



## Original Research Article

# Chemoselective oxidation of sulfides to sulfoxides using a novel Zn-DABCO functionalized Fe<sub>3</sub>O<sub>4</sub> MNPs as highly effective nanomagnetic catalyst

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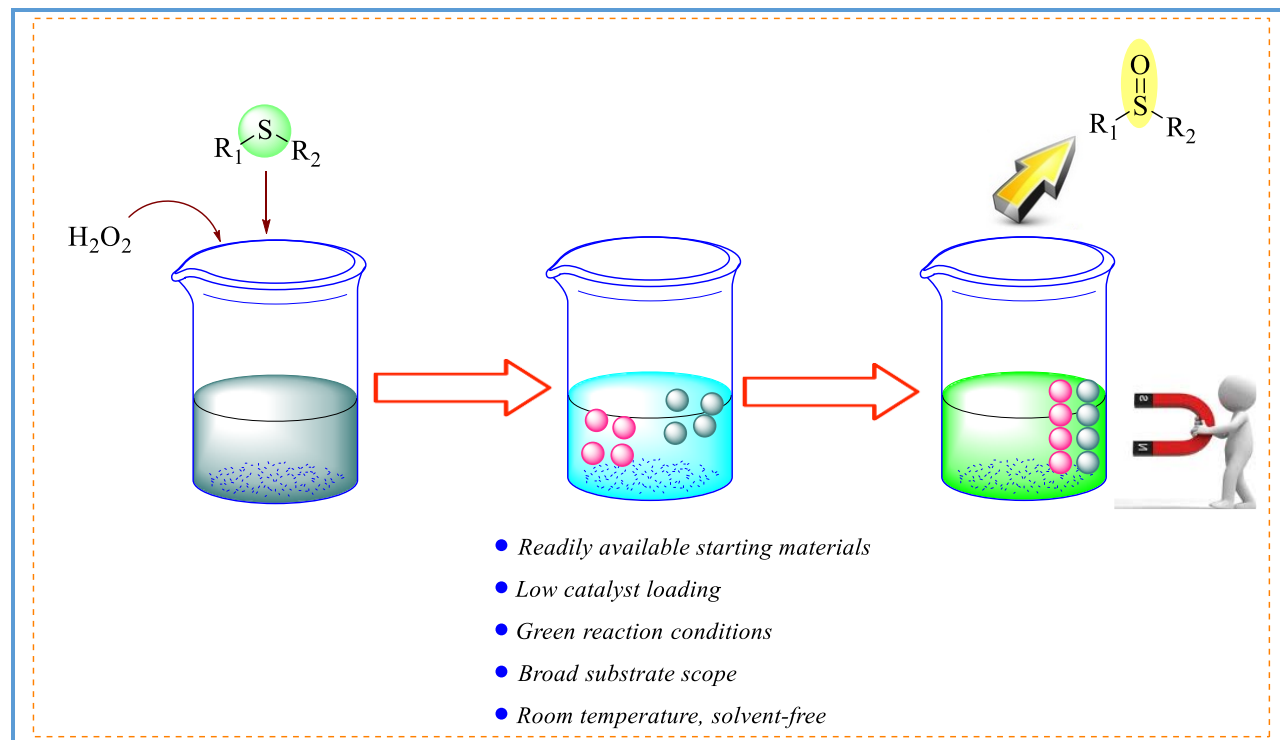
### KEYWORDS

Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub>  
Green chemistry  
Oxidation  
Hydrogen peroxide  
Lewis acid

### ABSTRACT

In the present study, Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> with a high surface area and readily was synthesized. The morphology, particle size distribution, and phase analysis of the Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> nanopowders were characterized using the scanning electron microscope (SEM), transmission electron microscope (TEM), vibrating sample magnetometer (VSM) and X-ray diffraction (XRD) analysis. The prepared Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> nanoparticle was utilized as efficient catalyst for selective oxidation of sulfides to sulfoxides using 30% H<sub>2</sub>O<sub>2</sub> as oxidant. The products were achieved with good to excellent yields at room temperature with no over-oxidation of sulfoxides and disulfides to unexpected by-products. This catalyst can be magnetically recovered by applying an external magnet and reused for eight continuous cycles in both oxidation reactions without a notable loss in its catalytic activity. Furthermore, the oxidation of various sulfides was highly chemoselective, but *O,O*-acetals remained intact under the described reaction conditions.

