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Catalytic synthesis of cyclic carbonates from epoxides and carbon dioxide by magnetic UiO-66 under mild conditions

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Email: stanges@sci.ui.ac.ir; moghadamm@sci.ui. ac.ir; mirkhani@sci.ui.ac.ir The catalytic activity of UiO-66@Fe₃O₄@SiO₂ catalyst was investigated in the fixation of carbon dioxide with epoxides under mild conditions. In this manner, a facile magnetization of UiO-66 was achieved simultaneously by simply mixing this metal– organic framework and silica-coated Fe₃O₄ nanoparticles in solution under sonication. The prepared catalyst was characterized using Fourier transform infrared and UV–visible spectroscopies, X-ray diffraction, transmission and field emission scanning electron microscopies, N₂ adsorption and inductively coupled plasma atomic emission spectroscopy. This new heterogeneous catalyst was applied as a highly efficient catalyst in the coupling of carbon dioxide with epoxides at mild temperatures and pressures. Furthermore, it could be easily recovered with the assistance of an external magnetic field and reused three consecutive times without significant loss of activity and mass.

KEYWORDS

CO₂ fixation, magnetization, metal–organic frameworks, UiO-66, UiO-66@Fe₃O₄@SiO₂

1 | INTRODUCTION

Carbon dioxide, an available, inexpensive, non-toxic and renewable carbon resource, has received much attention in recent decades from both economic and environmental points of view. Global warming is primarily a problem of accumulation of CO_2 as an important greenhouse gas in the atmosphere. So its capture, storage and utilization have received much attention.^[1,2]

One of the most promising methodologies in this area for chemical fixation of CO_2 is its conversion to cyclic carbonates via the reaction of it with epoxides. Cyclic carbonates have been widely used as synthetic intermediates, aprotic polar solvents and in many biomedical applications.^[3,4]

In recent decades numerous catalytic systems including metal oxides, ionic liquids, alkali metal salts, Lewis acids, transition metal complexes, porphyrins, polyoxometalates and Schiff bases have been developed for this transformation.^[5–14] Generally, these homogeneous catalysts provide high turnover number and high activity but they often suffer from difficulty of recycling and separation from products,

which limits the wide application of these catalysts. In order to overcome the separation problem, many heterogeneous catalysts have been developed.

Metal–organic frameworks (MOFs), which are metal–oxo clusters linked by tunable organic linkers,^[15–17] have attracted increasing attention because of their porosity, tunable cavities, various topologies and extraordinary surface areas, as well as their numerous potential applications in gas sorption or storage,^[18] luminescence,^[19] drug release,^[20] optoelectronics,^[21] chemical sensing^[22] and catalysis.^[23–29]

One of the most stable MOF structures, which has been synthesized by Lillerud and co-workers, is UiO-66.^[30] This MOF, which also has high surface area and nanometre pore size useful for catalysis, has been directly synthesized by the reaction of the corresponding metals with benzenedicarboxylate via a conventional solvothermal method.

Magnetic nanoparticles such as Fe_3O_4 nanoparticles have been widely used in the collection and separation of bioactive molecules, biomedical applications and targeted drug delivery. Combination of MOFs and magnetic nanoparticles has obvious advantages in adsorption and separation.^[31–34] The separation of magnetic nanoparticles using a magnet is typically more effective than filtration or centrifugation and which prevents the loss of catalyst. This separation is economical, simple and promising for industrial applications.^[35] Zhao et al. reported an efficient strategy for fabricating a magnetic MOF as sorbent for removing organic compounds from simulated water samples.^[31] Yuan and co-workers reported a facile and environmentally friendly fabrication of a novel type of magnetic porous MOF-based nanocomposites that can be potentially used for targeted drug delivery.^[36]

In the work reported in this paper, we successfully synthesized magnetic UiO-66 as a new hybrid catalyst. The catalytic activity of this new heterogeneous catalyst was investigated in the chemical fixation of CO_2 with epoxides in the presence of LiBr as co-catalyst at ambient pressure and temperature (Scheme 1). This catalyst was highly efficient, stable and reusable in the conversion of epoxides with CO_2 into cyclic carbonates.

