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Highly efficient synthesis of cyclic carbonates from CO_2 and epoxides catalyzed by KI/lecithin

Jinliang Song, Binbin Zhang, Peng Zhang, Jun Ma, Jinli Liu, Honglei Fan, Tao Jiang, Buxing Han*

Beijing National Laboratory for Molecular Sciences, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100190, China

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ABSTRACT

Development of efficient, cheap and non-toxic catalysts for the cycloaddition of CO_2 with epoxides to produce five-membered cyclic carbonates is a very attractive topic. In this work, cycloaddition of CO_2 with propylene oxide (PO) to propylene carbonate (PC) catalyzed by potassium halides (KCl, KBr, and KI) in the presence of lecithin, which is renewable, non-toxic, environmentally benign and biocompatible, was studied. It was discovered that potassium halides and lecithin had excellent synergetic effect in promoting the reaction, and KI/lecithin catalytic system was the most efficient among them. The effects of temperature, pressure, reaction time, and amount of catalysts on the reaction were investigated using KI/lecithin as the catalyst, and the reaction conditions were optimized. At the optimal conditions, the KI/lecithin catalytic system was also very active and selective for the cycloaddition of CO_2 with other epoxides, such as glycidyl phenyl ether, epichlorohydrin, styrene oxide, 1,2-epoxyhexane, and glycidyl methacrylate. The mechanism for the synergetic effect was also studied.

1. Introduction

Conversion of CO_2 into value-added chemicals has attracted increasing interest because it is a non-toxic, abundant, relatively inexpensive and renewable C1 source. Many useful organic compounds have been synthesized using CO_2 as a building block [1–5], such as dimethyl carbonate [6–8], cyclic carbonates [9–11], N,N′-disubstituted ureas [12,13], urethanes [14–16], formic acid [17–20], methyl formate [21]. In particular, one of the most successful routes is the cycloaddition of CO_2 with epoxides to produce five-membered cyclic carbonates (Scheme 1), which can be used as aprotic polar solvents, precursors for producing polycarbonates, fine chemical intermediates, etc. [22–26].

Up to now, various catalysts have been developed for the coupling reaction of CO_2 and epoxides to produce cyclic carbonates, such as alkali metal salts [27–30], metal oxides [31,32], transition metal complexes [33–40], Schiff base [41,42], ion-exchange resins [43], functional polymers [44–46], quaternary ammonium and phosphonium salts [47–49], ionic liquids [50–53], lanthanide oxychloride [54,55], SiO_2 modified by quaternary ammonium or phosponium salts [56–59], gold nanoparticles supported on resins [60], CO_2 adducts of N-heterocyclic carbenes [61]. It has been reported that chitosan-supported zinc chloride exhibited high activity and selectivity for the synthesis of cyclic carbonates from cycloaddition between carbon dioxide and epoxides in the presence of [Bmim]Br as co-catalyst [62], and cyclodentrin could promote the reaction through hydrogen bonding [63]. As an abundant and inexpensive catalyst, alkali metal salts are among the most promising catalysts for the coupling reactions. However, co-catalysts are generally needed to enhance the catalytic efficiency due to the low activity of alkali metal salts when used alone [27–30]. For instance, Endo and co-workers reported that alkali metal salts could be used as catalyst for the synthesis of cyclic carbonates using crown ether as co-catalyst [28]. Huang and Shi found that alkali metal salts could catalyze cycloaddition of CO_2 with epoxides effectively in the presence of PPh_3 and phenol [29]. Cellulose could also improve the coupling reaction of CO_2 and epoxides catalyzed by KI [30]. Although these catalytic systems were effective, exploration of cheap and non-toxic co-catalysts is still highly desirable.

Lecithin, which is renewable, non-toxic, environmentally benign and biocompatible, contains a diglyceride, a phosphate group and a choline group (Scheme 2). As a non-toxic, abundant and cheap substance, lecithin is widely used in human food, animal feed, paint, pharmaceutical and industrial applications. In addition, as an excellent biosurfactant, lecithin has emulsification and lubricant properties [64–71].

