

# Synthesis of cyclic carbonates from carbon dioxide and epoxides over betaine-based catalysts

Yinxi Zhou, Suqin Hu, Xiumin Ma, Shuguang Liang, Tao Jiang\*, Buxing Han\*

*Beijing National Laboratory for Molecular Sciences (BNLMS), Centre for Molecular Science,  
Institute of Chemistry, Chinese Academy of Sciences, Beijing 100080, China*

Received 11 October 2007; received in revised form 29 December 2007; accepted 3 January 2008

Available online 16 January 2008

## Abstract

A series of betaine-based salts containing quaternary ammonium ion and carboxylic acid group were synthesized by direct protonation of anhydrous betaine using different Brønsted acids. The catalytic activity of these salts for the synthesis of cyclic carbonates via cycloaddition reaction of CO<sub>2</sub> with epoxides was studied. The effects of anions of the salts, reaction temperature, pressure, reaction time, and the amount of catalyst used on the reaction were studied. It was demonstrated that the carboxylic acid group in the catalyst had synergetic effect with halide anion, and high yield of cyclic carbonates and excellent selectivity could be achieved at optimized condition.

© 2008 Elsevier B.V. All rights reserved.

**Keywords:** Betaine-based salts; Carbon dioxide; Cyclic carbonates; Epoxides; Cycloaddition

## 1. Introduction

CO<sub>2</sub> is a renewable carbon resource, which is cheap, safe, abundant, and non-toxic. As an important C1 building block, its chemical conversion into useful organic compounds has attracted intense attention [1–4]. Synthesis of cyclic carbonates via the cycloaddition of CO<sub>2</sub> with epoxides is one of the most attractive routes. Cyclic carbonates have been widely used not only as polar aprotic solvents in organic and polymeric synthesis, but also as ingredients for pharmaceutical/fine chemicals in biomedical synthesis [5–8]. Various homogeneous and heterogeneous catalysts have been developed to catalyze the reaction, including alkali metal salts [9–11], metal oxides [12–15], quaternary ammonium salts [16–18], Schiff base [19–20], transition metal complex [21–24], ion-exchange resins [25]. Ionic liquids (ILs) have also been used as the catalysts [26–30], and it has been demonstrated that [C<sub>8</sub>-mim]<sup>+</sup>[BF<sub>4</sub>]<sup>−</sup> was very active and selective for the reaction [27].

The processes to employ homogeneous catalysts, such as quaternary ammonium salts (e.g., NEt<sub>4</sub>Br) or alkali halides (e.g.,

NaI or KI) that are dissolved in cyclic carbonate-rich phase, are undesirable in industry because separation of the catalysts from the reaction mixtures is complex, energy consumable and may result in the decomposition of the catalysts. Although several heterogeneous catalysts have been reported, the development of more efficient catalysts for the synthesis of cyclic carbonates under mild conditions is still an interesting topic.

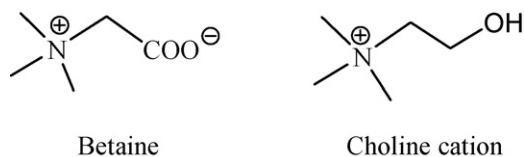
In many previous researches, quaternary ammonium salts or alkali metal halides are typically used as the homogeneous catalysts for cyclic carbonate synthesis [18,31,32]. Shi et al. found that phenol could act as a Brønsted acid to accelerate the ring-opening reaction of epoxides through hydrogen bonding in the presence of NaI/PPh<sub>3</sub>/phenol without using any organic solvents [10]. In our previous work, we found that choline chloride/urea could be utilized to catalyze the cycloaddition of epoxides and CO<sub>2</sub>, and the hydroxyl group in choline chloride could activate the epoxides through hydrogen bonding [33].

Betaine (Bet, Scheme 1) or trimethylglycine is one of the quaternary ammonium alkaloids that have a zwitterionic structure, and it is an inner salt. Its chemical name is 1-carboxy-*N,N,N*-trimethylmethanaminium inner salt. Betaine has a sweet taste and is soluble in water. It exists widely in plants and animals. Betaine is a metabolite of choline and a substrate in the recycling routes that convert homocysteine to L-methionine. Betaine-based salts, which are formed through

\* Corresponding authors. Tel.: +86 10 62562821; fax: +86 10 62562821.

