

Dicationic ionic liquid grafted with silica-coated nano-Fe₃O₄ as a novel and efficient catalyst for the preparation of uracil-containing heterocycles

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Abstract

Bisulfate-functionalized dicationic ionic liquid grafted with silica-coated nano- Fe_3O_4 (nano- $[Fe_3O_4@SiO_2@BDIL]$ or NFSBDI) was synthesized as a novel nanomagnetic material, and its structure was confirmed by EDS, FE-SEM, FT-IR, VSM, XRD and TGA. Afterward, it was utilized as an efficient, magnetically recyclable and dual-functional catalyst for the preparation of an important class of uracil-containing heterocycles, namely pyrimido[4,5-*b*]quinolines. The one-pot multicomponent reaction of dimedone, arylaldehydes and 6-amino-1,3-dimethyluracil in the presence of NFSBDI under solvent-free conditions afforded pyrimido[4,5-*b*]quinolines in high yields and short times.

Graphical abstract



Keywords Bisulfate-functionalized dicationic ionic liquid grafted with silica-coated nano-Fe₃O₄ · Nano-[Fe₃O₄@SiO₂@BDIL] (NFSBDI) · Nanomagnetic catalyst · Uracil-containing heterocycle · Pyrimido[4,5-b]quinoline

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Introduction

Ionic liquids (ILs) are of great significance in different scientific and industrial fields [1-8]; the benefits of these salts have been mentioned in the literature [1-8]. They have been especially utilized as efficient homogeneous catalysts in organic synthesis [5-8]. Heterogenization of ionic liquids through grafting with solid supports (nano or bulk materials) can convert them to catalysts which have simultaneously the advantages of ILs and heterogeneous catalysts. Handling, isolating, recycling and reusing heterogeneous catalysts are easier than homogeneous ones [9-16].

Currently, preparation and application of nanomagnetic materials in scientific, medicinal and industrial fields are the theme of growing interest [17–27]. Nano-Fe₃O₄ is the best candidate to use as core for preparation of nanomagnetic materials, since it has several unique characteristics, consisting of thermal and chemical stability, non-toxicity, low cost, simple isolating and recycling, superparamagnetic property, large specific surface area and capacity to graft with various functional groups (for different uses) [21–27]. Nanomagnetic materials have been applied as efficacious catalysts to promote organic transformations [21–27].

Solvent-free conditions and multicomponent reactions are practical, useful, efficient and well-known techniques which have been used for production of a wide range of organic, medical and complex materials; the advantages of these techniques have been mentioned in the literature [28–33].

Nitrogen-bearing heterocycles have been found in the structures of many bioactive and pharmaceutical compounds. Uracil, pyrimidine, quinoline and pyrimidoquinoline are nitrogen-containing heterocycles which are present in the structures of numerous biological compounds, e.g., anticancer [34, 35], antimicrobial [36], antifolate [37], anti-allergic [38], anti-inflammatory [39], analgesic [39], DNA intercalating carrier [40], antiviral [41], antifungal [42] and antagonists [43] agents.

An important class of uracil-containing heterocycles is pyrimido[4,5-b]quinolines which could synthesized through the one-pot multicomponent reaction of dimedone, arylaldehydes and 6-amino-1,3-dimethyluracil. Some catalysts have been reported for this transformation [44-52]; nevertheless, it has not been comprehensively studied in the literature. Hence, there is still an increasing interest to introduce new catalysts for the reaction of dimedone, arylaldehydes and 6-amino-1,3-dimethyluracil.

Considering the benefits of grafting ILs with solid supports, nanomagnetic materials, solvent-free conditions, multicomponent reactions and pyrimido[4,5-b]quinolines, we have reported here synthesis of a novel nanomagnetic material, namely bisulfate-functionalized dicationic ionic liquid grafted with silica-coated nano-Fe₃O₄ (nano-[Fe₃O₄@SiO₂@BDIL] or NFSBDI), and its characterization using EDS (energy-dispersive X-ray spectroscopy), FE-SEM (field emission scanning electron microscopy), FT-IR, VSM (vibrating sample magnetometry), XRD (X-ray diffraction), TG (thermal gravimetric) and DTG (differential thermal gravimetric). Then, we have reported the solvent-free preparation of pyrimido[4,5-b]quinolines through the one-pot multicomponent reaction of dimedone, arylaldehydes and 6-amino-1,3-dimethyluracil using NFSBDI as an efficient, magnetically recyclable and dual-functional catalyst.

