# Synthesis of 1,3,5-Trisubstituted Pyrazoles and Hydrazones Using Fe<sub>3</sub>O<sub>4</sub>@CeO<sub>2</sub> Nanocomposite as an Efficient Heterogeneous Nanocatalyst

H. Hassani<sup>*a*,\*</sup> and Z. Jahani<sup>*a*</sup>

<sup>a</sup> Department of Chemistry, Payame Noor University, Tehran, 19395-4697 Iran \*e-mail: hassaniir@yahoo.com

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Abstract—Pyrazoles and hydrazones, as two significant kinds of potentially bioactive compounds, were produced with good to excellent yields by condensation of  $\beta$ -dicarbonyl compounds with hydrazines in aqueous media in the presence of Fe<sub>3</sub>O<sub>4</sub>@CeO<sub>2</sub> nanocomposite as an efficient heterogeneous nanocatalyst. The magnetic nanocatalyst can readily be separated using an external magnet and reused at least six times without significant loss in activity. The products were characterized by IR and <sup>1</sup>H and <sup>13</sup>C NMR spectra.

Keywords: Fe<sub>3</sub>O<sub>4</sub>@CeO<sub>2</sub> nanocatalyst, nanocomposite, pyrazole, hydrazone

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Acid catalysts such as nitric, sulfuric, phosphoric, and hydrochloric acids play an important role in organic chemistry. However, inability to reuse, difficult separation, and corrosive properties are disadvantages of these homogeneous catalysts. Therefore, the design of novel recyclable heterogeneous catalysts tolerating a wide range of temperatures and pressures and minimizing waste product is necessary [1–3]. Among heterogeneous catalysts, magnetic nanoparticles containing Fe<sub>3</sub>O<sub>4</sub> are highly regarded because of their desirable magnetic properties, high recycling capability, easy handling, low cytotoxicity, chemically modifiable surface, and high commercially availability [4, 5].

Due to the increasing interest in the extension of environmentally safe reactions, multicomponent reactions (MCRs) were presented for merging environmental aspects to economic aspects of new reactions. Multicomponent reactions are one-pot reactions involving three or more reactants to produce a favorable product in a single flask and are characterized by mild reaction condition, high yield, atom economy, simplicity, and cost efficiency [6, 7].

One example of MCRs is the synthesis of pyrazoles. Pyrazoles and their derivatives have gained much attention in organic synthesis, because they have significant therapeutic and biological values. In particular, they are introduced as anti-inflammatory, antitumor, antiparasitic, and antibacterial agents [8–11]. Pyrazole derivatives are part of the core of various biologically active compounds. Analgesic, antidiabetic, antiphlo-



Scheme 1.

**1**,  $R^1 = R^2 = Me$  (**a**),  $R^1 = Me$ ,  $R^2 = OEt$  (**b**),  $R^1 = R^2 = Ph$  (**c**); **2**,  $R^3 = H$  (**a**), Ph (**b**),  $4\text{-ClC}_6H_4$  (**c**); **3**,  $R^1 = R^2 = Me$ ,  $R^3 = H$  (**a**), Ph (**b**),  $4\text{-ClC}_6H_4$  (**c**); **a**,  $R^1 = R^2 = Me$ ,  $R^3 = H$  (**a**),  $R^1 = Me$ ,  $R^3 = Ph$  (**e**),  $4\text{-ClC}_6H_4$  (**f**);  $R^1 = R^2 = R^3 = Ph$  (**g**),  $R^1 = R^2 = Ph$ ,  $R^3 = 4\text{-ClC}_6H_4$  (**h**); **5**,  $R^1 = R^2 = Me$  (**a**),  $R^1 = Me$ ,  $R^2 = OEt$  (**b**),  $R^1 = R^2 = Ph$  (**c**).



Fig. 1. Vibrating sample magnetometry pattern of  $Fe_3O_4@CeO_2$  magnetic nanoparticles.



Fig. 2. EDS pattern of Fe<sub>3</sub>O<sub>4</sub>@CeO<sub>2</sub> magnetic nanoparticles.



Fig. 3. XRD pattern of Fe<sub>3</sub>O<sub>4</sub>@CeO<sub>2</sub> magnetic nanoparticles.



