

Fe₂O₃@MCM-BP Hybrid Nanocomposite as an Alkaline Magnetic Catalyst in the One-pot Preparation of Various Derivatives of Pyrano [2,3-C] Pyrazole under Solvent-free Conditions

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In this paper, bi-pyridinium chloride supported on the core-shell mesoporous substrate, Fe₂O₃@MCM was prepared as an organic-inorganic magnet nanocomposite. The prepared hybrid nanocomposite, Fe₂O₃@MCM-BP, was characterized using FT-IR spectroscopy, X-ray diffraction, scanning electron microscopy, transmission electron microscopy, thermal gravimetric analysis, and vibrational sample magnetometry techniques. The efficacy of the nanocomposite as an alkaline heterogeneous magnetic catalyst in the one-pot multicomponent condensation reaction for preparation of various derivatives of pyrano [C-2,3] pyrazole under solvent-free conditions was investigated. High efficiency, ease of preparation and reusability are some the features of this catalyst.

Keywords: Organic-inorganic nano-magnetic composite, MCM-41, Fe₂O₃@MCM-BP, Pyrano [C-2,3] pyrazole

INTRODUCTION

Pyranopyrazoles are heterocyclic compounds resulting from the conjugation of Pyran and Pyrazole units. These compounds have been the subject of expert attention for medicinal properties such as anti-allergic, anti-microbial [1], anti-cancer and anticoagulant, diuretic, anti-spasmodic drugs [2], and biological applications such as insecticides [3] since the 1980s [4]. This class of hetrocyclic compounds are synthesized through two-component condensation reactions [5], three-component condensation reactions [6] as well as four-component condensation reactions involving malononitrile, aryl aldehydes, ethyl acetoacetate and hydrazine [7].

Various catalysts have been previously reported for the preparation of Pyranopyrazoles including Cs-Pr-Me [8], Ag/TiO₂ [9], DABCO [10], triethyl ammonium acetate [11], zwitterionic sulfamic acid functionalized nanoclay [12], morpholine triflate [13], [nano-Fe₃O₄SiO₂(CH₂)₃-imidazole-

SO₃H]Cl [14], magnetized silica with Ni-Fe₂O₃ nanoparticles (NPs) on H₃PW₁₂O₄ [15], triphenyl phosphine [16], γ -alumina [17], PEG-400 [18], ZnAl₂O₄ NPs [19] and cerium ammonium nitrate [20]. Although the methods reported provide good results, the long reaction time, high temperature, harsh reaction conditions as well as the difficult separation of the catalyst from the reaction mixture are limitations of the above methods.

MCM-41 is a group of mesoporous materials that their application as an inert shell for coating magnetic NPs has flourished due to its prominent properties such as having regular cavities, high pores and surface area, large number of hydroxy groups, and high thermal, chemical and mechanical stability [21]. Considering the MCM-41's properties and the importance of performing multicomponent reactions in the synthesis of heterocyclic compounds [22-24], in this study, we examine the ability of the Fe₂O₃@MCM-BP hybrid nanocomposite that we previously demonstrated its catalytic ability in one pot Hantzsch condensation reaction [25], in one pot four condensation reaction of aromatic aryl aldehydes,

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phenylhydrazine or hydrazine, malononitrile and ethyl acetoacetate for the synthesis of pyrano [C-2,3] pyrazoles under solvent-free conditions.

EXPERIMENTAL

Chemical Materials and Apparatus

All the materials were bought from Merck and Aldrich chemical companies. The reaction progress was followed by thin-layer chromatography. The Fourier transform infrared spectra (FT-IR) were recorded by a FT-IR spectrometer (Perkin-Elmer BX-II). The Philips X-ray diffractometer (Model PW 1840) was used to record X-ray diffraction (XRD) patterns. The scanning electron microscope (SEM, Philips-X230) and transmission electron microscope (TEM, model Philips CM200) were utilized to assess particles' size and shape. A vibrating sample magnetometer (Meghnatis Daghigh Kavir Co Iran) was applied to estimate magnetization. Thermal gravimetric analysis (TGA) was determined using a Perkin Elmerpyris System. In addition, the products were detected using FT-IR, melting points and the nuclear magnetic resonance (NMR-Bruker-Avance-300 MHz).

Preparation of the Magnetic Fe₃O₄ Nanoparticles

In this study, iron oxide NPs were selected as the magnetic core and prepared by chemical coprecipitation method. To prepare super-paramagnetic iron oxide NPs, to a solution of FeCl₂·4H₂O (3.1736 g, 16 mmol) in deionized water (320 ml), FeCl₃·6H₂O (7.57 g, 28 mmol) was added. After 1 h stirring, the mixture under the nitrogen atmosphere at 80 °C, 40 ml ammonium hydroxide (25%) was rapidly added to the reaction mixture. The solution was stirred in the presence of nitrogen flux at 80 °C for 1 h and then cooled to room temperature. The precipitated particles were washed five times with hot water and collected with an external magnet. Lastly, the nano-Fe₃O₄ was dried under vacuum at 70 °C.

Preparation of MCM- 41 Coated Magnetic Nanoparticles (Fe₂O₃@MCM NPs)

In a 250 ml flask, to the solution of CTAB (1.67 g) in deionized water (33 ml), aqueous ammonia solution (140 mmol, 25%) and ethanol (40 ml) were added and stirred to give a uniform solution. After 20 min, nano-Fe₃O₄

(0.5 g) and tetraethylortosilicate (3.14 g) were added to the reaction mixture and stirred for 2 h in an ultrasonic bath at ambient temperature. The obtained precipitate was then filtered and dried at 100 °C for 48 h in an oven and was calcined for 6 h at 540 °C in atmospheric pressure (Fig. 1).

