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Sulfamic acid heterogenized on functionalized magnetic Fe₃O₄ nanoparticles with diaminoglyoxime as a green, efficient and reusable catalyst for one-pot synthesis of substituted pyrroles in aqueous phase

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Surface functionalization of magnetic nanoparticles is an elegant way to bridge the gap between heterogeneous and homogeneous catalysis. We have conveniently loaded sulfonic acid groups on amino-functionalized Fe_3O_4 nanoparticles affording sulfamic acid-functionalized magnetic Fe_3O_4 nanoparticles (MNPs/DAG-SO₃H) as an active and stable magnetically separable acidic nanocatalyst, which was characterized using X-ray diffraction, Fourier transform infrared and energy-dispersive X-ray spectroscopies, scanning and transmission electron microscopies, vibrating sample magnetometry and elemental analysis. The catalytic activity of MNPs/DAG-SO₃H was probed through one-pot synthesis of *N*-substituted pyrroles from γ -diketones and primary amines in aqueous phase at room temperature. The heterogeneous catalyst could be recovered easily by applying an external magnet device and reused many times without significant loss of its catalytic activity. Copyright © 2014 John Wiley & Sons, Ltd.

Keywords: magnetic nanoparticle; nanocatalyst; sulfamic acid; magnetic recovery; pyrroles

Introduction

The design of novel magnetically separable systems has attracted a great deal of attention in recent years as an interesting alternative for improving the efficient separation of heterogeneous (nano)catalysts from solution upon reaction completion by applying a simple magnet, providing improved reusability of the designed systems as well as enhanced properties depending on the selected approach.^[1] Following the successful applications of magnetic nanoparticles in the design of new catalytic supports,^[2] there is now a growing interest in the further development of this type of catalyst support. So, magnetic catalytic systems have been introduced as very powerful and clean recoverable supports for a variety of catalytic reactions. The use of nanoparticles as catalysts has some problems. One of them is their highly active surface which leads to the agglomeration of the catalyst particles. Coating the catalyst surface with an organic or an inorganic shell is an appropriate strategy for preventing this agglomeration.^[3–7]

The immobilization of homogeneous catalysts on solid supports opens up new avenues for design and engineering of new and interesting catalysts. These structures are easily separable from the reaction mixture, allowing the recovery of the solid and eventually its reuse. The highest level of evolution in the surface immobilization strategy is the covalent attachment of an organic compound or ligand to the solid surface.^[8] The incorporation of organic moieties with pendant attached chains, either on the external part or on the internal surface, is a useful method for modifying the physical and chemical properties of natural or synthesized materials. It can improve

the ability of new multifunctional materials – considered as 'inorganic–organic hybrids' – to act in a variety of academic or technological activities.^[9] An obvious advantage of such hybrids is the favourable combination of both organic and inorganic properties in one nanomaterial.^[9]

Acid catalysts are used in a variety of organic transformations, including aldol condensations, hydrolyses, acylations, nucleophilic additions, etc. However, several drawbacks such as separation problems, reactor corrosion, waste neutralization and inability to be reused are associated with the reactions catalysed by soluble liquid acids.^[10] Consequently, there has been considerable interest in the development of stable, reusable and highly active solid acids^[11-13] as environmentally benign replacements for their homogeneous counterparts. During the last few years, sulfamic acid (NH₂SO₃H), a dry non-hygroscopic, non-volatile and odourless solid, has been considered as an efficient heterogeneous catalyst substitute for conventional acidic catalysts.^[14–17] Ketal formation or acetalization,^[18] esterification,^[14] nitrile formation of β -ketoesters^[21] are among the reactions carried out in the presence of sulfamic acid.