2 | EXPERIMENTAL

2.1 | Reagents and methods

All materials were of commercial reagent grade and were obtained from Merck or Fluka. Fourier transform infrared (FT-IR) spectra were obtained with potassium bromide pellets in the range 400–3500 cm⁻¹ with a JASCO 6300 spectrophotometer. Field emission scanning electron microscopy (FE-SEM) was conducted with a Hitachi S-4700 instrument. X-ray diffraction (XRD) patterns were recorded using a Bruker D₈ Advance X-ray diffractometer equipped with nickel monochromatized Cu K_{α} radiation ($\lambda = 1.5406$ Å). Specific surface area was measured by adsorption-desorption of N₂ gas using an Micromeritics ASAP 2000 instrument. Inductively coupled plasma (ICP) analyses were carried out with a PerkinElmer Optima 7300 DV spectrometer. GC experiments were performed with a Shimadzu GC-16 A instrument using a 2 m column packed with silicon DC-200 or Carbowax 20 M. In the GC experiments, n-decane was used as the internal standard. An alternating gradient force magnetometer (AGFM model, Kavir) was used. UiO-66 was prepared based on the procedure reported by Lillerud and co-workers.^[30] The Fe₃O₄ magnetic nanoparticles (MNPs) were synthesized and coated with silica as reported in the literature.^[37]



SCHEME 1 Chemical fixation of CO_2 with epoxides catalysed by UiO-66@Fe₃O₄@SiO₂

2.2 | Magnetization of UiO-66

In a typical and very simple procedure, $Fe_3O_4@SiO_2$ (100 mg) and UiO-66 (60 mg) in methanol were placed in a 25 ml glass vial under ultrasonication for 2 h for the magnetization of UiO-66 in the form of UiO-66@Fe₃O₄@SiO₂ microspheres. Then, an external magnet was attached to the outside bottom of the vial, so that the prepared catalyst was gathered to the bottom of the vial, and the supernatant discarded, and dried in an air oven at 75 °C.

2.2.1 | General procedure for CO₂ fixation

First, the reaction parameters including the type of solvent, amount of catalyst, the kind and amount of co-catalyst and temperature were optimized in the reaction of CO2 with 1,2-epoxyoctane. Under the optimized reaction conditions, the reaction of various epoxides (linear and cyclic) with CO_2 was investigated in the presence of UiO-66@Fe₃O₄@SiO₂. To a solution of epoxide (1 mmol) in dimethylformamide (DMF; 3 ml) were added lithium bromide (2 mmol) and the catalyst UiO-66@Fe₃O₄@SiO₂ (40 mg, 0.023 mmol). Carbon dioxide gas was bubbled into the solution at atmospheric pressure. The progress of the reaction was monitored by GC. At the end of each reaction, the catalyst was separated from the reaction mixture using an external magnet, washed with DMF and diethyl ether, and reused. The catalyst was consecutively reused several times without detectable catalyst leaching or significant loss of its activity. The amount of Zr and Fe leached in the filtrates was determined by ICP analysis. The stability of UiO-66@Fe₃O₄@SiO₂ was investigated using XRD and FT-IR spectroscopy.

3 | RESULTS AND DISCUSSION

3.1 | Characterization of catalyst

The crystallinity of the framework and nanoparticles was studied using XRD analysis. The XRD pattern of UiO-66 (Figure 1A) is the same as the profile of UiO-66 synthesized previously by Lillerud et al.^[30] Also, the XRD pattern of Fe₃O₄@SiO₂ (Figure 1B) is the same as that of Fe₃O₄@SiO₂ in the literature.^[37,38] As can be seen in Figure 1(C), the crystallinity of UiO-66 and Fe₃O₄@SiO₂ is well retained after the magnetization reaction, because all diffraction peaks of the MOF and MNPs can be readily indexed to the pattern of UiO-66@Fe₃O₄@SiO₂. Thus the XRD pattern of the new nanocatalyst indicates that the basic lattice structure of UiO-66 is well retained. Also, the sharp peaks indicate the excellent crystallinity of the framework.

The FT-IR reflectance spectra of the samples are shown in Figure 2. The FT-IR spectrum of UiO-66 synthesized according to the literature is shown in Figure 2(A). The peak at 1550–1630 cm⁻¹ corresponds to C=O of carboxylates coordinated with the metal centres by oxygen during the



FIGURE 1 XRD patterns: (A) UiO-66; (B) $Fe_3O_4@SiO_2$; (C) UiO-66@Fe_3O_4@SiO_2; (D) recovered catalyst



FIGURE 2 FT-IR spectra: (A) UiO-66; (B) $Fe_3O_4@SiO_2$; (C) UiO-66@Fe_3O_4@SiO_2; (D) recovered catalyst

deprotonation process.^[27,28] In each spectrum a weak peak in the region 1450–1580 cm⁻¹ belongs to C–C in the aromatic compound of the organic linker. The strong peak at around 1400 cm⁻¹ is ascribed to C–O bond of C–OH group of carboxylic acid.