As discussed above, many catalysts have been developed for the coupling reactions of CO_2 and epoxides. However, it is still very interesting to develop efficient catalytic systems for the cycloaddition of CO_2 with epoxides using cheap and non-toxic reagents. In this work, we conducted the reactions using potassium halides as the catalysts in the presence of lecithin at solvent-free condition. It

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* Corresponding author. Tel.: +86 10 6256 2821; fax: +86 10 6256 2821.
E-mail address: hanbx@iccas.ac.cn (B. Han).

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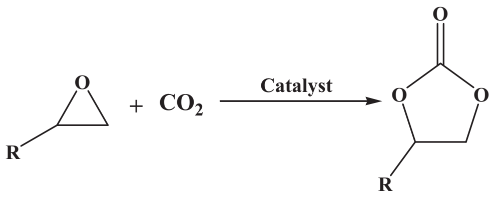
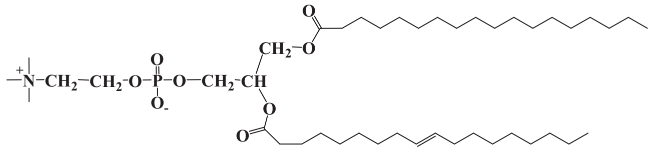


Scheme 1. Cycloaddition of CO_2 with epoxides.



Scheme 2. The chemical structure of lecithin.

was discovered that the salts and lecithin had excellent synergetic effect to catalyze the reactions. To our knowledge, this is the first work to combine these two kinds of compounds for the reactions. We believe that the simple, cheaper, and ecologically safer route to synthesize cyclic carbonates has great potential in industrial application.

2. Experimental

2.1. Materials

CO_2 was supplied by Beijing Analytical Instrument Factory with a purity of 99.95%. Propylene oxide, epichlorohydrin, potassium iodide, potassium chloride, potassium bromide were analytical grade and produced by Beijing Chemical Reagents Company. Lecithin (Soybean) was purchased from Alfa Aesar. Other epoxides were purchased from Acros Organics. All chemicals were used as received.

2.2. Charaterization

XPS measurements were carried out on an ESCAL Lab 220i-XL spectrometer at a pressure of $\sim 3 \times 10^{-9}$ mbar using Al Kα as the excitation source ($h\nu = 1486.6$ eV) and operated at 15 kV and 20 mA.

2.3. Cycloaddition reaction

All the cycloaddition reactions were conducted in a 22 mL stainless steel reactor equipped with a magnetic stirrer. We describe the procedures for the cycloaddition of propylene oxide (PO) because the procedures for the reactions of other epoxides were similar. In the experiment, desired amounts of catalyst, lecithin and PO were added into the reactor. The reactor was sealed and placed in a constant-temperature air bath of desired temperature. CO_2 was then charged into the reactor until desired pressure was reached, and the stirrer was started. After a certain time, the reactor was placed into ice water and CO_2 was released slowly passing through a cold trap containing ethyl acetate to absorb the trace amount of reactant and product entrained by CO_2. After depressurization, ethyl acetate in the cold trap and internal standard dodecane were added into the reactor. The reaction mixture was analyzed by GC (Agilent 6820) equipped with a flame-ionized detector. The purity and structure of the product at some typical experimental conditions were also checked by ^1H NMR and GC-MS. Spectral characterizations of the products (2b–f) are as follows:

4-phenyloxymethyl-1,3-dioxolan-2-one (2b). ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 4.16 (dd, J = 3.5, 10.5 Hz, 1H), 4.24 (dd, J = 4.4, 10.5 Hz, 1H), 4.54 (dd, J = 6.0, 8.4 Hz, 1H), 4.62 (t, J = 8.4 Hz, 1H), 5.00–5.05 (m, 1H), 6.91 (d, J = 8.4 Hz, 2H), 7.02 (t, J = 7.4 Hz, 1H), 7.31 (t, J = 8.2 Hz, 2H);

Table 1
Coupling of CO_2 and PO catalyzed by different catalysts.[a]

Entry	Catalyst	Yield (%)[b]
1	None	0
2	Lecithin	0
3	Betaine	0
4	KCl	0
5	KBr	0.3
6	KI	4.5
7	Betaine + KI	11.8
8	Lecithin + KCl	2.8
9	Lecithin + KBr	21.3
10	Lecithin + KI	97.7

[a] Typical reaction conditions: a stainless reactor of 22 mL, 20 mmol PO, CO_2 pressure 2 MPa, 0.25 mmol catalyst, reaction temperature 100 °C, reaction time 4 h.

