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Efficient synthesis of 3,3'-bisindoles catalyzed by Fe₃O₄@MCM-48-OSO₃H magnetic core-shell nanoparticles

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1. Introduction

The chemistry of indoles has been and continues to be one of the most active areas of heterocyclic chemistry [1,2]. Various indole derivatives are pharmacologically and biologically active compounds [3–7]. For example, topsentin A (Fig. 1) is a 3,3'-bisindole derivative originating from natural sources like the marine sponge *Topsentina genitrix* [8] that acts as an anti-tumor agent [9].

In recent years, many studies have focused on the functionalization of indoles at the C-3 position because it is the most nucleophilic site. A large number of 3-substituted indoles, especially 3,3'-bis(indolyl)methanes, have been reported [10–14]. However, little attention has been paid to the reactions that involve more than one nucleophilic center, namely C-3, C-2 and N, of the indole ring system. Some exciting reports on this subject include Jiang's work on multicomponent domino reactions resulting in polyfunctionalized indoles [15–17]. Our involvement in the study of indoles and solid acid catalysts [18–20], along with a recent report on three-component formation of bis-indole derivatives by Tu's group [21], prompted us to investigate the use of functionalized magnetic nanoparticles in their protocol to overcome its associated limitations such as acidic media and need for specialized instruments, and the obtained results were satisfying. Functionalized magnetic nanoparticles were selected because they could be easily separated from the reaction mixture by an external magnet. Herein, we report sulfuric acid-functionalized MCM-48 coated on mag-



Fig. 1. Structure of topsentin A.

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ABSTRACT

Magnetite nanoparticles coated with sulfuric acid-functionalized mesoporous MCM-48 were synthesized and used as a catalyst in three-component domino reactions of indoles, arylglyoxal monohydrates and *N*-arylenaminones to furnish the desired 3,3'-bisindoles by formation of two C–C and one C–N bonds in a smooth cascade with good yields under mild reaction conditions. The catalyst was recovered easily and maintained activity in successive runs.

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Scheme 1. Fe₃O₄@MCM-48-OSO₃H-catalyzed three-component reaction of indoles, phenylglyoxal monohydrates and N-arylenaminones.

netite nanoparticles with a core-shell structure (Fe₃O₄@MCM-48-OSO₃H, where Fe₃O₄ nanoparticles serve as a core for a mesoporous MCM-48 shell functionalized with sulfuric acid) as an efficient solid acid nanocatalyst for three-component reaction of indoles, phenylglyoxal monohydrates and *N*-arylenaminones under mild reaction conditions (Scheme 1).

2. Experimental

2.1. Materials

Fe₃O₄ nanoparticles were synthesized according to our previous report [22]. To synthesize Fe₃O₄@MCM-48, the as-prepared Fe₃O₄ nanoparticles (1.5 g) and ammonia solution (5 mL, 25%) were mixed with distilled water (50 mL) in a glass reactor and sonicated for 2 min at 40 °C. Tetraethylorthosilicate (10.0 mL), NaOH (0.90 g) and NaF (0.19 g) were added and then the mixture was stirred for 2 h. Cetyltrimethylammonium bromide (7.0 g) was added to the mixture, and it was stirred at 40 °C for 2 h. The magnetic composite was then hydrothermally treated at 120 °C for 48 h in an autoclave. The resulting solid was filtered, washed with distilled water and dried at 60 °C. Finally, the template was removed by calcination of the synthesized particles for 3 h at 300 °C. SO₃H functionalization of Fe₃O₄@MCM-48 was carried out according to the method of Kiasat et al. [23]. Fe₃O₄@MCM-48 (2.0 g) was charged into a suction flask equipped with a constant-pressure dropping funnel, and dispersed in CH₂Cl₂ (75 mL) by ultrasound for 10 min. Chlorosulfonic acid (2.92 g, 25 mmol) in CH₂Cl₂ (20 mL) was added dropwise over a period of 30 min at room temperature. The mixture was then stirred for 1.5 h, and evolved HCl was removed by suction. Fe₃O₄@MCM-48-OSO₃H was then separated from the reaction mixture using an external magnet, washed several times with CH2Cl2, and then dried under vacuum at 60 °C. The calculated sulfonic acid loading was 2.3 mmol SO₃H per g of catalyst according to a literature method [23].