Figure 2(B) shows the FT-IR spectrum of the synthesized MNPs which exhibits characteristic bands at 580 cm⁻¹ (Fe–O), 950 cm⁻¹ (Si–OH) and 1091 cm⁻¹ (Si–O–Si),^[37,38] which are seen in the spectrum of UiO-66 after the magnetization reaction (Figure 2C), proving the successful synthesis of UiO-66@Fe₃O₄@SiO₂.



FIGURE 3 FE-SEM images: (A) UiO-66; (B) UiO-66@Fe₃O₄@SiO₂



FIGURE 4 SEM–EDX spectrum of UiO-66@Fe₃O₄@SiO₂ (Al is from the sample holder)

To investigate the surface morphology of the MOF and synthesized magnetic MOF, the samples were characterized using FE-SEM (Figure 3). FE-SEM images show a small cubic inter-grown architecture for UiO-66 (Figure 3A), and, as can be seen in Figure 3(B), the MNPs are homogeneously dispersed on the surface of UiO-66. The energy-dispersive X-ray (EDX) results, obtained from FE-SEM analysis, for UiO-66@Fe₃O₄@SiO₂ (Figure 4) clearly show the presence of C, O, Si, Zr and Fe in the nanocatalyst.

The porous structure of UiO-66 and the magnetic MOF were investigated using nitrogen physisorption measurements (Figure 5), and the textural parameters are presented in Table 1. The results clearly show a predictable decrease in pore volume (from 0.558 to 0.411 cm³ g⁻¹) and in the BET surface area (from 1315 to 532 m² g⁻¹) which can be attributed to the blocking of UiO-66 cavities by surface-located iron MNPs due to the magnetization reaction.

The magnetic properties of the samples are shown in Figure 6. The magnetic saturation (M_S) values of Fe₃O₄, Fe₃O₄@SiO₂ and UiO-66@Fe₃O₄@SiO₂ are about 69.4, 39.4 and 8.1 emu g⁻¹, respectively. The results indicate that magnetic property of Fe₃O₄ decreases as a result of silica coverage and magnetization of UiO-66, but the saturation



FIGURE 5 N_2 adsorption–desorption isotherms: (A) UiO-66; (B) UiO-66@Fe₃O₄@SiO₂ (O, adsorption; •, desorption)

TABLE 1 Textural parameters of UiO-66 and the prepared catalyst

Sample	Pore volume (cm ³ g ⁻¹)	Specific surface area $(m^2 g^{-1})$
UiO-66(Zr)	0.558	1315
UiO-66@Fe ₃ O ₄ @SiO ₂	0.411	532.3

magnetization value of 8.1 emu g^{-1} for UiO-66@Fe₃O₄@SiO₂ is enough to make it susceptible to a magnetic field and easy to isolate from reaction media.

3.2 | Catalytic experiments

The catalytic activity of UiO-66@Fe₃O₄@SiO₂ was evaluated in the coupling reaction of epoxides and CO₂ to generate relevant cyclic carbonates. All effective parameters such as solvent, amount of catalyst, kind of co-catalyst and temperature were optimized. At first, the effect of catalyst amount on the formation of 1-octene carbonate was investigated using various amounts of UiO-66@Fe₃O₄@SiO₂ in the presence of 1,2-epoxyoctane with CO₂ under atmospheric pressure at 80 °C. The best results are obtained using 40 mg (0.023 mmol) of UiO-66@Fe₃O₄@SiO₂. As evident from Table 2, no product is obtained in the absence of the catalyst. Also the selectivity for all reactions is almost 100% and no by-product is detected by GC analysis.

The effect of solvent was also investigated in the model reaction. The results, which are summarized in Table 3, show that the solvent is an important factor in these reactions. In methanol, only trace amounts of the corresponding carbonate are obtained in the presence of the catalyst, while in DMF the yield increases moderately. The positive effect of the solvent reported by Aresta et al.^[39] can be attributed to an increase of the nucleophilicity of the oxygen atom of the epoxide or CO_2 in DMF.