Fig. 4. (a) TEM and (b) FESEM patterns of  $Fe_3O_4@CeO_2$  magnetic nanoparticles.

gistic, and many other modern drugs contain a pyrazole ring as structural segment [12–14].

Imines are important intermediates in the synthesis of biologically active N-heterocyclic compounds such as fragrances,  $\beta$ -lactams, agricultural chemicals, pharmaceuticals, and dyes [15–18]. Due to the presence of lone electron pair on the imine nitrogen atom, particularly when this atom is located at the *ortho* position of an aromatic heterocycle, imines can coordinate to metals and the resulting complexes can be used as catalysts [19, 20].

In continuation of our studies of the synthesis of recyclable and effective nanocatalysts and their use in the preparation of biologically important compounds [21-25], in this work we surveyed the effect of Fe<sub>3</sub>O<sub>4</sub>@CeO<sub>2</sub> nanocomposite in one-pot synthesis of pyrazoles and hydrazones by the condensation of hydrazines/hydrazides with dicarbonyl compounds. The proposed method does not require removal of water and the use of any drying agent because the reaction is carried out in aqueous solution. Eventually, pyrazole and hydrazone derivatives were produced with good to excellent yields (80–98 %) in aqueous medium (Scheme 1).

The catalyst,  $Fe_3O_4@CeO_2$  magnetic nanoparticles, was prepared in two steps: (1) Colloidal iron oxide magnetite nanoparticles ( $Fe_3O_4$  MNPs) were prepared by the reaction of  $FeCl_2 \cdot 4H_2O$  and  $FeCl_3 \cdot 6H_2O$  with sodium hydroxide in deionized water; (1) Cerium(IV) oxide was incorporated as functional groups on the surface of ferrite nanoparticles by treatment of  $Fe_3O_4$  MNPs with Ce(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O for 12 h to give  $Fe_3O_4@CeO_2$  MNPs.

The catalyst was characterized by several techniques. Figure 1 shows the magnetization plot versus magnetic field for  $Fe_3O_4@CeO_2$  nanoparticles, which was obtained by vibrating sample magnetometry (VSM) at room temperature (300 K). The  $Fe_3O_4@CeO_2$  nanoparticles exhibited superparamagnetic behavior with a saturation magnetization (Ms) of 28.97 emu/g and without magnetic hysteresis area coercivity and remnant magnetization.

The energy-dispersive X-ray spectroscopy (EDS) pattern of  $Fe_3O_4@CeO_2$  MNPs was recorded to investigate their elemental composition (Fig. 2). It showed peaks of cerium, oxygen, and iron of asprepared nanoparticles.

The X-ray powder pattern recorded for a sample of  $Fe_3O_4@CeO_2$  MNPs is shown in Fig. 3. It contains several relatively strong peaks in the region 20–80°, which are quite similar to those observed for other

Fe<sub>3</sub>O<sub>4</sub> nanoparticles,  $2\theta = 30.1$ , 35.5, 43.1, 57.1, 62.7, 66, 74.1, and 75.4°. These peaks were assigned to the (523), (1000), (260), (342), and (598) faces. The pattern indicates a crystalline structure with the (184), (144), and (157) crystallographic faces of magnetite consistent with the standard pattern. Extra reflections are not detected in the XRD pattern. The average crystallite size *D* was estimated at 11.96–14 nm by using Scherrer's equation  $D = K\lambda/(\beta \cos \theta)$ , where K = 0.9 is a shape factor,  $\beta$  is the full width at half maximum (FWHM) of the peaks of all planes in the XRD pattern,  $\lambda = 1.5406$  Å is the wavelength of Cu target, and  $\theta$  is the Bragg angle.

The TEM and FESEM images of  $Fe_3O_4@CeO_2$ MNPs are shown in Fig. 4. The results showed that the average particle size of  $Fe_3O_4@CeO_2$  MNPs is 19–28 nm which is consistent with the XRD data.

Figure 5 shows the thermal behavior of  $Fe_3O_4@CeO_2$  nanoparticles. The TGA curve showed weight loss in the region between 0 and 800°C. Weight loss in the region between 24 and 445°C was attributed to decomposition of organic compounds and evaporation of absorbed water. The first weight loss in the region 24–221°C corresponds to vaporization of water adsorbed on the surface of the composite material. The second weight loss takes place between 221 and 445°C due to vaporization of gaseous nitrates and ammonia from the material. There was no weight loss above 440°C. Thus, the prepared  $Fe_3O_4/CeO_2$  material is quite thermally stable above that temperature.

