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Short Communication

Nanosized MCM-41 supported protic ionic liquid as an efficient novel catalytic system for Friedlander synthesis of quinolines

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ABSTRACT

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1. Introduction

Recently, room temperature ionic liquids have attracted considerable attention due to their special characteristics including unique solvation properties, high thermal stability, ease of handling, wide liquid range and negligible vapor pressure (10^{-8} bar) [1,2]. These compounds have been used as both catalyst and solvent in various kinds of reactions. These applications require large amounts of ionic liquids as expensive materials [3,4]. In addition, the high viscosity of ionic liquids can induce mass transfer limitations if the chemical reaction is fast, in which case the reaction takes place only within the narrow diffusion layer and not in the bulk of the ionic liquid. This problem can be avoided by immobilizing a thin film of ionic liquid on the surface and/or pores of a high surface area support [4]. The supported catalyst thus combines the advantages of ionic liquids and heterogeneous support. This process can transfer the desired catalytic properties of the ionic liquids to solid catalyst.

Various mineral oxides such as silica [5], alumina [6], titania [7] and zirconia [8] have been used as the support for preparation of solid catalyst. Since the discovery of mesoporous molecular sieves in 1992 [9], the use of nanosized mesoporous materials has attracted considerable attention due to their highly ordered porous structure and very high surface area. Among the various types of mesoporous silica, MCM-41 exhibits a highly ordered hexagonal array of one-dimensional mesopores with a pore diameter of 15–100 Å, high

Nanosized MCM-41 has been synthesized by sol-gel method. *n*-Butanesulfonic acid pyridinium hydrogensulfate as protic ionic liquid has been dispersed on the MCM-41 nanoparticles. The morphology of the MCM-41 and MCM-41 supported ionic liquid has been studied by SEM, XRD, BET and FT-IR techniques. The catalytic performance of the nanosized MCM-41 supported ionic liquid was investigated in the Friedlander synthesis of quinolines.

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surface area (>1000 m² g⁻¹) and high thermal stability (ca. 900 °C) [10]. This compound has been used as support in the preparation of solid acid catalysts for organic transformations [11,12]. Recently, several reports have been published describing the use of the immobilized ionic liquids on mesoporous silica of MCM-41 [13,14].

In this research we report the preparation, characterization and catalytic application of nanosized MCM-41 supported *n*-butanesulfonic acid pyridinium hydrogensulfate ((BSPY)HSO₄/MCM-41), as catalyst in Friedlander synthesis of quinolines (Scheme 1). The novel multi SO₃H functionalized ionic liquids have been synthesized as protic ionic liquid and employed in many organic transformations [15–18]. However, less attention has been paid to the immobilization of this type of protic ionic liquids on the solid support.

2. Experimental methods

2.1. Materials and methods

All chemicals were commercial products. All reactions were monitored by TLC and all yields refer to isolated products. ¹H and ¹³C NMR spectra were recorded in CDCl₃ on a Bruker DRX-500 AVANCE (500 MHz for ¹H and 125.72 MHz for ¹³C) spectrometer. Infrared spectra of the catalysts and reaction products were recorded on a Bruker FT-IR Equinax-55 spectrophotometer in KBr with absorption in cm⁻¹. XRD patterns were recorded on a Bruker D8 ADVANCE X-ray diffractometer using nickel filtered Cu K α radiation (λ = 1.5406 Å). The 20 angles were scanned from 1.5° to 10°. The morphology was studied using a Philips XL30 scanning electron microscopy. The BET surface area was performed using Micromeritics model Gemini 2375V

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 R^2 , R^3 = alkyl and aryl with various functional groups

Scheme 1. The Friedlander synthesis of quinolines in the presence of 23% (BSPY)HSO₄/MCM-41.

4.02 from the nitrogen adsorption isotherms at -170 °C. All samples were degassed at 130 °C under flowing nitrogen for 1.5 h. Specific surface area (S_{BET}) was calculated from adsorption data using BET equation at P/P° = 0.1–0.3, and pore volume (V_{pore}) was calculated based on Barret–Joyner–Halenda (BJH) method.

2.2. Preparation of MCM-41 nanoparticles

The synthesis of nanosized MCM-41 was carried out using tetraethyl orthosilicate (TEOS) as the Si source, cetyltrimethylammonium bromide (CTAB) as the template and ammonia as the pH control agent with the gel composition of SiO₂:CTAB:NH₄OH:H₂O = 22.5:2.74:53.5:11.11.

