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Incorporation of Metal Ions into Silica-Grafted Imidazolium-Based Ionic Liquids to Efficiently Catalyze Cycloaddition Reactions of CO_2 and Epoxides

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Abstract A variety of imidazolium-based ionic liquids (ILs) have been widely developed to effectively catalyze the cycloaddition reactions of CO_2 and epoxides. To further improve the catalytic performance of those IL catalysts, we incorporated various metal chlorides ($CoCl_2$, $NiCl_2$, $CuCl_2$, $ZnCl_2$, and $MnCl_2$) into silica-grafted 1-methyl-3-[(triethoxysilyl)propyl] imidazolium chloride to produce a series of heterogeneous catalysts. Catalytic reaction tests demonstrated that the incorporation of such metal ions can significantly enhance the catalytic reactivity of the silica-grafted ILs towards cycloaddition reactions of CO_2 and epoxides that produce cyclic carbonates in solvent-free conditions. In addition, the effects on the catalytic reactivity of reaction parameters including reaction time, reaction temperature and CO_2 pressure were investigated.

Keywords Cyclic carbonate · CO_2 · Heterogeneous catalysts · Ionic liquids · Metal ions

1 Introduction

Recently, the development of an efficient process for chemical fixation of CO_2 has attracted tremendous interest. CO_2 is a main greenhouse gas responsible for global warming. On the other hand, it provides an abundant, cheap and non-toxic carbon source for various organic reactions. As one of the most promising strategies, cycloaddition reactions between CO_2 and epoxides that produce cyclic carbonates have been extensively explored [1–4], since cyclic carbonates are important industrial products with wide-ranging applications as both polar aprotic solvents and versatile ingredients in organic synthesis [5, 6]. In view of the chemical inertness of CO_2, catalysts are indispensable in the production of cyclic carbonates via cycloaddition reactions. For this purpose, a number of catalysts, such as alkali metal salts [7, 8], transition metal complexes [9, 10], Schiff bases [11, 12], and ionic liquids (ILs) [13–17], have been developed so far. Among them, ILs have received more attention owing to their excellent catalytic performance towards cycloaddition reactions, in addition to a number of other intrinsic advantages including environmental friendliness, non-volatility, low melting temperatures, and the fact that they are easy to separate.

Considering the increasing demands on new catalysts in terms of reuse, ease of operation, and high catalytic performance with minimal of catalyst consumption, it is quite necessary to graft ILs onto suitable substrates to generate heterogeneous catalysts towards the chemical fixation of CO_2 with epoxides. To this end, various heterogeneous catalysts have been developed by directly grafting imidazolium-based ILs onto the surfaces of silica or polymer materials. For example, in our group, we have synthesized a series of catalysts by grafting various imidazolium-based ILs bearing alkyl motifs with different chain lengths and different halide counter-anions onto the surfaces of commercial silica and polystyrene beads. The resulting heterogeneous catalysts exhibited excellent catalytic performance towards the cycloaddition reaction of CO_2 and allyl glycidyl ether (AGE) [18, 19].

Inspired by the sustained interest in the efficient synthesis of cyclic carbonates via cycloaddition reactions, two pioneering strategies for the development of novel catalytic systems have been explored recently. One strategy has

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focused on introducing a certain amount of co-catalysts (such as various Lewis acids, water, various alcohols, and carboxylic acids) into the traditional IL catalysts [19, 20]. These new catalytic systems tend to give an enhanced yield of the cyclic carbonates compared with the bare ILs. The other efficient alternative to improve the reactivity of ILs is to tailor the alkyl chains of the ILs with functional end moieties. Our recent research revealed that the incorporation of –COOH and –OH groups at the chain ends of imidazolium-based ILs can significantly improve their catalytic activity and selectivity [21].

More recently, Qiao et al. [22] reported that combining zinc ions with polystyrene-immobilized imidazolium ILs also can effectively promote the cycloaddition reaction of styrene oxide and CO_2. According to previous reports, imidazolium-based ILs can interact with various metal ions, forming a variety of complexes with catalytic functions toward various versatile reactions, such as the Heck reaction [23–25], the Kharash reaction [26, 27], hydrodimerization reactions [28–30], and hydrogenation reactions [31–33]. However, it still remains little systematic investigation into the effects of the incorporation of different metal ions into imidazolium-based ILs on the cycloaddition reaction of CO_2 and epoxides.