E-mail addresses: [Jiangt@iccas.ac.cn](mailto:Jiangt@iccas.ac.cn) (T. Jiang),

[Hانبx@iccas.ac.cn](mailto:Hانبx@iccas.ac.cn) (B. Han).



Scheme 1. The structures of betaine and choline cation.

the reaction of the zwitterionic betaine with Brønsted acids, contain several functional groups, such as the quaternary ammonium cation, nucleophilic anion and carboxylic acid. The chemical name of betaine hydrochloride is 1-carboxy-*N,N,N*-trimethylmethanaminium chloride. Both betaine and betaine hydrochloride are available as dietary supplements. Betaine is a biodegrade, harmless and cheap raw material.

The hydroxyl group in choline cation is replaced by a carboxylic acid group in betaine (Scheme 1). Compared with hydroxyl group, the carboxylic acid group is undoubtedly a stronger Brønsted acid and hydrogen bonding donor. In this paper, we report several efficient betaine-based catalysts for the cycloaddition of CO<sub>2</sub> with epoxides. These catalysts are composed of betaine cation and Cl<sup>-</sup>, Br<sup>-</sup>, I<sup>-</sup>, BF<sub>4</sub><sup>-</sup>, or PF<sub>6</sub><sup>-</sup>, which can be prepared simply by protonation of anhydrous betaine. The effects of anions and reaction parameters on the yields and selectivity were studied. The results demonstrated that the carboxylic acid group of the catalysts could accelerate the ring opening of epoxides and showed a co-operative effect with halide anions.

## 2. Experimental

### 2.1. Materials and instruments

CO<sub>2</sub> was purchased from Beijing Analytical Instrument Factory with a purity of 99.995%. Propylene oxide, hydrochloric acid, hydrobromic acid, hydroiodic acid, tetrafluoroboric acid and hexafluorophosphate acid were A.R. grade and produced by Beijing Chemical Reagents Company. Other epoxides were purchased from ACROS ORGANICS. Anhydrous betaine was purified by recrystallization with anhydrous ethanol. Styrene oxide was purified by distillation under reduced pressure. Other chemicals were used without further purification.

<sup>1</sup>H NMR spectra were recorded on a Bruker 400 MHz NMR spectrometer with TMS as the internal standard. Decomposition temperature (*T*<sub>dec</sub>) was determined by NETZSCH STA 409 PC/PG thermogravimetric analysis (TGA) system in N<sub>2</sub> atmosphere with a heating rate of 10 °C/min. FTIR spectra were determined using a Bruker Tensor 27 spectrometer. The samples were prepared by KBr pellet method. The products were analyzed by a gas chromatography (GC, Agilent 4890D) equipped with a flame-ionized detector.

### 2.2. Preparation of betaine-based salts

The betaine-based salts were prepared by protonation of anhydrous betaine [34]. We only describe the procedures to

synthesize betaine hydroiodide (HBetI) because those to prepare other betaine-based salts are similar. The main difference was that corresponding acids were used when preparing other salts. In a typical experiment, 15 mL of anhydrous ethanol and 1.172 g of anhydrous betaine (10 mmol) were loaded into a 50-mL flask at room temperature. Then, 12 mmol of HI dissolved in 5 mL of ethanol was added slowly into the flask under vigorous stirring. The reaction lasted for 2 h. The reaction mixture was evaporated under reduced pressure. The remaining solid was washed with ethyl acetate until the color became white, then the product was dried under vacuum for 6 h at 30 °C. The total yield of HBetI was 93.1%. TGA study showed that the decomposition temperature (*T*<sub>dec</sub>) of HBetI was 245 °C. Other characterizations of HBetI were provided as follows: IR, 1641, 1738, 2835, 2968, and 3438 cm<sup>-1</sup>. <sup>1</sup>H NMR (400 MHz, D<sub>2</sub>O); δ (ppm), 4.16–4.18 (m, 2H), 3.41 (s, 9H).

