

# Catalytic study of the copper-based magnetic nanocatalyst on the aerobic oxidation of alcohols in water

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# Abstract

A copper-based magnetic nanocatalyst has been prepared by co-precipitation method and characterized by FESEM, EDS, TEM, XRD, XRF, ICP–OES, FTIR, and BET analysis. This new nanocatalyst displays a good activity toward the aerobic oxidation of a wide range of alcohols in water. Moreover, it is recyclable up to five following runs by simple filtration without any significant loss of its catalytic activity.

Keywords Magnetic nanocatalyst  $\cdot$  Fe $_3O_4@SiO_2 \cdot$  Aerobic oxidation  $\cdot$  Alcohols  $\cdot$  Carbonyl compounds

## Introduction

The oxidation of alcohols into the corresponding aldehydes and ketones is a significant conversion in organic chemistry that has both academic and industrial importance [1, 2]. To achieve this conversion, a variety of procedures have been reported. Conventionally, alcohol oxidation was accomplished by using stoichiometric oxidants, such as manganese oxides, chromium salts, and the Dess–Martin reagent, which produced large amounts of inorganic and organic wastes [3]. From the viewpoint of economy and environmental protection, aerobic oxidation of alcohols with molecular oxygen or air as the oxidant illustrated superior consideration in organic chemical synthesis. The main reason for this methodology is attributed to the point that oxygen is a green oxidant leading to water as the by-product [4].

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Various metal-catalyzed systems have been reported for the aerobic oxidation of alcohols that among them, copper-based catalysts have received significant attention because of their low cost compared to conventional expensive metals [5–20]. Moreover, it is found that the copper nanoparticles supported exhibit suitable activity and selectivity, because of their high surface area, catalyst loading capacity, and good dispersion [21, 22].

Cu is a 3d transition metal and has some exciting physical and chemical properties. Cu-based compounds can promote and undergo a variety of reactions due to having different oxidation states. Because of their unique characteristics and properties, Cu-based nanocatalysts have found many applications in nanotechnology, including catalytic organic transformations, electrocatalysis, and photocatalysis [23]. Under oxidizing conditions, copper exists as  $Cu^{2+}$ , while reducing conditions favor the  $Cu^+$  form. Different performances of the two states may be traced to variances in their hardness: Whereas the  $Cu^{2+}$  ion is classified as a borderline Lewis acid, the reduced complement, the  $Cu^+$  ion, is an model soft acid. Hard N- and O-donors lead the coordination chemistry of divalent state, whereas the  $Cu^+$  ion favors ligands with soft donor atoms, such as phosphorus, sulfur, or iodine [24].

Therefore, copper nanoparticles supported on various supports such as cellulose,  $SiO_2$ , carbon-based materials, or polymers were developed as reactive and selective catalytic systems [23].

The highly oxygenated supports such as  $Fe_3O_4@SiO_2$  as the O-donor support provide good dispersions of active sites, mainly of metal nanoparticles, leading to better reactivity. These supports have great potential as stable inorganic ligands for coordination with metal such as copper nanoparticles, opposing from organic ligands [25].  $Fe_3O_4@SiO_2$  contains multiple hydroxyl groups that perform as nucleation sites for the metals such as copper nanoparticles and provide necessary support for the stabilization of the nanoparticles. The catalysts obtained in this approach display distinctive properties and find their application in varied areas [26]. Moreover, the nanomagnetic core–shell structure has attracted support because of the easy preparation, good stability, high surface area, and low toxicity. The hydroxyl functional groups of SiO<sub>2</sub> provide a chemically modifiable surface which facilitates the immobilizing of other different metal catalysts [27].

Due to the fact that  $Fe_3O_4@SiO_2$  can act as a suitable support for the metal nanoparticles, in continuation of our researches on the improvement of new nanocatalysts [28–34], herein, we wish to report the use of  $Fe_3O_4@SiO_2$  to prepare an efficient copper-based magnetic nanocatalyst ( $Fe_3O_4@SiO_2/CuO$  nanocatalyst) for the chemoselective aerobic oxidation of alcohols in water (Scheme 1).