Functionalization of Fe₂O₃@MCM with n-Propyl Chloride (Fe₂O₃@MCM-Cl)

To the suspension of Fe₂O₃@MCM-41 (2 g) in anhydrous toluene (30 ml), 3-chloropropyl triethoxysilane (4 ml) was added and refluxed for 24 h. After the reaction was completed, the precipitate was filtered and washed with toluene, ethanol, and deionized water and was dried at 100 °C for 24 h in vacuum oven (Fig. 2).

Preparation of Fe₂O₃@MCM-BP Nanocomposite

To the solution of 4,4'-bipyridine (1.8 mmol) in anhydrous toluene (30 ml), 2 g of Fe₂O₃@MCM-Cl was added and refluxed for 24 h. The precipitate was filtered and washed with anhydrous toluene and dried in vacuum oven at 50 °C for 6 h (Fig. 3).

A General Method for the Preparation of Dihydropyrano [C-2,3] Pyrazoles Derivatives

A mixture of aromatic aldehydes (1 mmol), phenylhydrazine or hydrazine (1 mmol), ethyl acetoacetate (1 mmol), malononitrile (1 mmol) and 0.005 g of Fe₂O₃@MCM-BP catalyst was stirred at 100 °C in oil bath. The reaction progress was followed by thin-layer chromatography. After completion of the reaction, 10 ml of ethanol was added to the reaction mixture and heated for 2 min. The catalyst was extracted by an external magnet and the solvent was evaporated. The precipitate was recrystallized with water-ethanol mixture and characterized using techniques such as melting point, FT-IR, ¹H NMR and ¹³C NMR (Tables 4 and 3).

RESULTS AND DISCUSSION

The Fe₂O₃@MCM-BP nanocomposite was prepared and characterized according to our previously reported method [25]. At the first step of the Fe₂O₃@MCM-BP nanocomposite preparation, super-paramagnetic iron oxide NPs were prepared by co-precipitation reaction of Fe²⁺ and Fe³⁺ salts in ammonia solution [22]. Then, Fe₂O₃

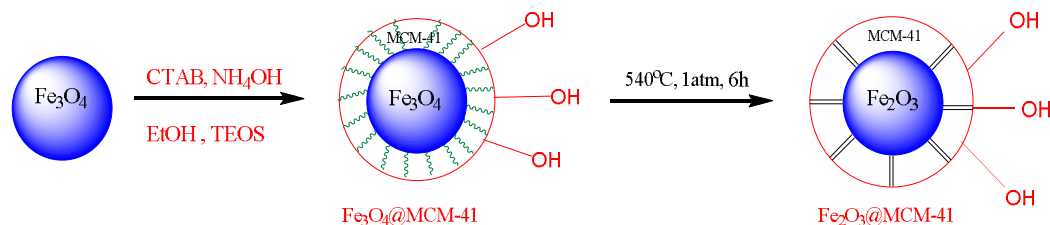


Fig. 1. Preparation of Fe₂O₃@MCM- 41.

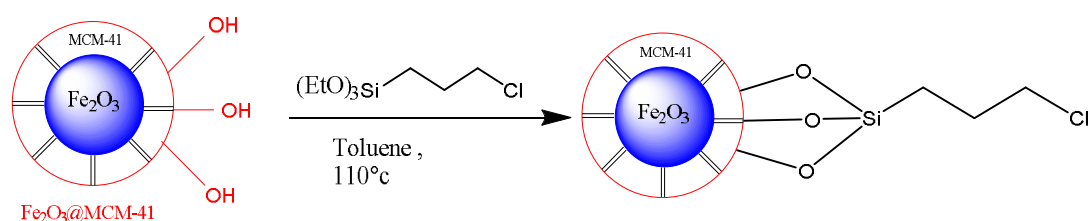


Fig. 2. Preparation of Fe₂O₃@MCM-Cl.

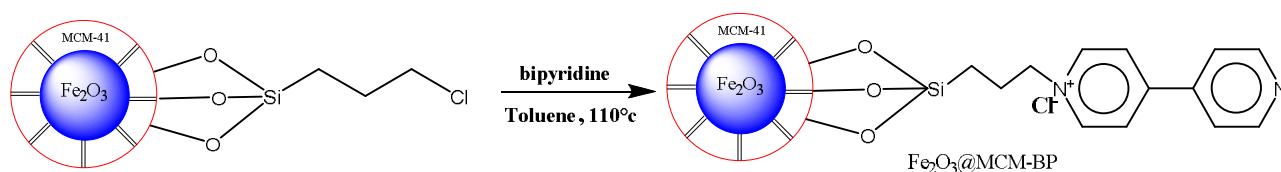


Fig. 3. Preparation of Fe₂O₃@MCM- BP.

nanoparticles was coated by MCM-41 during sol-gel process by polycondensation reaction of tetraethoxysilane (TEOS) as the silicate source in the presence of surface active template (CTAB) in the alkaline media (Fig. 1).

After elimination of CTAB template by calcination, in the next step, 3-chloropropyltriethoxysilane reacted with hydroxyl groups on the surface of MCM-41 shell (Fig. 2).