In this study, we have incorporated various metal chloride ions into silica-grafted imidazolium-based ILs, and used them as heterogeneous catalysts to catalyze cycloaddition reactions of CO_2 and epoxides. In addition, the effects of reaction parameters including the reaction temperature, CO_2 pressure and reaction time were also investigated.

2 Experimental

2.1 Materials

Anhydrous toluene, 1-methylimidazole (>99%) and 3-triethoxysilylpropyl chloride were purchased from Fluka. Anhydrous $ZnCl_2$, $NiCl_2$, $CoCl_2$, $CuCl_2$, $MnCl_2$, and silica (surface area: 500 m^2/g, average pore size: 60 Å) and other chemicals were purchased from Aldrich. All chemicals except the silica were used as received without further purification.

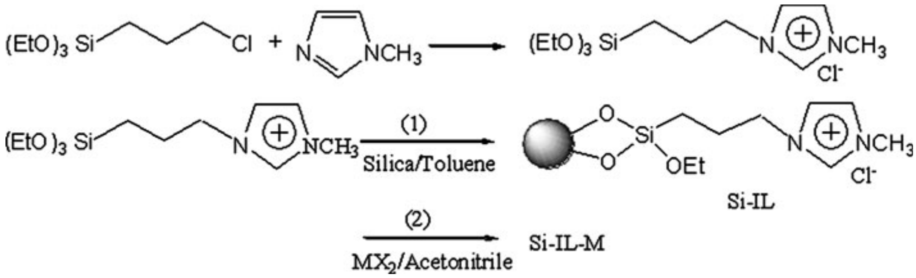
2.2 Silica-Grafted Metal Ion-Containing IL Catalyst Preparation

The catalyst preparation procedures are schematically illustrated in Scheme 1. All processes were conducted under nitrogen atmosphere. To synthesize 1-methyl-3-[(triethoxysilyl)propyl] imidazolium chloride, 1-methylimidazole (10 mL) and (3-chloropropyl) triethoxysiliane (80 mL) were mixed in a dry 250 mL flask under nitrogen flow. The mixture was stirred at 70 °C for 48 h, after which the reaction mixture was cooled to room temperature. Then, the unreacted reactants were removed by extraction with diethyl ether. The residual ether was evaporated under reduced pressure. Finally, IL was obtained with 95% yield. ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.58 (t, $J = 8.1$ Hz, 2H), 1.18 (t, $J = 6.9$ Hz, 9H), 1.98 (p, $J = 7.6$ Hz, 2H), 3.79 (q, $J = 6.9$ Hz, 6H), 4.09 (s, 3H), 4.30 (t, $J = 7.2$ Hz, 2H), 7.32 (s, 1H), 7.62 (s, 1H), 10.58 (s, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): δ 6.8, 18.0, 23.9, 36.1, 51.3, 58.0, 121.9, 124.0, 138.1 ppm.

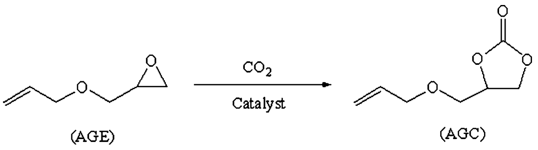
Prior to immobilization, commercial silica was purified with hot Piranha solution (H_2O_2 and H_2SO_4) and then rinsed in water and dried under a stream of nitrogen. The pretreated silica and IL were co-dispersed in anhydrous toluene in a flask. The mixture was refluxed for 24 h under nitrogen atmosphere. The resultant product was filtered and washed with dichloromethane to remove excess IL and obtain Si–IL. In the following step, Si–IL was added to an acetonitrile solution of MCl_2 (i.e., $ZnCl_2$, $NiCl_2$, $CoCl_2$, $CuCl_2$, or $MnCl_2$) in a 50-mL Schlenk tube for 24 h. Acetonitrile was removed by Soxhlet extraction with acetone for 48 h. The prepared silica-grafted metal ion-containing ILs are designed as Si–IL–M (i.e., Si–IL–Zn, Si–IL–Ni, Si–IL–Co, Si–IL–Cu, or Si–IL–Mn).

