A Brønsted Acid Ionic Liquid Immobilized on Fe₃O₄@SiO₂ Nanoparticles as an Efficient and Reusable Solid Acid Catalyst for the Synthesis of 2,3-Dihydroquinazolin-4(1*H*)-ones

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Abstract—In the present study, an efficient and magnetically recoverable Brønsted acid ionic liquid, 1-methyl-3-[3-(triethoxysilyl)propyl]-1*H*-imidazol-3-ium hydrogen sulfate, immobilized on the surface of $Fe_3O_4@SiO_2$ magnetic nanoparticles ($Fe_3O_4@SiO_2$ -ImHSO_4) has been used for a high-yield synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones through the condensation of anthranilamide with aldehydes and ketones in EtOH at room temperature or under reflux. The significant features of the present protocol are short reaction times, high yields of products, ecofriendly reaction conditions, simple work-up, and reusability of the catalyst. The catalyst can be simply magnetically recovered and reused at least five times without considerable loss of its catalytic activity.

Keywords: magnetic nanoparticles, supported ionic liquids, 2,3-dihydroquinazolin-4(1*H*)-ones, anthranilamide, cyclocondensation

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Ionic liquids (ILs) are considered as one of the most important alternatives to organic solvents in the development of green chemistry. They offer such advantages as low melting points and vapor pressures, low coordinating ability, and excellent thermal and chemical stability and can replace usual organic solvents in a wide range of chemical processes [1, 2]. Imidazolium-based ILs showed a high catalytic activity and received significant interest as an eco-friendly catalyst in organic transformations [3, 4]. However, these ILs suffer from high viscosity and, as a result, mass transfer of molecules in the ILs is limited; furthermore, because of their homogeneous nature, ILs are more difficult to separate from the final reaction product [5, 6]. One of the main ways to overcome these disadvantages is to immobilize ILs on solid supports such as polymers [7], amorphous silica [8], mesoporous silica [9], carbon nanotubes [10], or magnetic nanoparticles (MNPs) [11].

At present, MNPs have attracted special attention as prominent catalyst supports due to their vast surface area-to-volume ratio, low cost, facile synthesis, easy functionalization, and simple separation be means of an external magnet [12]. Due to these advantages, in recent years, magnetic nanoparticles and especially ferrite ones have found wide application for immobilization of homogenous ionic liquids. Nanomagnetic-supported ILs combine the benefits of ILs and heterogeneous catalysts, such as high designability, high "solubility" of catalytic site, ease of handling, separation, and recycling [13–15].

Quinazolinones are an important class of nitrogenous heterocycles, which exhibit potential biological and pharmaceutical activities [16, 17]. 2,3-Dihydroquinazolin-4(1H)-ones are quinazolinone derivatives that have attracted much attention due to their antibacterial, antitumor, antifungal, and monoamine oxidaze inhibitory activity [18–20]. Furthermore, these compounds can be easily oxidized to quinazolin-4(3H)-ones [21], which, too, are biologically active compounds and exist in a variety of natural products, pharmaceutical candidates, and agrochemicals [22]. Several methods have been reported for the synthesis of 2,3-dihydroquinazolin-4(1H)-ones, including desulfurization of 2-thioxoquinazolin-4(3H)-ones [23], reductive cyclization of o-nitrobenzamides or o-azidobenzamides [24], condensation of o-aminobenzamides with benzil [25], reduction of quinazolin-4(3H)-ones [26], and cyclocondensation of anthranilamide (2-aminoben-



zamide) with aldehydes or ketones in the presence of various catalysts [27–45]. However, most of the reported procedures feature one or more disadvantages, such as low product yields, prolonged reaction time, difficult work-up, use of hazardous solvents, and lack of catalyst reusability. Therefore, search for effective, simple, and mild synthetic approaches to 2,3-dihydroquinazolin-4(1H)-ones remains a relevant task.