[b] Yields were determined by GC versus an internal standard.

4-*n*-butyl-1,3-dioxolan-2-one (2c). ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 0.95 (t, J = 7.2 Hz, 3H), 1.26–1.48 (m, 4H), 1.64–1.87 (m, 2H), 4.09 (dd, J = 7.3, 8.0 Hz, 1H), 4.52 (t, J = 8.0 Hz, 1H), 4.67–4.76 (m, 1H);

4-methacryloyloxymethyl-1,3-dioxolan-2-one (2d). ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 1.96 (s, 3H), 4.31–4.38 (m, 2H), 4.44 (dd, J = 3.1, 12.6 Hz, 1H), 4.62 (t, J = 8.7 Hz, 1H), 4.96–5.02 (m, 1H), 5.67 (s, 1H), 6.16 (s, 1H);

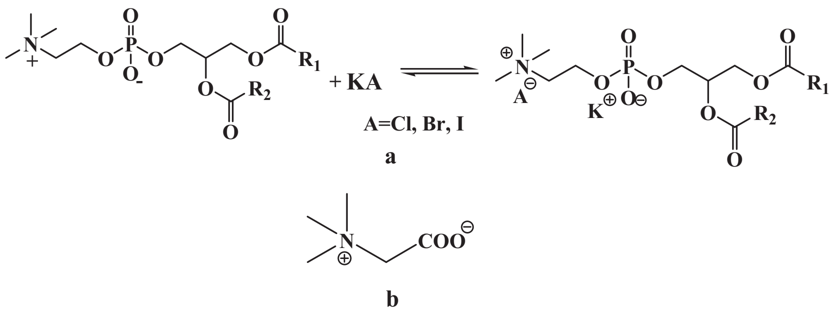
4-phenyl-1,3-dioxolan-2-one (2e). ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 4.34 (t, J = 8.2 Hz, 1H), 4.80 (t, J = 8.4 Hz, 1H), 5.68 (t, J = 8.0 Hz, 1H), 7.35–7.45 (m, 5H);

4-chloromethyl-1,3-dioxolan-2-one (2f). ^1H NMR ($CDCl_3$, 400 MHz) δ (ppm) 3.75 (dd, J = 3.6, 12.3 Hz, 1H), 3.84 (dd, J = 4.9, 12.3 Hz, 1H), 4.42 (dd, J = 5.8, 8.8 Hz, 1H), 4.61 (t, J = 8.4 Hz, 1H), 5.00–5.06 (m, 1H).

3. Results and discussion

3.1. Activity of various catalysts

The activity of various catalysts was tested using the reaction of PO and CO_2 to produce propylene carbonate (PC), and the results are summarized in Table 1. No product was detected without catalyst or when lecithin was used as the catalyst only (entries 1, 2). Potassium halide could catalyze the cycloaddition alone, but the yield of PC was very low (entries 4–6). When lecithin was added, the yield of PC was enhanced (entries 8–10). This could result from the formation of quaternary ammonium halide from potassium halide and lecithin because lecithin has a zwitterionic structure (Scheme 3a). This reduced the interaction between K^+ and A^- (A = Cl, Br, I) effectively, which is proved by the binding energy of $I(3d_5)$ for KI (619.3 eV) and KI/lecithin (618.3 eV) as shown in Fig. 1. This favoured the activity of KI for catalyzing the reaction. As a result, the KA/lecithin catalytic system was more active for the reaction. In order to confirm this effect, we used betaine as the co-catalyst, which also had the similar zwitterionic structure (Scheme 3b), to conduct the reaction. It was shown that betaine could also increase the activity of KI (entries 3, 6 and 7), although it was not as effective as lecithin. The lower efficiency of betaine than lecithin (entries 7 and 10) may result mainly from the difference of the chain lengths of the two compounds, which results in different solubility of the compounds in PO and behavior of the anions [40]. The order of the activity of potassium halides was KI > KBr > KCl (entries 4–6, 8–10), which is consistent with the order of the nucleophilicity of the halide ions. Furthermore, the leaving ability of the halide anions is another important factor to influence the catalytic activity, and the activity increases with the leaving ability. The



Scheme 3. (a) Formation of quaternary ammonium halide between potassium halide and lecithin. (b) Structure of betaine.

