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Magnetic nanoparticle supported guanidine as a new sorbent for preconcentration of trace amounts of Pb^{2+}, Cd^{2+}, Zn^{2+}, Ni^{2+} and Cu^{2+} ions in water samples

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Abstract

Pre-concentration of very small amount of heavy metal ions is a key process for detection the inorganic pollutant in water samples.

There are many methods of preconcentration and separation, such as liquid liquid extraction (LLE),[1] ion-exchange techniques,[2] coprecipitation,[3] sorption on the various adsorbents, such as activated carbon,[4] Amberlite XAD resins,[5] and other sorbent.

Among them, SPEs are widely employed due to their simplicity, high concentration factor and low consumption of harmful organic solvents. In this work, magnetic nanoparticle (Fe_3O_4) has been chemically functionalized with guanidine groups. The resulting material has been characterized and employed as solid phase extractant for preconcentration of Pb^{2+}, Cu^{2+}, Zn^{2+} , Ni^{2+} and Cd^{2+} ions. The time and the optimum amount of the sorbent, pH and minimum amount of EDTA for elution was investigated. The pre-concentration ions were identified by flame atomic absorption spectrometry (FAAS). The enrichment factor of the proposed method was 50 for all cations and detection limits were found to be 26.7, 4.78, 7.6, 14.16 and 1.08 ng mL^{-1} for Pb^{2+}, Cu^{2+}, Zn^{2+} , Ni^{2+} and Cd^{2+} respectively. The maximum capacity of the adsorbent was found to be 25/2 (±1.7) mg, 15/70 (±0.7) mg, 16/20 (±0.9) mg , 12/61 (±1.6) mg and 13/60 (±0.8) mg of Pb^{2+}, Cu^{2+}, Zn^{2+} , Ni^{2+} and Cd^{2+} per gram of functionalized magnetic nanoparticle, respectively. In conclude, the proposed method is a facile and simple way for very fast pre-concentration of transition metal ions in water samples.

Keywords: Magnetic solid phase extraction, MNPs-guanidine, Pre-concentration, Water samples

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Investigation of azo dye modified carbon paste electrode for voltammetric detection of indigo carmine

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Abstract:

In recent years, substantial efforts have been devoted to the field of electrochemical methods based on modified electrodes, for their enhanced selectivity and sensitivity.[1] Among the wide range of electrodes, carbon paste electrode has permitted its easy modification with wide variety of modifiers. Chemically modified carbon paste electrodes have been extensively employed in various electrochemical investigations due to their many advantages such as simplicity, easy fabrication, facile renewable surface, biocompatibility, nontoxicity, stability in various solvent and relatively low-background characteristics.[2] To date, various materials have been used to make modified electrodes. Among the various mediators used for electrode modification, azo dyes attracted the attention of electrochemists as excellent electron transfer mediators.[3]

In this study, a new synthetic azo dye was used as modifier and electron mediator in electrochemical determination of indigo carmine using a modified carbon paste electrode. The experimental parameters like supporting electrolyte, the effect of pH of solution, the amount of modifier, the number of electrons involved in the rate determining step and diffusion coefficient were studied. The modified electrode showed high electrocatalytic activity toward indigo carmine in phosphate buffer solution (pH 2.4). The differential pulse voltammetric response of the modified electrode was linear against the concentration of indigo carmine in the range of 1 to 100 µM. The limit of detection was found to be 0.36 µM.

Keywords: Modified carbon paste electrode; Azo dye; Voltammetric determination; Indigo carmine

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Synthesis and characterization of Fe_3O_4@PPy-CNT nanocomposites as a novel sorbent, and its optimization by combining experimental design and artificial neural network for the determination of PAHs compounds in environmental samples

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Abstract:

Recently, magnetic solid phase extraction (MSPE) has been proposed from traditional solid phase extraction (SPE) and widely used due to its high recovery and ease of operation.[1-4] By taking advantage of both magnetic and non-magnetic adsorbent material, the MSPE method exhibits excellent adsorption efficiency and rapid separation from the matrix by an external magnetic field. The most important component of the MSPE technique is the adsorbent material, which dominates the sensitivity and selectivity of the method.[5-7]