Herein, we report a versatile procedure for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones through the cyclocondensation of various aldehydes and ketones with anthranilamide, catalyzed by 1-methyl-3-(3-triethoxysilylpropyl)-1*H*-imidazol-3-ium hydrogen sulfate immobilized on the surface of Fe₃O₄@SiO₂ MNPs (Fe₃O₄@SiO₂-ImHSO₄) as an efficient and magnetically recyclable Brønsted acid ionic liquid (Table 1).

The Brønsted acid ionic liquid immobilized on Fe₃O₄@ SiO₂ MNPs was prepared as shown in the Scheme 1. Magnetite nanoparticles were easily prepared by the coprecipitation of FeCl₂ with FeCl₃ in an ammonia solution. The surface of Fe₃O₄ MNPs was successfully coated with silica by the ammonia-catalyzed hydrolysis of tetraethyl orthosilicate (TEOS) to obtain core-shell Fe₃O₄@SiO₂ MNPs. The reaction between 1-methylimidazole and 3-(chloropropyl)triethoxysilane at 80°C gave a 1-methyl-3-[3-(triethoxysilyl)propyl]-1*H*-imidazol-3-ium chloride ionic liquid, which was immobilized on the surface of Fe₃O₄@SiO₂ MNPs by the reaction of surface OH groups in the latter and the organosilyl group in the ionic liquid. Finally, ion exchange of the Cl⁻ anions for HSO⁻₄ was performed by treatment with conc. H₂SO₄.

The catalyst was characterized by scanning electron microscopy (SEM), energy dispersive X-ray (EDAX)

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200 nm

Fig. 1. SEM image of Fe₃O₄@SiO₂-ImHSO₄ MNPs.

spectroscopy, Fourier-transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD) analysis, vibrating sample magnetometry (VSM), and thermogravimetric analysis (TGA).

The particle size distribution was measured and morphological characterization of Fe₃O₄@SiO₂-ImHSO₄ was performed by SEM on a Philips XL30 scanning electron microscope. As shown in Fig. 1, Fe₃O₄@SiO₂-ImHSO₄ MNPs have a spherical shape, and their dimensions range from 20 to 30 nm.

The components of Fe₃O₄@SiO₂-ImHSO₄ MNPs were analyzed using EDAX spectroscopy. The EDAX



Fig. 3. FTIR spectra of (1) Fe_3O_4 , (2) Fe_3O_4 @SiO₂, and (3) Fe₃O₄@SiO₂-ImHSO₄ MNPs.



Fig. 2. EDAX spectra of Fe₃O₄@SiO₂-ImHSO₄ MNPs.

spectrum is consistent with our expectations and confirms the presence of carbon, nitrogen, oxygen, iron, silica, and sulfur in Fe₃O₄@SiO₂-ImHSO₄ (Fig. 2).

The FTIR spectra of Fe₃O₄, Fe₃O₄@SiO₂, and Fe₃O₄@SiO₂-ImHSO₄ are shown in Fig. 3. The FTIR spectrum of Fe₃O₄ (Fig. 3) displays a strong band at low frequencies (578 cm⁻¹) due to Fe–O vibrations and a broad band at 3200–3600 cm⁻¹, which can be attributed to surface –OH groups. The FTIR spectrum of Fe_3O_4 (2)SiO₂ (Fig. 3) shows three additional bands around 460, 980, and 1080 cm⁻¹, which are assignable Si-O-Si bending, symmetric stretching and asymmetric stretching vibrations, respectively. These peaks confirm the presence of the silica shell on Fe₃O₄ MNPs. The introduction of the linker is confirmed by the observation in the FTIR spectrum of Fe₃O₄@SiO₂-ImHSO₄ of bands at 1400-1500 and 2850-2950 cm⁻¹ assignable to C–H bending and stretching vibrations, respectively. The band around



Fig. 4. XRD patterns of (1) Fe₃O₄ and (2) Fe₃O₄@SiO₂-ImHSO₄ MNPs.



1600 cm⁻¹ is due to imidazole C=N bending and imidaz- temperature

ole ring stretching vibrations (Fig. 3).