## Graphical Abstract



## Introduction

Nano magnetic based heterogeneous catalytic systems have a high surface-to-volume ratio, which guarantees the catalytic system's high activity. Also, these catalysts can be easily separated from the reaction media by applying a simple external magnet. These features make nano magnetic based heterogeneous catalytic systems as a great option for both industrial and academic chemists [1–7]. The magnetic nanoparticles can be utilized in various organic reactions such as synthesis of  $\alpha$ -amino nitriles [8], nucleophilic substitution reactions of benzyl halides [9], Suzuki coupling reactions [10], esterifications [11], Knoevenagel reaction [12], hydrogenation of alkynes [13], epoxidation of alkenes [14],  $\text{CO}_2$  cycloaddition reactions [15], and three-component condensations [16]. In addition, MNPs were emerged widely for various areas applications including, waste-water treatment [17],

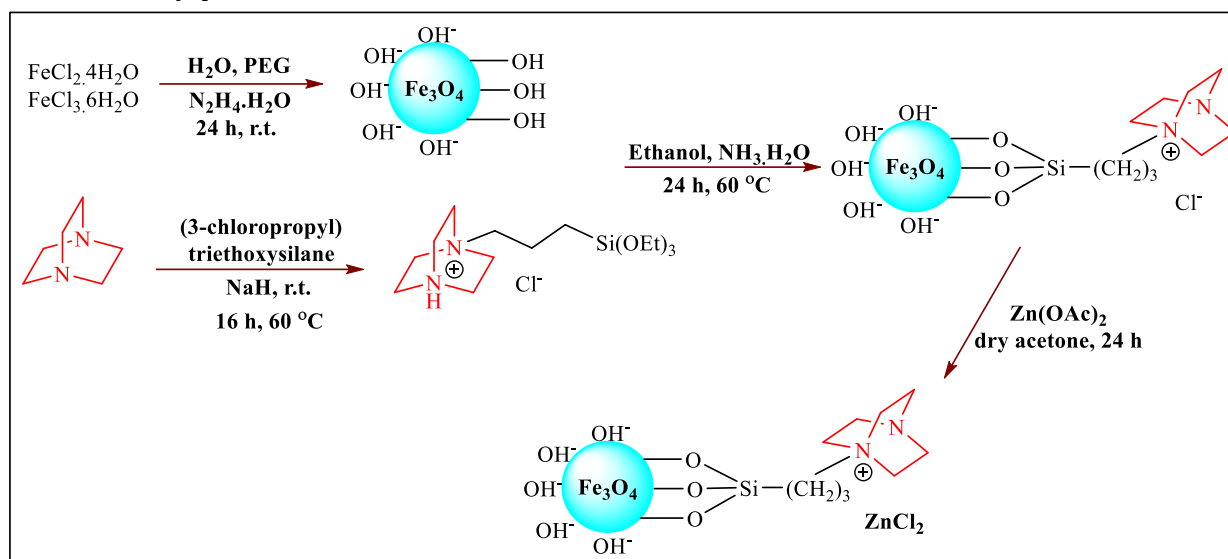
sonochemical approach [18], electrochemical storage of hydrogen [19], magnetic resonance imaging [20], drug delivery [21], biology and medical applications [22], and magnetic fluids [23].

The selective oxidation of sulfides into the corresponding sulfoxides has taken up an outstanding part in modern synthetic organic chemistry [24]. Organic sulfoxides play an essential role in synthesizing chemically useful and biologically active compounds, including drugs, flavours, germicides, and catabolism regulators [25]. In recent years, some procedures have been reported for the oxidation of sulfides to sulfoxides such as  $\text{H}_2\text{O}_2/\text{Mn(III)}$  [26], Amberlyst 15/ $\text{H}_2\text{O}_2$  [27], vanadium(IV) complexes of dibromo- and diiodo-functionalized chiral Schiff bases [28], NBS/b-cyclodextrine [29], silica-based tungstate/ $\text{H}_2\text{O}_2$  [30], Organic-Inorganic Polyoxometalate-Based Frameworks [31],

