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CHEMISTRY

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مجموعه مقالات
شیمی آلی

دومین کنفرانس دانشجویی
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رشت ، دانشگاه گیلان
۱۴ الی ۱۶ مهرماه ۹۴

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Silica Xerogel as a novel nano catalyst for the synthesis of 1,4-dihydropyrano[2,3-c]pyrazol-5-yl cyanides derivatives

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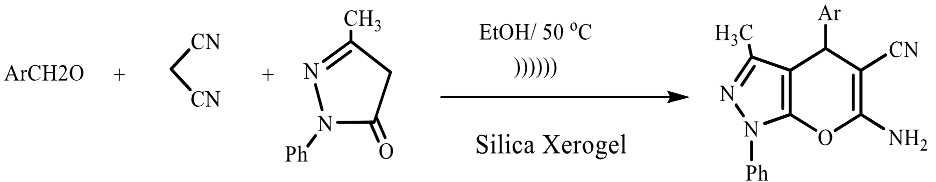
Abstract:

Sol–gel chemistry is an efficient tool to control the morphology and reactivity of solids. Efforts have been made to develop alternative gel synthesis procedures ,This method, that is based on hydrolysis and polycondensation of silicon or metal alkoxides, allows the preparation of materials with different morphological characteristics by varying experimental synthetic conditions. Inorganic materials possessing high surface area have been largely studied due to their possible application as sorbents, catalysts, sensors, molecular sieves.*porous silica xerogel* were synthesized with high surface area 624/3 m^2/g.

A facile and versatile procedure has been explored for the synthesis of 1,4-dihydropyrano[2,3-c]pyrazol-5-yl cyanides. This protocol employs the one-pot three-component condensation of aromatic aldehydes and malononitrile with 3-methyl-1-phenyl-2-pyrazolin-5-ones in ethanol under the catalytic effect of Silica Xerogel and ultrasonic-irradiation conditions. Simple manipulation, high reaction rates, improved yields, use of inexpensive and non-toxic catalyst, and also use of ethanol as a relatively environmentally benign solvent are the main advantages of this protocol.

Keywords: Silica Xerogel, 1,4-dihydropyrano[2,3-c]pyrazol-5-yl cyanides, ultrasonic-irradiation conditions.



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Solvent-free benzilic acid rearrangement using montmorillonite K10 and microwave irradiation

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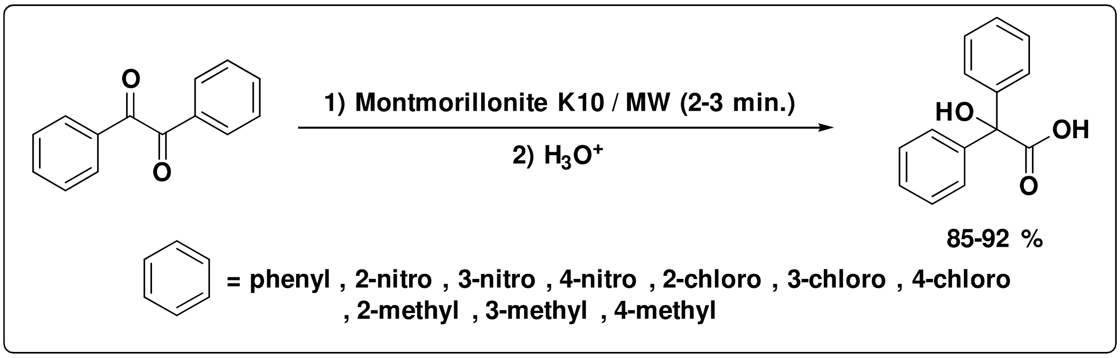
Abstract:

α-Diketones undergo a base catalyzed (such as KOH) rearrangement called benzil-benzilic acid rearrangement.[1] This reaction occurs in different conditions. For example, use of alkoxide ion instead of hydroxide or use of different metal catalysts such as cerium, rhodium and cupper salts reported.[2-5] First benzilic acid rearrangement in solvent free condition reported in 1990 and in 2001 by microwave irradiation.[6-7]

In this work, we report benzilic acid rearrangement in solvent free condition and grinding in presence of montmorillonite K10 as a *Lewis* acid catalyst and microwave irradiation (different powers) with yields 85-92% and reaction times 2-3 minutes.