In the last step, 4,4'-bipyridine supported on the surface of the nanocomposite by simple nucleophilic substitution reaction (Fig. 3).

Instrumental techniques such as infrared spectroscopy (FT-IR), X-ray diffraction (XRD), images of electron microscopy (TEM and SEM), thermal gravimetric analysis (TGA), and vibrational sample magnetometry were used to confirm the structure of the Fe₂O₃@MCM-BP nanocomposite.

The FT-IR spectra of the Fe₂O₃@MCM-41 and

Fe₂O₃@MCM-BP are shown in Fig. 4. The adsorption bands at 400 to 580 cm⁻¹ and 1627 cm⁻¹ are related to the bending vibrations of Fe-O and absorbed water, respectively. The observed peaks at 469 cm⁻¹, 801 cm⁻¹ and 1098 cm⁻¹ ranges are respectively related to the bending, symmetric and asymmetric stretching vibrations of the Si-O-Si bonds of the silicate coating around the Fe₂O₃ magnetic nanoparticles [23].

In addition, the observed adsorptions at 1443 and 1640 cm⁻¹ are related to C=C and C=N vibrations of bipyridine unites, respectively, and the adsorption of right side of 3000 cm⁻¹ are related to the aliphatic C-H stretching vibrations and the left side of 3000 cm⁻¹ are related to the aromatic C-H stretching vibrations.

Figure 5 shows the X-ray diffraction spectra in the low and high angles for the Fe₂O₃@MCM-BP nanocomposite. The broad peak appeared at d₁₀₀ = 2.7 demonstrated the lack

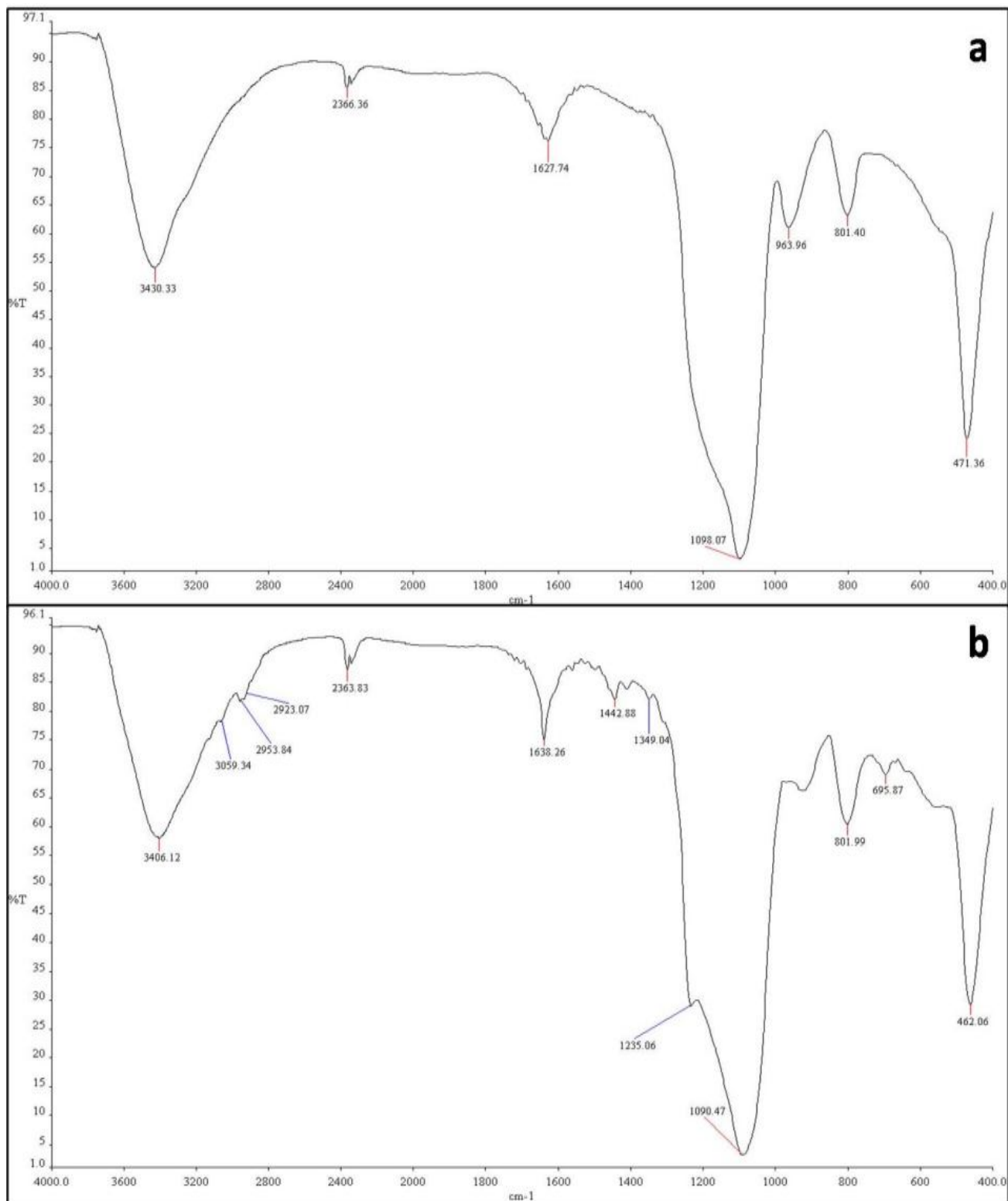


Fig. 4. FT-IR spectra of (a) Fe₂O₃@MCM-41 and (b) Fe₂O₃@MCM-BP.