In a typical procedure, to a solution of CTAB (1 g, 2.74 mmol) in deionized water (200 mL), aqueous ammonia (25 wt.%) was added until the pH of the solution was adjusted to 10.5. Then TEOS (5 mL) was added dropwise at 70 °C for 1 h and the mixture was allowed to cool to room temperature and was stirred for 12 h. The solid was separated by centrifuge and washed with distilled water (20 mL) and EtOH (2×10 mL) respectively. The solid was dried in an oven at 120 °C for 1 h and then calcined at 550 °C for 4 h.

2.3. Preparation of 23 wt.% (BSPY)HSO₄/MCM-41

(BSPY)HSO₄ as protic ionic liquid was synthesized according to reported procedure [18]. To a suspension of the MCM-41 (60 mg) in acetone (1 mL), (BSPY)HSO₄ (0.0626 g) was added and the mixture was stirred for 12 h. The catalyst was separated by centrifuge, washed with acetone (4×2 mL) and dried in an oven at 120 °C for 2 h.

2.4. Catalytic activity of (BSPY)HSO₄/MCM-41 in Friedlander synthesis

A mixture of *o*-aminobenzophenone (1 mmol), ketone or β -diketone (1.2 mmol) and (BSPY)HSO₄/MCM-41 (70 mg) was heated at 100 °C. After completion of the reaction (monitored by TLC, eluent; *n*-hexane:EtOAc, 8:2), EtOH (2 mL) was added, the catalyst was separated and washed with EtOH (3×2 mL). After addition of water, the product was precipitated with high purity. Further purification was achieved by recrystallization in EtOH. The pure quinolines were obtained in 78–95% yields.

2.5. Physical and spectroscopic data for selected compound

2.5.1. Ethyl-2-methyl-4-phenylquinoline-3-carboxylate (3a)

MP: 100–101 °C (Lit. [19], 100–101 °C). IR (KBr): v_{max} : 2978, 1711, 1561, 1444, 1402, 1297, 1237, 1182, 1130, 1070, 767, 700, 623, 603 cm⁻¹. ¹H NMR (500 MHz, CDCl₃): δ =1.00 (t, 3H, J=7.1 Hz),

Table 1

Preparation of (BSPY)HSO₄/MCM-41 with various loading amounts of (BSPY)HSO₄/MCM-41.

Entry	Mole ratio of (BSPY)/HSO ₄ :MCM-41	Amount of (BSPY)/HSO ₄ on the MCM-41 (wt.%)
1	1:20	5
2	1:10	14
3	1:5	23
4	1:2	28

2.84 (s, 3H), 4.11 (q, 2H, J=7.1 Hz), 7.41 (m, 2H), 7.47 (dt, 1H, J= 0.8, 7.2 Hz), 7.53 (m, 3H), 7.62 (d, 1H, J=8.2 Hz), 7.77 (dt, 1H, J=1.3, 7 Hz), 8.13 (d, 1H, J=8.5 Hz). ¹³C NMR (125.7 MHz, CDCl₃): δ =14.06, 24.15, 61.78, 125.61, 126.9, 126.95, 127.87, 128.66, 128.91, 129.165, 129.80, 130.76, 136. 14, 146.84, 155.05, 168.82.

3. Results and discussion

3.1. The catalyst preparation

Nanosized MCM-41 was prepared by sol-gel method. The (BSPY)HSO₄/MCM-41 catalysts with various loading amounts of (BSPY)HSO₄ were prepared. For this purpose, suspensions of the MCM-41 and (BSPY)HSO₄ with various molar ratios of (BSPY)HSO₄:MCM-41 in a constant amount of acetone were stirred for 12 h. The suspensions were centrifuged and washed many times with acetone and then dried in an oven at 120 °C. The details of conditions for catalyst preparation are listed in Table 1.

As shown in Table 1, loading amounts of $(BSPY)HSO_4$ present in the MCM-41 were found to be 5, 14, 23 and 28 wt.%.

3.2. Characterization of materials

The MCM-41 and the prepared catalysts were characterized by SEM, XRD, FT-IR and BET techniques.

The SEM image of MCM-41 (Fig. 1) shows spherical nanoparticles with the size of less than 100 nm.

The FT-IR spectra of MCM-41, (BSPY)HSO₄ and (BSPY)HSO₄/MCM-41 with different loading amounts of (BSPY)HSO₄ are shown in Fig. 2. As shown in Fig. 2a, pure (BSPY)HSO₄ exhibited characteristic peaks of alkyl sulfonate and hydrogensulfate at 1230, 1170, 1058, 1028, 885, 854 and 594 cm⁻¹ and the peaks of pyridine ring at 1489, 1651 and 684 cm⁻¹. FT-IR spectrum of MCM-41 (Fig. 2b) shows typical IR bands at 1080 and 812 cm⁻¹ corresponding to stretching Si–O and 460 cm⁻¹ corresponding to bending Si–O–Si. In the FT-IR spectra of 5–23% (BSPY)HSO₄/MCM-41 (Fig. 2c–f), apart from the main peaks of MCM-41, additional peaks corresponding to functional group of



Fig. 1. The SEM image of MCM-41.