2.3 Catalytic Reactions

All reactions were carried out in a 55 mL stainless-steel reactor with a magnetic stirrer. Allyl glycidyl carbonate (AGC) was synthesized via the coupling reaction between AGE and CO_2 in the presence of Si–IL–M. In a typical reaction process, 0.5 g of catalyst was introduced into a reactor containing 40 mmol of AGE (Scheme 2). The



Scheme 1 Schematic illustrations for the synthetic procedures of Si–IL–M catalysts



Scheme 2 Synthesis of AGC via a cycloaddition reaction of CO_2 and AGE in the presence of Si–IL–M catalyst

reaction was carried out under a preset pressure of carbon dioxide at different temperatures. After the reaction completed, the reactor was put into iced water to cool down to the ambient temperature. The obtained product was analyzed with a gas chromatograph (Agilent HP 6890 A) equipped with a capillary column (HP-5, 30 m × 0.25 µm) using a flame-ionized detector and gas chromatography/mass spectrometry (GC–MS, Micromass, UK) analysis.

2.4 Characterization

The elemental analysis (EA) was carried out using a Vario EL III. Each 2 mg samples was heated to 1,100 °C and sulfanilic acid was used as a standard. Solid-state NMR was carried out with ^{29}Si and ^{13}C frequencies of 79.5 and 100.6 MHz, respectively, on a BRUKER-400 WB MAS probe. ^{29}Si magic-angle spinning (MAS) spectra were measured at room temperature with the following conditions: MAS at 5 kHz, a $\pi/2$ pulse of 6.5 µs, a repetition delay of 60 s, and a total of 3,928 scans that were referenced to tetramethylsilane. ^{13}C cross-polarization spectra were measured with a recycle delay of 5 s for a total of 1,024 scans with the following conditions: MAS at 5 kHz, and a л/2 pulse of 7 µs. Inductively coupled plasma-optical emission spectrometry (ICP-OES) was conducted with an Activa spectrometer manufactured by JY Horiva. The Raman spectrum was measured with a Vertex 80 V spectrometer, Bruker, Germany. The AGE concentration was determined by gas chromatography (HP 6890 A) equipped with a HP-5 capillary column.