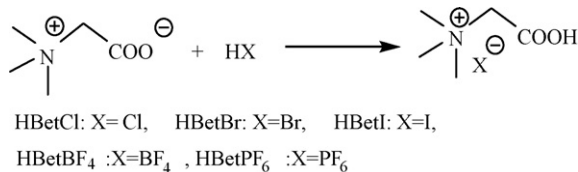
### 2.3. Typical cycloaddition procedure

All cycloadditions were conducted in a 6-mL stainless steel autoclave equipped with a magnetic stirrer. The desired amount of catalyst and epoxide were added into the autoclave. After being sealed, the reactor was put into a constant temperature air bath. CO<sub>2</sub> was then introduced into the reactor using a high-pressure pump until desired pressure was reached, and the stirrer was started. After the reaction was completed, the reactor was cooled to 0 °C quickly in ice water and depressurized by releasing the gas slowly through an absorbing tube (also put in ice water) containing *N,N*-dimethyl formamide (DMF) to capture the reactants and products entrained by CO<sub>2</sub>. After depressurization, the catalyst was separated from the reaction mixture by centrifugation. Then DMF in the absorbing tube was mixed with the reaction mixture. The mixture was analyzed by GC using *n*-butyl alcohol as the internal standard. The purity and structure of the product obtained at some typical experimental conditions were also checked by <sup>1</sup>H NMR and GC–MS. The solid products of other epoxides were analyzed at room temperature on a Bruker 400 MHz NMR spectrometer using CDCl<sub>3</sub> as the solvent. In the experiments to test the reusability of the catalyst, the catalyst was recovered by distillation at reduced pressure, and was used for the next run. Spectral characterizations of the products (cyclic carbonates **2b–d**) were provided as follows:

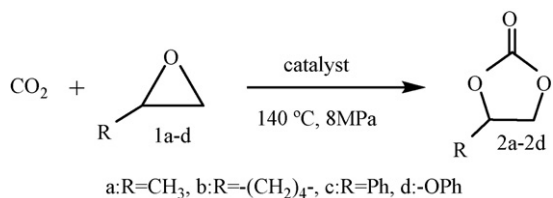
Hexahydro-benzo[1,3]-dioxolan-2-one (**2b**): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz); δ (ppm): 1.38–1.46 (m, 2H), 1.58–1.63 (m, 2H), 1.88–1.92 (m, 4H), 4.66–4.71 (m, 2H);

4-Phenyl-1,3-dioxolan-2-one (**2c**): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz); δ (ppm): 4.35 (t, *J* = 8.2 Hz, 1H), 4.80 (t, *J* = 8.3 Hz, 1H), 5.68 (t, *J* = 8.0 Hz, 1H), 7.35–7.38 (m, 2H), 7.43–7.48 (m, 3H);

4-Phenylloxymethyl-1,3-dioxolan-2-one (**2d**): <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz); δ (ppm): 4.15 (dd, *J* = 3.6/10.5 Hz, 1H), 4.24 (dd, *J* = 4.3/10.6 Hz, 1H), 4.72 (dd, *J* = 5.9/8.5 Hz, 1H), 4.61 (t, *J* = 8.5 Hz, 1H), 5.00–5.05 (m, 1H), 6.91 (d, *J* = 8.7 Hz, 2H), 7.02 (t, *J* = 7.4 Hz, 1H), 7.30 (t, *J* = 8.0 Hz, 1H).



Scheme 2. Preparation of betaine-based salts (protonation): Bet (1.172 g, 10 mmol), HX (12 mmol), anhydrous ethanol, room temperature, 2 h.



Scheme 3. Coupling of epoxides and CO<sub>2</sub>.

### 3. Results and discussion

#### 3.1. Synthesis of propylene carbonate (2a) from CO<sub>2</sub> and propylene oxide (1a)

The betaine-based solid catalysts were facilely prepared via the reactions of anhydrous betaine with several Brønsted acids (Scheme 2). The as-prepared salts were first used to catalyze the cycloaddition reaction of CO<sub>2</sub> with propylene oxide (PO) at 140 °C for 8 h to produce propylene carbonate (PC) (Scheme 3). The results were summarized in Table 1. Product was not detected without catalyst or as anhydrous betaine (Bet) was used as the catalyst (Table 1, Entries 1 and 2). When betaine-based salts were used to catalyze the cycloaddition, in which the anions were Cl<sup>-</sup>, Br<sup>-</sup>, I<sup>-</sup>, BF<sub>4</sub><sup>-</sup>, and PF<sub>6</sub><sup>-</sup>, moderate to good yields were achieved, especially when the Cl<sup>-</sup> or I<sup>-</sup> were anions (Table 1, Entries 3 and 5). In the meantime, the catalysts had high selectivity, and no by-product was detected by GC–MS.