The crystal structure of Fe_3O_4 @SiO₂–ImHSO₄ MNPs was studied in the 2 θ range 0–80°. The XRD pattern displays peaks at 30.26° (30.31°), 35.63° (35.69°), 43.39° (43.32°), 53.72° (53.81°), 57.33° (57.26°) and 62.85° (62.92°) and is quite similar to the XRD pattern of Fe₃O₄ (the respective peak positions are given in parentheses) (Fig. 4).

The magnetic properties of the catalyst were studied by the VSM method (Fig. 5). As seen, the saturation magnetization value decreased from 42 emu/g in Fe₃O₄@ SiO₂ to about 27 emu/g in Fe₃O₄@SiO₂-ImHSO₄. This decrease is explained by that in the latter case magnetic nanoparticles have a nonmagnetic ionic liquid coating.

The TGA analysis of $Fe_3O_4@SiO_2$ -ImHSO₄ was performed by heating the sample to 600°C at a rate of 10°C min⁻¹ in air (Fig. 6). Three main weight loss stages were observed. The first, small (~2%) weight loss at below 130°C was assigned to a loss of physically adsorbed water, as well as dehydration of the surface OH groups. The main weight losses at 130–250°C (~8%) and 250– 430°C (~7%) are probably associated with the breakdown and decomposition of 1-methyl-3-propylimidazolium groups grafted on the Fe₃O₄@SiO₂ surface.

The catalytic activity of $Fe_3O_4@SiO_2$ -ImHSO₄ MNPs was studied in the condensation reaction of benzaldehyde with anthranilamide under various reaction conditions (Table 1). Among the screened solvents, including ethanol, water, acetonitrile, dichloromethane, chloroform, diethyl ether, toluene, acetone, ethyl acetate and polyethylene glycol, the best results were obtained in ethanol (Table 1, entries 1–10). The reaction at room temperature, as well as under reflux conditions gave the desired product with an excellent yield in a short reaction time (Table 1, entries 9 and 10). In the absence of catalyst, the reaction was not complete after 2 h in refluxing ethanol (Table 1, entry 11). According to the results, the highest yields of 2,3-dihydro-2-phenylquinazolin-4(1*H*)-one were obtained in the presence of 0.05 g of Fe₃O₄@ SiO₂–ImHSO₄ in ethanol and at room temperature or under reflux (93% or 91%, respectively; Table 1, entries 9 and 10).

To explore the universality of the developed procedure, a variety of aldehydes and cyclic ketones were reacted with anthranilamide in the presence of Fe₃O₄@ SiO₂-ImHSO₄ under the optimal conditions (Table 2). Arylaldehydes with both electron-donor and electronacceptor substituents reacted with anthranilamide to give the corresponding 2-aryl-2,3-dihydroquinazolin-4(1*H*)-ones in excellent yields (Table 2, entries 2–12). The reactions with cinnamaldehyde and 2-furaldehyde under same reaction conditions, too, produced the desired products in high yields (Table 2, entries 13 and



Fig. 6. TGA curve of Fe₃O₄@SiO₂-ImHSO₄ MNPs.

Experiment no.	Catalyst amount, g	Solvent	Thermal conditions	Reaction time, min	Yield, %
1	0.05	H ₂ O	rt	120	80
2	0.05	MeCN	rt	120	traces
3	0.05	CH_2Cl_2	rt	120	traces
4	0.05	CHCl ₃	rt	120	traces
5	0.05	Et ₂ O	rt	120	traces
6	0.05	PhMe	rt	120	traces
7	0.05	EtOAc	rt	120	60
8	0.05	PEG-400	rt	120	traces
9	0.05	EtOH	rt	15	93
10	0.05	EtOH	reflux	10	91
11	None	EtOH	reflux	120	traces
12	0.04	EtOH	rt	60	89

Table 1. Optimization of conditions of the cyclocondensation of anthranilamide with benzaldehyde in the presence of the $Fe_3O_4@SiO_2$ -ImHSO₄ nanocatalyst

14). Cyclic ketones and diketones were also condensed with anthranilamide successfully, and the corresponding spiroquinazolinones were obtained in good yields and short reaction times (Table 2, entries 15–19). Noteworthy is the formation a dispiro compound in the reaction of cyclohexane-1,4-dione with anthranilamide in a 1 : 2 ratio (Table 2, entry 20).