Experimental

Materials and instruments

The utilized starting materials and solvents were bought from Merck or Fluka Chemical Companies. Observation of the reactions progress was done by thin-layer chromatography (TLC) using silica gel SIL G/UV 254. For measurement of melting points, a Buchi B-545 instrument was utilized. FT-IR spectra were run using a Shimadzu IR-60 apparatus. ¹H and ¹³C NMR spectra were recorded by a Bruker Avance DPX FT-NMR spectrometer. EDS was performed by a SAMx-EDS (France system) device. A field emission scanning electron microscope (model MIRA3TES-CAN-XMU) was used to determine sizes and morphologies of the particles. VSM analysis was carried out by a MDK (Meghnatis Daghigh Kavir) apparatus (Iran) at room temperature. XRD analysis was performed using Cu K α radiation (λ = 1.5408, model: X'Pert PRO MPD, PANalytical, the Netherlands). TGA was performed by Bahr STA 504 (Germany) apparatus, at 25–600 °C, with temperature increase rate of 10 °C min⁻¹ in an argon atmosphere.

Preparation of nano-[Fe₃O₄@SiO₂@BDIL] (NFSBDI) (Scheme 1)

Nano-Fe₃O₄ was synthesized using the reported procedure [53, 54]. A mixture of nano-Fe₃O₄ (0.75 g), Si(OEt)₄ (2.25 mL), EtOH (60 ml), H₂O (15 mL) and ammonia (2.4 mL) was stirred and refluxed for 12 h to furnish intermediate I (which was isolated from the reaction mixture by centrifuging and decanting). Afterward, a mixture of (3-chloropropyl)trimethoxysilane (1.38 mL, 7.5 mmol) and intermediate I in dry toluene (60 mL) was stirred under flowing nitrogen gas and reflux conditions for 12 h; the resulting mixture was centrifuged, decanted, washed by dry toluene (twice) and Et₂O and dried under vacuum at 90 °C to give II [54, 55]. *N*,*N*,*N*',*N*'-tetramethylethylenediamine (1.12 mL, 7.5 mmol) was added to intermediate II in toluene (45 mL), and the resulting mixture was stirred in reflux conditions for 12 h.



Scheme 1 The synthesis of NFSBDI

The solid was separated from the solution by centrifuging and decanting, washed by toluene and dried under vacuum at 90 °C to produce III. Finally, H_2SO_4 (0.42 mL, 7.5 mmol) was added gradually to III at room temperature and stirred for 5 h at the same temperature and 1 h at 60 °C; the residue was washed by distilled water and dried under vacuum at 90 °C to afford NFSBDI.

General procedure for the synthesis of pyrimido[4,5-b]quinolines

A mixture of dimedone (0.140 g, 1 mmol), aldehyde (1 mmol), 6-amino-1,3-dimethyluracil (0.155 g, 1 mmol) and NFSBDI (0.048 g) was stirred vigorously by a small rod at 120 °C. When the reactants were consumed (as observed by TLC), the reaction mixture was cooled to room temperature, ethyl acetate (40 mL) was added, stirred and refluxed for 2 min, and the catalyst was isolated magnetically (washed by ethyl acetate and dried). Ethyl acetate of the remained solution was evaporated, and the residue was recrystallized from EtOH (95%) to give the pure product.

Note: Selected spectral data and original spectra of the synthesized pyrimido[4,5*b*]quinolines have been given in supplementary material.

Results and discussion

Characterization of the nanomagnetic material

The structure of nano-[Fe₃O₄@SiO₂@BDIL] (NFSBDI) was verified by EDS, FE-SEM, FT-IR, VSM, XRD and TGA.

Iron, silicon, oxygen, carbon, nitrogen, sulfur and chlorine elements are present in the structure of NFSBDI; the peaks pertaining to these elements were observed in the EDS spectrum (Fig. 1), and no extra peak related to impurity was seen. Morphologies and sizes of the particles were determined by FE-SEM (Fig. 1). As it can be seen in Fig. 1, the particles had different crystalline and amorphous forms, and their sizes were below 100 nm (i.e., NFSBDI is a nanomaterial); the interpretations are in agreement with the literature [10].