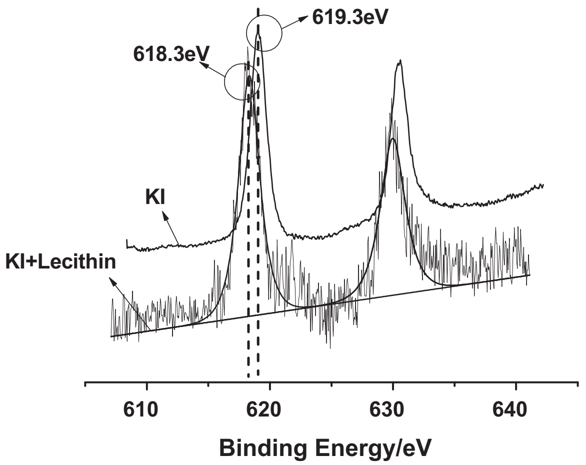


Fig. 1. X-ray photoelectron spectra of I(3d_5) for KI and KI/lecithin.

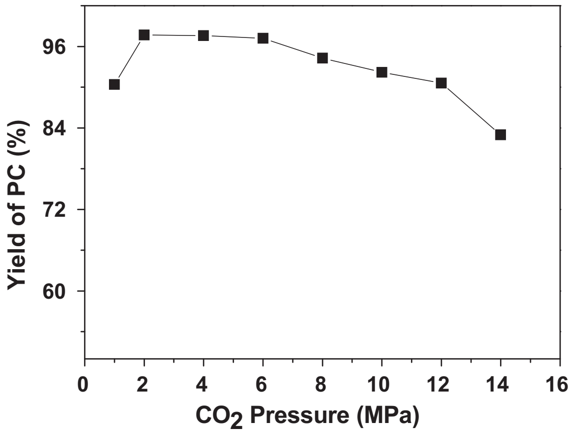


Fig. 3. Effect of CO_2 pressure on PC yield. Reaction conditions: 20 mmol PO, 0.25 mmol KI, 0.25 mmol lecithin, reaction temperature 100 °C, reaction time 4 h.

leaving ability of the halide anions is $I^- > Br^- > Cl^-$. The order is the same as that of the nucleophilicity of these anions [28]. Therefore, KI was selected as the catalyst to study the effect of reaction conditions on the coupling reaction in the presence of lecithin.

The catalytic activity of the lecithin/KI in the cycloaddition reaction of PO and CO_2 was compared with those of other quaternary ammonium and phosphonium salts reported in the literature and the results are listed in Table 2. It can be known from the data in the table that lecithin/KI had better activity.

3.2. *The effect of the catalyst amount*

The amount of KI and lecithin in the reaction system had strong effect on the coupling reaction (Fig. 2). Increasing the amount of KI and/or lecithin could enhance the activity of the catalyst system (Fig. 2a) because the amount of quaternary ammonium iodide formed from KI and lecithin (Scheme 3a) would increase, resulting in higher yield of the desired product, as shown in Fig. 2b.

3.3. *Effect of CO_2 pressure*

The partition behavior of substrates between phases may affect the reaction rate of a biphasic reaction system [72]. The effect of CO_2 pressure on the catalytic activity of KI/lecithin system was studied at 100 °C in pressure range of 1–14 MPa, and the results are shown in Fig. 3. PC yield increased with increasing pressure in the range of 1–2 MPa, and nearly unchanged with pressure in the range of 2–6 MPa. However, the yield decreased as the pressure changed from 6 to 14 MPa. This can be explained qualitatively by