Table 1  
Coupling of CO<sub>2</sub> and propylene oxide catalyzed by different catalysts<sup>a</sup>

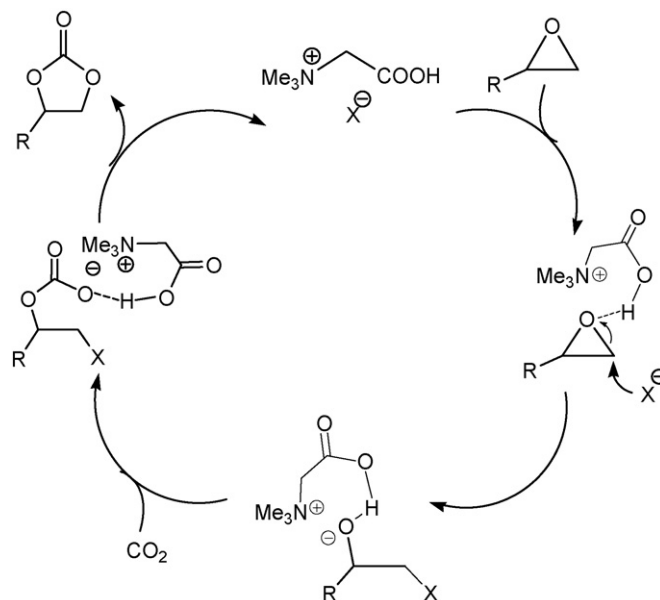
Entry	Catalysts	Yield (%)
1	None	0
2	Bet	0
3	HBetCl	94
4	HBetBr	76
5	HBetI	98
6	HBetBF <sub>4</sub>	74
7	HBetPF <sub>6</sub>	20
8	ChoCl	83
9	N(Bu) <sub>4</sub> Br	88
10	N(Me) <sub>4</sub> Cl	2
11 <sup>b</sup>	N(Bu) <sub>4</sub> Br + CH <sub>3</sub> COOH	91
12 <sup>c</sup>	HBetI	99
13 <sup>d</sup>	HBetI	96

<sup>a</sup> Typical reaction conditions: a stainless steel autoclave of 6 mL; 10 mmol of PO with 2.5 mol% catalyst; temperature, 140 °C; pressure, 8 MPa; time, 8 h.

<sup>b</sup> 2.5 mol% CH<sub>3</sub>COOH.

<sup>c</sup> The 2nd use of the catalyst in Entry 5.

<sup>d</sup> The 3rd use of the catalyst in Entry 5.



Scheme 4. The assumed reaction mechanism for the cycloaddition of epoxide with CO<sub>2</sub> catalyzed by HBetX.

The yields of PC decreased in the order of Cl<sup>-</sup> > BF<sub>4</sub><sup>-</sup> > PF<sub>6</sub><sup>-</sup> (Table 1, Entries 3, 6, and 7), which is consistent with the order of the nucleophilicity of the anions. Interestingly, the order of activity of the catalysts containing halide elements was found to be HBetI > HBetCl > HBetBr (Table 1, Entries 3–5), which is different from the order of nucleophilicity of halide anions. Better nucleophilic anion will attack the epoxide ring more effectively to form reactive intermediate shown in Scheme 4. The higher activity of HBetCl than HBetBr can be explained partially by the difference in miscibility of the catalysts with the reaction mixtures. After the reaction being completed, the catalyst precipitated from the product when HBetI or HBetBr was used, while the catalyst did not precipitate when HBetCl was used even the reaction mixture was centrifuged at high speed (Fig. 1). In this case, the system was homogenous, which was favorable to the reaction (Table 1, Entry 3). Another important factor is the leaving ability of the anions, and the activity increases with the leaving ability. The leaving ability of the anions is in the order I<sup>-</sup> > Br<sup>-</sup> > Cl<sup>-</sup>, which is consistent with the nucleophilicity of these anions.