The recyclability and reusability of the Fe₃O₄@SiO₂– ImHSO₄ nanocatalyst was tested in the cyclocondensation of benzaldehyde with anthranilamide in EtOH at room temperature. After the reaction had been complete (15 min), Fe₃O₄@SiO₂–ImHSO₄ was separated by means of an external magnet, washed thoroughly with water and ethanol, dried in a vacuum oven at 60°C, and then reused in another catalytic cycle. We found that the catalyst could be magnetically recovered and reused without any appreciable loss of activity in five consecutive catalytic cycles: yield, % (cycle no.): 93 (1), 91 (2), 90 (3), 87 (4), 83 (5).

Comparison of the efficacy of the $Fe_3O_4@SiO_2$ -ImHSO₄ nanocatalyst and some of those reported in the literature in the synthesis of 2-phenyl-2,3-dihydroquinazolin-4(1*H*)-one (**3a**) is presented in Table 3. As seen, the best results, both in terms of reaction time and product yield, were obtained with $Fe_3O_4@SiO_2$ -ImHSO₄.

In conclusion, the present paper described the synthesis of 2,3-dihydroquinazolin-4(1H)-one derivati-

ves using an efficient and magnetically recyclable Brønsted acid ionic liquid, $Fe_3O_4@SiO_2$ –ImHSO₄. This developed catalytic synthesis offers several advantages, including environmental friendliness, high yield, short reaction time, mild reaction conditions, simple work-up procedure, ease of separation, and recyclability of the catalyst and can be considered as a new and suitable addition to the present methodologies in this area.

EXPERIMENTAL

All of the commercially available chemicals were purchased from Merck, Fluka, and Aldrich and used with no further purification. Fe₃O₄, Fe₃O₄@SiO₂, and Fe₃O₄@SiO₂–ImHSO₄ were prepared pursuant to the reported procedures [45–47]. All of the products were characterized by their physical properties and comparison with authentic samples. The reactions were monitored by TLC on Polygram SIL G/UV 254 plates. The melting points were measured in open capillary tubes and on an Electrothermal 9200 apparatus. The IR spectra were run on a Shimadzu IRPresting-21 spectrophotometer in the range of 4000–400 cm⁻¹ in KBr pellets. The ¹H and ¹³C NMR spectra were recorded in DMSO- d_6 solutions on a Bruker Advanced DPX 400 MHz spectrometer using TMS as internal reference.

Synthesis of 1-methyl-3-(3-triethoxysilylpropyl)-1*H*imidazol-3-ium chloride. A mixture of 1-methylimid-





Entry	\mathbf{R}^{1}	R ²	Room temperature		Reflux		mp, °C	
no.			time, min	yield, %a	time, min	yield, %a	measured	published
1	C ₆ H ₆	Н	15	93	10	91	220–222	220–222 [44]
2	$4-CH_3C_6H_4$	Н	20	90	10	87	231–233	231–233 [44]
3	4-OCH ₃ C ₆ H ₄	Н	25	92	20	88	187–189	191–193 [44]
4	$4-OHC_6H_4$	Н	15	87	10	88	270–272	275–277 [36]
5	2-ClC ₆ H ₄	Н	35	95	30	87	207–209	207–209 [44]
6	3-ClC ₆ H ₄	Н	75	85	40	87	185–187	185–187 [44]
7	4-ClC ₆ H ₄	Н	90	80	60	83	205–207	205–207 [44]
8	4-BrC ₆ H ₄	Н	100	82	60	81	202–204	198–200 [36]
9	$2-NO_2C_6H_4$	Н	35	87	25	89	189–191	190–193 [36]
10	$3-NO_2C_6H_4$	Н	45	84	40	81	183–184	183–184 [44]
11	$4-N(CH_3)_2C_6H_4$	Н	100	89	80	88	209–210	209–210 [44]
12	2,5-(OCH ₃) ₂ C ₆ H ₃	Н	70	89	30	87	189–189	_
13	C ₆ H ₅ CH=CH	Н	35	91	30	90	240–243	240–243 [44]
14 CHO		90	90	50	84	166–168	165–167 [44]	
15	-(CH ₂) ₄ -		125	88	75	85	260–262	260–262 [44]
16 –(CH ₂) ₅ –		20	91	10	92	217–219	217–219 [44]	
17	17 –(CH ₂) ₆ –		150	88	90	85	197–200	197–200 [44]
18	$8 \qquad \qquad$		125	88	80	83	215–217	215–217 [44]
19	0		80	87	55	86	> 300	> 300 [44]