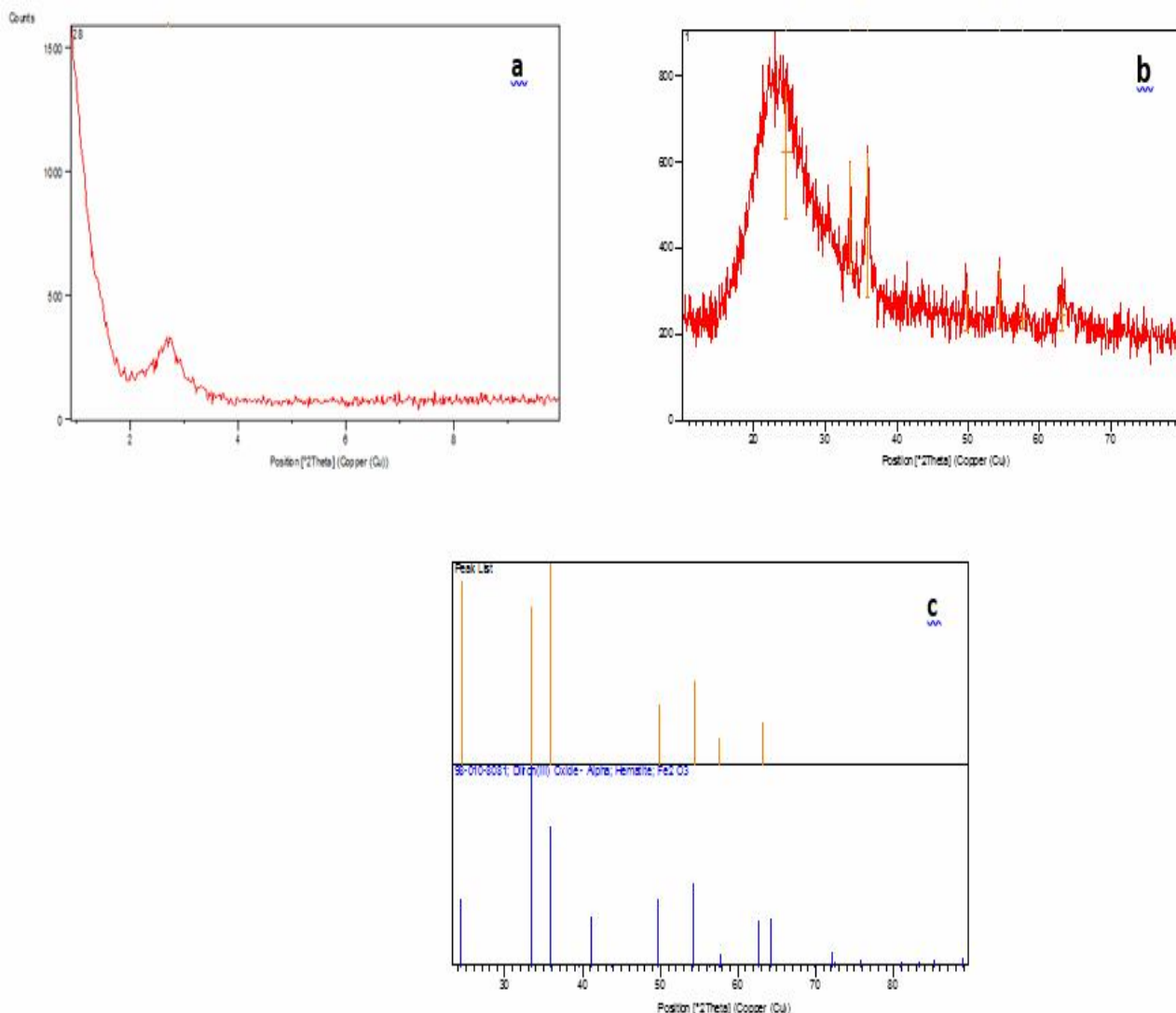


Fig. 5. XRD spectra of Fe₂O₃@MCM-BP nanocomposites (a) at low angle, (b) at high angle, and (c) its comparison with reference Fe₂O₃.

of crystalline structure in the nanocomposite. Since no peaks were observed at d_{110} and d_{200} , the catalyst should be a hexagonal amorphous with spherical shape, leading to the conclusion that the silicate coating around the Fe₂O₃NPs remains unchanged after calcination [24].

According to Fig. 5c, the distinct diffraction peaks at 2θ values 63.26, 57.67, 54.42, 49.82, 36.93, 33.49 and 24.49 were parallel well with the standard XRD data for magnetite (JCPDS, file no. 8081-010-98), and therefore, the presence of Fe₂O₃ in the nanocomposite is confirmed.

Figure 6 shows the SEM and TEM images of Fe₂O₃@MCM-BP which signifies that the synthesized nanocomposite is spherical in shape. According to the SEM image, however, the particles have aggregated due to the magnetic dipolar interaction; the size of the nanoparticles is under 100 nm. The darker spots at TEM images also appear in the presence of Fe₂O₃ NPs core inside the SiO₂ shell.