Heterocyclic small molecules play an important role in the search for new therapeutic and drug candidates. Pyrroles are an important

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class of heterocyclic compounds and are structural units found in a vast array of natural products, synthetic materials and bioactive molecules, such as heme, vitamin B12 and cytochromes.^[22] One of the most common approaches to pyrrole synthesis is the Paal-Knorr reaction in which 1,4-dicarbonyl compounds are converted to pyrroles in the presence of primary amines. Many catalysts have been used for this conversion, such as montmorillonite, KSF clay,^[23] microwave irradiation,^[24,25] Bi(NO₃)₃·5H₂O,^[26] Sc(OTf)₃,^[27] ToISO₃H,^[28] layered zirconium phosphate and zirconium sulfophenylphosphonate,^[29] titanium,^[30] TiCl₄/Et₃N^[31] and silica sulfuric acid.^[32] Although these protocols represent considerable progress, recovery and reuse of catalysts are difficult. Achieving a green aspect requires the use of a more effective recovery method for these types of catalysts. In this paper, we report immobilization of sulfamic acid groups on synthesized magnetic Fe₃O₄/diaminoglyoxime (DAG) nanoparticles to achieve sulfamic acid-functionalized magnetic nanoparticles (MNPs/DAG-SO₃H) as an active and stable magnetically separable acidic nanocatalyst, for the one-pot synthesis of N-substituted pyrroles at room temperature in aqueous phase.

Experimental

Preparation of Magnetic Fe₃O₄ Nanoparticles (MNPs)

Naked Fe₃O₄ nanoparticles were prepared by chemical coprecipitation of Fe³⁺ and Fe²⁺ ions with a molar ratio of 2:1. Typically, FeCl₃·6H₂O (5.838 g, 0.0216 mol) and FeCl₂·4H₂O (2.147 g, 0.0108 mol) were dissolved in 100 ml of deionized water at 85 °C under nitrogen atmosphere and with vigorous mechanical stirring (500 rpm). Then, 10 ml of 25% NH₄OH was quickly injected into the reaction mixture in one portion. The addition of the base to the Fe²⁺/Fe³⁺ salt solution resulted in the immediate formation of a black precipitate of MNPs. The reaction continued for another 25 min and the mixture was cooled to room temperature. Subsequently, the resultant ultrafine magnetic particles were treated using magnetic separation and washed several times with deionized water.

Preparation of Fe₃O₄/DAG

The obtained MNP powder (500 mg) was dispersed in 50 ml of ethanol–water (1:1 v/v) solution using sonication for 20 min, and then DAG (250 mg) was added to the mixture. After mechanical agitation at 40 °C for 5 h, the suspended substance was separated with centrifugation. The settled product was re-dispersed in ethanol using sonication and then was isolated with magnetic decantation five times. The precipitated product (DAG-MNPs) was dried at room temperature under vacuum.

Preparation of Fe₃O₄/DAG-SO₃H

The DAG-MNPs (500 mg) were dispersed in CH_2CI_2 (50 ml) using an ultrasonic bath for 30 min. Subsequently, chlorosulfonic acid (0.8 ml in 10 ml of CH_2CI_2) was added dropwise over a period of 30 min and the mixture was stirred for 2 h at room temperature. Hydrogen chloride gas was evolved immediately from the reaction vessel. Then, the final product was separated using magnetic decantation and washed twice with dry CH_2CI_2 , EtOH and CH_2CI_2 successively to remove the unattached substrates.

General Procedure for Synthesis of Pyrroles in Presence of $Fe_3O_4/DAG-SO_3H$

To a solution of amine (1 mmol) and hexan-2,5-dione (1 mmol) in EtOH–H₂O (2 ml, 1:1) at room temperature was added catalyst (0.02 g). The mixture was stirred at this temperature for the required period of time. The reaction was monitored using TLC (3:1 *n*-hexane–acetone). After completion of the reaction, 10 ml of ethanol was added, and the catalyst was removed using an external magnet. Further purification was achieved with TLC using *n*-hexane–acetone (70:30) as the solvent system to afford the pure pyrroles.

Results and Discussion

Scheme 1 presents the preparation steps for fabricating MNPs/DAG-SO₃H: naked MNPs are prepared through a chemical co-precipitation method, and subsequently are coated with DAG to achieve amino-functionalized MNPs. Ultimately, the reaction of amino groups with chlorosulfonic acid leads to MNPs/DAG-SO₃H. Elemental analysis shows that a S loading of 0.7 mmol g⁻¹ is obtained. Various amounts of SO₃H were immobilized on the surface of Fe₃O₄/DAG and, in each case, the extent of loaded SO₃H was measured through a back titration process. Briefly, 0.2 g of catalyst was dispersed in 2.0 ml of distilled water and then to this solution was added NaOH (1 N) until the pH of the solution changed to basic condition. Eventually, the excess amount of base was titrated with 1 M acid titrasol HCI. Finally, the calculations show that for 20 mg of catalyst, 0.75 mmol H⁺ is released.