Fig. 2. FT-IR spectra of (a) (BSPY)HSO₄, (b) MCM-41, (c) 5 wt.% (BSPY)HSO₄/MCM-41, (d) 14 wt.% (BSPY)HSO₄/MCM-41, (e) 23 wt.% (BSPY)HSO₄/MCM-41, and (f) 28 wt.% (BSPY)HSO₄/MCM-41.

ionic liquid appeared in the patterns. It is noticeable that because of low intensity, all of the characteristic peaks of (BSPY)HSO₄ in the spectra of the catalysts with lower loading amounts of (BSPY)HSO₄ (Fig. 2c and d) did not appear. However, in the spectra of 23% and 28% (BSPY)HSO₄/MCM-41, more of (BSPY)HSO₄ peaks appeared. In these spectra, the

Table 2

Textural properties of MCM-41 a	nd (BSPY)HSO ₄ /MCM-41 catalysts
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Entry	Catalyst	$S_{BET} (m^2 g^{-1})$	Total pore volume $(mL g^{-1})$
1	MCM-41	1153	0.84
2	23% (BSPY)HSO ₄ /MCM-41	444	0.39
3	28% (BSPY)HSO ₄ /MCM-41	110	0.17
4	Recovered 23% (BSPY)HSO ₄ /MCM-41	948	0.62

peaks at 1651, 1489, 727 and 684 cm^{-1} are assigned to the pyridine ring of (BSPY)HSO₄, while the characteristic peaks of sulfate around 1230, 1170, 1058 and 1028 cm⁻¹ may overlap with the strong peak of MCM-41 around 1080 cm⁻¹ resulting in increase of intensity and broadening of this peak of MCM-41. The peaks at 885 and 854 cm⁻¹ are assigned to a stretching frequency of C – S and S – O bond of sulfate. These results indicated that ionic liquid is dispersed on the surface of MCM-41.

Fig. 3 shows the XRD pattern of MCM-41 and 5–23 wt.% (BSPY)HSO₄/MCM-41. As shown in Fig. 3a, MCM-41 exhibited a strong peak at $2\theta = 2.32^{\circ}$ which corresponded to the characteristics of hexagonal ordering and two weak peaks at $2\theta = 4.04$, 4.64° . In the XRD patterns of 5–23 wt.% (BSPY)HSO₄/MCM-41 (Fig. 3b–c), intensity of the peak at $2\theta = 2.32^{\circ}$ was decreased and its width was increased with an increase in loading amount of (BSPY)HSO₄, which means that the hexagonal mesostructures are less ordered due to dispersion of ionic liquid molecules in the mesoporous channels of MCM-41.

The textural properties of MCM-41, 23 wt.% and 28 wt.% (BSPY)HSO₄/MCM-41 and recovered 23 wt.% (BSPY)HSO₄/MCM-41 are listed in Table 2. As shown in Table 2, the surface area and pore volume of MCM-41 are 1153 m² g⁻¹ and 0.84 mL g⁻¹ respectively, in accordance with literature data [20]. The surface area and pore volume



Fig. 3. XRD patterns of (a) MCM-41, (b) 5 wt.% (BSPY)HSO₄/MCM-41, (c) 14 wt.% (BSPY)HSO₄/MCM-41, and (d) 23 wt.% (BSPY)HSO₄/MCM-41.



Fig. 4. Nitrogen adsorption isotherms of (a) MCM-41, (b) 23% (BSPY)/HSO₄/MCM-41, and (c) 28% (BSPY)/HSO₄/MCM-41.

Table 3

Table 4

Reaction of ethylacetoacetate and 2-aminobenzophenone for the synthesis of quinolines in the presence of various amounts of (BSPY)HSO₄/MCM-41.

Entry	Catalyst amount (mg)	(BSPY)HSO ₄ loading on the MCM-41 (wt.%)	(BSPY)HSO ₄ /MCM-41 wt.% related to reactants	Time (min)	Conversion (%)
1	70	5	21	120	100
2	70	14	21	100	100
3	70	23	21	70	100
4	70	28	21	180	<50
5	50	23	15	130	100
6	100	23	30	65	100

decrease after supporting the (BSPY)HSO₄ on the MCM-41 surface area (Table 2, entries 2, 3).