In order to compare the catalytic activity of the betaine-based salts with those of the conventional catalysts, the catalytic activity of *n*-tetrabutyl ammonium bromide (Table 1, Entry 9) was also examined. The result indicated that HBetI had higher catalytic activity than *n*-tetrabutyl ammonium bromide; Choline chloride (ChoCl) is also a quaternary ammonium alkaloid and was tested for the same reaction (Table 1, Entry 8). The results indicated that HBetCl had higher catalytic activity than ChoCl. The difference between choline and betaine-based salts is that the hydroxyl group in choline cation is replaced by a carboxylic acid group in betaine-based salts. It can be deduced that carboxylic acid group is more beneficial to activate the ring of epoxide than hydroxyl group.

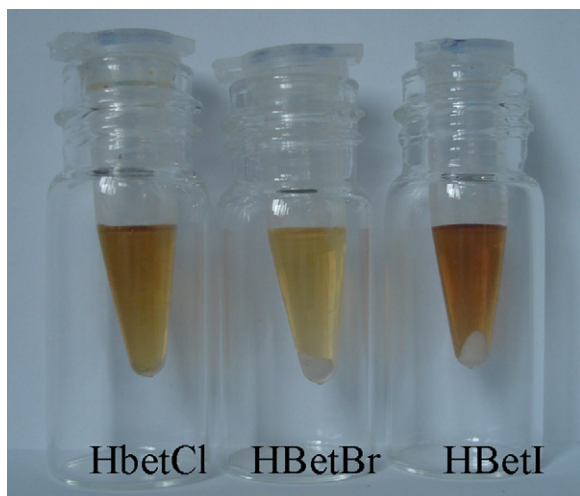


Fig. 1. The photographs of the reaction mixtures after the cycloadditions of PO with CO<sub>2</sub> catalyzed by betaine-based salts.

Based on the above results, HBetI was selected as the catalyst to investigate the effects of reaction parameters on the coupling of CO<sub>2</sub> with PO. Fig. 2 showed the effect of CO<sub>2</sub> pressure, reaction temperature, reaction time and the amount of HBetI on the

yield of PC. The yield increased with the reaction temperature up to 140 °C (Fig. 2(a)). However, further increase in temperature caused a decrease in the yield, which was possibly due to the side reactions because all PO was converted and usually higher temperature results in side reaction. Fig. 2(b) demonstrated that the yield increased with pressure up to 8 MPa. The main reason may be that too much CO<sub>2</sub> in the reaction mixture might decrease the interaction between the epoxide and the catalyst [35], and resulted in a low concentration of epoxide in the vicinity of the catalyst. The amount of catalyst also influenced the yield (Fig. 2(c)). When the molar ratio of HBetI to PO was increased from 1.5 mol% to 2.5 mol%, the yield of PC increased from 84% to 98%. However, slight reduction in the yield was observed as the amount of the catalyst was increased further. The influence of reaction time on the yield was given in Fig. 2(d). The results indicated that a reaction time of 8 h was necessary for the complete conversion of propylene oxide under the selected conditions. Longer reaction time may result in side reactions and decreased the yield. Therefore, the optimized conditions of the reaction are 140 °C, 8 MPa, 2.5 mol% HBetI, and 8 h.

In addition, a series of catalytic cycles were run to investigate the reusability of the catalyst HBetI, and the results were also given in Table 1 (Entries 10 and 11). The catalyst can be