#### Experimental

All chemical materials were purchased from Sigma-Aldrich or Merck Chemical Companies. Products were characterized by comparison of their spectroscopic data (<sup>1</sup>H NMR, IR) and physical properties with those reported in the literature. X-ray diffraction (XRD) patterns were obtained using a diffractometer APD2000 using Cu K<sub> $\alpha$ </sub> ( $\lambda = 1.5406$  Å) radiation. The ICP–OES analysis was achieved by a Varian Vista-Pro



 $R^1$ ,  $R^2 = H$ , Alkyl, Aryl

Scheme 1 Aerobic oxidation of alcohols catalyzed by  $Fe_3O_4@SiO_2$ -CuO nanocatalyst

7410 device. Field emission scanning electron microscopy (FESEM) analysis was performed using a MIRA 3 TESCAN instrument equipped with an EDS analytical system. Transmission electron microscope (TEM) images were recorded on a TEM microscope Zeiss EM10C at an accelerating voltage 80 kV. Magnetic saturation was carried out using a vibrating sample magnetometer (VSM/model LBKFB, Kavir Magnet Co., Kashan, Iran) at room temperature. The Brunauer–Emmett–Teller (BET) analysis was obtained using a gas adsorption–desorption analyzer (BEL-SORP-mini 2). The XRF study was done by a Philips Venus 100 minilab instrument.

## Preparation of Fe<sub>3</sub>O<sub>4</sub> magnetic nanoparticles

A mixture of FeCl<sub>3</sub>·6H<sub>2</sub>O (5.2 g), FeCl<sub>2</sub>·4H<sub>2</sub>O (2.0 g), and HCl 12 M (0.85 mL) was dissolved in 25 mL of deionized water under N<sub>2</sub> atmosphere with vigorous stirring. The obtained solution was added dropwise into NaOH 1.5 M (250 mL) under a continuous N<sub>2</sub> flow for 15 min, while the reaction media were being stirred vigorously. The precipitated black Fe<sub>3</sub>O<sub>4</sub> MNPs were separated by an external magnet, washed with deionized water, and then dried in the oven [35].

## Synthesis of silica-coated $Fe_3O_4$ magnetic nanoparticles ( $Fe_3O_4@SiO_2$ )

1.2 g of Fe<sub>3</sub>O<sub>4</sub> MNPs was dispersed in 20 mL ethanol and sonicated for 15 min, followed by the addition of 1 mL triethylamine. Then, 0.8 mL tetramethyl orthosilicate (TMOS) was added dropwise into the reaction solution under violent stirring. The resulting dispersion was stirred for 30 min and sonicated again for 15 min. The precipitate was removed by an external magnet, washed with deionized water and EtOH, and dried at 80 °C.

## Preparation of Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>/CuO nanocatalyst

At first, Na<sub>2</sub>CO<sub>3</sub> solution (50 mL, 0.05 M) was added dropwise into a mixture of Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O (50 mL, 0.025 M) and Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub> (0.1 g) under vigorous stirring for 24 h at 50–60 °C. Then, the precipitate was separated by an external magnet,

washed with deionized water, and dried for 2 h at 100 °C followed by calcination at 300 °C for 3 h to afford the  $Fe_3O_4@SiO_2/CuO$  nanocatalyst.

#### **General procedure**

A mixture of alcohol (1 mmol), Na<sub>2</sub>CO<sub>3</sub> (2 mmol), and Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>/CuO nanocatalyst (0.04 g) in water was stirred under oxygen at reflux condition. After reaction completion, the catalyst was separated from the reaction mixture by external magnetic field, washed with hot EtOAc ( $2 \times 5$  mL), and dried for consecutive reaction runs. Then, the filtrate was cooled to room temperature, quenched with 2 M HCl aqueous solution, filtered, and extracted with dichloromethane. The solvent was evaporated and the organic layer dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. Evaporation of the solvent followed by column chromatography on silica gel (*n*-hexane/ethyl acetate 9:1 as v/v%) afforded the pure products.

# **Results and Discussion**

## **Catalyst characterization results**

Figure 1 exhibits the XRD images of  $Fe_3O_4@SiO_2$  and  $Fe_3O_4@SiO_2/CuO$  nanocatalyst. From Fig. 1a, the diffraction peaks located at  $30.28^\circ$ ,  $35.54^\circ$ ,  $43.48^\circ$ ,  $53.44^\circ$ ,  $57.20^\circ$ ,  $62.92^\circ$ , and  $74.48^\circ$  can be attributed to the crystal planes (220), (311), (400), (422), (511), (440), and (533) of cubic  $Fe_3O_4$  with inverse spinel structure, respectively [36]. It can be seen from Fig. 3b that the diffraction peaks at  $35.79^\circ$ ,  $39.02^\circ$ ,  $48.83^\circ$ ,  $57.60^\circ$ ,  $63.04^\circ$ ,  $66.29^\circ$ , and  $75.43^\circ$  are verified the CuO forming



Fig. 1 The XRD patterns of  $Fe_3O_4@SiO_2(a)$  and  $Fe_3O_4@SiO_2$ -CuO nanocatalyst (b)

with monoclinic phase on the surface of  $Fe_3O_4@SiO_2$  support [37]. Furthermore, after immobilization of the CuO and the next calcination, no phase change observed, which revealed that the crystalline structure of the nanocatalyst has been retained.