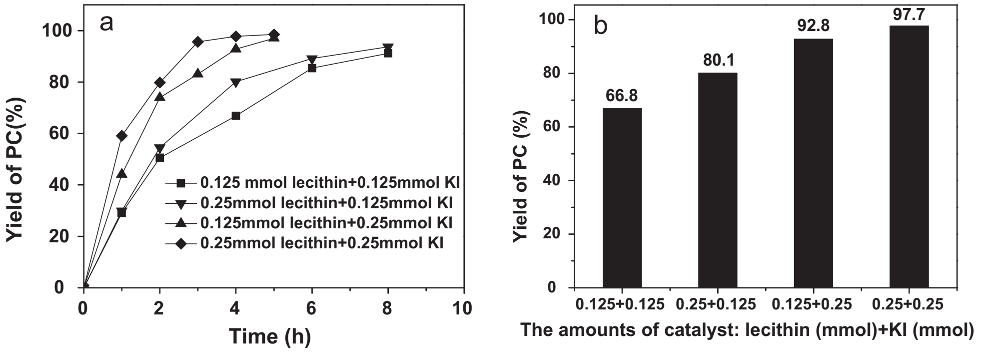


Fig. 2. Effect of catalyst amounts on PC yield. (a) Reaction conditions: 20 mmol PO, reaction temperature 100 °C, CO_2 pressure 2 MPa. (b) Reaction conditions: reaction time 4 h, the others was the same as (a).

Table 2
Comparison of the activity of lecithin/KI with other quaternary ammonium and phosphonium salts in the cycloaddition reaction of PO and CO_2.

Catalyst (mol%)	Time (h)	Pressure (MPa)	Temperature (°C)	Yield (%)	Ref.
n-Bu_4NBr/SiO_2 (1)	10	8	150	87	[47]
n-Bu_4NBr (1)	10	8	150	95	[47]
n-Bu_4NI (1)	10	8	150	85	[47]
$[(n\text{-}C_7H_{15})_4N]_6[\alpha\text{-}SiW_{11}O_{39}Co]$ (0.1)	2	3.5	150	97	[48]
$[(n\text{-}C_7H_{15})_4N]_6[\alpha\text{-}SiW_{11}O_{39}Mn]$ (0.1)	2	3.5	150	96	[48]
$(C_6F_{13}C_2H_4)_3MePI$ (0.1)	24	14	100	93	[49]
$(C_8F_{17}C_2H_4)_3MePI$ (0.1)	24	14	100	92	[49]
$(C_4F_9C_2H_4)_4PI$ (0.1)	24	14	100	83	[49]
$(C_6F_{13}C_2H_4)_4PI$ (0.1)	24	14	100	89	[49]
Bu_4PI (0.1)	24	14	100	90	[49]
Lecithin + KI (1.25)	4	2	100	97.7	This work

the effect of pressure on the concentrations of CO_2 and epoxides in the two phases. In this work, we observed the phase behavior of the reaction system using a view cell reported previously [73]. There were two phases in the reaction system at the reaction condition. The upper phase was vapor phase and the bottom phase was the liquid phase. The reaction took place mainly in the liquid phase because the catalyst was dispersed in this phase. In the system, the pressure of CO_2 affects the reaction in two opposite ways. Firstly, the solubility of CO_2 in PO increases with increasing pressure, which favoured the reaction considering that CO_2 was a reactant. Secondly, the solvent power of CO_2 to extract the reactant increases with increasing CO_2 pressure. In other words, more PO is extracted into the vapor phase as pressure rises, which reduces the reaction rate. At the low pressure, the yield increased as pressure rose, indicating that the first factor was dominant. The two factors compensated each other in the pressure range of 2–6 MPa and the effect of pressure on the yield was not considerable. When the pressure is higher than 6 MPa, the second factor became dominant and the yield decreased with increasing pressure.

3.4. Influence of reaction temperature

Fig. 4 demonstrates the dependence of the yield of PC on temperature at CO_2 pressure of 2 MPa in the temperature range of 50–110 °C, and the reaction time was 4 h. The figure shows that the activity of the catalyst was strongly affected by reaction temperature. The yield of PC increased with increasing temperature, and reached 97.7% at 100 °C, then the PC yield kept almost constant with further increase of temperature, hinting that 100 °C is the optimal temperature for the reaction at the reaction conditions we used.