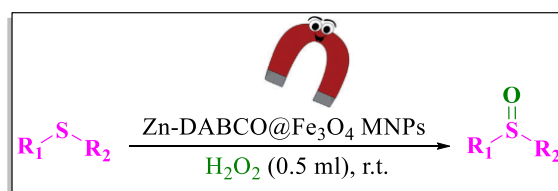
MNPs-supported acidic catalysts [32], SBA-15 Based Tungstate/H<sub>2</sub>O<sub>2</sub> [33], Cu Salen-Fe<sub>3</sub>O<sub>4</sub>/H<sub>2</sub>O<sub>2</sub> [34], pre-formed manganese complex/H<sub>2</sub>O<sub>2</sub> [35],  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>@HAp-Ag NPs [36], molybdate-based catalyst/ H<sub>2</sub>O<sub>2</sub> [37], and metalloporphyrins immobilized into montmorillonite/H<sub>2</sub>O<sub>2</sub> [38]. Unfortunately, some methods based on sulfides' oxidation require hazardous and toxic reagents or include over-oxidation of sulfides and thiols to undesired by-products. Therefore, further

progress in eco-friendly selective oxidation of sulfides to sulfoxides using less poisonous catalysts, oxidants, and solvents is still needed.

In this work, Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> was synthesized (Scheme 1). The prepared Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> nanoparticles were fully characterized using various physicochemical methods and successfully used for selective oxidation of sulfides to sulfoxides at room temperature (Scheme 2).



**Scheme 1.** The synthesis of Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> MNPs



**Scheme 2.** Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> MNPs catalyzed oxidation of sulfides to sulfoxides at room temperature

## Experimental

### Materials and methods

All reagents and used materials were purchased from the Sigma-Aldrich and Merck, and were utilized any additional purification. Products were separated and purified by

different chromatographic techniques and were identified by the comparison of their IR, NMR, and melting point with those reported for the authentic samples. All the melting points were recorded by open capillary method and are uncorrected. <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on a Bruker Avance (250 MHz)

spectrometer using  $\text{CDCl}_3$  as the solvent and TMS as an internal standard. Thin-layer chromatography (TLC) was performed on pre-coated aluminium plates (silica gel 60 F254, Merck). IR spectra of the compounds were obtained on a Perkin Elmer spectrometer version 10.03.06 using a KBr disk. The phases present in the magnetic materials were analyzed using a powder XRD, Philips (Holland), model X0 Pert with X' Pert with  $\text{CuK}\alpha 1$  radiation ( $\lambda = 1.5401 \text{ \AA}$ ), and the X-ray generator was operated at 40 kV and 30 mA. Diffraction patterns were collected from  $2\theta = 20^\circ$ – $80^\circ$ . The mean size and the surface morphology of the nanoparticles were characterized by FT-IR, TEM, SEM, VSM, and XRD.

#### *Synthesis of Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub>*

DABCO@Fe<sub>3</sub>O<sub>4</sub> was prepared *via* the condensation of Fe<sub>3</sub>O<sub>4</sub> MNPs with *n*-propyl-4-aza-1-azoniabicyclo[2.2.2]octane according to the literature procedure [39]. A mixture of DABCO@Fe<sub>3</sub>O<sub>4</sub> and Zn(OAc)<sub>2</sub> in dry acetone (20 mL) was stirred at room temperature for 24 h. The solid product was filtered by suction, washed with acetone, distilled water and acetone successively and dried under vacuum at 60 °C for 4 h to give complex of Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> (2.03 g).

#### *General procedure for oxidation of sulfides to sulfoxide*

In a typical procedure, 15 mg of the prepared Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> nanocatalyst, 1 mmol of sulfide and 0.5 mL of 30% H<sub>2</sub>O<sub>2</sub> were mixed at room temperature for the time specified in Table 3. This mixture was magnetically stirred at room temperature and the reaction progress was checked by thin-layer chromatography (TLC). After completion of the reaction, the catalyst was magnetically collected and was

washed with  $\text{CH}_2\text{Cl}_2$  and dried. Lastly, to achieve the pure product, it was extracted with  $\text{CH}_2\text{Cl}_2$  ( $3 \times 10 \text{ mL}$ ) and purified on a silica-gel column and products were obtained in 82–99% yield.