The FT-IR spectrum of the novel bisulfate-functionalized dicationic ionic liquid grafted with silica-coated nano-Fe₃O₄ is shown in Fig. 2, and the respective data are summarized in Table 1. All expected peaks relevant to the bonds and functional groups of NFSBDI were seen in the spectrum (Fig. 2 and Table 1); the obtained data are in accordance with the literature [12, 54]. In another study to prove successful functionalization of the ionic liquid on Fe₃O₄@SiO₂, the FT-IR data of nano-[Fe₃O₄@SiO₂@BDIL] were compared with those reported for Fe₃O₄@SiO₂ [56]. Two additional peaks (2925 and 2700–3734 cm⁻¹) were observed in the FT-IR spectrum of NFSBDI when compared with the Fe₃O₄@SiO₂ spectrum; these additional peaks are related to the grafted ionic liquid. It should be mentioned that in the FT-IR spectrum of Fe₃O₄@SiO₂, there was a peak at about 3100–3680 cm⁻¹ (corresponded to the OH groups on silica surface); however, in the NFSBDI spectrum, a



Fig. 1 The EDS spectrum and FE-SEM micrographs of NFSBDI



Fig. 2 The FT-IR spectrum of nano-[Fe₃O₄@SiO₂@BDIL]

Peak (cm ⁻¹)	Bond or functional group				
466	Rocking of Si–O				
~648	Stretching of S-O				
590	Fe–O				
805	Symmetric stretching vibrations of Si-O-Si				
1089	Asymmetric stretching vibrations of Si-O-Si				
1633	Bending of O–H groups of H ₂ O adsorbed on silica surface				
2925	Stretching vibration of C-H				
2700–3734	Stretching of OH group of bisulfate and OH groups on silica surface				

peak at 2700–3734 cm^{-1} was observed (corresponded to the OH group of bisulfate and the OH groups on silica surface).

VSM experiment was utilized for magnetic measurement of the nanomagnetic catalyst (Fig. 3). Considering the VSM results, saturation magnetization of NFS-BDI was 13.5 emu g⁻¹. Saturation magnetization of the used nano-Fe₃O₄ for the synthesis of NFSBDI has been reported 52 emu g⁻¹ in the literature [54]. (We prepared nano-Fe₃O₄ according to the reported procedure in the paper cited in reference [54].) The silica layer coated on Fe₃O₄ nanoparticles and bisulfate-functionalized dicationic ionic liquid grafted with the silica surface have caused the decreasing in saturation magnetization of Fe₃O₄ nanoparticles. Nevertheless, proper magnetization was observed for NFSBDI, and it could be readily separated from the reaction mixture by an external magnet; Fig. 4 shows this subject.

The XRD diagram of NFSBDI showed two broad peaks (at $2\theta \approx 16.6^{\circ}-32.0^{\circ}$ and $41.9^{\circ}-45.9^{\circ}$) and some sharp diffraction lines (at $2\theta \approx 9.9^{\circ}$, 11.4° , 18.9° , 21.3° , 22.0° , 25.2° , 26.5° , 28.8° , 30.5° , 32.2° , 35.9° , 37.6° , 38.6° , 39.4° , 46.4° ,



Fig. 3 The VSM diagram of NFSBDI

Table 1 The FT-IR results of

NFSBDI

Fig. 4 The images of an aqueous solution of NFSBDI before (a) and after (b) applying magnetic field



47.9°, 52.5°, 54.2°, 57.5°, 59.5°, 61.1°, 63.2°, 67.6°, 71.9° and 74.8°) (Fig. 5). The observed peak at $16.6^{\circ}-32.0^{\circ}$ belongs to SiO_2 [10, 12], and the peaks appeared at 30.5° , 35.9° , 46.4° , 57.5° , 63.2° , 71.9° and 74.8° are related to Fe₃O₄ [54]. The other peaks can be attributed to the grafted ionic liquid with silica-coated nano-Fe₃O₄. According to the XRD diagram, the nanoparticles were in amorphous and crystalline forms (the literature verified this conclusion [10, 12]); this topic was also observed in the FE-SEM micrographs (Fig. 1).