The present study is the first report on the synthesis of multi-walled carbon nanotube oxide that is immobilized on the pyrrole magnetic nanoparticles. Application of this newly designed material in the MSPE of polycyclic aromatic hydrocarbons (PAHs), as model analytes, in the environmental samples was investigated.Determination of the analytes were performed with gas chromatography–mass spectrometry (GC–MS). The structure and morphology of Fe_3O_4@PPy-MWCNT were characterized by Fourier transform infrared (FT-IR) spectroscopy, scanning electron microscopy (SEM), thermo gravimetric analysis (TGA), and vibrating sample magnetometer (VSM). Many parameters have to be taken care of when developing an MSPE method because its performance is mainly affected by extraction time, sorbent amount, sample solution volume, and eluent type and volume. In this study, the best possible performance of MSPE has been achieved using experimental design and ANN modeling. The ANN used is a multilayer perceptron (MLP) trained with the standard error back propagation algorithm. Under the optimum extraction conditions (200 mL sample solution, extraction and desorption times of 3 and 1 min, respectively, 250 µL of 2-propanol as eluent, and 13 mg of adsorbent), a linear range between 0. 5 and 250 µg L^{-1} ($R^2 > 0.994$), preconcentration factors from 232 to 403 and limits of detection ranging from 0.1 to 0.3 µg L^{-1} were obtained. Relative standard deviations (RSDs) for intra- and inter-day precision were 3.3–5.1% and 3.7–5.6%, respectively. In addition, feasibility of the method was demonstrated with extraction and determination of PAHs from some real samples containing tap water, hookah water as well as soil samples with relative recovery of 85.4–106.8%. This new MSPE method provides several advantages, such as high extraction efficiency, minimum sorbent for extraction of the analytes from high sample volumes, convenient extraction procedure, and short analysis times.

Keywords: Magnetic solid phase extraction, ANN modeling, Gas chromatography–mass spectrometry.

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Extraction and separation of zirconium from hafnium using nano structured supramolecular solvent microextraction followed by flow injection inductively coupled plasma-optical emission spectrometry

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Abstract:

Recentely, a new mode of liquid-phase microextraction, namely supramolecular solvent microextraction (SUPRAS-ME), has been developed.[1,2] SUPRAS-ME is an extraction technique that consumes less organic solvents and provides very high pre-concentration factors. supra molecular solvents (SUPRASs) are nano-structured liquids generated from amphiphiles through a sequential self-assembly process occurring on two scales, molecular and nano.[3-5] The application of SUPRAS to analytical extraction was first proposed by Watanabe *et al.*.[6] Zirconium and hafnium have similar atomic radii, 1.45 and 1.44 A°, respectively, and similar configuration of the valence electrons $4d^2\ 5s^2$ and $5d^2\ 6s^2$ for Zr and Hf, respectively. Due to their similarity, complicated and expensive separation methods are required to remove the Hf impurity.

In this study, a simple versatile separation method using SUPRASs for extraction of trace levels of zirconium and hafnium is proposed. The extraction of analytes from aqueous samples was performed in the presence of cyanex 272 as chelating agent. After phase separation, the surfactant-rich phase was diluted with 50% (v/v) 2-propanol solution. Then, the enriched analytes in the surfactant-rich phase were determined by inductively coupled plasma-optical emission spectrometry (ICP-OES). Many parameters have to be taken care of when developing SUPRASs method because its performance is mainly affected by pH sample solution, amount of decanoic acid, ultrasonic time, salt effect and the complexation. In this study, the best possible performance of SUPRASs has been achieved using experimental design. Under the optimum conditions, the linear dynamic range were obtianed in the range of 0.5–100 $\mu g\,l^{-1}$ and 2–100 $\mu g\,l^{-1}$ with detection limits (DLs) of 0.21 and 0.62 $\mu g\,l^{-1}$ for Zr and Hf, respectively. The intraday ($n = 5$) and interday standard deviations were calculated by extracting the Zr and Hf from water through supramolecular solvent microextraction. Interday RSDs% 6.3% and intraday RSDs% 5.7% were obtained. Also the selectivity factor of 4.1 was obtained. Finally, the proposed method was successfully utilized for the separation and extraction of Zr and Hf in water samples.