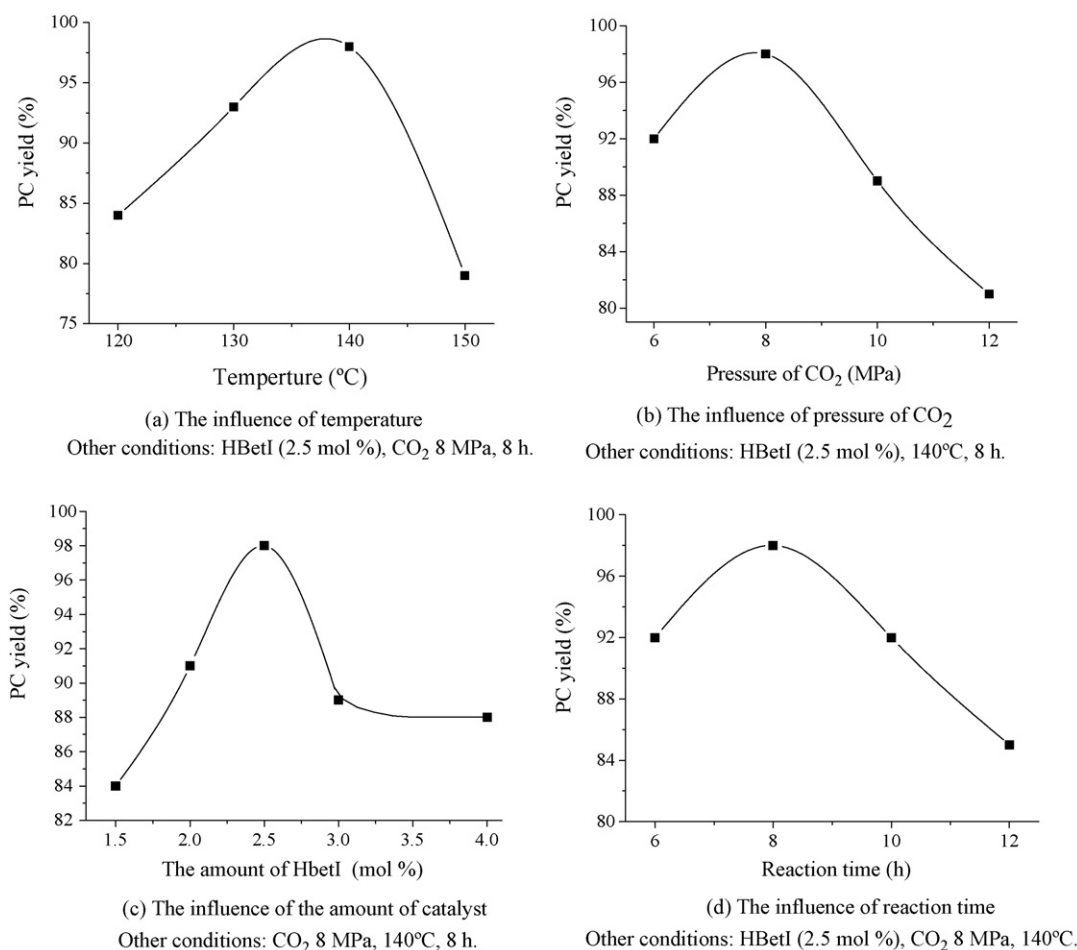


Fig. 2. The effects of different parameters on the yields of PC catalyzed by HBetI. (a) The influence of temperature. Other conditions: HBetI (2.5 mol%), CO<sub>2</sub> 8 MPa, 8 h. (b) The influence of pressure of CO<sub>2</sub>. Other conditions: HBetI (2.5 mol%), 140 °C, 8 h. (c) The influence of the amount of catalyst. (d) The influence of reaction time. Other conditions: CO<sub>2</sub> 8 MPa, 140 °C, 8 h. Other conditions: HBetI (2.5 mol%), CO<sub>2</sub> 8 MPa, 140 °C.



Table 2  
Reactions of different epoxides with CO<sub>2</sub> in the presence of HBetI<sup>a</sup>

Entry	Epoxides	Products	Time (h)	Yield (%)
1			8	98
2			8 24	34 85
3			8	96
4			8	99

<sup>a</sup> Reaction conditions: epoxide, 10 mmol; catalyst/epoxide, 2.5 mol%; temperature, 140 °C, pressure, 8 MPa; time, 8 h.

reused for at least two times without considerable loss of catalytic activity. After the reaction mixture was centrifugated, the up layer appeared brown which indicated some I<sup>-</sup> was dissolved in the reaction mixture. But the product obtained from distillation under reduced pressure was colorless, which showed that there was no I<sup>-</sup> in the product. Due to catalyst partly dissolved in reaction mixture, it is difficult to recycle the catalyst by simple filtration. Therefore, the catalyst was recovered by distillation under reduced pressure.