The FESEM micrographs of  $Fe_3O_4@SiO_2$  and  $Fe_3O_4@SiO_2/CuO$  are shown in Fig. 2. The FESEM images exhibit that nanoparticles are almost uniform in spherical shape and possess particle diameters less than 40 nm. The lower aggregation of nanoparticles in the  $Fe_3O_4@SiO_2/CuO$  in comparison with  $Fe_3O_4@SiO_2$  can be confirmed by precipitation of CuO nanoparticles on the surface of  $Fe_3O_4@SiO_2$ .

The evaluation of the EDS analysis, as shown in Fig. 2c and d, presented the Cu content of about 13.2% ww<sup>-1</sup> in accordance with the obtained results by ICP–OES (13.4% ww<sup>-1</sup>) and XRF analysis (13.5% ww<sup>-1</sup>). From the TEM images, CuO nanoparticles are spread on the surface of Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub> support. The nanoparticles are almost uniformly in spherical shape with core–shell structure at about 13 nm (Fig. 3).

According to the IUPAC classification, the  $N_2$  adsorption-desorption isotherms showed typical IV isotherms with a type H1 hysteresis loop that is characteristic of the highly ordered mesoporous material (Fig. 4).

The BET analysis showed that the specific surface area of  $Fe_3O_4@SiO_2$  and  $Fe_3O_4@SiO_2/CuO$  was 106 m<sup>2</sup> g<sup>-1</sup> and 122 m<sup>2</sup> g<sup>-1</sup>, respectively (Table 1). Also, mean pore diameter and total pore volume of  $Fe_3O_4@SiO_2$  were found to be 7.821 nm and 0.183 cm<sup>3</sup> g<sup>-1</sup>, respectively.



Fig. 2 The FESEM images of  $Fe_3O_4@SiO_2$  (a)  $Fe_3O_4@SiO_2$ -CuO nanocatalyst (b), the EDS analysis of  $Fe_3O_4@SiO_2$  (c), and  $Fe_3O_4@SiO_2$ -CuO nanocatalyst (d)



Fig. 3 The TEM images of the  $Fe_3O_4@SiO_2$ -CuO nanocatalyst



Fig. 4 The  $N_2$  adsorption–desorption isotherms of  $Fe_3O_4@SiO_2$  and  $Fe_3O_4@SiO_2/CuO$  nanocatalyst

Table 1 Structural properties of the Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> and Fe <sub>3</sub> O <sub>4</sub> @ SiO <sub>2</sub> /CuO nanocatalyst (BET analysis)	Sample	BET surface area $(m^2 g^{-1})$	Average pore size (nm)	Pore vol- ume (cm <sup>3</sup> $g^{-1}$ )
	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub>	106	7.821	0.183
	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> /CuO	122	6.907	0.239



Fig. 5 The pore diameter size distribution of  $Fe_3O_4@SiO_2$  and  $Fe_3O_4@SiO_2/CuO$  nanocatalyst

The mean pore diameter and total pore volume of the  $Fe_3O_4@SiO_2/CuO$  were found to be 6.907 nm and 0.239 cm<sup>3</sup> g<sup>-1</sup>, respectively (Fig. 5). All of the results indicated that the incorporation of CuO on the surface of  $Fe_3O_4@SiO_2$  and calcination leads to surface area increasing as well as pore volume increasing.

The magnetic property of nanoparticles was evaluated by vibrating a sample magnetometer using model 7410 series, LBKFE at ambient temperature with field sweeping from -15,000 to +15,000 Oe. The magnetic saturation was measured to be 47.51 emu/g<sup>-1</sup> and 23.25 emu/g<sup>-1</sup> for Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub> and Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>/CuO



Fig. 6 The magnetic hysteresis loops of (a) Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub> and (b) Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>/CuO nanocatalyst

(Fig. 6). Almost any hysteresis loop was not seen for both samples. These results indicated that both catalysts are super paramagnetic. A decrease in  $Fe_3O_4@SiO_2/CuO$  is related to CuO grafting to the surface of  $Fe_3O_4@SiO_2$ .