FIGURE 6 Magnetic hysteresis: (A) Fe_3O_4 microspheres; (B) $Fe_3O_4@SiO_2$; (C) UiO-66@Fe_3O_4@SiO_2

TABLE 2 Effect of catalyst amount on formation of 1-octene carbonate from reaction of 1,2-epoxyoctane with CO_2^a

Entry	Amount of catalyst (mg)	Yield after 10 h $(\%)^b$
1	Without catalyst	0
2	Fe ₃ O ₄ @SiO ₂	30
3	30	87
4	40	98
5	50	98
6	60	98

^aReaction conditions: 1,2-epoxyoctane (1 mmol), LiBr (0.2 mmol), DMF (3 ml), CO₂ atmospheric pressure, T = 80 °C.

^bGC yield based on starting epoxide.

TABLE 3 Effect of solvent on formation of 1-octene carbonate from reaction of 1,2-epoxyoctane with CO_2^a

Entry	Solvent	Yield after 10 h (%) ^b	<i>T</i> (°C)
1	Dimethylformamide	98	80
2	Dichloromethane	55	30
3	Acetonitrile	60	50
4	Methanol	8	45
5	Tetrahydrofuran	51	60

^aReaction conditions: 1,2-epoxyoctane (1 mmol), LiBr (0.2 mmol), solvent (3 ml), CO₂ atmospheric pressure, UiO-66@Fe₃O₄@SiO₂ (0.023 mmol).

^bGC yield based on starting epoxide.

TABLE 4 Effect of temperature on formation of 1-octene carbonate from reaction of 1,2-epoxyoctane with CO_2^a

Entry	Temperature (°C)	Yield after 10 h (%) ^b	
1	Room temperature	20	
2	50	45	
3	70	83	
4	80	98	
5	90	98	

^aReaction conditions: 1,2-epoxyoctane (1 mmol), LiBr (0.2 mmol), UiO-66@Fe₃O₄@SiO₂ (0.023 mmol), DMF (3 ml), CO₂ atmospheric pressure. ^bGC yield based on starting epoxide.

TABLE 5 Effect of co-catalyst on formation of 1-octene carbonate fromreaction of 1,2-epoxyoctane and CO_2^a

Entry	Co-catalyst	Yield after 10 h $(\%)^{b}$
1	Without co-catalyst	5
2	Tetrabutylphosphonium bromide	86
3	Tetrabutylammonium bromide	73
4	Sodium chloride	20
5	Sodium bromide	65
6	Lithium bromide	98

^aReaction conditions: 1,2-epoxyoctane (1 mmol), co-catalyst (0.2 mmol), UiO-66@Fe₃O₄@SiO₂ (0.023 mmol), DMF(3 ml), CO₂atmospheric pressure, T = 80 °C.

^bGC yield based on starting epoxide.

TABLE 6 Effect of co-catalyst amount on formation of 1-octene carbonate from 1,2-epoxyoctane and CO₂^a

Entry	Amount of co-catalyst (mmol)	Yield after 10 h (%) ^b
1	Without co-catalyst	5
2	0.05	83
3	0.1	90
4	0.2	98
5	0.3	98

^aReaction conditions: 1,2-epoxyoctane (1 mmol), LiBr as co-catalyst, UiO-66@Fe₃O₄@SiO₂ (0.023 mmol), DMF (3 ml), CO₂ atmospheric pressure, T = 80 °C.

^bGC yield based on starting epoxide.

Temperature is another factor that influences the coupling reaction. As evident from Table 4, the highest yield of 1-octene carbonate is obtained at 80 °C, and increasing the temperature to 90 °C does not affect the yield or reaction time.

The results show that in the absence of catalyst or cocatalyst no reaction progress is observed which indicates that the presence of the catalyst as Lewis acid (electrophile) and co-catalyst as Lewis base (nucleophile) is necessary for obtaining the highest yield.^[14,40] Since Zr(IV) is a good Lewis acid,^[41,42] various Lewis bases as co-catalyst were checked in the reaction of 1,2-epoxyoctane with CO₂ in the presence of this catalyst (Table 5). LiBr as a co-catalyst shows higher activity than quaternary halide salts (ammonium and phosphonium) in this reaction, probably due to less of a steric effect.