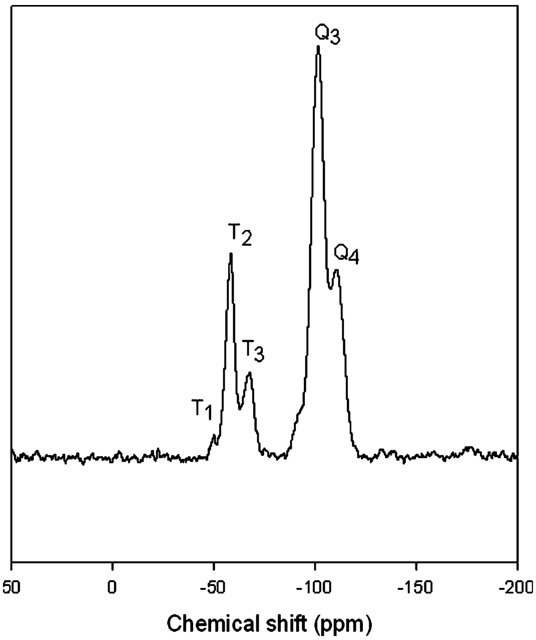


Fig. 1 ^{29}Si NMR spectrum of Si–IL

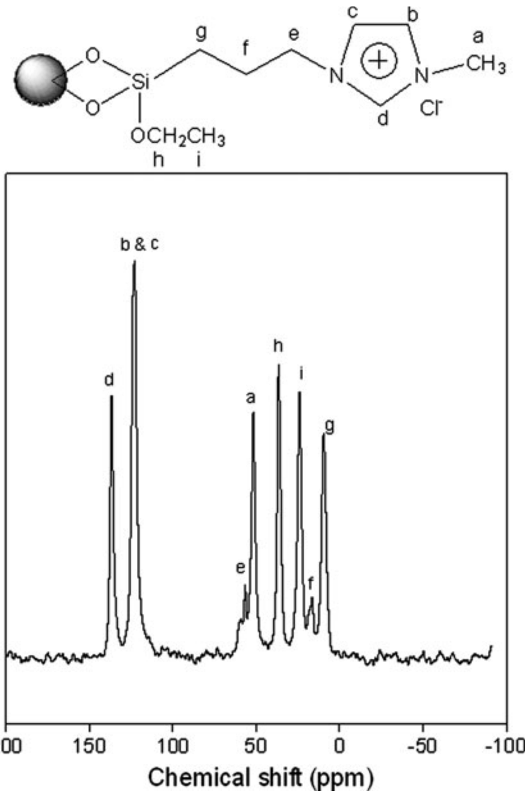


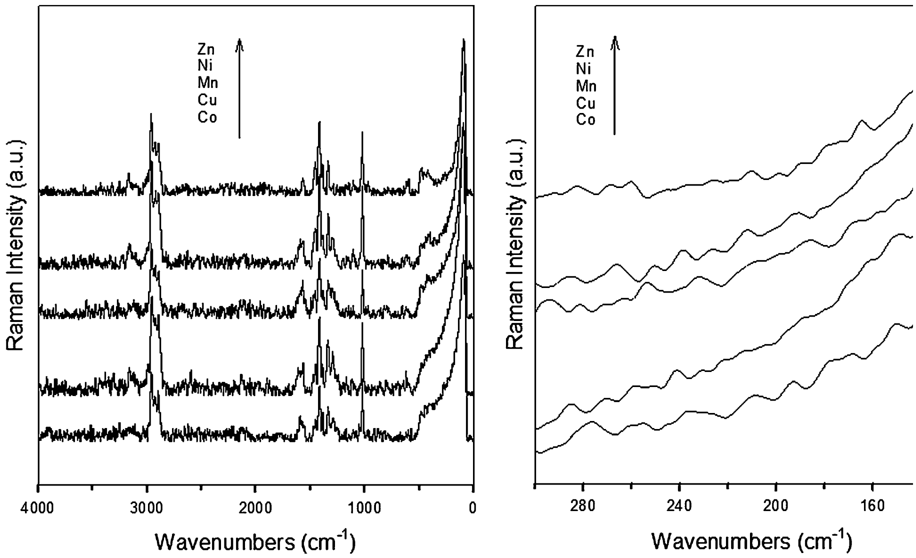
Fig. 2 Solid ^{13}C NMR spectrum of Si–IL

3 Results and Discussion

3.1 Characterization of Catalysts

The synthesized silica-grafted 1-methyl-3-[(triethoxysilyl)propyl] imidazolium chloride (Si–IL) was characterized using solid-state ^{29}Si and ^{13}C MAS-NMR analysis. As shown in Fig. 1, there were two peaks centered at −101.8 and −110.7 ppm in the ^{29}Si MAS-NMR spectrum corresponding to Q_3 ($Si(OSi)_3OH$) and Q_4 ($Si(OSi)_4$) silicon atoms, respectively. The peaks situated at −67.8, −8.4, and −50.1 ppm were assigned to T_3 ($Si(OSi)_3R$), T_2 ($Si(OSi)_2ROH$) and T_1 ($Si(OSi)_1R(OH)_2$) organosiloxane, which indicates the presence of organic functionalization moieties in the silica. Figure 2 shows the ^{13}C MAS-NMR spectrum of the Si–IL. The peaks at chemical shifts of 122.8 and 136.7 ppm correspond to the three imidazole ring carbon atoms. The signal at 65 ppm is attributed to the carbon

Fig. 3 Raman spectra of synthesized Si–IL–M heterogeneous catalysts



atoms connected to imidazole ring and the other carbon atoms give peaks from 5.3 to 58.4 ppm. These results further evidenced the successful graft of the IL onto the silica surface. As determined by EA, the amount of IL grafted onto the silica in the resultant Si–IL is 1.32 mmol/g (9.46 wt% C; 3.52 wt% H; 3.70 wt% N).