2.2. Instrumentation

Powder X-ray diffraction (XRD) measurements were performed on a Philips diffractometer with monochromatized Cu K_{α} radiation. Fourier transform infrared (FT-IR) spectra were recorded on a Shimadzu FTIR-8400S spectrometer. ¹H NMR spectra were obtained on a Bruker DRX-400 Advance spectrometer and ¹³C NMR spectra were recorded on a Bruker DRX-100 Advance spectrometer. Chemical shifts of ¹H and ¹³C NMR spectra were expressed in ppm downfield from tetramethylsilane. Melting points were measured on a Büchi Melting Point B-540 instrument and were not corrected. Elemental analyses were performed using a Carlo-Erba EA1110 CNNO-S analyzer and agreed with the calculated values. TEM images were obtained on a transmission electron microscope (TEM; PHILIPS MC 10) with an acceleration voltage of 80 kV. Vibrating sample magnetometry (VSM) curves were obtained on a vibrating sample magnetometer (JDM-13) at room temperature. Analytical gas chromatographic (GC) evaluations of product mixtures were carried out on a Varian CP-3800 chromatograph using a split/splitless injector, CP Sil 8CB column, and FID assembly.

2.3. Typical procedure to prepare 3,3'-bisindoles 1a-1i

In a three-necked round-bottom flask equipped with a reflux condenser, indole (1.00 mmol), phenylglyoxal monohydrate (1.00 mmol), and *N*-arylenaminone (1.00 mmol) were dissolved in refluxing ethanol (10 mL). Fe₃O₄@MCM-48-OSO₃H (0.1 g) was added and the resulting mixture was mechanically stirred until the starting indole completely disappeared (monitored by TLC). After completion of the reaction, the catalyst was removed by an external magnet. The resulting hot solution was quenched with water. The solidified product was filtered, rinsed with a cold mixture of ethanol and water (70:30) and then dried under high vacuum overnight to provide pure product. The recovered catalyst was washed three times with CH₂Cl₂ and then dried under vacuum at 60 °C overnight.

2.4. Characterization data for 6,7-dihydro-6,6-dimethyl-3-(2methyl-1H-indol-3-yl)-1,2-diphenyl-1H-indol-4(5H)-one (**1a**)

White solid, Yield 80%; m.p. 255–256 °C; FT-IR (KBr): v_{max} = 3275, 1640, 1598, 1498, 1460, 1366, 1123, 1075, 787, 739, 706 cm⁻¹. ¹H NMR (400 MHz, DMSO- d_6 , 25 °C) δ = 10.72 (s, 1H, NH), 7.36–7.43 (m, 3H, ArH), 7.28 (s, 2H, ArH), 7.18 (d, *J* = 7.6 Hz, 1H, ArH), 6.87–6.97 (m, 5H, ArH), 6.71–6.77 (m, 3H, ArH), 2.65 (d, *J* = 16.8 Hz, 1H, CH₂), 2.58 (d, *J* = 16.9 Hz, 1H, CH₂), 2.28 (s, 2H, CH₂), 1.90 (s, 3H, CH₃), 1.09 (s, 3H, CH₃), 1.08 (s, 3H, CH₃). ¹³C NMR (100 MHz, DMSO- d_6 , 25 °C) δ = 193.3, 143.7, 137.9, 135.5, 133.8, 133.0, 131.9, 130.0, 129.3, 129.1, 128.0, 127.6, 126.2, 125.9, 120.3, 119.7, 119.2, 118.8, 113.2, 110.1, 106.1, 53.1, 35.0, 29.1, 28.3, 12.4; Anal. Calc for C₃₁H₂₂N₂O (%): C 83.75, H 6.35, N 6.30; Found (%): C 83.81, H 6.39, N 6.29.

All of the other products are known compounds, and their spectroscopic and physical data were identical to those de-

scribed in the literature [21].

3. Results and discussion

Mesoporous MCM-48 was selected as the shell for Fe₃O₄ magnetite nanoparticles because of its three-dimensional channel system and numerous external hydroxyl groups. Deposition of the shell onto the surface of Fe₃O₄ nanoparticles was performed by a simple, low-cost method. Subsequent treatment of the core-shell nanocomposite with chlorosulfonic acid dichloromethane provided the solid acid catalyst in Fe₃O₄@MCM-48-OSO₃H, which was characterized by FT-IR spectroscopy, TEM, XRD and VSM. In the FT-IR spectrum of Fe₃O₄@MCM-48-OSO₃H nanoparticles (Fig. 2(2)), basic characteristic vibrations of Fe-O at 588 cm-1 and Si-O-Si asymmetric stretching, symmetric stretching and bending vibrations at 1072, 795 and 453 cm⁻¹, respectively, were observed. Characteristic bands of the sulfonyl groups were observed at 1229 and 1120 cm⁻¹, along with simultaneous disappearance of the Si-OH peak of Fe₃O₄@MCM-48 at 900 cm⁻¹ [23].