Scheme 1. Benzil-Benzilic acid rearrangement with various phenyl groups

Keywords: Benzilic acid rearrangement, Solvent Free, Microwave, Montmorillonite.

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Aluminide coatings and role of modifying with silicon on high temperature corrosion : A review

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Abstract:

The components of a gas turbine operate in an aggressive environment where the temperature of service varies from ambient to near melting point of materials which introduce a variety of degradation on the components. Hot gases that are produced by burning the contaminated fuel in the combustion chamber will cause oxidation and corrosion on their passage. Sevsral approach have been employed to control hot corrosion of gas turbine components. This approaches include proper selection of alloys, application of coatings and etc. coating is preferred approach and numerous variants of high-temperature coatings that are in use today. Aluminide diffusion coatings are the most widely used to protect nickel- and cobalt-based superalloys for gas turbine applications.Diffusion coatings canwithstand hot corrosion evenmore effectively, if certain elements like platinum, chromiumor silicon are added.Besides aluminium and chromium, silicon is one of the most favourable protective scale-forming or scale-modifying elements.

In this paper we shall try to summarize the present basic understanding for Aluminide coatings and the role of silicon in this form of coatings.

Keywords: Turbine**,** Aluminide coatings, Silicon, High temperature corrosion.

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دانشگـاه گیلان - مهرماه ۹۴

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An efficient and convenient method for one-pot synthesis of β-acetamido ketones using Nano-cerium oxide as a catalyst

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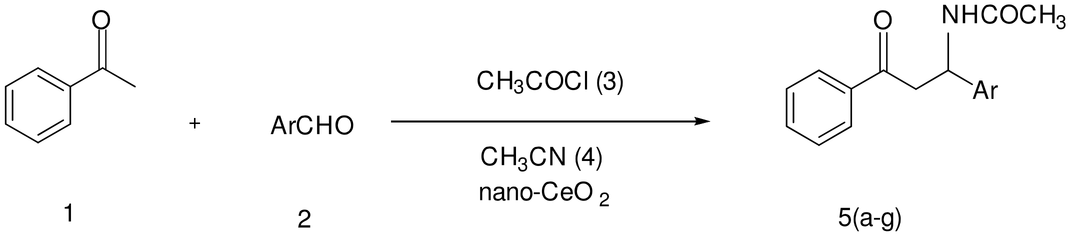
Abstract:

β-Acetamido ketones are versatile intermediates, in that their skeletons exist in a number of biologically or pharmacologically active compounds.[1] They could easily be converted to 1,3-amino alcohols,[2] which are utilized for the synthesis of several antibiotics.[3]

β-Acetamido ketones are usually prepared through acylation of β-aminoketones,[4] Michael addition to α,β-unsaturated ketones[5] or photoisomerisation of phthalimides.[6] Although some of these methodologies are efficient and provide the practical means for the synthesis of β-acetamido ketones, some of the reported methods suffer from drawbacks such as longer reaction times, tedious work up, higher temperatures, expensive catalysts, lower yields and requiring an inert atmosphere.