The thermal gravimetric analysis (TGA) of catalyst was also used to determine the content of organic functional groups of the sample and its thermal stability (Fig. 7). The

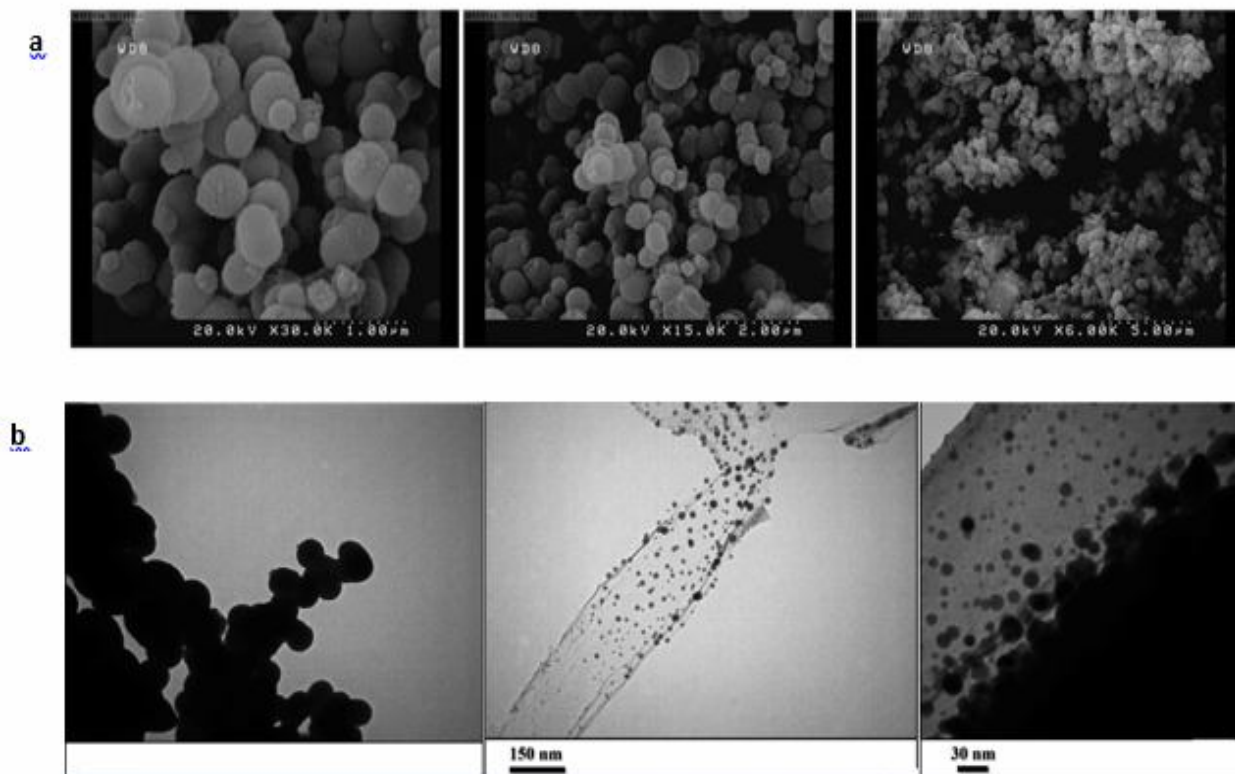


Fig. 6. (a) SEM and (b) TEM of Fe₂O₃@MCM-BP nanocomposite.

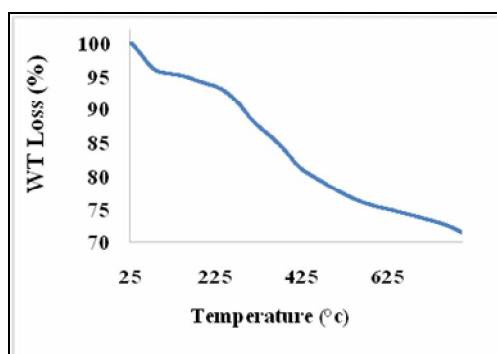


Fig. 7. TGA diagram of Fe₂O₃@MCM-BP nanocomposite.

TGA curve showed that the emission mass fraction of water was about 6.5% when the temperature was less than 200 °C and the other decrease in weight was 20% in the temperature range from *ca.* 200 to 470 °C should be ascribed to the decomposition of organic groups grafted to the MCM surface during hydrothermal treatment. It could

easily be seen that the total content of the organic units in Fe₂O₃@MCM-BP is about 0.2 g g⁻¹ of the nanocomposite (0.78 mmol of organic unit per g of nanocomposite).

The magnetic property of Fe₂O₃@MCM-BP nanocomposite has been investigated by VSM at ambient temperature (Fig. 8), showing that the saturated

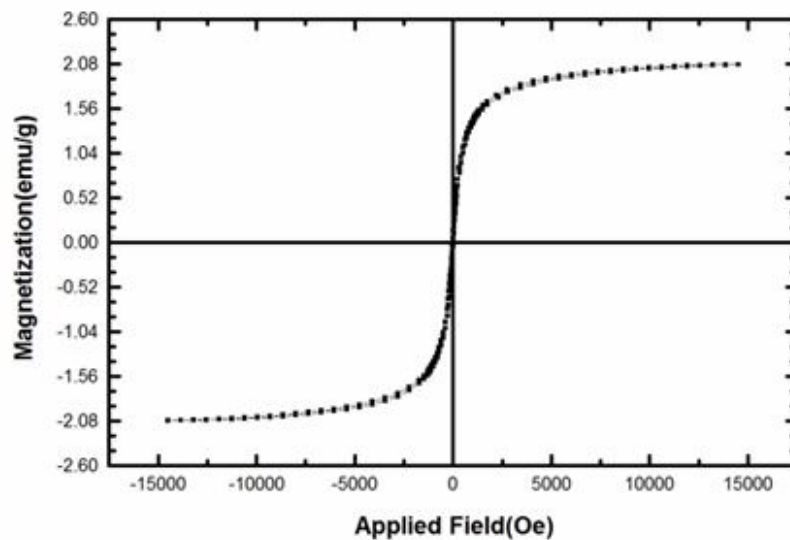


Fig. 8. VSM of Fe₂O₃@MCM-BP nanocomposite.