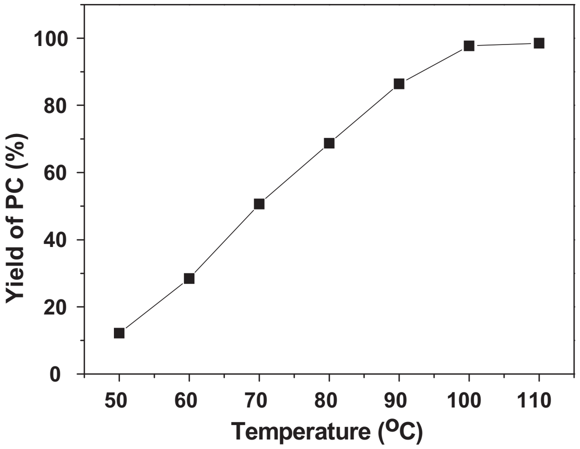


Fig. 4. Influence of reaction temperature on PC yield. Reaction conditions: 20 mmol PO, 0.25 mmol KI, 0.25 mmol lecithin, CO_2 pressure 2 MPa, reaction time 4 h.

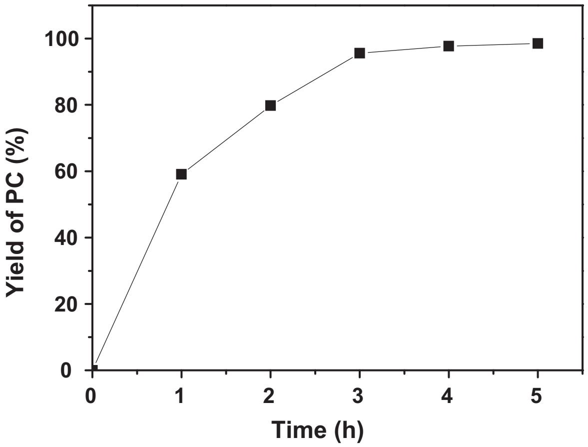


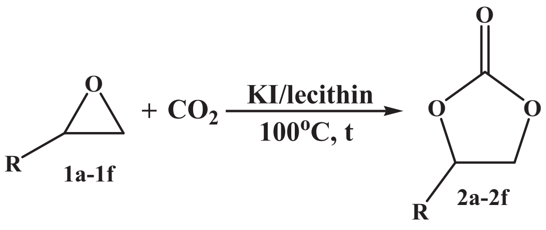
Fig. 5. The dependence of PC yield on reaction time. Reaction conditions: 20 mmol PO, 0.25 mmol KI, 0.25 mmol lecithin, CO_2 pressure 2 MPa, reaction temperature 100 °C.

3.5. Influence of reaction time

The dependence of the PC yield on reaction time is presented in Fig. 5. The reaction was performed in the presence of 1.25 mol% KI and 1.25 mol% lecithin at 100 °C under CO_2 pressure of 2 MPa. In a relatively short reaction time, the conversion of PO was incomplete and the PC yield was low. It can be seen that a yield of 97.7% could be achieved at 4 h. No further increase in the yields of PC was observed with prolonged reaction time. Therefore, the reaction time of 4 h was suitable for this system.

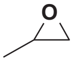
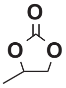
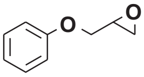
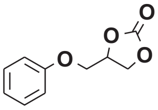
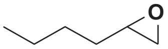
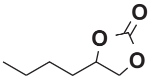
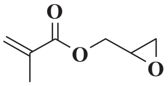
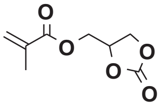
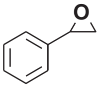
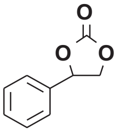
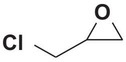
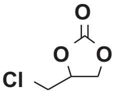
3.6. Other substrates

The performance of KI/lecithin to catalyze the cycloaddition of CO_2 with other epoxides (Scheme 4) were also studied at 100 °C and 2 MPa without using any solvent, and the results are summarized in Table 3. The catalyst system was applicable to a variety of terminal epoxides, producing the corresponding cyclic carbonates with excellent yields. Epichlorohydrin (1f) showed less activity and required a longer time (12 h) to give product 2f in a high yield