## **Results and Discussion**

The successful preparation of novel acidic nanomagnetic catalyst was investigated and confirmed by applying several skills such as X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscope (SEM) and vibrating sample magnetometer (VSM) analysis. In the following, the obtained data were investigated in detail.

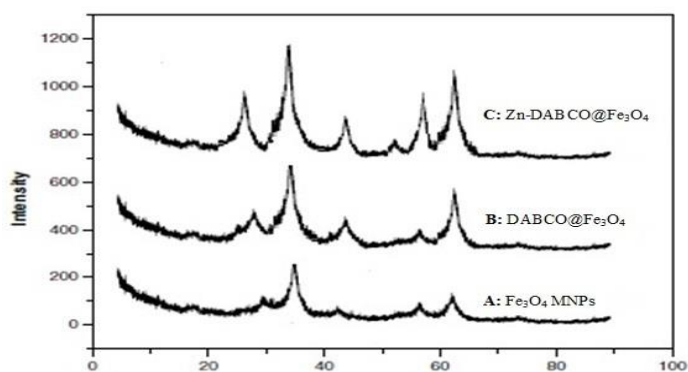
#### *Characterization of Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> MNPs*

The structural properties of synthesized Fe<sub>3</sub>O<sub>4</sub> MNPs (A), DABCO supported with Fe<sub>3</sub>O<sub>4</sub> MNPs (B), and Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> (C) were analyzed by XRD (Figure 1). As seen in Figure 1, the XRD patterns of the synthesized Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> display several relatively strong reflection peaks in the  $2\theta$  region of  $20^\circ$ – $80^\circ$ , which is quite similar to those of Fe<sub>3</sub>O<sub>4</sub> MNPs, DABCO supported with Fe<sub>3</sub>O<sub>4</sub> MNPs reported by other group. The XRD patterns of the particles demonstrated characteristic peaks at (220), (311), (400), (422), (511), and (440), which match well with the database of magnetite (JCPDS Card: 19-629) file.

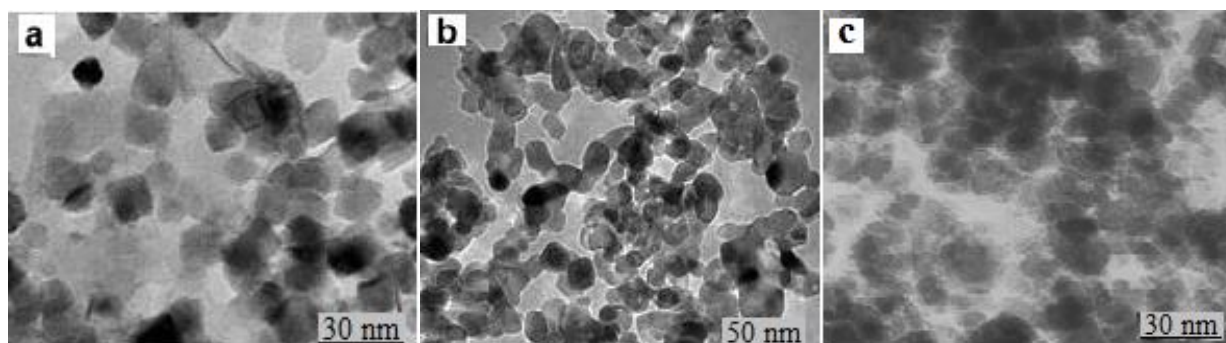
The nanostructure of the Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> MNPs was confirmed by transmission electron microscopy (TEM) (Figure 2c). The TEM micrograph clearly proved that the particles were in nanosize. The average particle size of the Fe<sub>3</sub>O<sub>4</sub> MNPs was found to be 25–30 nm (Figure 2a). After being coated with a DABCO, the typical core-shell structure of the Fe<sub>3</sub>O<sub>4</sub>-DABCO can be observed, and the average size increases to about 45–50 nm (Figure 2b). Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> NPs (Figure 2c) consist of relatively small, nearly spherical particles, with an average size of 30 nm, much smaller than the sizes obtained from the XRD measurements.

The scanning electron microscope (SEM) micrographs of the  $\text{Fe}_3\text{O}_4$  MNP (Figure 3a), DABCO supported with  $\text{Fe}_3\text{O}_4$  MNPs (Figure 3b), and Zn-DABCO@ $\text{Fe}_3\text{O}_4$  NPs (Figure 3c) revealed that the particles of the catalyst were observed in nanosize.