Figure 1 shows the Fourier transform infrared (FT-IR) spectrum for MNPs/DAG-SO₃H. The FT-IR bands at low wavenumbers (\leq 700 cm⁻¹) come from vibrations of Fe–O bonds of iron oxide, which for the bulk Fe₃O₄ samples appear at 570 cm⁻¹ but for Fe₃O₄ nanoparticles are blue-shifted at 624 and 572 cm⁻¹, due to the size reduction.^[33,34] The broad bands at 3170 and 3450 cm⁻¹ are assigned to the N–H and O–H stretching vibrations and the band at 1440 cm⁻¹ is assigned to the C=N stretching vibration.^[35] Reaction of MNPs/DAG with chlorosulfonic acid produces MNPs/DAG-



Scheme 1. Preparation steps for fabricating sulfamic acid-functionalized magnetic Fe_3O_4 nanoparticles.



Figure 1. FT-IR spectrum of Fe₃O₄/DAG-SO₃H.

SO₃H in which the presence of sulfonyl moiety is indicated by the 1221 and 1125 cm⁻¹ bands in the FT-IR spectrum.

The X-ray diffraction (XRD) pattern of Fe₃O₄/DAG-SO₃H is shown in Fig. 2. Diffraction peaks at $2\theta = 30.2^{\circ}$, 35.6° , 42.3° , 53.6° , 57.1° and 62.6°, corresponding to (1 1 1), (2 2 0), (3 1 1), (4 0 0), (4 4 0) and (5 1 1), are readily recognized from the XRD pattern, which can be indexed to a cubic phase of Fe₃O₄. This result indicates that the



Figure 2. XRD pattern of Fe₃O₄/DAG-SO₃H.



Direct Mag = 40000x AMT Camera System

AMT Camera System HV = 100 kV





Figure 4. EDX data for the catalyst.

crystal structure of Fe₃O₄ MNPs is not changed after modification with DAG-SO₃H.

Transmission electron microscopy (TEM) images provide more accurate information on the particle size and morphology of MNPs and Fe₃O₄/DAG-SO₃H (Fig. 3). The naked Fe₃O₄ nanoparticles show slight agglomeration, which can be related to the absence of surfactants and the lack of any repulsive force between the MNPs. For both the pristine and SO₃H-functionalized nanoparticles, the average diameter of the core is around 8 nm with an approximate spherical shape, which is in accord with the XRD pattern. There is no detectable outer shell within the sensitivity limit of TEM. Energydispersive X-ray spectroscopy (EDX) data for the obtained nanomaterials (Fig. 4) also indicate the presence of the expected elements in the structure of the catalyst, namely iron, oxygen, sulfur and carbon.

Figure 5 shows scanning electron microscopy (SEM) images of the synthesized DAG-SO₃H-loaded magnetite nanoparticles. It is confirmed that the catalyst is made up of uniform nanometre-sized particles.

The magnetic properties of the synthesized nanocatalyst (Fe₃O₄/DAG-SO₃H) were characterized using vibrating sam-

> ple magnetometry (VSM). Figure 6 shows a typical magnetization curve. It has been reported that M_s (saturation magnetization) of bare Fe₃O₄ nanoparticles is 73.7 emu g^{-1} , and M_s of the nanocatalyst prepared in this study is ~60.03 emu g $^{-1}$. The decrease of the saturation magnetization suggests the presence of sulfamic acid groups on the surface of the magnetic supports. Even with this reduction in the saturation magnetization, the catalyst can still be efficiently and easily separated from solution using an external magnetic force.

> In continuation of our interest in environmentally benign chemical processes,[37-40] after characterization of the prepared nanocatalyst, we investigated the use of Fe₃O₄/DAG-SO₃H as an active and stable



Figure 5. SEM images of Fe₃O₄/DAG-SO₃H.