The XRD pattern of $Fe_3O_4@MCM-48-OSO_3H$ (Fig. 3(b)(2)) contained peaks that could be indexed to both mesoporous structure and Fe_3O_4 nanoparticles. The Fe_3O_4 nanoparticles



Fig. 2. FT-IR spectra of (1) Fe $_3O_4@MCM-48$ and (2) Fe $_3O_4@MCM-48-OSO_3H$.



Fig. 4. (a) TEM image of Fe₃O₄@MCM-48-OSO₃H nanoparticles and (b) corresponding particle size distribution.

exhibited peaks at $2\theta = 29.72^{\circ}$, 35.57° , 43.17° , 57.15° and 62.77° , consistent with pure magnetite (Fig. 3(b)(1)) and matched well with the standard XRD pattern of Fe₃O₄ (JCPDS No. 19-692) [24]. The siliceous mesoporous structure exhibited four peaks at $2\theta = 1.5^{\circ}-10^{\circ}$ (Fig. 3(a)(2)) from the (211), (220), (420) and (332) planes, which are characteristic peaks of MCM-48.

The nanoscale size of the composite was confirmed by TEM observation, as shown in Fig. 4(a). An average particle size of 20 nm was determined for $Fe_3O_4@MCM-48-OSO_3H$ according to a particle size distribution analysis (Fig. 4(b)). TEM image indicated that the surface of $Fe_3O_4@MCM-48-OSO_3H$ consisted of an agglomeration of many ultrafine spherical particles with dark magnetite cores each surrounded by a mesoporous shell.

The magnetic hysteresis curves of the $Fe_3O_4@MCM-48-OSO_3H$ magnetic nanoparticles were also obtained. Bare Fe_3O_4 and $Fe_3O_4@MCM-48-OSO_3H$ nanoparticles exhibited typical superparamagnetic behavior. The large saturation magnetization of bare Fe_3O_4 decreased from 82 to 49 emu/g for the $Fe_3O_4@MCM-48-OSO_3H$ nanoparticles because of their non-magnetic mesoporous shell (Fig. 5).

The porosity of the Fe₃O₄@MCM-48-OSO₃H nanoparticles was evaluated by N₂ adsorption-desorption isotherm measurements, which showed a characteristic type-IV curve (Fig. 6). This curve exhibited a distinct hysteresis loop in the relative pressure (p/p_0) range of 0.6–0.9, indicating the presence of mesopores with a narrow size distribution. Other physicochemical properties of the Fe₃O₄@MCM-48-OSO₃H nanoparti-



Fig. 3. XRD patterns of (1) Fe₃O₄ nanoparticles and (2) Fe₃O₄@MCM-48-OSO₃H nanoparticles. (a) Low-angle; (b) High-angle.



Fig. 5. Hysteresis curves of (1) Fe_3O_4 and (2) $Fe_3O_4@MCM-48-OSO_3H$ nanoparticles measured at 27 °C.



Fig. 6. N_2 adsorption-desorption isotherms measured at -196 °C for Fe₃O₄@MCM-48-OSO₃H nanoparticles with core-shell structure.

cles determined from the N₂ adsorption-desorption measurements are a Brunauer-Emmett-Teller surface area of 412.5 m^2/g (calculated in the $p/p_0 = 0-0.5$), mean Barrett-Joyner-Halenda pore diameter of 2.6 nm, total pore volume of 0.417 cm³/g and mean pore volume of 0.401 cm³/g.

Thermogravimetric (TG) analysis of the as-synthesized product (Fig. 7) contained distinct parts: (1) loss of physisorbed water from 80 to 180 °C, which corresponded to the

 $\begin{array}{c}
100 \\
80 \\
40 \\
20 \\
0 \\
100 \\
200 \\
0 \\
100 \\
200 \\
300 \\
400 \\
500 \\
600 \\
Temperature (°C)
\end{array}$

Fig. 7. TG curve for the Fe₃O₄@MCM-48-OSO₃H nanoparticles.

water molecules adsorbed on the external surface and those hosted in the pores, (2) loss of template molecules from 200 to 350 °C and (3) collapse of the $-SO_3H$ functional groups or condensation of silanol groups from 350 to 600 °C. The TG analysis reveals that the obtained catalyst has sufficient thermal stability to endure recycling processes or harsh reaction conditions up to 300 °C.

