogen atom in cyclohexane ring resulting in the formation of a radical which is similar to that for the oxidation of methyliminobibenzyl[2]. On the reverse sweep from +1.3 to -0.1 V, a reduction peak (R2) at 0.32 V is observed due to the formation of a dimer [Fig. 1], which gets subsequently oxidized (O2) in the second cycle at 0.74 V. The appearance of the oxidation peak (O1) is due to a two-electron and one proton pseudo-reversible oxidation of the DES radical and that the R2/O2 redox peak is caused by the reversible one electron and one proton oxidation and reduction of the dimer. Electrochemical ECE mechanism proposed based on voltammetry and coulometry diagnostic criteria.

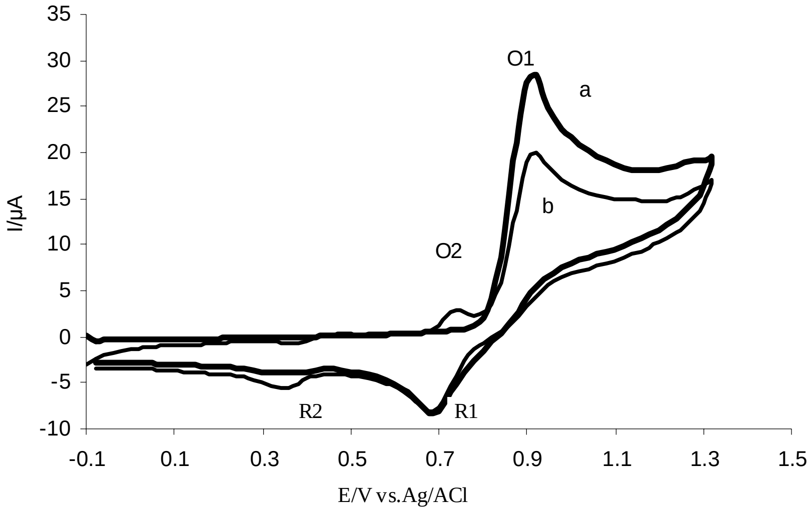

Fig. 1 Two repeated CV(s) for 1mM DES at glassy carbon electrode in 0.2 M phosphate Buffer (pH=2). First scan(a)and second scan(b)at a scan rate of 500mVs^{-1}.

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Voltammetry studies for simultaneous determination of methylparaben and propylparaben at the surface of glassy carbon electrode assisted by chemometric methods

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Preservatives are added to pharmaceutical, cosmetics formulation and also foodstuff to control microbial contamination. The most common preservative system contains 0.3% methylparaben (MP) and 0.1% propylparaben (PP).

Darber has detected the existence of parabens in human breast tumors [1]. Also parabens have estrogenic activity and they have been shown to estrogen receptors from different sources [2]. So it is important to control the amount of parabens in products.

In this research simultaneous determination of MP and PP in alcoholic solution and sulfate buffer using cyclic voltammetry (CV) and differential pulse voltammetry (DPV) techniques at the surface of glassy carbon electrode are investigated. Central composite design (CCD) and response surface methodology (RSM) were used to optimize the influencing parameters such as scan rate, H-step, pulse height and pH. The obtained optimum values of each factors are 0.06 V, 0.009 V, 0.06 V and 7, respectively.

In order to simultaneous determination of MP and PP with a very high degree of overlapping, multivariate calibration methods were used. After optimization and resolution, the linear least squares calibration curve based on the area under voltammograms of the two analytes was provided. Finally the ability of the proposed method was investigated by determination of the two analytes in Moistural cream as a real sample with satisfactory recovery results. These results show a general and promising way for simultaneous determination of MP and PP in real samples.

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Zinc nanoparticles matrix solid-phase dispersion combined with dispersive liquid-liquid microextraction for the determination of thirteen n-alkanes in soil samples