IR spectroscopy was used to monitor successful loading of CeO<sub>2</sub> on the surface of Fe<sub>3</sub>O<sub>4</sub>. As shown in Fig. 6a, the peak about 3500 cm<sup>-1</sup> corresponds to vibrations of OH groups. The peak at 609 cm<sup>-1</sup> is attributed to Fe–O bending vibrations. The FTIR spectrum of Fe<sub>3</sub>O<sub>4</sub>@CeO<sub>2</sub> (Fig. 6b) showed a peak at 431 cm<sup>-1</sup> due to Ce–O stretching and bending vibrations of CeO<sub>2</sub>.

To investigate the catalytic activity of  $Fe_3O_4@CeO_2$ nanocomposite, substituted hydrazines (acetohydrazide) (1 mmol) were reacted with diketones/ $\beta$ -keto esters (1 mmol) in water in the presence of the nanocomposite (0.005 g) as catalyst at room temperature to produce pyrazoles and imines (Scheme 1). To define the optimal conditions, the synthesis of 3,5-dimethyl-1*H*-pyrazole (**3a**) as model was carried out in various solvents (Table 1) and in the presence of various amounts of the catalyst (Table 2). All reactions were stopped at 5 min, and the approximate yields were measured by TLC. The results showed that water is the







**Fig. 6.** FT-IR spectra of (a)  $Fe_3O_4$  and (b)  $Fe_3O_4@CeO_2$ .

 Table 1. Synthesis of 3,5-dimethyl-1*H*-pyrazole (3a) in different solvents (reaction time 5 min)

Solvent	Yield, %
Water	98
Ethanol	95
Methanol	95
Ethyl acetate	90
Dimethyl sulfoxide	90
Dimethylformamide	85
1,2-Dichloroethane	85
Tetrahydrofuran	80
Chloroform	80
Methylene chloride	75
None (solvent-free)	40

**Table 2.** Synthesis of 3,5-dimethyl-1*H*-pyrazole (**3a**) in water in the presence of different amounts of  $\text{Fe}_3\text{O}_4@\text{CeO}_2$  NPs (amounts of the reactants 1 mmol, room temperature, reaction time 5 min)

Amount of Fe <sub>3</sub> O <sub>4</sub> @CeO <sub>2</sub> , g	Yield, %
0.001	80
0.005	98
0.007	98
0.010	98
No catalyst	40

best solvent. The optimal amount of the catalyst was estimated at 0.005 g per mmol of the substrate.

To define the reaction scope, acetylacetone, ethyl acetoacetate, and dibenzoylmethane were reacted with hydrazine hydrate, arylhydrazines, and acetohydrazide under the optimized conditions (0.005 g of the catalyst  $Fe_3O_4@CeO_2$ , water, room temperature; Scheme 1, Table 3). Pyrazoles **3a–3h** were produced in good to excellent yields (80–98%) in a very short reaction time

(5–35 min). Acetylacetone reacted with hydrazines to afford pyrazoles **3a–3c** with high yields (Table 3, entry nos. 1–3). The reactions of hydrazines with dibenzoylmethane, which is a less reactive carbon electrophile than acetylacetone, also produced pyrazoles **3g** and **3h** with high yields but in a longer time (Table 3, entry nos. 7, 8). Ethyl 3-oxobutanoate reacted with hydrazine to give hydroxypyrazole **3d** (entry no. 4), whereas its reactions with phenyl- and 4-chlorophenylhydrazines afforded pyrazolones **3e** and **3f**, respectively (entry nos. 5, 6). In the reactions of  $\beta$ -dicarbonyl compounds **1a–1c** with acetohydrazide (4) under the same conditions (0.005 g of Fe<sub>3</sub>O<sub>4</sub>@CeO<sub>2</sub>, H<sub>2</sub>O, room temperature), we isolated the corresponding hydrazones **5a–5c** in 80 to 98% yield (Table 3, entry nos. 9–11).