In our attempts to develop new catalyst systems, Herein, we describe the use of this reaction to prepare β-acetamido ketones in a mild and facile manner in the presence of a catalytic amount of nano nano cerium oxide in high yields (Scheme 1)



Scheme 1. synthesis of β-acetamido ketones

Keywords: One-pot; β-acetamido ketones; Recyclable catalyst; Nano cerium oxide

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Second Iranian Student Chemistry Conference

دانشگـاه گیلان – مهرماه ۹٤

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2nd Iranian Student Chemistry Conference
University of Guilan, Rasht, 6-8 Oct. 2015

Preparing a conductive paint using an emulsion polymerization of aniline

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Abstract:

Conductive paint similar to ordinary pigmented paint system, is a suspension of a solid in a binder.[1] However, this kind of paints can be produced in other ways too, for example as what we did, they can be preparde by polymerization of a conducting polymer such as, polypyrol, polythiophene, polyacetylen, polyaniline, etc. Among these polymers polyaniline is a very promising conducting material due to its facile synthesis, low cost, remarkable environmental stability, relative high conductivity, and unique redox behavior.[2] These paints have different applications, they can be used in marine industuries as anti corrosion, electromagnetic (EMI) shielding devices, radar absorbing instruments, and, so many other things. conductive paints are applicable on any kind of substrates by spraying, dipping, printing, etc.[3,4] In this project a PANI conducting solution is made in a way which aniline is polymerized in an emulsion of HCL solution 3M and also xylene (weakly polar organic solvent). This polymerization is carried out in the presence of a functionalized protonic acid which is dodecylbenzenesulfonic acid (DBSA). DBSA acts simultaneously as a surfactant and as a protonating agent that makes the solution stay stable for the final PANI. The polymerization mechanism of ANI oxidized with APS is shown in Figure 1. For finding the optimum condition we designed 27 experiments in which the ratio of DBSA/ANI, oxidant/ANI (both: 0.4, 1, 1.5) and the temperature (0, -5, -10) were changing factors. For preparing the optimum solution ANI 2.5ml (0.027 mol) was added into the micellar solution containing different quantities of DBSA dissolved in 50 ml of two different solvents xylene and HCl solution 3M seperately. The solution was mixed and stirred and maintained in a cooling bath at selected temperatures. The APS solution which was made of different extents of oxidant (ammonium peroxidisulfate) dissolved in 50 ml HCl solution 1M and was added to the first solution dropwise in 30 minutes. The process took 4 hours. The best stability and the best viscosity occurred in xylene and HCl solution 3M respectively (Figure 2). The opimum condition is selected as followed: DBSA/ANI=1.5, oxidant/ANI=1, and, the temperature -5 degrees of centigrade. The best resistivity (57 ohm-meter) occurred in this condition which makes us reagard this condition as optimum condition the surface tension and viscosity of the optimum paint solution is 4.45 N/M and 61 centi poise respectively (Table1).

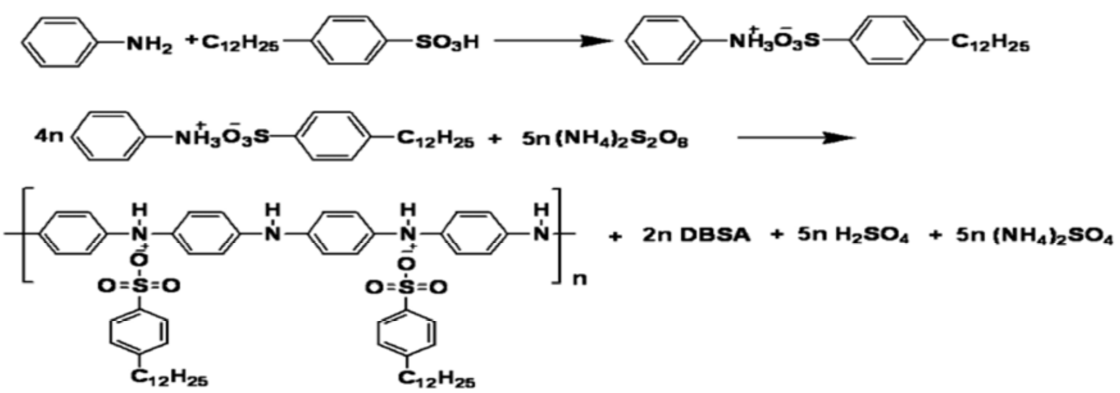

Figure 1. The polymerization mechanism of ANI oxidized with APS

(a) (b)

Figure 2. The paint solution made in xylene (a) and the HCl solution 3M in (b) after two weeks.

