Efficient fixation of CO_2 to cyclic carbonates and oxazolidinones with multi-hydroxyl bis-(quaternary ammonium) ionic liquids as catalysts under mild conditions

BAI Yumeng[1,2], YANG Haijian[2*]
(1 School of Environmental Engineering and Chemistry, Luoyang Institute of Science and Technology, Luoyang 471023, Henan China; 2 College of Chemistry and Materials Science, South-Central Minzu University, Wuhan 430074, China)

Abstract A series of multi-hydroxyl bis-(quaternary ammonium) ionic liquids (**Ils1−7**) was prepared as bifunctional catalysts for the chemical fixation of CO_2. All these ionic liquid compounds were efficient for the catalytic synthesis of cyclic carbonates and oxazolidinones via the cycloaddition reactions between CO_2 and epoxides or aziridines with excellent yield and high selectivity in the absence of co-catalyst, metal and solvent. Due to the synergistic effects of hydroxyl groups and halogen anion, the cycloaddition reactions proceeded smoothly either at atmospheric pressure or room temperature. The selectivity for substituted oxazolidinones at 5- and 4- positions can be tuned via changing the reaction conditions. Finally, possible mechanisms including the activation of both CO_2 and epoxide or aziridines were proposed based on the literatures and experimental results.

Keywords chemical conversion of CO_2; multi-hydroxyl bis-(quaternary ammonium); ionic liquids
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多羟基双(季铵)离子液体在温和条件下高效催化剂固定CO_2

白宇鹲[1,2],杨海健[1*]
(1 洛阳理工学院 环境工程与化学学院, 河南 洛阳 471023;2 中南民族大学 化学与材料科学学院, 武汉 430074)

摘 要 制备了一系列多羟基双(季铵)离子液体(**ILs1−7**)作为化学固定二氧化碳(CO_2)的双功能催化剂.这些离子液体化合物是高效的催化剂,可通过CO_2与环氧烷或氮丙啶之间在没有助催化剂、金属和溶剂的情况下,环加成反应合成环状碳酸酯和噁唑烷酮,具有良好的产率和高选择性.由于羟基和卤素阴离子的协同作用,环加成反应在高压和室温下都能顺利进行.5位和4位取代噁唑烷酮的选择性可以通过改变反应条件调节.最后,根据文献和实验结果,提出了CO_2与环氧烷和氮丙啶的活化机理.

关键词 二氧化碳的化学转化;多羟基双季铵;离子液体

As a renewable C1 source and greenhouse gas on the carpet for global warming, carbon dioxide (CO_2) has the advantages of being economical, abundant, nonflammable, non-toxic and capable of replacing fossil fuel sources[1]. The coupling reaction between CO_2 and epoxide is a 100% atom economical reaction and environment-friendly because of the efficient resource utilization[2]. The synthesis of cyclic carbonates from pure CO_2 or impure flue gas CO_2[3] and epoxides has attracted great interest both industrially and academically that are extensively used as polar aprotic solvents, lithium batteries electrolytes, fuel additives and chemical manufacture intermediates[4].

As we known, CO_2 can react not only with

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* **通信作者** 杨海健(1974-),男,教授,博士,研究方向:二氧化碳化学转化,E-mail:yanghaijian@vip.sina.com

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epoxides but also aziridines to form oxazolidinones, which are thought of as a kind of new synthetic antibacterial drugs against a variety of important human pathogenic bacteria[5-6]. Oxazolidinone also has important utilizations in the field of daily chemical industry, such as lubricants, fiber products, dyes, cosmetics, rust inhibitors, etc[7]. As three-membered ring compounds very similar to alkylene oxides, aziridines can also react with CO_2 to form high value-added chemical oxazolidinone via simple cycloaddition reaction with the same catalysts.

Considering that almost all CO_2 conversions always require a large energy input[8], appropriate and efficient catalysts are necessary to facilitate the reactions. Therefore, many scientists have been working on such catalysts, such as the bifunctional aluminum catalyst synthesized by YANG et al. to efficiently catalyze the cycloaddition reaction of epoxirane and aziridine[9]. Fabio and co-workers used diethylamine iodide as a metal-free catalyst to catalyze the cycloaddition reaction of aziridine with high efficiency[10]. YANG et al. summarized a series of metal organic frameworks (MOFs) catalysts for the coupling reactions of carbon dioxide with epoxide and aziridine[11]. His group designed and synthesized a series of polyethylene glycol (PEG) -functionalized ionic liquid catalysts that can catalyze aziridine for cycloaddition reactions efficiently[12]. Among all reported catalysts for carbon dioxide immobilization via coupling reactions, ionic liquids (ILs) are of interest for their high thermal and chemical stability, low vapor pressure, high solubility, ease of recovery, and especially tunable properties[13]. By functionalizing various anions and cations, a large number of suitable IL catalysts can be designed and synthesized for different chemical reactions. This inspired us to design a series of novel bifunctional polyhydroxylated ILs with Lewis bases as nucleophilic attack by halide ions and hydroxyl groups as Lewis acids within one molecule. In present work, a series of polyhydroxy bis(quaternary ammonium) ionic liquids (**ILs1－7**) was synthesized by a simple procedure and used to catalyze the formation of cyclic carbonates and also oxazolidinones via the cycloaddition reactions of CO_2 and epoxides or aziridines. The catalytic performance was systematically investigated and the effects of reaction parameters such as temperature, pressure, time and catalyst dosage were studied, and possible reaction mechanisms were also discussed.