The characterized catalyst was then used in one-pot three-component condensation reactions of indoles, phenylglyoxal monohydrates and *N*-arylenaminones. To optimize the reaction conditions with respect to catalyst type and loading, and examine the effect of solvent and temperature on the reaction yield, 2-methylindole, phenylglyoxal monohydrate and 5,5-dimethyl-3-(phenylamino)cyclohex-2-enone were used as model substrates and their reactions were carried out under different conditions. The results of these reactions are summarized in Table 1.

Table 1 shows that in the absence of catalyst, the desired product was not formed (entry 1). Although far from giving the maximum yield, it was proved that the optimum loading of Fe₃O₄@MCM-48-OSO₃H under ambient conditions was 0.1 g per mmol of indole (entry 5). The same loading of MCM-48 sulfuric acid (entry 4) was also tested under ambient conditions and gave a comparable yield. However, Fe₃O₄@MCM-48-OSO₃H has the advantage over MCM-48 sulfuric acid in terms of easy recycling. It was found that elevated temperature had a powerful effect on the reaction yield, and an excellent yield of

Table 1

Effect of various parameters on the three-component reaction of indole, phenylglyoxal monohydrate and 5,5-dimethyl-3-(phenylamino)cyclohex-2-enone.

Entry ^a	Catalyst	Solvent	Catalyst loading (g per mmol of indole)	Temperature (°C)	Yield (%)	
1	No catalyst	EtOH	0	25	0	
2	Fe ₃ O ₄	EtOH	0.1	25	0	
3	MCM-48	EtOH	0.1	25	0	
4	MCM-48-OSO ₃ H	EtOH	0.1	25	21	
5	Fe ₃ O ₄ @MCM-48-OSO ₃ H	EtOH	0.1	25	18	
6	Fe ₃ O ₄ @MCM-48-OSO ₃ H	EtOH	0.1	Reflux	80	
7	Fe ₃ O ₄ @MCM-48-OSO ₃ H	EtOH	0.05	Reflux	65	
8	Fe ₃ O ₄ @MCM-48-OSO ₃ H	EtOH	0.15	Reflux	83	
9	Fe ₃ O ₄ @MCM-48-OSO ₃ H	MeOH	0.1	Reflux	77	
10	Fe ₃ O ₄ @MCM-48-OSO ₃ H	1,4-Dioxane	0.1	Reflux	53	
11	Fe ₃ O ₄ @MCM-48-OSO ₃ H	1,2-DCE	0.1	Reflux	19	
12	Fe ₃ O ₄ @MCM-48-OSO ₃ H	Toluene	0.1	Reflux	14	

^a Each reaction was carried out according to the general experimental procedure.

80% was obtained under reflux (entry 6). Meanwhile, higher loads of the catalyst did not have a considerable effect on yield (entry 8). To evaluate the effect of particle size on catalyst performance, larger Fe₃O₄@MCM-48-OSO₃H nanoparticles with a diameter of about 50 nm (Fig 8) were synthesized by manipulation of the hydrothermal synthesis conditions (140 °C, 4 d). However, under the same reaction conditions, the larger particle size of the new catalyst had only a moderate effect on the reaction yield (73% vs 80%). This may be because the $-SO_3H$ functionality plays the main role in this catalytic system. It is noteworthy that the yield of the product depended on solvent, and the highest yields were obtained using ethanol and methanol. Ethanol was selected as the solvent because it is less toxic than methanol.

After determining the optimum reaction conditions (Scheme 1), a series of 2-substituted indoles, phenylglyoxal monohydrates and *N*-arylenaminones were used to investigate the generality and scope of the reaction. As shown in Table 2, the results are satisfying, because the same regioselectivity toward 3,3'-bisindoles was observed in each case.

Table 2 reveals that various indoles, phenylglyoxal monohydrates and *N*-arylenaminones participated in the reaction, and this catalytic system tolerated different functional groups. With regard to substituents, the electron-releasing nature of the aryl moiety in *N*-arylenaminones favors the formation of



Fig. 8. TEM image of larger Fe₃O₄@MCM-48-OSO₃H nanoparticles.

products. However, 1-methylindole did not yield the desired product. These observations may be explained by the following proposed reaction mechanism (Scheme 2).