Fig. 4 shows N_2 adsorption isotherms of MCM-41, and 23 wt.% and 28 wt.% (BSPY)HSO₄/MCM-41. The typical curve of MCM-41 could be

assigned to type IV isotherm. A mesoporous inflection was observed on the isotherm of MCM-41 at a relative pressure between $p/p^{\circ} =$ 0.3–0.4 due to capillary condensation. In the isotherm of 23 wt.% (BSPY)HSO₄/MCM-41, mesoporous isotherm (type IV) characteristic still could be observed, but the inflection becomes shorter corresponding to a reduction in the pore volume. This result is due to (BSPY)HSO₄ dispersion in pore walls that causes lower capillary condensation. However, in the isotherm of 28 wt.% (BSPY)HSO₄/MCM-41 (Fig. 4c) the condensation region disappeared. This may be due to extremely full blockage of mesoporous channels of MCM-41 with (BSPY)HSO₄ and the isotherm shows only multilayer adsorption on the external surface.

3.3. Catalytic activity of (BSPY)HSO₄/MCM-41

The catalytic performance of (BSPY)HSO₄/MCM-41 in the Friedlander synthesis of quinolines by the condensation reaction

Tuble I				
Reaction of ketones and 2-aminobenzo	ophenone for the synthesis of	quinolines in the presence	e of 23 wt.% of (BSP	Y)HSO ₄ /MCM-41.

Entry	Aminoarylketone	Ketone	Quinoline	Time	Yield ^a	Mp (°C)
	(1)	(2)	(3)	(min)	(%)	Found Lit. [ref]
a	O Ph NH ₂	OEt	Ph O OEt	70	93	100–101 100–101 [19]
b	Ph NH ₂		Ph O N	45	91	108–109 111–112 [19]
с	Ph NH ₂	Ph CH ₃	Ph O Ph Ph	50	90	139–140 133–134 [21]
d	Ph NH ₂		Ph N	90	87	142-143 138-141 [21]
e	Ph NH ₂		Ph N	210	82	133–135 133–135 [21]
f	Ph NH ₂	0,00	Ph 0	40	87	144-146 151-153 [21]
g	C1 Ph NH2	OEt	CI Ph O OEt	80	94	98-99 102-104 [21]
h	C1 Ph NH ₂		Cl Ph O N	70	88	155–156 154 [21]
i	C1 Ph NH ₂		Cl Ph	55	89	162–163 159 [19]
j	C1 Ph NH ₂		Cl Ph	80	91	97–98 100 [21]

^a Isolated yields.

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Table 5

comparison of the catalytic activity of (bsi 1)1504/ment 41 with other heterogenet	Comparison o	f the catalytic activity c	f (BSPY)HSO ₄ /MCM-41	with other heterogeneous catalysts.
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Entry	Catalyst	Amount of the catalyst	Solvent, reaction temp. (°C)	Reaction time (min)	Yield (%)	Ref.
1	(BSPY)HSO ₄ /MCM-41	70 mg	Solvent-free, 100	70	93	-
2	NaHSO ₄ /SiO ₂	200 mg	Solvent-free, 100	60	92	[22]
3	Amberlyst-15	10% (w/w)	EtOH, reflux	150	89	[23]
4	HClO ₄ /SiO ₂	200 mg	CH ₃ CN, 60	120	94	[24]
5	Nano Al ₂ O ₃	0.03 mmol	CHCl ₃ , reflux	180	98	[25]
6	Cellulose sulfuric acid	80 mg	Solvent-free, 100	20	82	[26]
7	Starch sulfuric acid	80 mg	Solvent-free, 100	30	81	[26]

of *o*-aminobenzophenones and ketones was investigated. Initially, the optimization experiments were performed in the reaction of *o*-aminobenzophenone and ethyl acetoacetate at 100 $^{\circ}$ C under solvent-free condition as the model reaction and the results are shown in Table 3. It is noticeable that the reaction times presented in Table 3 (except for entry 4) are the times for total conversion of reactants.

To investigate the effect of the loading amount of (BSPY)HSO₄ on the catalytic activity of (BSPY)HSO₄/MCM-41 in the Friedlander synthesis, the model reaction was performed in the presence of 70 mg of catalysts with 5–28 wt.% (BSPY)HSO₄. As shown in Table 3 (entries 1–4) the reaction times were reduced from 120 to 70 min with an increase in the loading amount of (BSPY)HSO₄ up to 23 wt.%. Further increase in loading up to 28 wt.% results in a drop in activity and increases the reaction time. These results show that in terms of reaction time the catalyst with 23 wt.% (BSPY)HSO₄ has the best catalytic activity. Low catalytic activity of 28 wt.% (BSPY)HSO₄/MCM-41 is in compliance with the results of BET experiments. This low activity of the former catalyst is due to high blockage of mesoporous channels of MCM-41 and thus reaction cannot take place in very low surface of the heterogeneous catalyst.