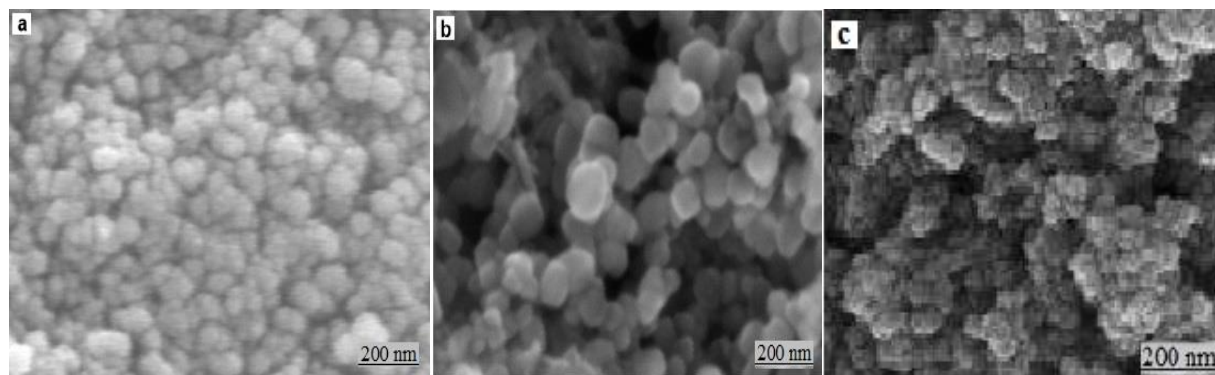
In another investigation, magnetic measurements of Zn-DABCO@ $\text{Fe}_3\text{O}_4$  MNPs were performed at room temperature using a vibrating sample magnetometer (VSM). The magnetization curve in Figure 4 gives a saturation magnetization value of 64 emu/g (Figure 4).



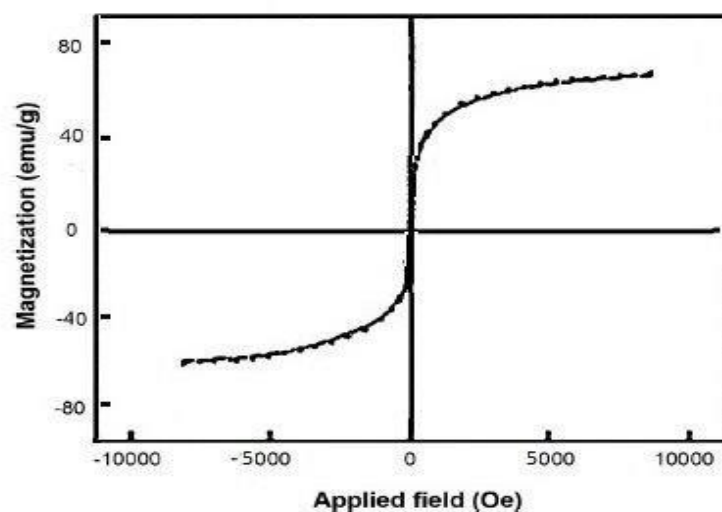
**Figure 1.** XRD patterns of a)  $\text{Fe}_3\text{O}_4$  MNP, b) DABCO supported with  $\text{Fe}_3\text{O}_4$  MNPs, and c) Zn-DABCO@ $\text{Fe}_3\text{O}_4$



**Figure 2.** TEM images of a)  $\text{Fe}_3\text{O}_4$  MNPs, and b) DABCO@ $\text{Fe}_3\text{O}_4$  and c) Zn-DABCO@ $\text{Fe}_3\text{O}_4$



**Figure 3.** The SEM micrographs of a)  $\text{Fe}_3\text{O}_4$  MNPs, b) DABCO@ $\text{Fe}_3\text{O}_4$ , and c) Zn-DABCO@ $\text{Fe}_3\text{O}_4$



**Figure 4.** Magnetization curve of Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> NPs

#### Scope of reaction

After characterization of the synthesized Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> NPs, its catalytic activity was evaluated in the oxidation of sulfides to provide sulfoxides to find the best reaction conditions, the effect of the different experimental parameters (e.g. effect of solvent and catalyst) on the reaction efficiency were studied. Evidently, in the lack of the catalyst, the reaction

could not progress by 120 min (Table 1, entry 1). The highest yield was obtained in the presence of 15 mg of Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> NPs. After wide screening, the highest yield, best reaction time, and reduction of undesired side products were obtained when the reaction was supported in the presence of 15 mg of the catalyst under solvent-free conditions at room temperature (Table 1, entry 5).