Keywords: Supramolecular solvent microextraction, Zirconium, Hafnium, Inductively coupled plasma-optical emission spectrometry.

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Effect of pH on photocatalytic degradation of direct red 107 azo dye catalyzed by POM@TiO_2 nanocomposite under visible light

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Abstract:

In this study the photocatalytic activity of the $K_8[SiW_{11}O_{39}]$ encapsulated to TiO_2 (abbreviated as POM@TiO_2) nanocomposite was investigated for degradation of an aqueous solution direct red 107 azo dye under visible light.

Azo dyes are resistant to aerobic degradation; however, under anaerobic conditions, the azo linkage is reduced to generate aromatic amines that are colorless but can also be toxic and potentially carcinogenic. New developments in advanced oxidation processes (AOPs) are attractive in providing a promising and competitive solution for the abatement of numerous hazardous compounds in wastewater including Fenton or photo-assisted Fenton process, ozone and/or peroxide photolysis and semiconductor photocatalysis process.[1]

Anatase TiO_2 and polyoxometalate (POM) are two kinds of widely investigated photocatalytic materials. They have very similar photocatalytic processes owing to their similar electronic attributes.[2] The shortcoming of these two materials is that they have to be photoactivated by <380 nm light due to their high energy band gap, which makes use of solar light difficult since the solar light is mainly visible in a wavelength range of 400–800 nm. Recently, numerous efforts have been made in order to improve the photoefficiency of TiO_2 or POM on waste water under visible light.[3] It has been found that the fast photogenerated charge-pair (h^+, e^-) recombination on the surface of TiO_2 can be retarded by mixing a homogeneous POM with a TiO_2 photocatalytic system. This results in the enhanced quantum efficiency of TiO_2 as a consequence of synergistic effect between TiO_2 and POMs.[4] Because POMs are strong electron acceptors, they are able to enhance the rate of conduction band electron transfer by accepting electrons from donors into its empty d orbits. Accordingly, the fast recombination rate of h^+, e^- pairs is retarded.

In this article, the authors report a new route, the combination of sol-gel chemistry and solvothermal treatment, to fabricate POM@TiO_2 nanocomposite catalysts with a high photocatalytic activity.

The results of pH effect on the photocatalytic degradation of direct red 107 has been presented here. The effect of initial pH on degradation rate was ascertained and optimized pH values for maximum degradation was determined. The impact of pH on the degradation of a 50 $mg.L^{-1}$ solution of this azo dye under visible light by POM@TiO_2 nanocomposite was studied. The results are shown in Fig.1. Decreasing pH had a positive influence on the reactivity of the system.

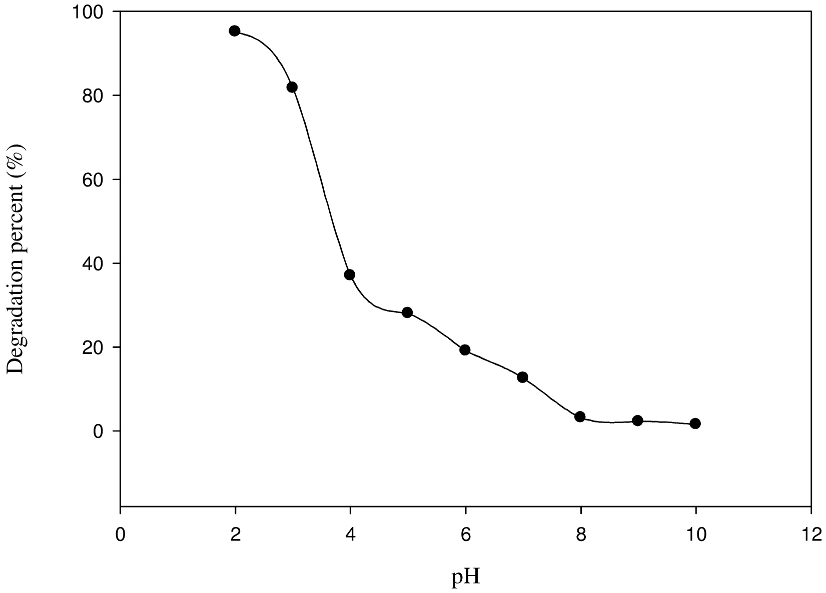

Fig.1. Influence of the initial pH on degradation percent of direct red 107 solution (50 mg.L^{-1}) in the presence of POM@TiO_2 (10 mg) under visible light

Keywords: Photocatalytic degradation, Nanocomposite, POM@TiO_2, Direct red 107, Azo dye, Visible light.