Entry no.	\mathbb{R}^1	R ²	Room temperature		Reflux		mp, °C	
			time, min	yield, %a	time, min	yield, %a	measured	published
20 ^b	0		30	84	20	84	> 300	> 300 [44]

Table 2. (Contd.)

^a Isolated yield.

^b Anthranilamide–ketone, 2 : 1.

azole (0.82 g, 10 mmol) and (3-chloropropyl)triethoxysilane (2.41 g, 10 mmol) was stirred at 80°C for 72 h under nitrogen. The resulting viscous yellow liquid was washed with diethyl ether (3×5 mL) to remove unreacted material and was dried under high vacuum to obtain the target product.

Synthesis of the $Fe_3O_4@SiO_2$ -ImHSO₄ nanocatalyst. A suspension of $Fe_3O_4@SiO_2$ nanoparticles (1 g) in CHCl₃ (30 mL) was dispersed under ultrasonic irradiation for 1 h, after which 1-methyl-3-(3-triethoxysilylpropyl)-1*H*-imidazol-3-ium chloride (0.28 g, 1 mmol) was added, and the reaction mixture was stirred under reflux for 48 h under nitrogen. The product was magnetically collected, washed with 3 portions of CHCl₃, and dried at 60°C in a vacuum oven. To exchange the Cl⁻ anion, the dry product (1 g) was suspended in dry CH_2Cl_2 (20 mL), the suspension was cooled to 0°C, and and conc. H_2SO_4 (0.07 mL, 1.3 mmol) was added to it dropwise with vigorous stirring. The mixture was then let to warm up to room temperature and refluxed for 48 h, cooled, filtered, and dried to give the target product.

General procedure for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones. The Fe₃O₄@SiO₂-ImHSO₄ nanocatalyst (0.05 g) was added to a solution of anthranilamide (1 mmol) and aldehyde or ketone (1 mmol) in ethanol (5 mL), and the reaction mixture was stirred at room temperature or under reflux conditions during the time shown in Table 2. After the reaction had been completion (by TLC in hexane–EtOAc, 3 : 1), the catalyst was removed with a magnet, the solution was filtered, and

Entry no.	Catalyst	Conditions	Time, min	Yield, %	References
1	[bmim]PF ₆	Solvent-free, 75°C	35	89	[27]
2	Sc(OTf) ₃	EtOH, 70°C	25	92	[28]
3	NH ₄ Cl	EtOH, rt	15	92	[29]
4	ZrCl ₄	EtOH/H ₂ O, MWI	25	95	[33]
5	H[Gly ₂ B]	Solvent-free, 60°C	10	90	[35]
6	Poly(VPyPS)-PW	EtOH, USI	6	95	[38]
7	Y(OTf) ₃	EtOH, rt	90	93	[41]
8	α-Chymotrypsin	EtOH, 60°C	20	91	[43]
9	Fe ₃ O ₄ @ SiO ₂ -SO ₃ H	EtOH, rt	20	92	[44]
10	Fe ₃ O ₄ @SiO ₂ -ImHSO ₄	EtOH, rt	15	93	This work
11	Fe ₃ O ₄ @SiO ₂ -ImHSO ₄	EtOH, reflux	10	91	This work

Table 3. Comparison of the efficiencies of different catalysts in the synthesis of 2-phenyl-2,3-dihydroquinazolin-4(1*H*)-one

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the filtrate was washed with cold ethanol to obtain the target product, which was purified by recrystallization from ethanol. All products are known compounds, and they were identified by comparing their physicochemical characteristics and IR and NMR spectra with those of authentic samples.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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