Figure 6. Room temperature magnetization curve of Fe₃O₄/DAG-SO₃H.



Scheme 2. Fe₃O₄/DAG-SO₃H-catalysed synthesis of *N*-substituted pyrroles.

magnetically separable acidic nanocatalyst for the one-pot synthesis of *N*-substituted pyrroles by Paal–Knorr condensation at room temperature (Scheme 2). Initially, we decided to explore the role of catalyst in the synthesis of 2,5dimethyl-*N*-benzylpyrrole as a model compound (Scheme 2) at room temperature using various solvents, namely CH_2CI_2 , CCI_4 , CH_3CN , H_2O and EtOH (Table 1). The results show that EtOH– H_2O is a better solvent (product yield of 98%) than the other solvents tested (Table 1, entry 6).

To investigate the effect of catalyst concentration, systematic studies were carried out in the presence of various amounts of the catalyst (0, 5, 10, 20 and 50 mg) in H₂O–EtOH, affording 2,5-dimethyl-*N*-benzylpyrrole in 10, 50, 75, 98 and 96% isolated yields, respectively (Table 2). Thus, the best yield is obtained in the presence of just 20 mg of Fe₃O₄/DAG-SO₃H (Table 2, entry 4). The use of a greater amount of catalyst (50 mg) does not improve the result to an appreciable extent (Table 2, entry 5).

These results prompted us to investigate the scope and generality of this new protocol for various

amines (aliphatic and aromatic) under optimized conditions. In the same manner, a variety of amines were coupled with hexan-2,5-dione in the presence of a catalytic amount of $Fe_3O_4/DAG-SO_3H$ at room temperature in order to give the corresponding pyrroles in good to excellent yields (Table 3).

Table 1. Effects of solvent on the synthesis of 2,5-dimethyl- <i>N</i> -benzylpyrrole at room temperature ^a				
Entry	Solvent	Time (min)	Yield (%) ^b	
1	CH_2CI_2	20	80	
2	CCl ₄	20	70	
3	CH₃CN	15	85	
4	EtOH	10	90	
5	H ₂ O	30	60	
6	EtOH-H ₂ O (1:1)	10	98	
7	Solvent-free	20	75	

^aReaction conditions: hexan-2,5-dione (1 mmol), benzylamine (1 mmol), catalyst (50 mg), solvent (2 ml).
^bIsolated yield.

Table 2.	Effects of	catalyst	concentration	on	the	synthesis	of	2,5-
dimethyl-	N-benzylpy	rrole at re	oom temperatu	rea				

Entry	Amount of catalyst (mg)	Time (min)	Yield (%) ^b
1	0	60	10
2	5	60	50
3	10	60	75
4	20	10	98
5	50	10	96

^aReaction conditions: hexan-2,5-dione (1 mmol), benzylamine (1 mmol), H_2O -EtOH (2 ml, 1: 1).

^blsolated yield.

Table 3. $Fe_3O_4/DAG-SO_3H$ -catalyzed synthesis of pyrroles ^[32] under aqueous conditions ^a				
	H_3C H_3C H_3C H_3C H_3C $H_2O/EtoH$ $H_2O/EtoH$ $r.t.$	$\begin{array}{c} -SO_{3}H \\ \hline (1:1) \end{array} \xrightarrow{H_{3}C} \begin{pmatrix} N \\ N \\ \dot{R} \end{pmatrix} CH_{3}$		
Entry	R	Time (min)	Yield (%)	
1	Ph-CH ₂	10	98	
2	p-MeO-Ph-CH ₂	10	96	
3	NH ₂ -CH ₂ -Ph-CH ₂	15	96	
4	Ph	20	95	
5	<i>p</i> -Cl–Ph	25	85	
6	<i>p</i> -MeO–Ph	20	95	
7	<i>p</i> -Me–Ph	18	90	
8	<i>m</i> -CF ₃ –Ph	50	80	
9	<i>p</i> -COOH–Ph	90	75	
10	<i>p</i> -NH ₂ –Ph	90	70	
11	<i>m</i> -NH ₂ –Ph	120	75	
12	o-NH ₂ –Ph	120	60	
13	1-Naphthyl	25	90	
14	2-Pyridyl	120	70	
15	3-Indole-CH ₂ -CH ₂	15	96	
16	Cyclohexyl	5	98	
17	NH ₂ -CH ₂ -CH ₂	5	98	
18	NH ₂ -CH ₂ -CH ₂ -N-CH ₂ -CH ₂	5	98	
19	$(NH_2-CH_2-CH_2-N)_2-CH_2-CH_2$	5	98	
^a Conditions: amine (1 mmol), hexan-2,5-dione (1 mmol), catalyst (20 mg), H ₂ O–EtOH (2 ml, 1:1), room temperature. Products were characterized from their physical properties, comparison with authentic samples and by spectroscopic methods.				