**Table 1.** Optimizing the reaction conditions for the methylphenyl sulfide

Entry	Amount of catalyst (mg)	Solvent	Time (min)	Yield (%) <sup>a</sup>
1	None	Solvent-free	120	10
2	1	Solvent-free	70	45
3	5	Solvent-free	50	55
4	10	Solvent-free	50	74
5	15	Solvent-free	15	96
6	20	Solvent-free	30	90
7	15	CH <sub>2</sub> Cl <sub>2</sub>	120	36
8	15	Hexane	120	Trace
9	15	CCl <sub>4</sub>	120	Trace
10	15	CH <sub>3</sub> OH	90	45
11	15	CH <sub>3</sub> CN	60	55
12	15	C <sub>2</sub> H <sub>5</sub> OH	60	61

<sup>a</sup> Isolated yields

As is evident from Table 2, all electron-donating substituents and electron-withdrawing substituents such as 4-bromo

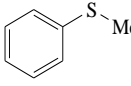
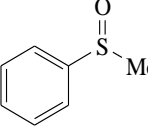
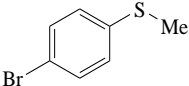
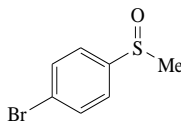
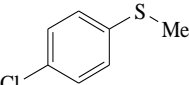
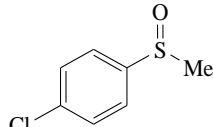
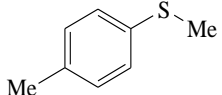
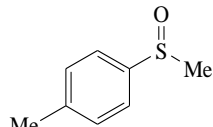
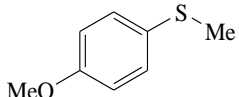
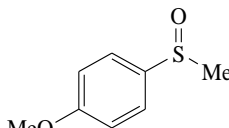
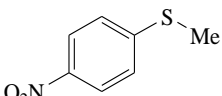
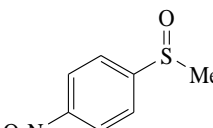
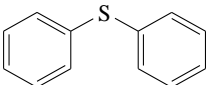
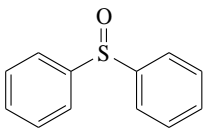
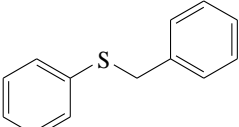
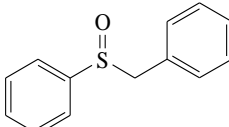

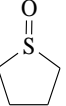
thioanisole (Table 2, entry 2), 4-chloro thioanisole (Table 2, entry 3), 4-methyl thioanisole (Table 2, entry 4), 4-methoxy

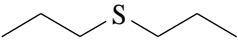
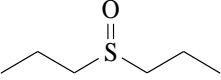
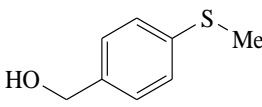
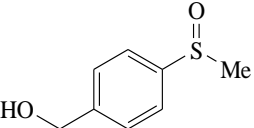
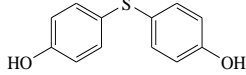
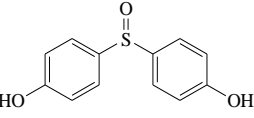
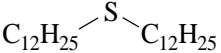
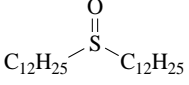
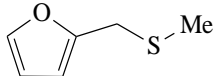
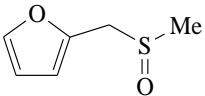
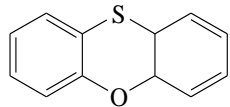
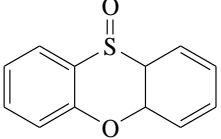


thioanisole (Table 2, entry 5) were converted into their corresponding sulfoxides in good to excellent yields and short reaction time. Also, 4-nitro thioanisole (Table 2, entry 6) was converted into their corresponding sulfoxides in excellent yield and the slightly longer reaction times. Interestingly, tetrahydrothiophene (Table 2, entry 9), dipropylsulfane (Table 2, entry 10), 4a, 10a-dihydrophenoxathiine (Table 2, entry 15) were

converted into their corresponding sulfoxides in short reaction time and good yields. Similarly, diallyl sulfides (Table 2, entries 7, 8 and 12) were oxidized to their corresponding sulfoxides in short reaction time and high yield. All the products listed in Table 2 are known and their structures were determined by  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR and IR which were in accordance with the literature.