#### **Catalytic performance**

Initially, to optimize the reaction conditions, we checked the aerobic oxidation of 4-chloro-benzyl alcohol in the presence of  $Fe_3O_4@SiO_2/CuO$  nanocatalyst under  $O_2$  at different conditions. The performance of various  $Fe_3O_4@SiO_2/CuO$  nanocatalysts containing 8, 13, and 20 wt. % CuO was studied on this reaction. The catalyst containing about 13 wt. % of CuO showed the highest yield of reaction of about 95%, in relatively lower reaction time. Moreover, the effect of various catalyst amounts was checked and found that using 0.04 g of the catalyst provided the best results. The model reaction was studied in different solvents and under solvent-free conditions. Also, the effect of various bases on the progress of the reaction has been examined. It is found that the catalytic activity of  $Fe_3O_4@SiO_2/CuO$  nanocatalyst in the presence of  $Na_2CO_3$  in water under reflux conditions is significantly higher than the other solvents and the solvent-free conditions (Table 2).

Under optimized conditions, aerobic oxidation of a wide range of alcohols was studied. The catalyst performance displayed a good activity on the aerobic oxidation of benzylic alcohols having electron-donating and electron-withdrawing groups. The oxidation of primary and secondary aliphatic alcohols was also investigated under similar reaction conditions, which showed that desired carbonyl products are obtained in high yields. Furthermore, aldehydes or ketones are the only products of the all reactions and no other products were identified in these processes that suggest the selectivity of the catalyst to these constituents. It is found that the oxidation of the bulky aliphatic alcohols such as menthol is slower than other aliphatic ones (Table 3). Moreover, all the products could be easily separated and purified by column chromatography in high yields.

Table 2   Optimization of the reaction conditions	Entry	Conditions	Catalyst amounts (g)	Time (min)	Yield (%) <sup>a</sup>
	1	EtOH/r.t	0.02	180	_
	2	EtOH/r.t	0.04	180	_
	3	EtOH/reflux	0.02	180	30
	4	EtOH/reflux	0.04	180	50
	5	H <sub>2</sub> O/r.t	0.02	180	-
	6	H <sub>2</sub> O/r.t	0.04	180	Trace
	7	H <sub>2</sub> O/r.t	0.06	180	Trace
	8	H <sub>2</sub> O/reflux	0.02	90	70
	9	H <sub>2</sub> O/reflux	0.04	60	93
	10	MeCN/reflux	0.02	180	Trace
	11	MeCN/reflux	0.04	180	Trace

<sup>a</sup> Isolated pure products

Catalytic study	of the	copper-	based	magnetic	nanocataly	/st
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Entry	Alcohol Product		Time (min)	Yield (%) <sup>a</sup>	
1	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> OH	C <sub>6</sub> H <sub>5</sub> CHO	45	92	
2	2-ClC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> OH	2-ClC <sub>6</sub> H <sub>4</sub> CHO	60	93	
3	4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> CH <sub>2</sub> OH	4-ClC <sub>6</sub> H <sub>4</sub> CHO	60	95	
4	2,4-ClC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> OH	2,4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> CHO	60	95	
5	4-(Me <sub>2</sub> N)C <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> OH	4-(Me <sub>2</sub> N)C <sub>6</sub> H <sub>4</sub> CHO	60	92	
6	3-OHC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> OH	3-OHC <sub>6</sub> H <sub>4</sub> CHO	60	89	
7	4-Cl-3-NO <sub>2</sub> C <sub>6</sub> H <sub>3</sub> CH <sub>2</sub> OH	4-Cl-3-NO <sub>2</sub> C <sub>6</sub> H <sub>3</sub> CHO	90	91	
8	2-BrC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> OH	2-BrC <sub>6</sub> H <sub>4</sub> CHO	90	89	
9	4-BrC <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> OH	4-BrC <sub>6</sub> H <sub>4</sub> CHO	60	94	
10	3-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> OH	3-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CHO	90	92	
11	4-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> OH	4-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CHO	90	95	
12	3,4,5-(MeO) <sub>3</sub> C <sub>6</sub> H <sub>2</sub> CH <sub>2</sub> OH	3,4,5-(MeO) <sub>3</sub> C <sub>6</sub> H <sub>2</sub> CHO	45	92	
13	$4-\text{MeOC}_6\text{H}_4\text{CH}_2\text{OH}$	4-MeOC <sub>6</sub> H <sub>4</sub> CHO	60	94	
14	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> CH <sub>2</sub> OH	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> CHO	80	91	
15	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> OH	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> CH <sub>2</sub> CHO	80	92	
16	$C_6H_4CH(Me)CH_2OH$	$C_6H_4CH(Me)CHO$	90	91	
17	CH <sub>3</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> OH	CH <sub>3</sub> CH <sub>2</sub> CH <sub>2</sub> CHO	90	92	
18	CH <sub>3</sub> CH(OH)CH <sub>3</sub>	CH <sub>3</sub> COCH <sub>3</sub>	90	89	
19	O-CO-OH		60	88	
20	но		90	89	
21	CH <sub>2</sub> OH	CHO	120	87	
22	OH		120	88	
23	HO		160	89	
24	ОН	C <sup>0</sup>	90	91	
25	HO HO O O Me O Me		180	87	