In order to optimize the amount of 23 wt.% (BSPY)HSO₄ in the model reaction, various amounts of the catalyst were used in the reaction and the best result in terms of catalyst loading amount and the yield of the product was obtained using 70 mg of the catalyst for the reaction of 1 mmol *o*-aminobenzophenone in the model reaction with a mole ratio of 1:1.2 for *o*-aminobenzophenone:ethyl acetoacetate (Table 3, entries 4–6).

The scope and generality of this methodology is illustrated with respect to various types of ketones and results are summarized in Table 4. According to the results of optimization experiments for the reaction conditions and the amount of the catalyst, the reaction of various diketones with o-aminobenzophenones was carried out in the presence of 23 wt.% (BSPY)HSO4/MCM-41 at 100 °C under solvent-free condition and the corresponding quinolines were obtained in good to excellent yields (Table 4, entries 3a-3c and 3g-3h). Cyclic ketones were also reacted with o-aminobenzophenones in the same reaction conditions to afford the corresponding tricyclic quinolines in good yields (Table 4, entries 3d–3f and 3i–3j). The difference between reaction times of various ketones in Table 4 is not clear. This may be due to the absence of regular functional groups on the ketones. However, a plausible explanation is that various reactants have different interactions with the surface of the catalyst in mesoporous channels of MCM-41.

The workup and catalyst recovery are very simple. All of the reactions have very high selectivity toward corresponding quinolines and TLC monitoring shows only trace amounts of byproducts. After the completion of the reaction (monitored by TLC, eluent; EtOAc:*n*hexane, 20:80), EtOH was added to the reaction mixture and the catalyst was separated by centrifuge and washed with EtOH. After evaporation of excess amount of EtOH the crude product was easily isolated in almost pure state by addition of water and filtration of the precipitate. Further purification was achieved by recrystallization from EtOH. All of the reported yields in Table 4 are isolated yields (by weight) after recrystallization.

All of the synthesized products are known compounds and were characterized by comparing their melting points (Table 4) and IR spectra. Many of the compounds have also been characterized by ¹H and ¹³C NMR spectroscopy. The characterization detail of selected compound is shown in Experimental methods section.

The efficiency of 23 wt.% (BSPY)HSO₄/MCM-41 was also compared with other similar heterogeneous catalytic systems in the same reaction at the same reaction temperature and solvent-free condition (Table 5). The obtained results indicate the superiority of the present catalyst in terms of catalyst amount, yield or reaction times.

To investigate reusability of the catalyst, the recovered catalyst from the model reaction was washed with EtOH and dried in an oven at 120 °C for 1 h. The recovered catalyst was used in the same reaction. The results show a decrease of catalyst activity in comparison to the first run. This result suggests that deactivation is due to the leaching of (BSPY)HSO₄ from the MCM-41. The leaching was studied by comparing the FT-IR of the fresh catalyst and recovered catalyst after the first run (Fig. 5). As shown in Fig. 5b the FT-IR spectrum of the recovered catalyst shows very weak characteristic peaks of ionic liquid in comparison to the fresh catalyst (Fig. 5c). This confirms partial leaching of the ionic liquid from the surface of MCM-41. These results were confirmed by the study of BET surface area and pore volume of recovered and fresh catalysts. As shown in Table 2 (entry 4) BET surface area and pore volume of recovered catalysts were decreased to 947 m^2/g and 0.62 mL/g from 444 m^2/g and 0.39 mL/g in fresh catalyst. However, the surface area and pore volume of recovered catalyst are less than pure MCM-41 and these results can confirm partial leaching of (BSPY)HSO₄ from the support.



Fig. 5. FT-IR spectra of (a) MCM-41, (b) 23 wt.% (BSPY)HSO₄/MCM-41 after reaction, and (c) fresh 23 wt.% (BSPY)HSO₄/MCM-41.

4. Conclusion

In conclusion, we found that (BSPY)HSO₄ as protic ionic liquid could be dispersed highly on the surface of MCM-41 nanoparticles. The obtained material ((BSPY)HSO₄/MCM-41) can be used as solid acid catalyst for the Friedlander synthesis of quinolines. Easy workup, green condition, high yield and simple purification make this method attractive for the synthesis of quinolines.

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