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Developing an electrochemical DNA biosensor on the basis of human interleukine-2 gene using curcumin as new electroactive indicator

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Abstract:

DNA hybridization biosensors, also known as genosensor.[1] In the past few years, the field of electrochemical biosensors has been speeding up dramatically.[2,3] Biosensors are devices combining a biological component with a detector component.There has been considerable interest in developing electrochemical DNA biosensors for the rapid and inexpensive diagnosis of genetic diseases, forensic analysis and other applications.[4,5] Electrochemical DNA hybridization detection methods are classified into major two direct and indirect protocols. In direct DNA hybridization detection strategy (label-free detection) oxidation of guanine moiety in DNA strands directly induces signal transduction. While indirect DNA hybridization detection method is based on incorporation of an electroactive indicator such as anticancer agents[6,7] organic dyes and metal complexes.[8,9]

Development of an electrochemical DNA biosensor based on, a pencil graphite electrode (PGE) as transducer and Curcmin (CU) as new electroactive label is described. The sensor relies on the immobilization of a 20-mer single stranded oligonucleotide probe on the electrode. The hybridization between the probe and its complementary and noncomplementary sequence as the target was studied by Didfferential Puls voltammetry (DPV) of CU accumulated on the PGE. In this approach the extent of hybridization is evaluated on the basis of the difference between DPV signals of CU accumulated on the probe-PGE and CU accumulated on the probe-target-PGE. Some experimental variables affecting the performance of the biosensor including: polishing of PGE, its electrochemical activation conditions (i.e., activation potential and activation time) and probe immobilization conditions on the electrodes (i.e., immobilization potential and time) were investigated and the optimum values of 1.80V and 300 s for PGE activation, and +0.5V and 400 s for the probe immobilization on the electrode were suggested and The diagnostic performance of the developed biosensor is described and the detection limit is found to be 12 pM.

Keywords: Hybridization Biosensors, Pencil Graphite Electrode, DNA, Curcumin

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The use of nanocomposite conductive polymer on the stainless-steel wire as the sorbent in electromembrane surrounded solid-phase microextraction

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Abstract:

In this study, for the first time, a new solid-phase microextraction (SPME) sorbent[1] based on electrochemical synthesis of nanocomposite conductive polymer on stainless steel wire is used in electromembrane surrounded solid phase microextraction (EM-SPME) methods.[2, 3] In order to evaluate the method performance, Cyproheptadine (CYP) and ketotifen (KET) were selected and extracted as model analytes. To increase the method selectivity and sample cleanup, the organic solvent was immobilized in the pores of hollow fiber (HF) wall and the SPME fiber was placed in its lumen which was initially filled with the aqueous acceptor phase. Then, 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. Impressive parameters such as composition of the organic liquid membrane, pH of donor and acceptor phases, applied voltage and extraction time were optimized and figures of merit of the method were investigated in human plasma and urine samples. Under the optimal conditions, extraction recoveries in the range of 26.8–46.9% and good detection limits (less than 1.1 ng mL^{-1}) were obtained. Linearity of the method was studied in the range of 0.3–200.0 ng mL^{-1} and 0.7–200.0 ng mL^{-1} for CYP and KET, respectively and the method offered coefficients of determination greater than 0.9946. Finally, the optimal conditions were applied for analysis of human urine and plasma samples and passable results were obtained.

Keywords: Electrochemical synthesis, Electromembrane surrounded solid phase microextraction, Gas chromatography, Nanocomposite conductive polymer, Biologycal samples, Stainless steel wire.