Fig. 5 The XRD diagram of the catalyst

For studying thermal stability of the nanomaterial, TG and DTG were used (Fig. 6). According to the obtained curves, weight losses of NFSBDI have occurred in two stages. The weight loss below 180 °C can be relevant to vaporization of the absorbed solvents (or water) on the silica surface. The second weight loss (180–600 °C) can be pertinent to decomposing the bisulfate-functionalized dicationic ionic liquid grafted with silica-coated nano-Fe₃O₄ (organic part of the catalyst) and the condensation of the silanol groups. These discussions were verified by the literature [10].

Catalytic performance of NFSBDI for the preparation of pyrimido[4,5-b]quinolines

Catalytic performance of the novel bisulfate-functionalized dicationic ionic liquid grafted with silica-coated nano-Fe₃O₄ (nano-[Fe₃O₄@SiO₂@BDIL] or NFSBDI) was examined for the synthesis of an important kind of uracil-containing heterocycles, i.e., pyrimido[4,5-*b*]quinolines. For this purpose, the reaction of dimedone (1 mmol), 4-chlorobenzaldehyde (1 mmol) and 6-amino-1,3-dimethyluracil (1 mmol) was chosen as a model (Scheme 2), and influence of the nanocatalyst amount and temperature on it was studied; the results are summed up in Table 2. As Table 2 illustrates, the best catalyst amount and temperature were 0.048 g and 120 °C, correspondingly (Table 2, entry 2). Increasing the amount of NFSBDI and the temperature did not improve the results (Table 2, entries 3 and 5).

After successful application of NFSBDI to promote the model reaction, to assess its effectiveness and scope for the production of diverse derivatives of pyrimido[4,5*b*]quinolines, dimedone was reacted with miscellaneous arylaldehydes and 6-amino-1,3-dimethyluracil; the attained reaction times and yields are shown in Table 3. As Table 3 indicates, all aldehydes, consisting of benzaldehyde and arylaldehydes



Fig. 6 The TG and DTG curves of NFSBDI



Scheme 2 The model reaction to obtain optimal conditions

Table 2Influence of the catalystamount and temperature on thereaction of dimedone (1 mmol)with 4-chlorobenzaldehyde(1 mmol) and 6-amino-1,3-dimethyluracil (1 mmol)	Entry	The catalyst amount (g)	Temp. (°C)	Time (min)	Yield (%)
	1 2	0.040 0.048	120 120	40 15	91 96 ^a
	3	0.060	120	15	96 ^a
	4	0.048	110	30	85
	5	0.048	125	15	96 ^a

^aThe reaction was completed

Table 3 The preparation of pyrimido[4,5-b]quinolines using NFSBDI



Compd. No	Ar	Time (min)	Yield ^a (%)	M.p. (°C), Found (reported)
1	C_6H_5	15	94	271–273 (267–270) [52]
2	$3-BrC_6H_4$	15	92	280–282 (281–283) [50]
3	$4-BrC_6H_4$	15	90	234–236 (227–230) [50]
4	$2-ClC_6H_4$	15	88	324–326 (> 300) [5 0]
5	$4-ClC_6H_4$	15	96	293–295 (292–294) [47]
6	2,4-Cl ₂ C ₆ H ₃	15	85	319–321 (> 300) [47]
7	4-Cl,3-O2NC6H3	15	91	296-297 (this work)
8	$3-O_2NC_6H_4$	30	87	289–291 (287–290) [50]
9	4-MeOC ₆ H ₄	15	94	304–306 (> 300) [45]
10	$4-\text{MeC}_6\text{H}_4$	15	93	307–309 (> 300) [47]
11	$4-HOC_6H_4$	30	81	310–312 (307–310) [46]

^aIsolated yield

having halogen, electron-attracting and electron-releasing substituents on ortho-, meta- or para-positions, afforded the corresponding pyrimido[4,5-*b*]quinolines in high yields and short times. Having the acquired results in mind, NFSBDI was a highly efficacious and general catalyst for the synthesis of pyrimido[4,5-*b*] quinolines.