In general, after activation of the phenylglyoxal monohydrate by the solid Brönsted acid catalyst, nucleophilic attack from the C-3 carbon atom of indole gives intermediates **I**. The next steps are nucleophilic attacks by the β -C atom of β -enaminone to give intermediate **II** and NH group of β -enaminone resulting in C–C and C–N bond formation *via* [3+2] cyclization to provide intermediate **III**, which then dehy-

Table 2

Fe₃O₄@MCM-48-OSO₃H-catalyzed regioselective three-component synthesis of 3,3'-bisindoles.

Entry	Indole	Ar, Ar'	Product ^a		Time (min)	Yield ^b (%)	Entry	Indole	Ar, Ar'	Product ^a		Time (min)	Yield ^b (%)
1	2- methylindole	C6H5, C6H5		1a	25	80	6	2- phenylindole	4-FC ₆ H ₄ , C ₆ H ₅	O N H Ph	1f	25	70
2	2- methylindole	4-ClC ₆ H ₄ , C ₆ H ₅		1b	25	70	7	2- phenylindole	4-MeC ₆ H ₄ , 4-MeC ₆ H ₄		1g	30	80
3	2- methylindole	4-MeC ₆ H ₄ , C ₆ H ₅		1c	25	88	8	2- phenylindole	4-ClC6H4, 4-BrC6H4		1h	25	82
4	2-methylindole	4-MeOC ₆ H ₄ , C ₆ H ₅		1d	30	75	9	2- phenylindole	4-MeC ₆ H ₄ , 4-BrC ₆ H ₄		1i	25	89
5	2-methylindole	4-FC ₆ H ₄ , C ₆ H ₅		1e	25	67	10	1- methylindole	C6H5, C6H5	No reaction		60	0

^a All products were characterized by ¹H NMR, ¹³C NMR and FT-IR spectroscopies. ^b Isolated yields.



Scheme 2. Proposed mechanistic pathway for the formation of 3,3'-bisindoles.

drates to form the product.

To evaluate reusability of the catalyst, the reaction of 2-methylindole, phenylglyoxal monohydrate and 5,5-dimethyl-3-(phenylamino)cyclohex-2-enone was carried out in the presence of the recycled catalyst in successive runs. For reaction runs 1, 2, 3, 4, 5 and 6, the yields were 80%, 80%, 77%, 70%, 63% and 38%, respectively. Therefore, after five runs, the efficiency of the catalyst decreased by 17%. This result shows that Fe₃O₄@MCM-48-OSO₃H can be used as a recyclable catalyst for the formation of 3,3'-bisindoles under moderate conditions. To confirm heterogeneity of the catalyst, the sulfonic acid

loading was determined for the recycled catalyst used in the third run. The calculated value was 2.3 mmol SO₃H/g, similar to that of the unused catalyst, which confirmed that considerable leaching did not occur during the course of the reaction. To further strengthen this assumption, the reaction of 2-methylindole, phenylglyoxal monohydrate and 5,5-dime-thyl-3-(phenylamino)cyclohex-2-enone was interrupted half-way during the normal reaction period (after 12.5 min) and the catalyst was removed by an external magnet. The reaction was then continued for the rest of the time (up to 25 min). At the end of this period, the yield was only 39%, and no considerable change in the concentration of residual 2-methylindole was observed according to GC analysis of the reaction mixture.

4. Conclusions

We developed a convenient method for one-pot three-component reaction of indoles, phenylglyoxal monohydrates and *N*-arylenaminones under mild conditions. Highlights of the present work are regioselective one-pot formation of two C–C and one C–N bonds in a smooth cascade, recyclable catalyst, which promises minimization of waste, ease of work-up and good efficiency in terms of solvent, temperature and yield. Further manipulation of this reaction is currently underway in our laboratory.

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Graphical Abstract

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Efficient synthesis of 3,3'-bisindoles catalyzed by Fe₃O₄@MCM-48-OSO₃H magnetic core-shell nanoparticles

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Magnetite nanoparticles coated with sulfuric acid-functionalized mesoporous MCM-48 are used as a catalyst in green synthesis of 3,3'-bisindoles *via* formation of two C–C and one C–N bonds in a smooth cascade with good yields under mild reaction conditions.

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