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Investigation of interaction between G-quadruplex and neutral red detected by TUCK-FMIN and fluorescence data

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Abstract:

Neutral Red (NR) is an efficient antioxidant agent[1] and anticancer drug of parent compound targeted to DNA[2]. NR belongs to drugs which are intercalating with double strand DNA. In this study spectroscopic fluorescence data recorded during titration experiments of quanine-rich strand DNA (G-quadruplex or G4) with NR and simultaneously analyzed by TUCK-FMIN.

The simultaneous analysis of several data matrices allowed the resolution of the system, removing most of the ambiguities related to factor analysis. Multi-way arrays represent a particularly rich source of information, because many signals are used to describe a single sample.

A new Tucker method (TUCK-FMIN) for the calculation of meaningful solutions in Tucker is proposed based on the minimization of an objective function defined directly from the constraints non-fulfilment. This method is extended of non-linear optimization techniques[3] to three-way. TUCK-FMIN represents a new approach for obtaining S, T and U rotation matrixes and its subsequent use for transforming the tucker3 loadings to meaningful solutions and obtaining sparse tucker core using non-linear optimization techniques.

Simulated and real fluorescence data were used to evaluate the feasibility of this method. Proposed method (TUCK-FMIN) almost is resolved and fit pure components from overlapped large size signals more better than usual three-way methods such as PARAFAC or closed-form Tucker3. From the results obtained, we report the evidence of the formation of two conformations NRG1 and NRG2 during experiments.

Keywords: TUCK-FMIN; G-quarduplex; Neutral Red; Non-linear optimization; Tucker3

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Response surface modeling of adsorptive removal of Rhodamine B from aqueous solutions using alkali treated tea waste

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Abstract:

Response surface modeling is an empirical statistical technique that uses quantitative data obtained from appropriately designed experiments to determine the optimum set of operational variables of the process.[1-2] In the present study, response surface method was employed to optimize adsorptive removal of Rhodamine B from aqueous solutions using alkali treated tea waste (ATW) as adsorbent. Treatment of the tea wastes was done by aqueous sodium hydroxide solution that breaks the covalent association between lignocellulose components, hydrolyzing hemicellulose and de-polymerising lignin.[3] The experiments were carried out based on a Bax-Behnken design with three input variables including adsorbent dose (0.02-0.06 g/L), contact time (30-120 minutes) and pH (3
t

^{2}) of 0.9973 and Fisher ratio of 206.6.
Moreover, a closely high adjusted R^2
model. Adequacy of the model was verified by lack of fit test and residual analysis. According to the model, values of the independent variables for optimization of RB removal efficiency using ATW were predicted as: adsorbent dose of 0.06 g/L, solution pH of 3 and contact time of 120 minutes. Experimental value of 89%, for dye adsorption onto ATW under the optimum conditions validated ability of the response surface modeling method for optimization of RB removal process by ATW. In this study, Langmuir and Freundlich adsorption isotherm models were also used to describe the experimental adsorption data. Fit of the Langmuir model for the process showed formation of a monolayer covering of the adsorbate at the outer surface of the adsorbent. In addition, kinetic studies showed that sorption of RB onto ATW follows the pseudo-second order model suggesting chemisorption.

Keywords: Tea waste, Rhodamine B, Response surface method, Bax-Behnken design**,** Kinetic study.

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Nitrate removal from aqueous solutions using amine functionalized Kit-6 magnetite mesoporous nanocomposite

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Abstract:

Nitrate pollution in groundwater has become a serious problem in most parts of the world during past decades due to discharge of domestic, industrial wastewater and increased usage of nitrogenous fertilizers.[1] High concentration of nitrate in drinking water would pose a serious threat to human health such as liver damage, cancers and methemoglobinemia.[2] Until now, many techniques based on reverse osmosis, adsorption and biological methods have been developed to remove nitrate from water.[3]

In this study, amine functionalized Kit-6 magnetite mesoporous nanocomposite was synthesized by chemical methods and used as a recoverable sorbent for removal of nitrate from aqueous solutions. The synthesized nanocomposites were characterized by XRD, BET, BJH and SEM methods. The effect of various experimental parameters on the efficiency of nitrate removal was studied using Taguchi experimental design method. The kinetic studies for nitrate adsorption showed rapid sorption dynamics by a second-order kinetic model with $R^2=0.9998$, suggesting chemisorption mechanism. Nitrate adsorption equilibrium data were fitted well to the Freundlich model. According to the results, Kit-6 magnetite nanocomposites can be used as a cheap and efficient adsorbent for removal of nitrate from aqueous solutions.