NFSBDI can be a dual-functional catalyst, because its anion (bisulfate) possess one acidic (hydrogen) and one weak basic (negative oxygen) sites; so, it can be particularly applied for performing reactions which need acidic and basic catalysts simultaneously, e.g., the synthesis of pyrimido[4,5-*b*]quinolines. These roles of NFSBDI are obviously displayed in the mechanism (Scheme 3). As it can be seen in Scheme 3, the acidic hydrogen of bisulfate has activated the carbonyl groups in steps 1, 3 and 5 (for accepting nucleophilic attacks) and catalyzed these steps; furthermore, it has catalyzed steps 2 and 6 through assisting removal of H₂O molecule. In the other hand, the basic site of bisulfate has activated the nucleophiles to catalyze steps 1, 3 and 5; it also helps remove H₂O in step 6. NFSBDI has also catalyzed tautomerization (steps 4 and 7). The proposed mechanism is in accordance with the literature [46, 49]. Dual functionality of the catalysts containing acidic and basic sites and the roles of the sites have been mentioned in the literature [8, 57].

To study recyclability of NFSBDI, the reaction of dimedone, 4-chlorobenzaldehyde and 6-amino-1,3-dimethyluracil was carried out, and the catalyst was recycled according to the mentioned procedure in the experimental section. NFSBDI was successfully reused for three times; nonetheless, the reaction times slightly increased, and the yields slightly decreased during reusing (Fig. 7). The slight deactivation can be attributed to wasting the catalyst during the recycling.

In another study, the reaction conditions and the results of NFSBDI for the synthesis of products 1, 5 and 9 were compared with those of some reported catalysts (Table 4). As the data of Table 3 illustrate, NFSBDI was superior than the other catalysts in terms of the reaction time and yield. Besides, our synthesis was performed in solvent-free conditions.

Conclusions

In summary, we have introduced a novel bisulfate-functionalized dicationic ionic liquid grafted with silica-coated nano-Fe₃O₄ as a nanomagnetic catalyst for the production of pyrimido[4,5-*b*]quinoline derivatives. The advantages of this work include efficacy and wide scope of the catalyst, the preparation of the products in high yields and short times, utilization of a few amount of the catalyst in the reaction, application of solvent-free and multicomponent reaction techniques, magnetic recyclability of the catalyst, no need to column chromatography for purifying the products, easy purification of the products by recrystallization and good agreement with green chemistry principles.



Scheme 3 The proposed reaction mechanism



Fig. 7 The results of recyclability of NFSBDI

Table 4	Comparing the	reaction	conditions	and t	the results	of	NFSBDI	with	those	of the	reported	cata-
lysts for	the synthesis of	products	1, 5 and 9									

Catalyst	Conditions	Time (min) of product 1/5/9	Yield (%) of product 1/5/9	References	
NFSBDI	Solvent-free, 120 °C	15/15/15	94/96/94	This work	
p-Toluenesulfonic acid	H ₂ O, 90 °C	180/150/180	85/89/84	[44]	
InCl ₃	H ₂ O, reflux	- ^a /60/60	- ^a /91/89	[45]	
SBA-15/PrN(CH ₂ PO ₃ H ₂) ₂	Solvent-free, 100 °C	20/15/- ^a	89/85/- ^a	[46]	
Catalyst-free	[bmim]Br ^b , 95 °C	- ^a /210/210	- ^a /90/95	[47]	
Nano-Fe ₃ O ₄ @SiO ₂ -SO ₃ H	H ₂ O, 70 °C	30/25/40	92/92/86	[48]	
[H ₂ -DABCO][ClO ₄] ₂ ^c	H ₂ O, 75 °C	30/25/45	90/95/93	[49]	
[dsim]HSO4 ^d	EtOH, 70 °C	- ^a /15/25	- ^a /91/85	[50]	
[TSSECM] ^e	Solvent-free, 125 °C	40/40/30	93/92/95	[51]	
Fe ₃ O ₄ NPs-cell ^f	H ₂ O, reflux	120/120/120	89/91/87	[52]	

^aIn the work, this product has not been synthesized

^b1-*n*-Butyl-3-methylimidazolium bromide

^c1,4-Diazabicyclo[2.2.2]octane-1,4-diium perchlorate

^d1,3-Disulfonic acid imidazolium hydrogen sulfate

^eN,N,N',N'-tetramethyl-N-(silica-n-propyl)-N'-sulfonic acid-ethylenediaminium chloride/mesylate

^fFe₃O₄ nanoparticles supported on cellulose

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