After incorporation of various metal ions into Si–IL, the resulting Si–IL–M catalysts were subjected to Raman analysis (Fig. 3). Compared with bare Si–IL, the metal-incorporated Si–ILs exhibited new bands centered at 275, 260, 280, 270 and 240 cm^{-1}, which are characteristic of the presence of $[ZnCl_4]^{2-}$, $[NiCl_4]^{2-}$, $[CoCl_4]^{2-}$, $[CuCl_4]^{2-}$ and $[MnCl_4]^{2-}$, respectively. This fact indicates the successful incorporation of metal ions into Si–IL. As calculated from ICP analysis results (Table 1), the metal loading amounts were 0.58, 0.29, 0.27, 0.23, and 0.50 mmol/g for Si–IL–Zn, Si–IL–Cu, Si–IL–Co, Si–IL–Ni and Si–IL–Mn, respectively.

Table 1 ICP-OES analysis of Si–IL–M catalysts

Catalyst	Metal (ppm)	Loading amount of metal (mmol/g)
Si–IL–Zn	37963.6	0.58
Si–IL–Cu	18496.7	0.29
Si–IL–Co	15694.5	0.27
Si–IL–Ni	13785.2	0.23
Si–IL–Mn	27785.8	0.50

3.2 Effects of Different Metal Ions on the Cycloaddition Reaction

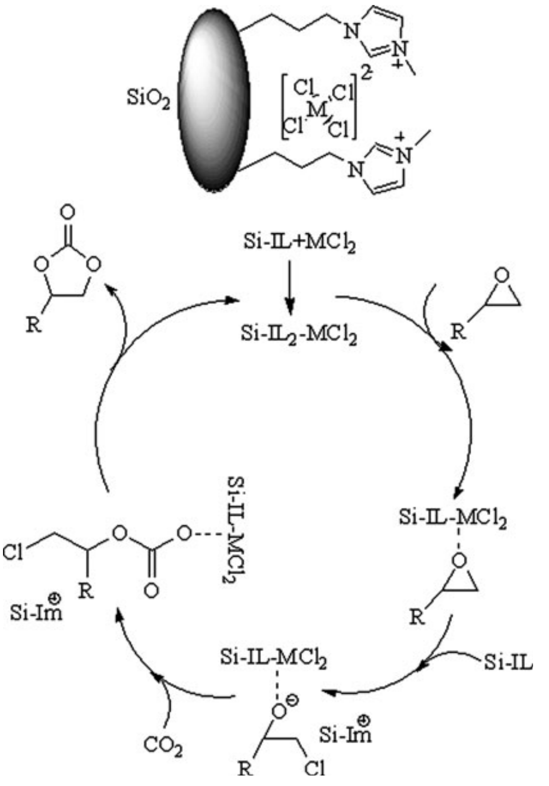
In order to investigate the effects of the incorporation of metal ions on the catalytic performance of the heterogeneous IL catalysts, a series of cycloaddition reactions between CO_2 and AGE was performed in the presence of the synthesized Si–IL and Si–IL–M catalysts under the same reaction conditions (110 °C, 0.86 MPa of CO_2 and 6 h of reaction time). As shown in Table 2, Si–IL exhibited 51.0% AGE conversion and 65.8% selectivity (entry 1). All

Table 2 Cycloaddition reactions of CO_2 and AGE in the presence of various Si–IL–M catalysts

Entry	Catalyst	Conversion (%)	Selectivity (%)	Yield (%)	TON
1	Si–IL	51.0	66	33.6	20
2	Si–IL–Zn	74.7	96	71.7	99
3	Si–IL–Ni	72.7	94	66.9	232
4	Si–IL–Mn	71.4	95	66.4	106
5	Si–IL–Cu	66.1	90	59.5	164
6	Si–IL–Co	59.1	93	55.0	163
7[a]	Si–IL/$ZnCl_2$	73.0	95	69.4	96