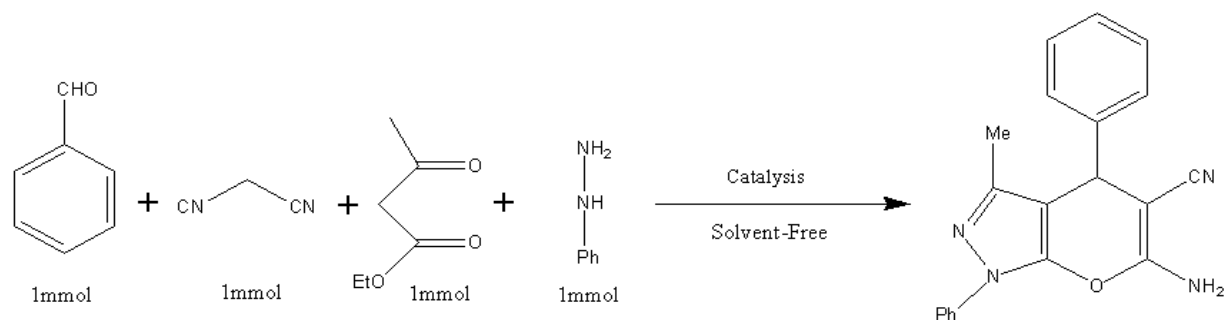


Fig. 9. Model reaction.

Table 1. Optimization Conditions for the Synthesis of Pyranopyrazoles under Solvent-free Conditions

Entry	Catalyst (g)	Temperature (°C)	Time (min)	Yield (%)
1	0.0001	60	50	68
2	0.0001	100	35	73
3	0.0005	60	30	78
4	0.0005	100	20	85
5	0.005	60	13	83
6	0.005	100	6	90
7	0.004	100	9	90

magnetization values is 2.1 emu g^{-1} . A diminish in saturation magnetization of Fe_2O_3 nanoparticle could be ascribed to the presence of nonmagnetic shell around the magnetic core.

After characterization of the desired nanocomposite structure, its catalytic activity in the one-pot multicomponent synthesis of pyrano [C-2,3] pyrazoles derivatives was investigated. In order to optimize the conditions, the reaction of benzaldehyde, malononitrile, ethyl acetoacetate and phenylhydrazine with molar ratios of 1:1:1:1 in the presence of different amounts of catalyst and at different temperatures under solvent free conditions was selected as the model reaction. According to the Table 1 and Fig. 9 the best conditions for completing the reaction were 0.005 g of $\text{Fe}_2\text{O}_3@\text{MCM-BP}$ nanocomposite containing 0.004 mmol of organic units (0.4 mol% of BP) at 100°C . As shown in Table 1, decreasing the catalyst amount and temperature leads to decrease in the product yield.

To explore the scope and generality of the present MCR protocol for the synthesis of pyrano [C-2,3] pyrazoles derivatives under the optimized conditions, a variety of aromatic aldehydes containing electron donating or electron withdrawing substituents were reacted with phenylhydrazine or hydrated hydrazine, ethyl acetoacetate and malononitrile. It was found that both electron-rich and electron-deficient aldehydes reacted well, mainly leading to good yields of target molecules (Tables 2 and 3).

The reusability of the catalyst was also tested. For doing so, after completion of the reaction, the $\text{Fe}_2\text{O}_3@\text{MCM-BP}$ nanocomposite was easily separated from the reaction mixture by means of an external magnetic field. The mixture was washed with water and diethyl ether and then dried and reused for five times. As shown in Fig. 10, the average chemical yield for five consecutive runs of the condensation reaction of benzaldehyde, malononitrile, ethyl

Table 2. Preparation of 6-Amino-4-aryl-5-cyano-3-methyl-1-phenyl-1,4-dihydropyrano [C-2,3] Pyrazol Compounds by Condensation Reaction between Arylaldehydes, Ethyl Acetoacetate, Malononitrile and Phenylhydrazine in the Presence of $\text{Fe}_2\text{O}_3@\text{MCM-BP}$ Nanocomposite under Solvent Free Conditions and at 100°C

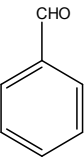
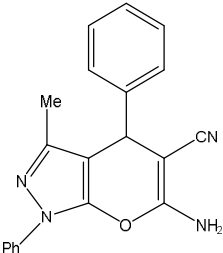
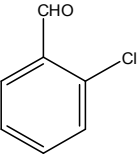
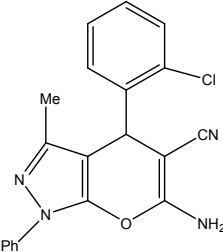
Entry	Aldehyde	Product	Time (min)	Yield (%)	M. P ($^\circ\text{C}$)	
					Observed	Reported [Rf.]
1			6	90	165-166	168-170 [35]
2			5	88	147-148	142-144 [36]