The amount of co-catalyst was also optimized. The results show that in the presence of 0.2 mmol of co-catalyst, the highest yield is obtained for UiO-66@Fe₃O₄@SiO₂



(0.023 mmol) after 10 h. Upon increasing the amount of co-catalyst, no improvement in yield is observed (Table 6). It is noteworthy that when LiBr is used as catalyst, no noticeable product is observed in the reaction mixture.

Under the optimized reaction conditions for the catalyst, the reaction of various epoxides (linear and cyclic) with CO₂ was investigated. All reactions were carried out under atmospheric pressure of CO2. The results are summarized in Table 7. Epoxides bearing aromatic, aliphatic, electronwithdrawing and electron-donating substituents are converted to their corresponding carbonates with 100% selectivity. The turnover frequencies (TOFs) for reaction of linear epoxides are higher than those for cyclic epoxides. The reactivity of a disubstituted epoxide such as cis-stilbene oxide was also tested and a yield of 23% of the corresponding cyclic carbonate is produced. It seems that the less the steric effect, the shorter the reaction time. It has been well documented that due to the porosity of MOFs, these materials can be used for storage of various gases. Therefore, they can increase the local concentration of CO₂ around active sites of the catalyst which in turn increases the catalytic activity.

3.3 | Catalyst reuse and stability

The reusability of a catalyst is an important factor from economic and industrial points of view. Therefore, we investigated the recyclability of UiO-66@Fe₃O₄@SiO₂ in the multiple sequential coupling reaction of 1,2-epoxyoctane with CO₂. After using the catalyst for three consecutive runs, the yield is 80% (Figure 7). Zr and Fe leaching was determined by analysing collected filtrates using the ICP method. The results show that after using the catalyst for three

TABLE 7 Results of coupling of various epoxides and CO₂ catalysed by UiO-66@Fe₃O₄@SiO₂^a

$\begin{array}{c} R \\ \swarrow \\ O \end{array} \xrightarrow{R'} \begin{array}{c} \text{UiO-66@Fe_3O_4@SiO_2} \\ \hline \\ CO_2, DMF, LiBr \end{array} \xrightarrow{R'} O \\ O \\ O \end{array}$					
Entry	R	R′	Yield (%) ^b	Time (h)	TOF (h^{-1})
1	CH ₃ (CH ₂) ₃	Н	98	5	8.7
2	CH ₃ (CH ₂) ₅	Н	98	10	4.3
3	CICH ₂	Н	98	6	7.2
4	Cyclohexyl	Н	98	14	3.0
5	(CH ₃) ₂ CHOCH ₂	Н	100	10	4.4
6	CH ₂ CHCH ₂ OCH ₂	Н	95	24	1.72
7	C ₆ H ₅	Н	98	30	1.4
8	(C ₆ H ₅)OCH ₂	Н	88	11	3.5
9	C ₆ H ₅	C ₆ H ₅	23	20	0.5

^aReaction conditions: epoxide (1 mmol), LiBr (0.2 mmol), UiO-66@Fe₃O₄@SiO₂ (0.023 mmol), DMF (3 ml), CO₂ atmospheric pressure, T = 80 °C. ^bGC yield.



FIGURE 7 Recyclability of UiO-66@ $Fe_3O_4@SiO_2$ in the formation of 1octene carbonate from 1,2-epoxyoctane with CO_2

consecutive times, the amount of Zr leached is about 22%, but no marked Fe is detected in the filtrates using ICP analysis. This means that the catalyst is stable under the reaction conditions, and can be recovered and reused. The nature of the recovered catalyst was investigated using FT-IR and XRD analyses. The XRD pattern indicates that the basic lattice structure of UiO-66@Fe₃O₄@SiO₂ is not altered after three cycles (Figure 1D). Also, by considering the FT-IR spectrum, it is proved that the catalyst has retained its nature during the reaction (Figure 2D).

4 | CONCLUSIONS

In summary, we have succeeded in designing a novel catalyst by facile magnetization of UiO-66 MOF for the synthesis of cyclic carbonates from epoxides and CO_2 . The product separation and catalyst recycling are possible using an external magnet. The prepared catalyst can be recovered and reused up to three times without significant loss of activity and mass. High activity, selectivity, easy work-up and extremely mild reaction conditions are other advantages of this new catalyst.

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