Reaction condition: catalyst = 0.5 g, AGE = 40 mmol, CO_2 pressure = 0.86 MPa, temperature = 110 °C, time = 6 h

[a] 0.58 mmol of $ZnCl_2$ was added to Si–IL as a co-catalyst



Scheme 3 Proposed mechanism for the coupling reaction of CO_2 and epoxide in the presence of Si–IL–M catalyst

Si–IL–M catalysts demonstrated activity and selectivity that were significantly enhanced compared to those of Si–IL. Upon the addition of metal ions into these Si–IL catalytic systems, the metal chloride firstly reacts with two adjacent IL moieties, forming a distorted metal imidazolium complex [L_2MCl_2], where L denotes an IL moiety (Scheme 3). The metal center serves as a Lewis acid, which enables the resultant L_2MCl_2 to coordinate with the oxygen of epoxide by forming a metal–O coordination bond as one L ligand dissociates. In the meantime, the dissociated IL nucleophilically attacks the less sterically hindered β-C of the coordinated epoxide, resulting in the formation of an intermediate (activated species). After the insertion of a CO_2 into the metal–O bond of the resulting intermediate, a metal carbonate active species is generated and finally produces a cyclic carbonate.

Further comparison of the activity and selectivity results indicated that the enhancement of the catalytic reactivity of the Si–IL–M heavily depended on the type of metal ions incorporated into the catalysts. From entry 2–7 in Table 2, the AGE conversion varied in the order of Si–IL–Zn > Si–IL–Ni > Si–IL–Mn > Si–IL–Cu > Si–IL–Co. Because the catalytic activity can depend on the amount of metal loading in the corresponding Si–IL–M catalyst, the turnover number (TON in mol AGC per mol metal ions) of each Si–IL–M catalyst was also calculated. As is shown in Table 2, the TON of Si–IL–M changed in the order of Si–IL–Ni > Si–IL–Co > Si–IL–Mn > Si–IL–Cu > Si–IL–Zn. Therefore, compared with zinc ions, the incorporation of the other investigated metal ions into Si–IL more strongly enhanced the cyclic carbonate yield. This interesting phenomenon is related to the intrinsic properties of the metal cations including their ionization potentials ($Mn^{2+} > Zn^{2+} > Co^{2+} > Cu^{2+} > Ni^{2+}$) and ionic radii ($Cu^{2+} > Ni^{2+} > Zn^{2+} > Co^{2+} > Mn^{2+}$). These different properties of the metal ions could produce a combined effect on their Lewis acidities [34] and thus determine their ability to coordinate with oxygen of the epoxide, the ability of the ligand to dissociate from the imidazolium–metal complex, and the stability of the intermediates.

For comparison, as a typical example, we also investigated the catalytic performance of the Si–IL catalyst in the presence of $ZnCl_2$ as a co-catalyst. Upon the direct addition of $ZnCl_2$ into the Si–IL catalytic system, it was found that AGE conversion significantly increased up to 73.0% with a dramatically enhanced selectivity of 95.0% (entry 7 in Table 2), which are much higher than those of bare Si–IL catalyst (51.0% of AGE conversion, 65.8% of selectivity, entry 1 in Table 2). However, it is slightly lower than that of Si–IL–Zn catalyst (74.7% AGE conversion, entry 2 in Table 2). A similar phenomenon was also observed in the reported homogeneous catalyst systems [35].

3.3 Effect of Reaction Parameter on the Cycloaddition Reaction

As a typical example, Si–IL–Zn was chosen as the catalyst in the following cycloaddition reactions to investigate the