Table 2. Continued

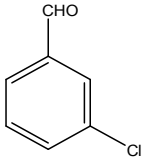
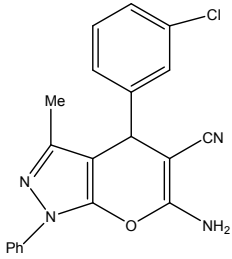
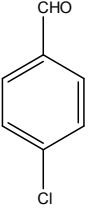
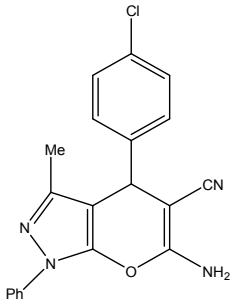
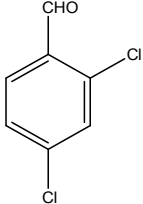
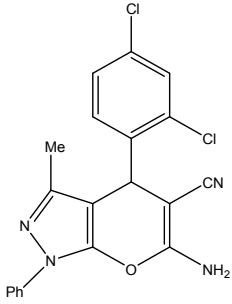
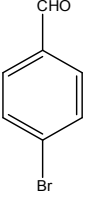
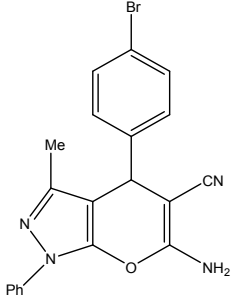
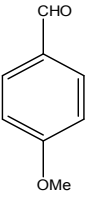
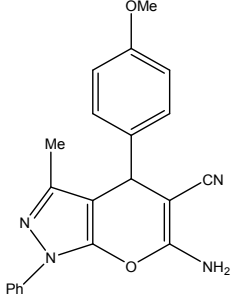
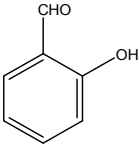
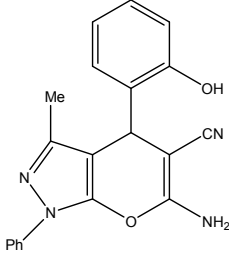
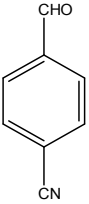
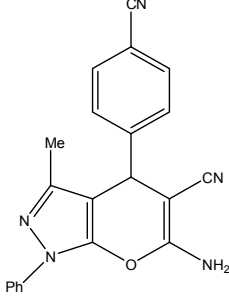
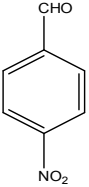
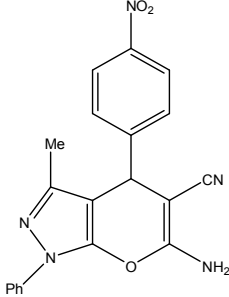
3			5	92	157-158	158-161 [37]
4			5	93	169-170	171-172 [37]
5			8	93	184-186	180-182 [38]
6			15	90	179-182	183-184 [35]
7			20	89	170-173	172-173 [39]

Table 2. Continued

8			25	88	201-204	216-217 [40]
9			6	93	195-197	212-214 [40]
10			5	93	198-202	188-190 [39]

acetoacetate and phenylhydrazine was 87% after 6 min which clearly demonstrates the practical recyclability of this catalyst.

In order to evaluate the stability of the catalyst in the reaction conditions, the FT-IR and SEM of recycled nanocomposite were compared with fresh nanocomposite. The FT-IR and SEM records of the recycled catalyst was exactly the same as the fresh catalyst.

To compare the specifications of the synthesized catalyst with the reported catalysts, we have shown the results and the reaction conditions in the condensation of benzaldehyde, hydrated hydrazine, ethyl acetoacetate and malononitrile in Table 4. Accordingly, Fe₂O₃@MCM-BP was superior, particularly regarding the reaction time and yield compared

to the other catalysts.

CONCLUSIONS

A novel, eco-friendly, efficient and reusable magnetically heterogeneous nanocatalyst, Fe₂O₃@MCM-BP, was efficiently produced, characterized and used to generate pyrano [C-2,3] pyrazoles. This method has advantages compared to previous methods of preparing pyrano [C-2,3] pyrazoles. Eco-friendly character and recyclability of the catalyst go towards the development of a greener product. Furthermore, this strategy renders distinct advantages, containing high yields, simplicity in operation, clean reaction conditions and being eco-friendly that make