Table 1. The best result reached under these conditions

DBSA/aniline	Oxidant/aniline	temp(c)	R (ohm-meter)	Viscosity(CP)	Surface tension (N/M)
1.5	1	0	112	34	2.45
1.5	1	-5	57	61	4.45
1.5	1	-10	68	61	4.45

Keywords: Conductive paint, Emulsion polymerization, Conducting polymers, Aniline.

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دانشگاه گیلان - مهرماه ٩٤

University of Guilan . Rasht . 6-8 october 2015

A green method for synthesis of 2-arylquinoline-3-carbamide in deep eutectic solvent

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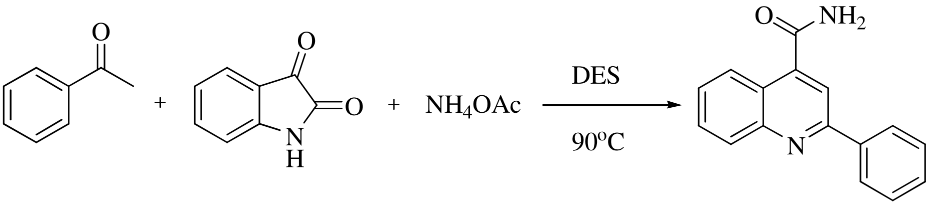
Abstract:

Solvents play an essential role in chemical processes serving to put reactants into contact by dissolution and also affecting rates of the reactions. Solvents are also used in the later stages of a reaction for extraction and purification of the products. Organic solvents are extensively used in organic synthesis and for this reason they are a matter of much concern due to characteristics such as high flammability, volatility, hazardness, and toxicity. Thus the search for environmentally benign substitutes for organic solvents has recently gained more attention in view of the increasing importance of green chemistry[1].

Room-temperature ionic liquids (ILs) are potential green alternatives to organic solvents for extractions, chemical reactions and biotransformations. Another new versatile reaction medium has emerged, known as deep eutectic solvents (DESs).[2] Abbott et al. pioneered the development of these solvents which are low-melting liquids derived from the mixture of a solid for correspondence organic salt and a suitable organic complexant, typically a hydrogen-bond donating species such as a polyol or urea derivatives.[3]

The purpose of this study is develop an efficient and convenient approach for the construction of the 2-arylquinoline-3-carbamide system. Thus, the reaction of isatin **1**, acetophenone derivatives **2**, and ammonium acetate **3** in the presence of Mannitol /citric acid /NaCl 70:20:10 as a highly efficient deep eutectic solvent at 90 °C in good yields is reported (Scheme 1). The reaction completion was monitored by TLC. At the end of the reaction about 5 mL water was added to reaction mixture. The products were recrystallized from ethanol to give pure compounds.



Scheme 1. Synthesis of 2-arylquinoline-3-carbamide in presence of DES.

Keywords: Deep eutectic solvent, Low melting mixtures, Ionic liquid.

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دانشگـاه گیلان – مهرماه ۹٤

University of Guilan . Rasht . 6-8 october 2015

Synthesis of isocyanoacetamide and its application in designing of novel multicomponent reactions