To better demonstrate the efficiency of the proposed catalyst, our results were compared with the reported results for other catalysts in the synthesis of pyrazoles (Table 4). The iron content of  $Fe_3O_4@CeO_2$  was measured by ICP-OES for a 0.01-g sample which was dissolved in a mixture of HNO<sub>3</sub> and HF, and the op-

Table 3. Synthesis of pyrazoles and imines in the presence of Fe<sub>3</sub>O<sub>4</sub>@CeO<sub>2</sub> nanocomposite<sup>a</sup>

Entry no.	Dicarbonyl compound	Hydrazine	Product	Time, min	Yield, <sup>b</sup> %
1	1a	2a	3a	5	98
2	1a	2b	3b	10	95
3	1a	2c	3c	10	90
4	1b	2a	3d	10	95
5	1b	2b	3e	15	90
6	1b	2c	3f	15	85
7	1c	2b	3g	30	90
8	1c	2c	3h	35	80
9	<b>1</b> a	4	5a	15	95
10	1b	4	5b	20	98
11	1c	4	5c	50	80

<sup>a</sup> Reaction conditions: 1 mmol of hydrazine/hydrazide, 1 mmol of  $\beta$ -dicarbonyl compound, 0.005 g of Fe<sub>3</sub>O<sub>4</sub>@CeO<sub>2</sub>; water, room temperature.

<sup>b</sup> Isolated yield.

**Table 4.** Comparison of  $Fe_3O_4@CeO_2$  nanocomposite with some reported catalysts in the synthesis of 1,3,5-triphenyl-1*H*-pyrazole (**3g**)

Catalyst and conditions	Time, min	Yield, %	TOF, h <sup>-1</sup>	Reference
Ir(ppy) <sub>3</sub> (2 mol %)/MeCN/hv	120	80	20	[27]
Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> @PDETSA (0.004 g)/H <sub>2</sub> O/r.t.	30	87	a	[28]
$[RuCl_2(p-cymene)]_2$ (5 mol %)/DMSO/base/1 atm O <sub>2</sub> /60–100°C	360	85	2.8	[29]
O <sub>2</sub> /activated carbon (50 mol %)/HOAc/120°C	150	91	0.73	[30]
Cellulose sulfuric acid (0.1 g)/solvent-free/r.t.	120	95	_a	[31]
Sc(OTf) <sub>3</sub> (2 mol %)/solvent-free/r.t.	20	94	134	[32]
Sulfamic acid (1 mol %)/solvent-free/r.t.	5	91	1137	[33]
Fe <sub>3</sub> O <sub>4</sub> @CeO <sub>2</sub> (0.0009 mol %)/H <sub>2</sub> O/r.t.	30	90	200 000	This work

<sup>a</sup> The catalyst amount was not given.

timized concentration was found to be 0.0009 mol %. Comparison of the turnover frequencies (TOFs) showed that our catalyst is one of the best catalysts for selective pyrazole synthesis.

The reusability of  $Fe_3O_4$  (a)  $CeO_2$  nanocomposite was studied in the reaction of acetylacetone with hydrazine hydrate to produce pyrazole 3a. The catalyst was separated using an external magnet, washed several times with deionized water, and then used to catalyze the reaction with fresh portions of the reactants in six successive runs. The yield of **3a** insignificantly decreased from 98% in the first run to 97, 94, 90, 88, and 83% in the second to sixth runs, respectively.

In conclusion, we have proposed a green and efficient method for the synthesis of pyrazoles and hydrazones in the presence of  $Fe_3O_4(a)CeO_2$  magnetic nanoparticles as a reusable and easily separable catalyst in water at room temperature with good to excellent yields (80–98%) and short reaction times (5–50 min).

## **EXPERIMENTAL**

The necessary reagents and solvents were purchased from Merck (Germany) and Fluka (Switzerland) and were used without further purification. The FT-IR spectra were recorded in the range 400–4000 cm<sup>-1</sup> using a Nicolet Avatar 370 spectrometer. The <sup>1</sup>H NMR spectra were measured on a Bruker Avance DPX-250 instrument at 300 MHz in CDCl<sub>3</sub> using tetramethylsilane (TMS) internal standard. The iron content of the catalyst was determined by inductively coupled plasma/optical emission spectroscopy (ICP-OES) using an Agilent 7700ce instrument.