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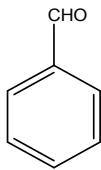
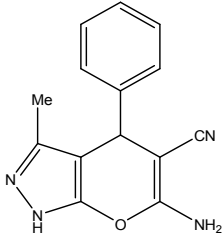
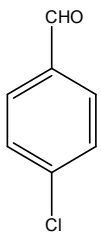
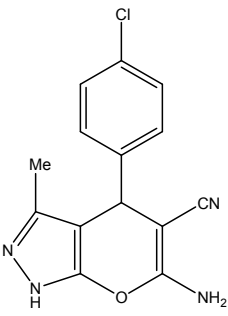
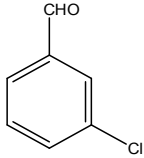
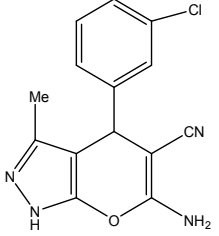
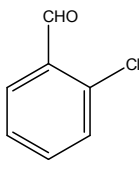
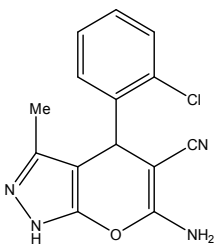
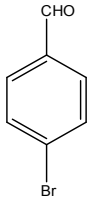
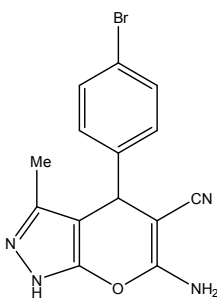
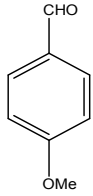
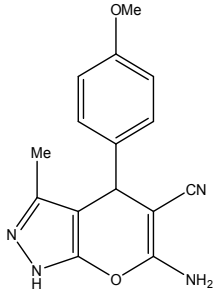
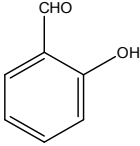
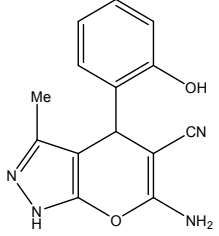
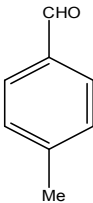
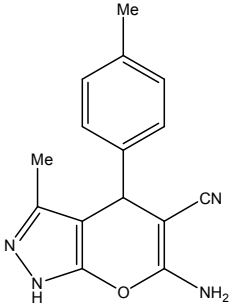
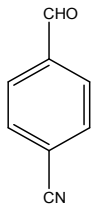
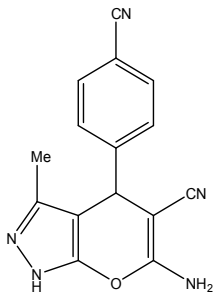
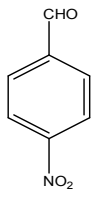
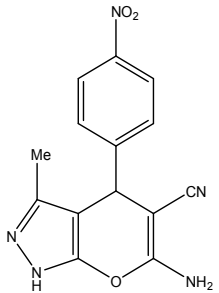
Entry	Aldehyde	Product	Time (min)	Yield (%)	M. P. (°C)	
					Observed	Reported [Rf.]
1			3	90	240-242	257-260 [41]
2			3	92	236-238	232-234 [39]
3			4	90	240-242	246-248 [42]
4			5	89	236-240	255 [36]
5			3	92	174-176	180-183 [43]

Table 3. Continued

6			10	98	213-215	210-212 [39]
7			8	91	202-204	208-210 [43]
8			6	95	208-210	207-209 [40]
9			3	91	215-220	230-240 [41]
10			2	93	248-250	250-252 [39]

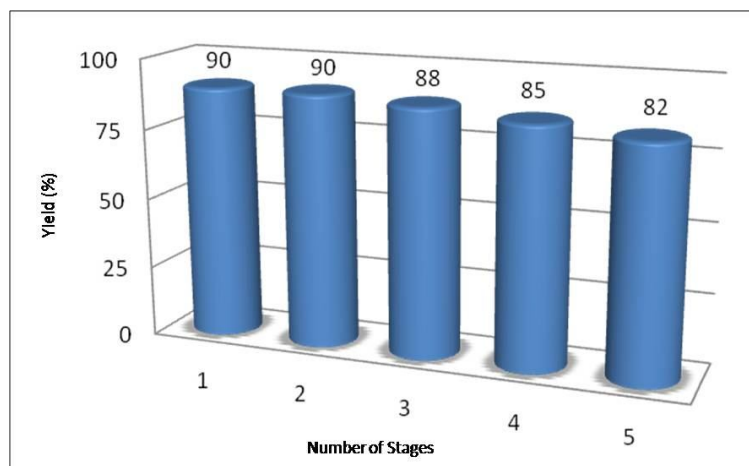


Fig. 10. Reusability study of nanocomposite in the condensation reaction of benzaldehyde, malononitrile, ethyl acetoacetate and phenylhydrazine.

Table 4. Comparing the Results and the Reaction Conditions of Fe₂O₃@MCM-BP in the Condensation of Benzaldehyde, Ethyl Acetoacetate, Malononitrile and Hydrazine

Entry	Catalyst (g)	Reaction Condition	Time (min)	Yield (%)	Ref.
1	Urea (10 mol%)	H ₂ O:EtOH (1:1), r.t.	480	86	[29]
2	Molecular sieves 4 Å	,EtOH/ultrasoni	30	85	[30]
3	Zwitterionic sulfamic acid functionalized nanoclay	S.F.90 °C	30	90	[12]
4	Uncapped SiO ₂ quantum dot	H ₂ O, r.t.	90	90	[31]
5	Cetyltrimethyl ammonium chloride (20 mol%)	H ₂ O, r.t.	340	80	[32]
6	Ag/TiO ₂	H ₂ O-EtOH, 70 °C	55	90	[9]
7	Triphenyl(3-sulfopropyl) phosphonium trinitromethanide	S.F.60 °C	20	90	[33]
8	Morpholine triflatet (10 mol%)	EtOH, H ₂ O reflux	60	70	[34]
9	Fe ₂ O ₃ @MCM-BP	S.F. 100 °C	3	90	This work

it a beneficial and effective technique for the synthesis of pyrazol scaffolds.

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