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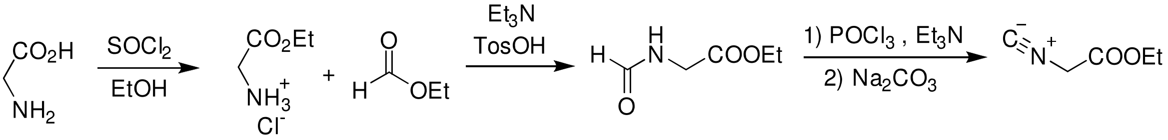
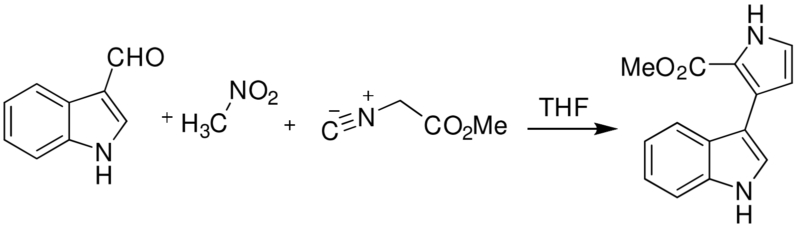
Abstract:

Multicomponent reactins (MCRs) have gained considerable interest for the synthesis of biologically active compounds. Designing the the new multicomponent and sequential reactions using readily available starting materials has gained widespread recognition among the organic chemists. This approach could be categorized based on combination of two or more distinct reactions to form molecular diversity and complexity in natural and biologically relevant systems. Herein, we report the synthesis of α-isocyanoacetamide (Scheme 1).



In the structure of this compound there are three active sites which could be used for designing of reactions. a) active methylene compound b) the methyl ester group c) existence of isocyanide moiety. The isocyanide contains formally a divalent carbon and has consequently a pronounced ability to undergo α-addition with electrophiles and nucleophiles at the same carbon. In continuation of our work, for the designing reaction based on nitro olefins (Scheme 2).



Three-component reaction of 3-formyl indole, nitroalkanes and isocyanoacetamide was investigated. This approach could be used for the synthesis some indole alkaloids. The products have nitro and ester group and an imine which could be used for further reactions. The details will be explained in the conference.[1-3]

Keywords: Ethyl isocyano acetate, Cyclization reactins, Domino reactions, Nitroolefines.

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Modification of ZSM-5 nanocatalyst with metal oxide and evaluation of its performance in the conversion of methanol to gasoline (MTG)

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Abstract:

Acid ZSM-5 catalyst is highly selective to gasoline cut and can improve the selectivity and quality of this product due to its unique properties.[1] In this study, the effect of molybdenum on the conversion and catalyst lifetime was investigated by preventing the coke formation during the reaction.[2,3] The conversion of methanol to gasoline (MTG) was performed in a fixed-bed reactor under atmospheric pressure, at 400 °C and WHSV of 4 h^{-1} over HZSM-5, and MoO-HZSM-5 catalysts. Synthesized catalysts were studied by XRD, SEM and BET methods. Aromatic yield was increased and catalyst lifetime was improved using MoO-HZSM-5.

In 1970s, Mobile researchers found that methanol could be converted to gasoline over mesoporous selective zeolites.Conversion of hydrocarbons from methanol using ZSM-5 has been investigated by Chang and Silvestri.[4] Doping metal oxides such as CuO into the HZSM-5 framework usually results in the generation of additional acidic sites, and thereby modifies the acidity of the parental material. It facilitates the dehydrating activity of the catalyst and also helps to form higher molecular weight hydrocarbons in the gasoline range. The objective of this study was to compare CuO/ZnO and CuO supported HZSM-5 and HZSM-5 under the same operating conditions such as temperature, pressure and flow rate, and to investigate the conversion and yield of hydrocarbons.

HZSM-5 (Si/Al = 15), the catalyst used for MTG. Amounts of MoO 2 wt.% was doped on the HZSM-5 and the effect of molybdenum oxide loading on HZSM-5 was studied. It was found that on increasing the MoO content, the yield and conversion increased. All these catalysts were prepared by standard impregnation technique.

Figure 1 shows the XRD pattern of MoO catalyst and a comparison with the starting HZSM-5 catalyst. XRD pattern of these catalyst were mostly amorphous (no sharp peaks of MoO) indicating the structure of zeolites remains intact after the different treatment procedures. However, the obtained intensities differ depending upon preparation methods and on MoO content. The higher intensity was observed for the starting zeolite (HZSM-5). The decrease in the intensities may be due to higher absorption coefficient of MoO.