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ABSTRACT

Dispersive liquid-liquid microextraction (DLLME) is a preconcentration method, which used a ternary system of solvents. The advantages of this technique are simplicity, fast, low cost, good efficiency and high enrichment factor. DLLME is a green extraction technique, due to the consumption of organic solvent is reduced to the microliter [1-3].In this study, zinc oxide nanoparticles was applied as a selective sorbent of miniaturized matrix solid phase dispersion combining with dispersive liquid-liquid microextraction (SPD-DLLME) for the simultaneous determination of thirteen n-alkanes (C8H18 to C20H42) in soil samples. The solid samples was directly blended with nanoparticles in SPD method and the eluent of SPD was used as the dispersive solvent of the followed DLLME for further purification and enrichment of the target compounds prior gas chromatography-flame ionization detector (GC-FID) analysis. Optimum condition were Under optimum condition, good linearity with correlation coefficients in the range 0.9991<r2<0.9995, suitable precision (1.9%<RSD<3.5%) and low detection limits (0.08 - 2.5 ngg-1) were achieved. The presented zinc oxide nanoparticles SPD-DLLME procedure combined the advantages of nanoparticles, SPD and DLLME, and could be applied for determination of n-alkanes in complicated soil samples and acceptable recoveries were obtained.

Key Words: n-Alkanes, Solid phase dispersion, Dispersive liquid-liquid microextraction,Gas chromatography, Soil samples.

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ZnO nanoparticle /carbon paste electrode as an electrochemical sensor for the simultaneous detection of dopamine , L-dopa and Ascorbic acid

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Abstract

Quantum dots (QDs) have received considerable attention due to their specific properties and applications in fields such as solar cells, light emitting diodes, and biological imaging. Zinc oxide (ZnO) is a typical II-VI group quantum dots and is used as an ingredient in cosmetics, and solar batteries. In recent years, the ZnO nanoparticles are attractive materials for a broad range of electronic, optical, and piezoelectric applications due to its direct band gap and excellent thermal, chemical, and piezoelectric properties [1]. An additional advantage emanates from chemical compatibility and stability of ZnO to create a high quality of interface in contact with other metal oxides[2]. Here we synthesized the ZnO nanoparticles from $Zn(NO_3)_2$ and NaOH in 2-propanol. ZnO nanoparticles was characterized by powder XRD, UV-visible absorption and Scanning electron microscopy (SEM). Then by incorporation of synthesized ZnO nanoparticles into carbon paste electrode, a new modified electrode was constructed for simultaneous determination of dopamine , L-dopa and Ascorbic acid using differential pulse voltammetry techniques (DPV). pH and different parameters such instrumental factors, were optimized. Under optimal conditions we take the advantage of the sensitive and rapid proposed method for simultaneous determination of dopamine , L-dopa and Ascorbic acid in aqueous solutions. The electrocatalytic peak current increases linearly with increase in their different concentrations . The ZnO NPs/ CPE enhanced peak current response and clearly resolved overlapped voltammetry signals for dopamine, L-dopa and Ascorbic acid.Also the carbon paste electrode was modified with ZnO and ZnO/polyglycine for further electrochemical investigation of them. The modified electrode shows good electrocatalytic activity towards the detection of dopamine , L-dopa and Ascorbic acid with a reduction in overpotential. References

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ZnO nanoparticles adsorbent for preconcentration and determination of methyl orange from water sample

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Using nonoparticles for separation and preconcentration in analytical chemistry may result in development of a new methodology that is faster, simpler and more precise than existig methodologies. The greatest advantage of nanoparticles is the large active surfase area for a given mass of particles, which causes them to be more reactive to some other molecules[1-3]. In this study, an easy and rapid method based ZnO nanoparticles solid phase extraction for pre-concentration and separation of trace amount of methyl orange (MO) from water samples prior to its determination using UV-Vis spectrophotometry was developed. The effects of various experimental parameters in extraction step were studied using Box-Behnken design. A five-factor, three-level Box-Behnken experimental design which combined the response surface modeling was used to optimize MO extraction. pH of solution, 4.3; mass of sorbent, 0.4 g; sample flow, 2 mL.min^{-1}; elution flow, 2 mL.min^{-1}; elution volume, 2 mL. The limit of detection was 0.3 µg/L^{-1}. the relative standard deviation (RSD%) was found to be 1.5%. The developed procedure was than used for the extraction of MO in water samples.