**Table 2.** Oxidation of sulfides to the sulfoxides in the presence of Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> MNPs

Entry	Sulfides	Sulfoxides	H <sub>2</sub> O <sub>2</sub> (mL)	Time (min)	Yield (%) <sup>a</sup>
1			0.5	15	96
2			0.5	20	93
3			0.5	20	95
4			0.5	25	90
5			0.5	20	92
6			1	30	94
7			1.5	20	95
8			1.2	15	90
9			0.5	10	85

10			0.5	15	83
11			0.5	25	89
12			0.5	25	90
13			1.2	15	91
14			0.5	10	90
15			0.5	10	85

<sup>a</sup> Isolated yields

To establish the catalytic activity of Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> MNPs, we compared our obtained results for the oxidation of methyl phenyl sulfide with the best of the well-known data from literature (Table 3). As seen in Table

3, oxidation of methyl phenyl sulfide in the presence of Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> MNPs as catalyst, the higher yields of products were obtained in shorter reaction time and under milder conditions.

**Table 3.** Comparison of various procedure used for the oxidation of methyl phenyl sulfide with different catalysts<sup>a</sup>

Entry	Catalyst	Conditions	Time (h)	Yield (%) <sup>c</sup>
1	Fe <sub>3</sub> O <sub>4</sub> /salen of Cu(II)	EtOH, H <sub>2</sub> O <sub>2</sub> (aq), 60 °C	3	83
2	Ti(O <sup>i</sup> Pr) <sub>4</sub>	(S)-1-phenylethylhydroperoxide, r.t.	6	79
3	Human hemoglobin	H <sub>2</sub> O <sub>2</sub> (aq), r.t.	30 <sup>b</sup>	96
4	n-Bu <sub>4</sub> NBr	NaOCl, CH <sub>2</sub> Cl <sub>2</sub> , 0 °C	30 <sup>b</sup>	93
5	HAuCl <sub>4</sub> .4H <sub>2</sub> O	H <sub>2</sub> O <sub>2</sub> (aq), r.t.	1	99
6	Fluorous thiourea	CH <sub>2</sub> Cl <sub>2</sub> , H <sub>2</sub> O <sub>2</sub> (aq), r.t.	12	95
7	Zn-DABCO@Fe <sub>3</sub> O <sub>4</sub> MNPs <sup>d</sup>	H <sub>2</sub> O <sub>2</sub> (aq), r.t.	15 <sup>b</sup>	96

<sup>a</sup> Reaction condition: methyl phenyl sulfide (1 mmol), H<sub>2</sub>O<sub>2</sub> (0.5 ml), Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> MNPs (15) mg, at r.t

<sup>b</sup> In minute

<sup>c</sup> Isolated yields

<sup>d</sup> Our work

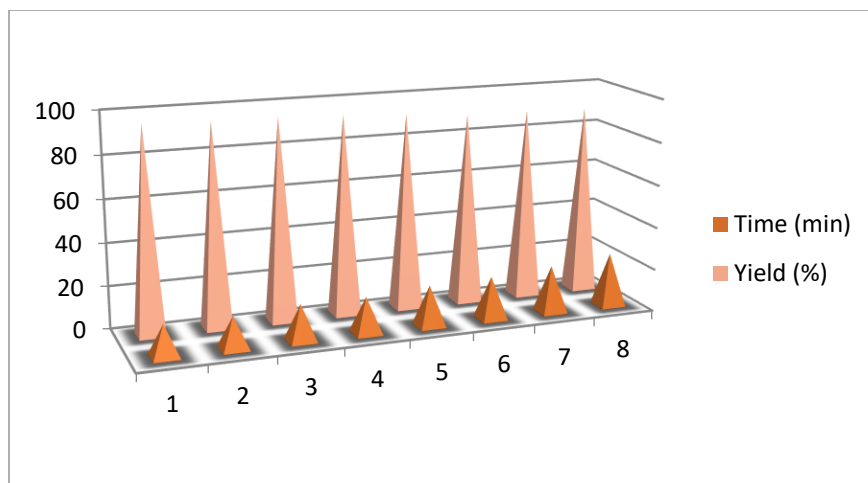
The recovery and reusability of catalysts are of great concern from economic, environmental

and industrial perspectives. In this regard, the reusability of the synthesized Zn-