Figure 2 shows a comparison of products distribution obtained with various metal oxide doped HZSM-5 catalysts at 400 °C and atmospheric pressure. Preliminary experiments carried out with the HZSM-5 catalyst at the same temperature, pressure and flow rate also showed that aromatic compounds are dominant in the products.

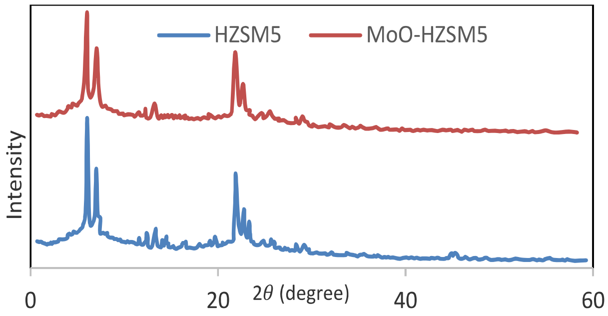

Figure 1. XRD patterns of the ZSM-5 zeolites

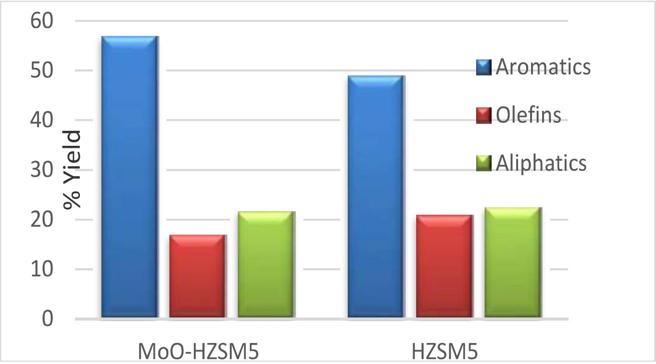

Figure 2. Product distribution of hydrocarbons

Keywords: Acid catalyst, ZSM-5, Metal oxide, Methanol to gasoline (MTG).

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Synthesis of dinuclear Al_2O_3 / ZrO_2 nano particles by complexing Sol-Gel method

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Abstract:

In this study, Alumina/ Zirconia nano particles are synthesized with complexing sol - gel process with 5,10,15,20% Zirconium nitrate. Synthesized nanoparticles were characterized by using X-ray diffraction (XRD), transmission electron microscopy(TEM), and Fourier transform infrared (FTIR) spectrometer. Some samples were calcined at different temperatures 200, 400, 700, 1100° C. The observed X-ray diffraction pattern within temperature range of 200 to 1100°C revealed that with the increase in temperature t-ZrO_2 phase shifted to m-ZrO_2. Moreover the morphological characteristic using FTIR, in corroboration with XRD, confirms the crystallization of corundum (α- Al_2O_3) as one of the alumina phase and cubic phase of zirconia at 1100°C. Alumina-zirconia crystals are average size using this method 1.14-4.37 nm.

Keywords: Nano composite, Sol-gel, Nano alumina, Nano zirconia.

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دانشگـاه گیلان – مهرماه ۹۴

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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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DABCO complex supported on Fe_3O_4 magneticnanoparticles: A new catalyst for synthesis of 1-*H*-pyrazolo[1,2-*b*]phthalazine-5,10-diones

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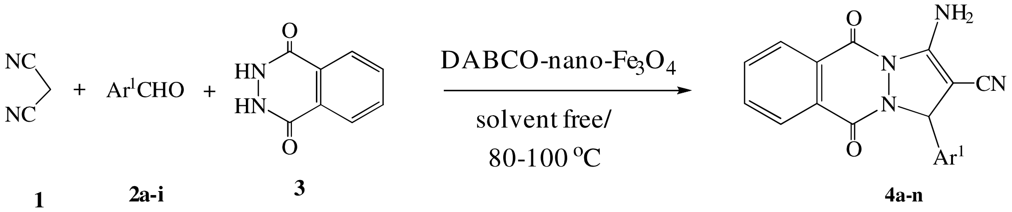
Abstract:

Heterogeneous catalysts have emerged as economically viable because of their high potent of recyclability.[1-4] Inparticular, nano-particles as heterogeneous catalysts have received considerable attraction in recent years owing to their interesting structures and outstanding catalytic activities. On the other hand, 1,4-diazabicyclo[2.2.2]octane (DABCO) is a medium-hindered tertiary amine with cage-like structure, which used as an in expensive, eco-friendly, highly reactive and non-toxic ligand or catalyst for various organic transformations. In this paper, the catalytic activity of DABCO complex supported on Fe_3O_4 magnetic nanoparticles has been explored in the synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives *via* a three-component coupling reaction between aromatic aldehydes, malononitrile, and phthalhydrazides. High yield, low reaction times, non-toxicity and recyclability of the catalyst, and easy work-up are the main merits of this protocol.

Keywords: DABCO, Fe_3O_4 magnetic nanoparticles, 1-*H*-pyrazolo[1,2-*b*]phthalazine-5,10-diones.



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[PVPH]HSO_4: Introduction of a new modified polymeric catalyst for the synthesis of 2*H*-indazolo[2,1-*b*]phthalazine-triones

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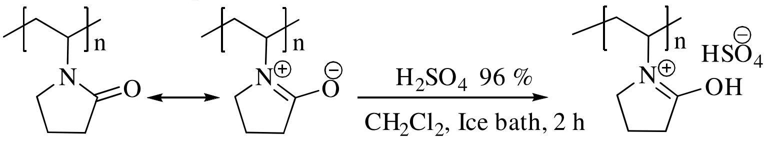
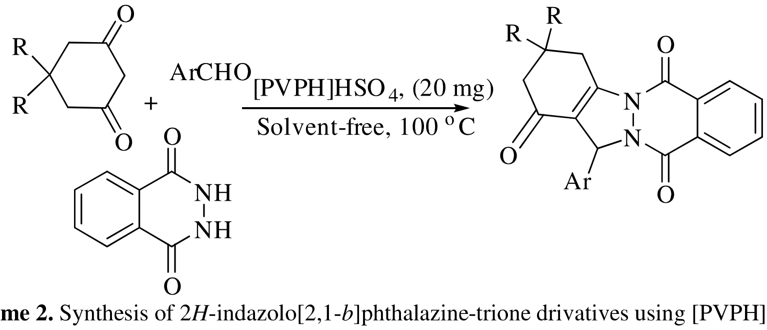
Abstract:

2*H*-indazolo[2,1-*b*]phthalazine-trione derivatives have occupied an important place in drug research because of their various biological and pharmacological activities such as an antimicrobial and anticancer activities.[1,2] In this article and in continuation of our previous report on the applicability of polymeric based reagents in organic reactions,[3] a one-pot efficient method for the synthesis of the above mentioned compounds *via* a multi-component reaction of aldehydes, phethalhydrazide and 1,3 -cyclic diketones using polyvinyl pyrrolidonium hydrogen sulfate ([PVPH]HSO_4) (Scheme 1) as a heterogeneous and reusable catalyst is reported. The most important advantages of this method are: simplicity, mild conditions, easy work-up, short reaction times, high yields of the products and reusability of the catalyst (Scheme 2).



Scheme 1. Preparation of [PVPH]HSO_4



Scheme 2. Synthesis of 2*H*-indazolo[2,1-*b*]phthalazine-trione drivatives using [PVPH]HSO_4

Keywords: ([PVPH]HSO_4), Poly(vinylpyrrolidone), 2*H*-indazolo[2,1-*b*]phthalazine-trione.

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