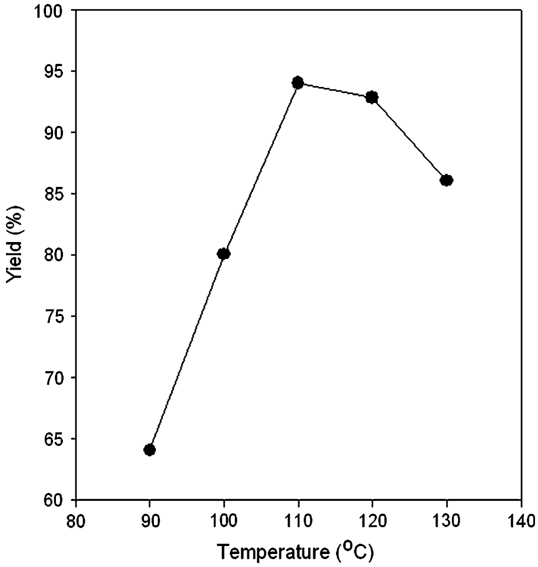


Fig. 4 Effects of reaction temperature on AGE conversion in the presence of Si–IL–Zn heterogeneous catalyst

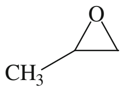
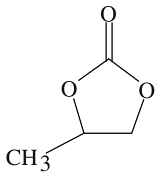
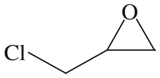
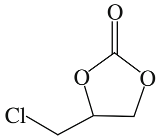
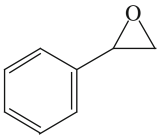
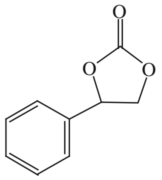
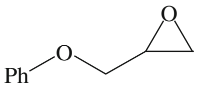
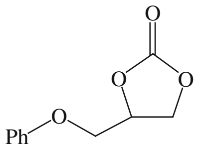
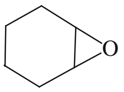
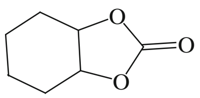
effects of the reaction parameters on the yield of AGC. As shown in Fig. 4, when the reaction temperature was increased from 90 to 110 °C, the AGE conversion dramatically increased from 64 to 94%. However, when the temperature was further increased to 130 °C, the AGC yield decreased to 86%. These changes in AGE conversion depending on the reaction temperature are attributed to two competitive factors including reaction efficiency and solubility of CO_2 gas phase in the reaction system. Higher reaction temperature directly leads to the increased reactivity, while simultaneously decreases the solubility of CO_2 gas phase [19]. When the catalytic reactions were carried out at lower temperature (90, 100 and 110 °C), the increased reactivity derived from the enhanced reaction temperature might play a dominant role to determine the overall reactivity. Nevertheless, when the reaction temperature reaches 130 °C, the lower solubility of CO_2 gas phase could be more dominant in the overall reactivity, leading to the decrease in AGE conversion. The effect of the CO_2 pressure on the AGC yield in the presence of Si–IL–Zn catalyst was also investigated (Table 3). As the CO_2 pressure increased from 0.86 to 1.76 MPa, the AGE conversion was significantly enhanced from 74.7 up to 95.8%. Nevertheless, further increasing the CO_2 pressure to 2 MPa, slightly decreased the AGE conversion to 94%. Such an effect of the CO_2 pressure on catalyst activity has also been observed in other catalytic systems [36, 37]. A high CO_2 pressure could enhance the absorption of CO_2 into the AGE solution, which would shift the equilibrium of the reaction towards the production of AGC, thus improving the AGE conversion. It has also been reported

Table 3 Effect of CO_2 pressure on the cycloaddition reactions of CO_2 and AGE in the presence of Si–IL–Zn catalyst

Pressure (MPa)	Conversion (%)	Selectivity (%)
0.86	74.7	96
1.34	83.0	98
1.76	95.8	98
2.00	94.2	97

Reaction conditions: catalyst (Si–IL–Zn) = 0.5 g, AGE = 40 mmol, temperature = 110 °C, time = 6 h

Table 4 Cycloaddition reactions of CO_2 and different epoxides react in the presence of Si–IL–Zn catalyst

Entry	Epoxides	Products	Temperature (°C)	Time (h)	Yield (%)
1			110	2	98
2			110	6	97
3			110	6	96
4			110	6	98
5			110	24	80

Reaction conditions: epoxide = 40 mmol, catalyst (Si–IL–Zn) = 0.5 g, CO_2 pressure = 1.76 MPa that a high CO_2 pressure increases the turnover rate of the CO_2/cyclohexene oxide cycloaddition reaction catalyzed by Cr–salen complexes [38]. However, when the CO_2 pressure reached 2.00 MPa, the dilution effect, which retards the interactions between AGE and the catalysts [18], slightly decreased the AGE conversion.