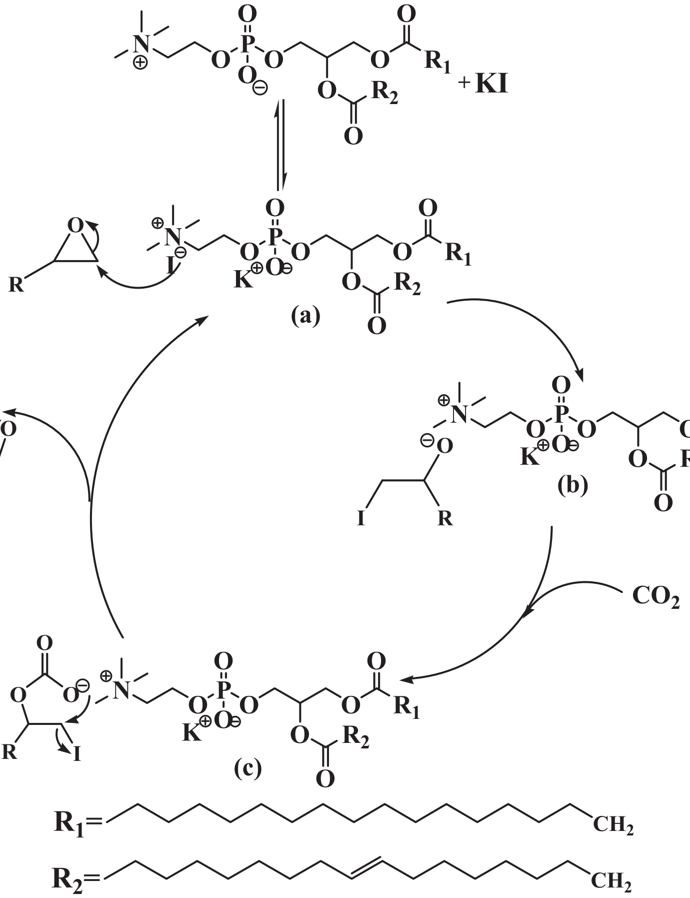


Scheme 4. Coupling of CO_2 with different epoxides in our catalytic system.

Table 3
Various carbonates synthesis catalyzed by KI in the presence of lecithin.[a]

Entry	Epoxides	Products	Time (h)	Yield (%)[b]	Selectivity (%)[c]
1	 1a	 2a	4	97.7	98
2	 1b	 2b	4	97.2	99
3	 1c	 2c	6	98.7	98
4	 1d	 2d	6	95.6	98
5	 1e	 2e	6	94.3	99
6	 1f	 2f	12	95.6	96

[a] Reaction conditions: 20 mmol epoxide, 0.25 mmol KI, 0.25 mmol lecithin, CO_2 pressure 2 MPa, reaction temperature 100 °C.
[b] Yields were determined by GC.
[c] Selectivity were determined by GC.



Scheme 5. The plausible reaction mechanism for the cycloaddition of CO_2 with epoxides catalyzed by KI and lecithin.

(95.6%), which may result from the reduced electron density of the epoxide oxygen atom caused by the electron-withdrawing CH_2Cl group.

3.7. Mechanism

Based on the results discussed above, we propose a possible mechanism for the formation of cyclic carbonates, which is shown in Scheme 5. Firstly, quaternary ammonium iodide (a in Scheme 5) is formed from KI and lecithin, which can increase the activity of I^- in KI. Secondly, the I^- anion attacks the less hindered carbon atom of the epoxy ring, followed by ring opening reaction step (b in Scheme 5). Then, the interaction between the oxygen anion of the opened epoxy ring and CO_2 occurred, and this can form an alkylcarbonate anion (c in Scheme 5). Finally, corresponding cyclic carbonate is formed through the intramolecular cyclic step, and meanwhile, the catalyst is regenerated. It can be known from the above discussion that there exists synergistic effect between KI and lecithin, which is responsible for the high catalytic activity of the KI/lecithin catalyst system.

4. Conclusions

The effect of lecithin on the cycloaddition of CO_2 with epoxides to produce five-membered cyclic carbonates catalyzed by KI has been studied for the first time. It is found that the KI/lecithin catalytic system is very active and selective for the reactions because KI and lecithin have excellent synergetic effect to catalyze the reactions. The main reason for the synergetic effect is that the zwitterionic structure of lecithin results in the formation of quaternary ammonium halide between potassium halide and lecithin. The greener, inexpensive, active and selective catalytic system has potential application for synthesizing cyclic carbonates from CO_2 and epoxides.

Acknowledgements

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