Preparation of Fe<sub>3</sub>O<sub>4</sub>@CeO<sub>2</sub> nanocomposite. Initially, magnetic Fe<sub>3</sub>O<sub>4</sub> nanoparticles were synthesized via a modified procedure based on the previously reported method [26]. For this purpose, a mixture of 2.0 g (10 mmol) of FeCl<sub>2</sub>·4H<sub>2</sub>O, 5.2 g (20 mmol) FeCl<sub>3</sub>·6H<sub>2</sub>O, 25 mL deionized water, and 0.85 mL of aqueous HCl was added dropwise to a solution of 15 g of sodium hydroxide in 25 mL of deionized water. The mixture was vigorously stirred, and a black solid separated. The mixture was heated on a water bath for 4 h at 80°C, and the magnetic product was separated by an external magnet, washed several times with ethanol and deionized water, and dried at 110°C for 5 h. A solution of 0.868 g (2.7 mmol) of  $Ce(NO_3)_3$   $GH_2O$  in a mixture of 17 mL of ethylene glycol and 34 mL of ethanol was stirred for 40 min, 0.347 g of as-prepared Fe<sub>3</sub>O<sub>4</sub> MNPs were added, and the mixture was sonicated for 30 min, heated on an oil bath for 5 h at

160°C, and allowed to cool to room temperature. The resulting magnetic catalyst was separated using a magnet, washed several times with ethanol and deionized water, and dried under reduced pressure at 70°C for 10 h.

General procedure for the synthesis of pyrazoles and hydrazones. A mixture of 0.005 g of  $Fe_3O_4@CeO_2$  as catalyst, hydrazine **2a–2c** or hydrazide 4 (1.0 mmol), and  $\beta$ -dicarbonyl compound 1a–1c (1 mmol) in water (2 mL) was stirred at room temperature for a time indicated in Table 3. The progress of the reaction was monitored by TLC on Polygram SILG/UV 254 plates. After completion of the reaction, the catalyst was separated using an external magnet and washed several times with ethanol and deionized water for subsequent use. The product was extracted with ethyl acetate (2×10 mL), the extract was dried over Na<sub>2</sub>SO<sub>4</sub> and evaporated, and the residue was purified by flash column chromatography (EtOAcn-hexane, 1:20). Spectral characteristics of some of the synthesized compounds are given below:

3,5-Dimethyl-1H-pyrazole (3a). IR spectrum (KBr), v, cm<sup>-1</sup>: 3397, 1620, 1531, 1492. <sup>1</sup>H NMR spectrum, δ, ppm: 2.36 s (CH<sub>3</sub>), 2.69 s (CH<sub>3</sub>), 5.91 s (4-H), 13.7 s (NH).

3,5-Dimethyl-1-phenyl-1H-pyrazole (3b). IR (neat), v, cm<sup>-1</sup>: 3084, 2903, 1615, 1573, 1537, 1459, 1395, 750, 678. <sup>1</sup>H NMR spectrum: 2.24 s (3H, CH<sub>3</sub>), 2.28 s (3H, CH<sub>3</sub>), 5.95 s (1H, 4-H), 7.31-7.34 m (5H, Ph).

1,3,5-Triphenyl-1H-pyrazole (3h). IR spectrum (KBr), v, cm<sup>-1</sup>: 3147, 3082, 1639, 1565, 1515, 1473, 1491, 1388, 1227, 1193, 1088, 1043, 976, 937, 759, 721. <sup>1</sup>H NMR spectrum, δ, ppm: 6.88 s (1H, 4-H), 7.35-7.99 m (15H, H<sub>arom</sub>).

N'-(4-Oxopentan-2-ylidene)acetohydrazide (5a). IR spectrum (neat), v, cm<sup>-1</sup>: 3013, 2953, 1745, 1612, 1421, 1390, 1341, 1295, 1165, 1101, 1056, 963, 821, 791. <sup>1</sup>H NMR spectrum, δ, ppm: 2.21 s (3H, CH<sub>3</sub>), 2.52 s (3H, CH<sub>3</sub>), 2.63 s (3H, CH<sub>3</sub>), 4.8 s (2H, CH<sub>2</sub>), 5.88 s (1H, NH).

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## CONFLICT OF INTERESTS

The authors declare the absence of conflict of interests.

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