Less basic aromatic amines require only slightly more time than more basic amino compounds, and both lead to high yields of the pyrrole products. As evident from Table 3, aromatic amines with electron-donating groups (entries 6, 7 and 11) or electron-withdrawing groups (entries 8 and 9) are both effective in the Paal–Knorr reaction. Heterocyclic amines (Table 3, entries 14 and 15) exhibit the same behaviour as



Scheme 3. A plausible mechanism for synthesis of pyrroles catalyzed by Fe_3O_4/DAG-SO_3H.

aromatic and aliphatic amines. The reaction conditions are also applicable to diamino or triamino substrates, in giving bipyrrole compounds (Table 3, entries 3, 11, 12, 17 and 18) or tripyrrole compounds (Table 3, entry 19) in excellent yields.

A plausible mechanism is shown in Scheme 3. The carbonyl groups are first activated by the catalyst to attach amine. Cyclization and dehydration of the intermediate give the final product and the catalyst is released for the next catalytic cycle.

The reusability of catalysts is one of the most important benefits and makes them useful for commercial applications. As shown in Fig. 7, the catalyst can be recycled up to seven runs without any significant loss of activity. In addition, one of the attractive features of this novel catalyst system is the rapid (within 15 s) and efficient separation of the catalyst



Figure 7. Recycling of the $Fe_3O_4/DAG-SO_3H$ catalyst for the synthesis of 2,5-dimethyl-N-benzylpyrrole.



Figure 8. Images showing that $Fe_3O_4/DAG-SO_3H$ can be separated using an applied magnetic field. A reaction mixture in the absence (left) or presence (right) of a magnetic field.

Table 4.	Reaction times and yields for previously published methods,
with benz	zylamine as substrate

Conditions	Reaction time	Yield (%)	Ref.
Fe ₃ O ₄ /DAG-SO ₃ H	10 min	98	This work
Montmorillonite, KSF	10 h	95	[41]
l ₂	0.5 h	92	[41]
Bi(NO ₃) ₃ ·5H ₂ O	10 h	95	[43]
Microwave	0.5 min	90	[42]
Sc(OTf) ₃	30 min	94	[44]
α -Zr(KPO ₄) ₂	2 h	78	[45]

(100%) using an appropriate external magnet (Fig. 8), which minimizes the loss of catalyst during separation.

We compared the current protocol with previously published methods for the synthesis of *N*-substituted pyrroles with benzylamine. The comparison is summarized in Table 4. These results, clearly demonstrate that $Fe_3O_4/DAG-SO_3H$ is a good catalyst for the preparation of *N*-alkyl- and *N*-aryl-2,5-dimethylpyrroles.

Conclusions

The preparation and characterization of sulfonic acid group-loaded amino-functionalized Fe₃O₄ nanoparticles (MNPs/DAG-SO₃H) as an active and stable magnetically separable acidic nanocatalyst were described. The prepared catalyst was characterized using XRD, FT-IR and EDX spectroscopies, SEM, TEM, VSM and elemental analysis. The catalytic activity of Fe₃O₄/DAG-SO₃H was probed through one-pot synthesis of *N*-substituted pyrroles from γ -diketones and primary amines in aqueous phase at room temperature. The heterogeneous catalyst could be recovered easily by applying an external magnet device and reused many times without significant loss of its catalytic activity.

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