Keywords: Kit-6 magnetite mesoporous**,** Nanocomposite, Amino-functionalization, Nitrate

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Elimination of dye pollutants by zeolite beta containing titania

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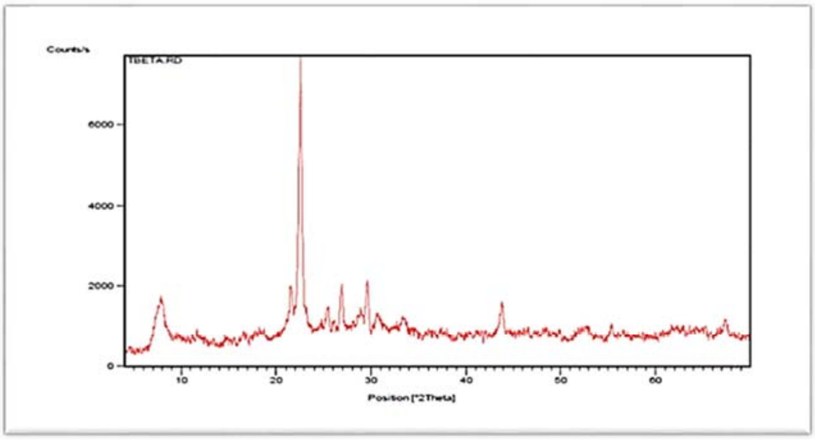
Abstract:

Zeolites one good supports for active species and also good adsorbents. In this work zeolite beta has been synthesized using hydrothermal methods.[1] It was establishes that a certain amount of the template (tetraethyl ammonium hydroxide) is necessary for successful synthesis of this zeolite.[2] TiO_2 was introduced into the structure of zeolite beta via ion exchange. Titanium isopropoxide was used as the raw material to be used for impregnation preparated of TiO_2-beta. Different amount of TiO_2 were introduced into the zeolite using different or by of amount of this compound.[3] A combination of different analytical methods including X-ray diffraction, BET, EDX and scanning electron microscopy were used to fully characterize the prepared catalysts. Our results show this material can eliminate moderate amount of methylen blue by adsorbing it. The work is continud to evalute its photocatalytic activities.



XRD Patterns of prepared Zeolite Beta

Keywords: Photocatalysis, Adsorbent, Beta zeolite, TiO_2, Methylene blue.

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Development of PES/Leonardie hybrid membrane and its application in removal of dye pollutants

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Abstract:

Polyethersulfone (PES)-based mixed matrix membranes (MMMs) with the incorporation of leonardite were prepared in this study and the resulting MMMs were characterized by XRD, SEM, and AFM. The influence of inorganic fillers were investigated in terms of hydrophilicity, permeation performance, membrane morphology and antifouling property.[1] Leonardite is a low-rank coal with large amounts of humic materials, mainly humic acids. Humic materials are complex organic molecules that contain different functional groups (carboxyl, hydroxyl and carbonyl).[2] Three different amounts of leonardite were introduced into the casting solutions to obtain the optimum value. The flux through a modified membrane was increased to a large extent compared to a virgin membrane. The contact angle measurements demonstrated that the hydrophilicity of modified membranes was enhanced by addition of leonardite in the casting solution. Besides, the antifouling properties of membranes were improved by changing the membrane surface from hydrophobic to hydrophilic after leonardite addition in the casting solution. The original porous PES membrane showed a very weak removal ability of dye from aqueous medium probably due to their poor wetting. The adsorption capacity of the PES/leonardite membrane was studied with the aqueous solutions of Malachite Green (MG) dye, and the efficiency of leonardite in PES casting solution was compared with that of the unmodified PES membrane.[3] According to the performance test, the membrane with 0.1 wt% nanoparticles indicated the highest MG removal (up to 99%).

Keywords: Leonardite, Polyethersulfone, Membrane, Malachite Green

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