### 3.2. Synthesis of cyclic carbonates from CO<sub>2</sub> and other epoxides

The above results indicated that HBetI was an effective catalyst for the cycloaddition of PO with CO<sub>2</sub> in solvent-free conditions. In order to survey the applicability of the catalyst to other epoxides, we also examined the reactions of other epoxides with CO<sub>2</sub> and the results were summarized in Table 2. HBetI was found to be applicable to a variety of epoxides to produce corresponding cyclic carbonates in high yields with almost 100% selectivity. When cyclohexene oxide (**1b**) was reacted with CO<sub>2</sub>, a yield of 85% was obtained in 24 h due to the higher steric hindrance of the reactant. The stereochemical structure of **2b** *cis*-hexahydro-benzo[1,3]-dioxolan-2-one can be deduced from the mechanism and <sup>1</sup>H NMR analysis, which was also demonstrated by other authors [36]. When other epoxides were used, the yields were in the range of 96–99% in 8 h.

### 3.3. Proposed mechanism of coupling reaction

It has been suggested by Huang and Shi [10] that Brønsted acid can accelerate the ring-opening reaction of epoxides

through forming hydrogen bonding. In our experiments, ChoCl and HBetCl had better catalytic activities than N(Me)<sub>4</sub>Cl (Table 1, Entries 3, 8 and 10). It can be concluded that hydroxyl group and carboxylic acid group can promote the reaction. Furthermore, when the simple mixture of acetic acid and N(Bu)<sub>4</sub>Br was used in the experiment (Table 1, Entry 11), PC yield is 91% which is higher than the one obtained by only N(Bu)<sub>4</sub>Br (88%). It further proved the positive effect of the carboxylic acid group. The proposed mechanism is shown in Scheme 4: the X anion (Lewis base) of catalyst opened the epoxy ring, which was activated by the betaine cation through hydrogen bonding with carboxylic acid group and electronic interaction with quaterammonium cation to give the intermediate. Then the intermediate further reacted with CO<sub>2</sub> to form the corresponding cyclic carbonate and regenerated the catalyst. So in our catalysis systems, the co-existence of hydrogen bonding donors (–COOH), quaterammonium cation and halide anion exhibited a synergetic effect to promote the reaction. And the synergistic effect was perhaps the main reason of the high catalytic activity and selectivity.

## 4. Conclusion

Betaine-based salts are effective catalysts for the synthesis of cyclic carbonates via the cycloaddition of epoxides with CO<sub>2</sub>. High yields with excellent selectivity can be achieved at optimized conditions. The carboxylate acid group in betaine cation can greatly accelerate the reactions and showed a synergetic effect with halide anions.

## Acknowledgements

We sincerely acknowledge the supports from National Natural Science Foundation of China (No. 20773144) and National Key Basic Research Project of China (2006CB202504).