DABCO@Fe<sub>3</sub>O<sub>4</sub> MNPs catalyst was investigated in the oxidation of dimethyl phenyl sulfoxide under the optimum reaction conditions. Since the catalyst can be separated from the reaction mixture using an external magnetic field, it was

recovered with a simple magnet after the dilution of the reaction mixture with water. As seen in Figure 5 the catalyst can be reused 8 times in both oxidation reactions with no considerable loss in its initial activity.



**Figure 5.** Reusability of Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> MNPs

In another study, to recognize the procedure's scalability, we assessed some reactions in 15 mmol of each reactant. The respective results are summarized in Table 4.

As demonstrated in Table 4, the reactions were successfully performed at the larger scale without significant loss of the yields.

**Table 4.** The scalability of the selective oxidation of sulfides to sulfoxides in larger scale (15 mmol of each reactants)

Entry	Sulfides	Time (min)	Yield (%) <sup>a</sup>
1		20	92
2		25	93
3		23	89
4		26	91

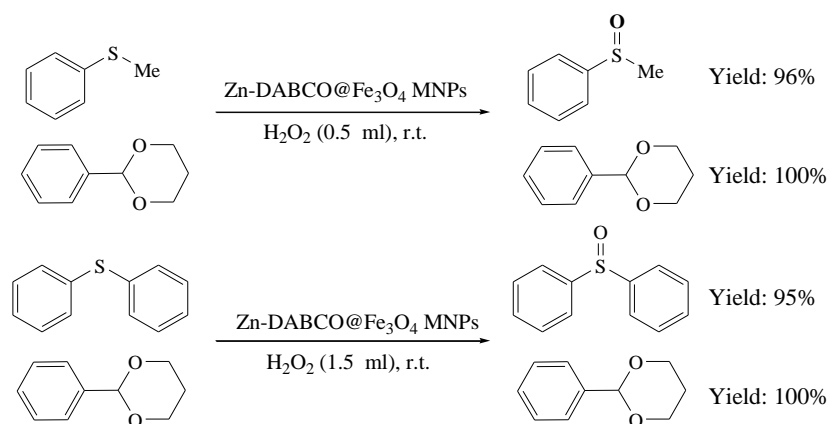
<sup>a</sup> Isolated yields

Finally, to demonstrate the high chemoselectivity of our method, we have

also conducted competitive oxidation of various sulfides with *O,O*-acetals using H<sub>2</sub>O<sub>2</sub> under

solvent-free conditions at room temperature (Scheme 3). As shown in Scheme 3, whereas various sulfides were converted to the

corresponding sulfoxides, *O,O*-acetals remained intact under the described reaction conditions.



**Scheme 3.** Selective oxidation of sulfides to sulfoxides in the presence of acetals at room temperature

## Conclusion

In this work, the magnetic heterogeneous Zn-DABCO@Fe<sub>3</sub>O<sub>4</sub> nanocatalyst was synthesized for the first time by DABCO on the magnetic Fe<sub>3</sub>O<sub>4</sub> nanoparticles. The synthesized catalyst was characterized using the XRD, SEM, TEM and VSM analysis. The results revealed that this novel catalyst has high catalytic activity in the selective oxidation of sulfides to sulfoxides under mild reaction conditions. The mild conditions and the use of a green, nontoxic, inexpensive, and reusable catalyst in high yields and in relatively short reaction time are some of the advantages of this method. Also, the involved catalyst can be magnetically separated from the reaction mixture and reused 8 times with no considerable change in its activity.

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## Supporting Information

Additional supporting information related to this article can be found, in the online version, at DOI: 10.48309/JMNC.2023.3.4.

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