Table 3 Aerobic oxidation of alcohols catalyzed by  $Fe_3O_4@SiO_2/CuO$  nanocatalyst

<sup>a</sup>Isolated yield. Products were characterized by comparison of their spectroscopic data and physical properties with those reported in the literature [4-11, 21, 26]

Table 4Recyclability studyof the $Fe_3O_4@SiO_2/CuO$ nanocatalyst	Run	1	2	3	4	5
	Time (min)	60	60	65	70	70
	Yield (%) <sup>a</sup>	95	94	94	92	89
	<sup>a</sup> Isolated yields	3				

 $\label{eq:comparison} \begin{array}{l} \mbox{Table 5} & \mbox{Comparison between $Fe_3O_4@SiO_2/CuO$ nanocatalysts with some other catalysts employed for the aerobic oxidation of benzyl alcohol \\ \end{array}$ 

Entry	Solvent	Catalyst	Time (h)	Yield (%) a	Reference
1	MeCN	POM/ZrO <sub>2</sub>	4	81	9
2	MeCN	CuBr <sub>2</sub> /NHPI	6	50.8	12
3	Toluene	Ru-DD3	3	97	16
4	DMF	Cu-AOOPs catalyst	2	90	17
5	MeCN	[L2CuBr]/TEMPO/NMI	24	99	18
6	$H_2O$	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> /CuO nanocatalyst	0.75	92	This work

<sup>a</sup> Isolated yield

The recyclability of the catalyst was also investigated on the aerobic oxidation of 4-chloro-benzyl alcohol under optimized reaction conditions. After reaction completion, the catalyst was recovered, was washed with hot ethanol, and after drying was reused in the consecutive reaction runs. Results exhibited a constant activity of the catalyst during the five runs without any significant loss in catalytic activity (Table 4). Moreover, the leaching test of the Cu into the reaction mixture by ICP–OES analysis was studied. It was observed that the difference between the Cu contents of the fresh catalyst and the reused catalyst (fifth run) was only 3.1%, which showed the low leaching amount of copper oxide catalyst into the reaction mixture.

In order to confirm the merit of this method, Table 5 shows the comparison of the results found from the aerobic oxidation of benzyl alcohol in the presence of  $Fe_3O_4@SiO_2/CuO$  nanocatalyst with some of the other reported catalysts. In comparison with other catalysts,  $Fe_3O_4@SiO_2/CuO$  nanocatalyst catalyzed the reaction effectively in water and provides the desired products at high yields in relatively short reaction times. Moreover, it is safe, is easy to handle, and can be recovered more than five runs by simple filtration. The reaction in the presence of some other catalysts has been performed in organic solvents such as DMF and MeCN. Moreover, other reported methods required longer reaction times and also performed in the presence of additional amounts of the base or acid. Furthermore, in some reactions, additive reagents such as 2,2,6,6-tetramethylpiperidine-*N*-oxyl (TEMPO) are needed.

# Conclusion

In summary,  $Fe_3O_4@SiO_2$ –CuO as an efficient recyclable magnetic nanocatalyst was prepared and studied on the aerobic oxidation of alcohols in water. The  $Fe_3O_4@SiO_2$ –CuO nanocatalyst exhibits a high performance with a superior selectivity. This nanocatalyst is safe, is easy to handle, and can be recovered more than five runs by simple filtration. Moreover, high yield of products, relatively short time of reactions, ease of workup, and clean procedure can represent this method as a suitable and applicable substitute to the common methods.

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