## References

- [1] D.H. Gibson, Chem. Rev. 96 (1996) 2063.
- [2] H. Arakawa, M. Aresta, J.N. Armor, M.A. Barteau, E.J. Beckman, A.T. Bell, J.E. Bercaw, C. Creutz, E. Dinjus, D.A. Dixon, K. Domen, D.L. DuBois, J. Eckert, E. Fujita, D.H. Gibson, W.A. Goddard, D.W. Goodman, J. Keller, G.J. Kubas, H.H. Kung, J.E. Lyons, L.E. Manzer, T.J. Marks, K. Morokuma, K.M. Nicholas, R. Periana, L. Que, J. Rostrup-Nielsen, W.M.H. Sachtler, L.D. Schmidt, A. Sen, G.A. Somorjai, P.C. Stair, B.R. Stults, W. Tumas, Chem. Rev. 101 (2001) 953.
- [3] P.T. Anastas, Green Chem. 5 (2003) G29.
- [4] C.S. Song, Catal. Today 115 (2006) 2.
- [5] A.-A.G. Shaikh, S. Sivaram, Chem. Rev. 96 (1996) 951.
- [6] J.H. Clements, Ind. Eng. Chem. Res. 42 (2003) 663.
- [7] J.P. Parrish, R.N. Salvatore, K.W. Jung, Tetrahedron 56 (2000) 8207.
- [8] J. Bayardon, J. Holz, B. Schäffner, V. Andrushko, S. Verevkin, A. Preetz, A. Börner, Angew. Chem. Int. Ed. 46 (2007) 5971.
- [9] M. Kihare, N. Hara, T. Endo, J. Org. Chem. 58 (1993) 6198.
- [10] J.W. Huang, M. Shi, J. Org. Chem. 68 (2003) 6705.
- [11] T. Zhao, Y. Han, Y. Sun, Phys. Chem. Chem. Phys. 12 (1999) 3047.
- [12] T. Yano, H. Yasuda, T. Koike, H. Ishiguro, H. Fujihara, M. Yoshihara, T. Maeshima, Chem. Commun. (1997) 1129.
- [13] K. Yamaguchi, K. Ebitani, T. Yoshida, H. Yoshida, K. Kaneda, J. Am. Chem. Soc. 121 (1999) 4526.
- [14] M. Tu, R.J. Davis, J. Catal. 199 (2001) 85.
- [15] B.M. Bhanage, S. Fujita, Y. Ikushima, M. Arai, Appl. Catal. A 219 (2001) 259.
- [16] V. Caló, A. Nacci, A. Monopoli, A. Fanizzi, Org. Lett. 4 (2002) 2561.
- [17] J.Q. Wang, D.L. Kong, J.Y. Chen, F. Cai, L.N. He, J. Mol. Catal. A: Chem. 249 (2006) 143.
- [18] H. Yasuda, L.N. He, T. Sakakura, C.W. Hu, J. Catal. 233 (2005) 119.
- [19] T. Aida, S. Inoue, J. Am. Chem. Soc. 105 (1983) 1304.
- [20] Y.M. Shen, W.L. Duan, M. Shi, Eur. J. Org. Chem. 14 (2004) 3080.
- [21] W.N. Sit, S.M. Ng, K.Y. Kwong, C.P. Lau, J. Org. Chem. 70 (2005) 8583.
- [22] J.L. Jiang, F. Gao, R. Hua, X. Qiu, J. Org. Chem. 70 (2005) 381.
- [23] H.S. Kim, J.J. Kim, S.D. Lee, M.S. Lah, D. Moon, H.G. Jang, Chem. Eur. J. 9 (2003) 678.
- [24] S.D. Allen, D.R. Moore, E.B. Lobkovsky, G.W. Coates, J. Am. Chem. Soc. 124 (2002) 14284.
- [25] Y. Du, F. Cai, D.L. Kong, L.N. He, Green Chem. 7 (2005) 518.
- [26] J.J. Peng, Y.Q. Deng, New J. Chem. 25 (2001) 639.
- [27] H. Kawanami, A. Sasaki, K. Matsui, Y. Ikushima, Chem. Commun. 7 (2003) 896.
- [28] H.Z. Yang, Y.L. Gu, Y.Q. Deng, F. Shi, Chem. Commun. 3 (2002) 274.
- [29] H.B. Xie, S.H. Li, S.B. Zhang, J. Mol. Catal. A: Chem. 250 (2006) 30.
- [30] H.B. Xie, H.F. Duan, S.H. Li, S.B. Zhang, New J. Chem. 29 (2005) 1199.
- [31] C.H. McMullen, J.R. Nelson, B.C. Ream, J.A. Sims, Jr., US Patent No. 4,314,945 (1982) (to Union Carbide Corporation).
- [32] B.C. Ream, US Patent No. 4,877,886 (1989) (to Union Carbide Chemicals and Plastics Company Inc.).
- [33] A.L. Zhu, T. Jiang, B.X. Han, J.C. Zhang, Y. Xie, X.M. Ma, Green Chem. 9 (2007) 169.
- [34] P. Nockemann, B. Thijs, K. Driesen, C.R. Janssen, K.V. Hecke, L.V. Meervelt, S. Kossmann, B. Kirchner, K. Binnemans, J. Phys. Chem. B. 111 (2007) 5254.
- [35] X.B. Lu, J.H. Xiu, R. He, K. Jin, L.M. Luo, X.J. Feng, Appl. Catal. A: Gen. 275 (2004) 73.
- [36] T. Iwasaki, N. Kihara, T. Endo, Bull. Chem. Soc. Jpn. 73 (2000) 713.