Keyword: Methyl orange; ZnO nanoparticle; Solid phase extraction; Response surface methodology; Water samples

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ZnO nanoparticles matrix solid-phase dispersion combined with dispersive liquid-liquid microextraction for the determination of BTX in water samples

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Abstract

Dispersive liquid-liquid microextraction (DLLME) is a preconcentration method, which used a ternary system of solvents. The advantages of this technique are simplicity, fast, low cost, good efficiency and high enrichment factor. DLLME is a green extraction technique, due to the consumption of organic solvent is reduced to the microliter [1-3]. In this study, zinc oxide nanoparticles was applied as a selective sorbent of miniaturized matrix solid phase dispersion combining with dispersive liquid-liquid microextraction (SPD-DLLME) for the simultaneous determination of benzene, toluene and xylenes (BTX) in water samples. The water samples was directly blended with nanoparticles in SPD method and the eluent of SPD was used as the dispersive solvent of the followed DLLME for further purification and enrichment of the target compounds prior gas chromatography-flame ionization detector (GC-FID) analysis. Optimum condition were dispersive solvent, acetone; extraction solvent, chloroform; volume of dispersive solvent, 1 ml; volume of extraction solvent, 200 µl; extraction time, 3 min and mass of adsorbent, 0.5 g. Under optimum condition, good linearity with correlation coefficients in the range $0.9992<r^2<0.9996$, suitable precision (2.2%<RSD<3.4%) and low detection limits (0.1 – 0.6 $\mu g l^{-1}$) were achieved. The presented zinc oxide nanoparticles SPD-DLLME procedure combined the advantages of nanoparticles, SPD and DLLME, and could be applied for determination of BTX in complicated water samples and acceptable recoveries were obtained.

Key Words: BTX, ZnO nanoparticles, Solid phase dispersion, Dispersive liquid-liquid microextraction, Gas chromatography, water samples.

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Abstract

Dispersive liquid-liquid microextraction (DLLME) is a preconcentration method, which used a ternary system of solvents. The advantages of this technique are simplicity, fast, low cost, good efficiency and high enrichment factor. DLLME is a green extraction technique, due to the consumption of organic solvent is reduced to the microliter [1-3]. In this study, zinc oxide nanoparticles was applied as a selective sorbent of miniaturized matrix solid phase dispersion combining with dispersive liquid-liquid microextraction (SPD-DLLME) for the simultaneous determination of benzene, toluene and xylenes (BTX) in water samples. The water samples was directly blended with nanoparticles in SPD method and the eluent of SPD was used as the dispersive solvent of the followed DLLME for further purification and enrichment of the target compounds prior gas chromatography-flame ionization detector (GC-FID) analysis. Optimum condition were dispersive solvent, acetone; extraction solvent, chloroform; volume of dispersive solvent, 1 ml; volume of extraction solvent, 200 μl; extraction time, 3 min and mass of adsorbent, 0.5 g. Under optimum condition, good linearity with correlation coefficients in the range $0.9992<r^2<0.9996$, suitable precision (2.2%<RSD<3.4%) and low detection limits (0.1 – 0.6 $\mu g l^{-1}$) were achieved. The presented zinc oxide nanoparticles SPD-DLLME procedure combined the advantages of nanoparticles, SPD and DLLME, and could be applied for determination of BTX in complicated water samples and acceptable recoveries were obtained.

Key Words: BTX, ZnO nanoparticles, Solid phase dispersion, Dispersive liquid-liquid microextraction, Gas chromatography, water samples.

References

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ZnO/CNTs nanocomposite as a sensor and a novel aminophenol derivative as a mediator for determination of hydroxylamine in environmental samples

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Abstract: Hydroxylamine, a derivative of ammonium, is known as a kind of reducing agent widely used in industry and pharmacy. It is one of the intermediate products of nitrogen-cycle and plays an important role in life science. For example, hydroxylamine is a well-known mutagen, which induces highly specific mutations with the nucleic acid cytosine. Modest levels of hydroxylamine can be toxic to humans, animals and plants, which have been known to cause both reversible and irreversible physiological changes. Moreover, some hydroxylamine derivatives also constitute a great part of anticancer drugs [1]. So, determination of this compound is very important. In this study, a new aminophenol-derivative compound was synthesized and used to construct a modified-ZnO/CNTs carbon paste electrode. The electro-oxidation of hydroxyamine at the surface of the modified electrode was studied using cyclic voltammetry, chronoamperometry, and electrochemical impedance spectroscopy. Using square wave voltammetric (SWV) method, the electrocatalytic oxidation peak current of hydroxylamine shows a linear calibration curve in the range 0.04 – 300 µM of hydroxylamine concentration. The detection limit (3σ) was determined as 0.02 µM by SWV method. The RSD% for 0.4 and 10.0 µM hydroxylamine was 1.8% and 1.9%, respectively. Finally, the suggestion sensor was successfully used for determination of hydroxylamine in some real samples such as water and waste water samples.

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