3.4 Synthesis of Cyclic Carbonate from Other Epoxides and CO_2

The Si–IL–M catalysts developed in this paper demonstrated efficient catalytic activity toward the cycloaddition of AGE and CO_2 under solvent-free conditions. To investigate the applicability of these catalysts to reactions producing other cyclic carbonates, various epoxides were utilized to chemically fix CO_2 via cycloaddition reactions in the presence of Si–IL–Zn catalyst under the optimized reaction conditions (110 °C, and 1.76 MPa of CO_2 pressure). As is shown in Table 4, Si–IL–Zn was found to be applicable to reaction of CO_2 and a variety of epoxides producing the corresponding cyclic carbonates. Compared with the other cycloaddition reactions, the reaction of cyclohexene oxide with CO_2 exhibited a much lower reaction rate owing to the hindrance effects of cyclohexene oxide [8, 39]. This relatively lower yield of cyclic carbonate occurred even when the reaction took a much longer time (80% conversion, for 24 h, entry 5 in Table 4). The stereochemical structure of *cis*-hexahydro-benzo [1, 3]-dioxolan-2-one can be deduced from the mechanism and ^1H NMR analysis, which was also demonstrated by other authors [39]. In contrast, when other epoxides were used to chemically fix CO_2, much higher yields of cyclic carbonates ranging from 96 to 99% were achieved.

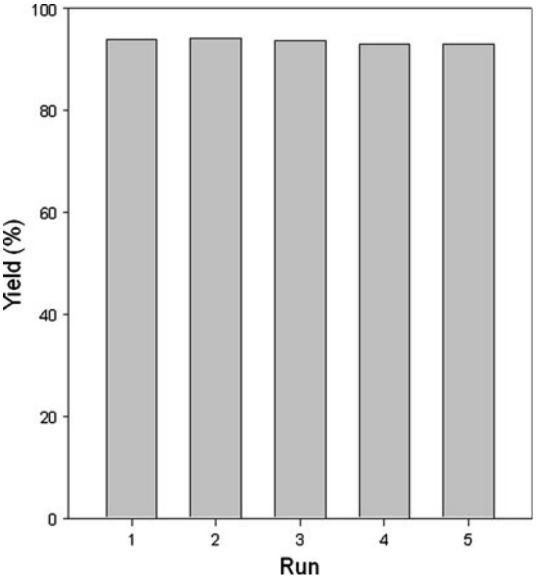


Fig. 5 The recycling test of Si–IL–Zn catalyst

3.5 Recycling Test

To examine the reusability of the prepared catalysts, Si–IL–Zn was used as candidate catalyst for the cycloaddition reaction of AGE and CO_2 (110 °C, 1.76 MPa of CO_2 pressure, and a reaction time of 6 h). After each reaction cycle, the catalyst was recovered by filtration and directly reused for another cycle of reaction. The AGC yields from five consecutive runs are shown in Fig. 5. Only A slight decrease in AGE conversion was observed after four recycling runs. Meanwhile, the selectivity for the AGC product remained constant (98%) in each run. These results indicated that Si–IL–Zn exhibits excellent reusability and stability for repetitive use in the cycloaddition reaction.

4 Conclusions

In this study, a series of heterogeneous catalysts have been successfully synthesized by incorporation of various metal ions into silica-grafted ILs. The resultant catalysts demonstrated significantly enhanced activity towards the production of cyclic carbonates via cycloaddition reaction of CO_2 and epoxides. Compared with the reported Si–IL–Zn system, the investigated Si–IL–M catalysts containing other metal ions (Mn^{2+}, Co^{2+}, Cu^{2+} and Ni^{2+}) exhibited much higher catalytic efficiencies (TON). The investigation on the effect of reaction parameters indicated that highest yield of cyclic carbonate can be achieved when the reaction is performed at 110 °C under 1.76 MPa of CO_2 for 6 h. In addition, the Si–IL–Zn catalyst exhibited excellent reusability.

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