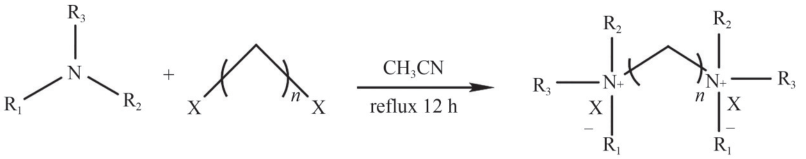
1 Experimental section

1.1 Chemicals and analytical methods

Carbon dioxide was purchased from Wuhan Steel Co. (mass fraction purity of 99.9%). All the epoxides were purchased from Sinopharm Chemical Reagent Wuhan Co. and distilled with CaH2. The starting materials 2-Dimethylaminoethanol, Triethanolamine, 1, 2-Dibromoethane, were purchased from Shanghai Darui Fine Chemical Co., Ltd. 1,2-Diiodoethane, 1,2-Dichloroethan, 1,4-Diiodobutane, 1,6-Diiodohexane were purchased from Bailingwei Technology Co., Ltd. N-Methyldiethanolamine was purchased from Wuhan Changcheng Chemical Technology Development Co., Ltd. All the other reagents and solvents used in the experiments were purchased from J&K Chemical Tech. and used without further purification. ^1H and ^{13}C NMR spectroscopy was performed on a Bruker Al-400 MHz instrument using TMS as internal standard. Fourier transform infrared (FT-IR) spectra were recorded on a Nicolet 6700 FT-IR spectrometer in the range of 600–4000 cm^{-1} with the samples pressed into KBr. All spectra were recorded at room temperature. All the known compounds were identified by comparison of their physical and spectral data with those in previous reports.

1.2 Preparation of ILs1–7

A series of new multi-hydroxyl bis-(quaternary ammonium) ionic liquids (**ILs1–7**) was synthesized via simple procedure showing in Fig.1. 2-Dimethylaminoethanol (3 mL, 0.03 mol), 1,2-Diiodoethane (2.6 g, 0.01 mol) and acetonitrile (30 mL) were added to a 50 mL three-necked flask equipped with a magnetic stirrer under the atmosphere of nitrogen. After 24 h reflux, a brown viscous liquid was precipitated after adding petroleum ether and ethyl acetate to the solution. Then the mixture was extracted by a separating funnel, and the



IL1:n=2，X=I，R_1=R_2=—CH_3,，R_3=—C_2H_4OH；**IL5**:n=6，X=I，R_1=R_2=—CH_3，R_3=—C_2H_4OH；
IL2:n=2，X=Cl，R_1=R_2=—CH_3，R_3=—C_2H_4OH；**IL6**:n=2，X=I，R_1=R_2=—C_2H_4OH，R_3=—CH_3；
IL3:n=2，X=Br，R_1=R_2=—CH_3，R_3=—C_2H_4OH；**IL7**:n=2，X=I，R_1=R_2=R_3=—C_2H_4OH.
IL4:n=4，X=I，R_1=R_2=—CH_3，R_3=—C_2H_4OH；

Fig. 1　Synthesis of catalyst **ILs1–7**

图 1　催化剂 **ILs1–7** 的合成

residual solvent was removed by rotary evaporation. The brown viscous liquid was collected **IL1** and dried under vacuum for 24 h.

ILs1–7 were prepared using the same procedure employed for the preparation of **IL2**, except replacing 1,2-Diiodoethane with the appropriate alkyl halides or replacing 2-Dimethylaminoethanol with the *N*-Methyl diethanolamine or Triethanolamine.（1,2-Dichloroethane for IL2，1,2-Dibromoethane for **IL3**，1,4-Diiodobutane for **IL4**，1，6-Diiodohexane for **IL5**，*N*-Methyl diethanolamine for **IL6** and Triethanolamine for **IL7**）. The coupling reaction of CO_2 and epoxides or aziridine sat at both atmospheric and high pressure were carried out by employing the identical procedure reported in our published papers[14].

IL1，brown viscous liquid. Yield：72%. ^{1}H NMR（400 MHz，DMSO-d_6）δ 5.30（s，1H），3.69（q，J = 5.1，4.5 Hz，2H），3.42（dd，J = 14.3，7.4 Hz，2H），3.13（dq，J = 6.6，2.9 Hz，2H），2.77（d，J = 2.1 Hz，7H）. ^{13}C NMR（101 MHz，DMSO-d_6）δ 58.82，55.60，43.11，40.97. Selected IR peaks（KBr，cm^{-1}）：σ 3379，2964，2725，1651，1467，1073.

IL2，brown viscous liquid. Yield：51%. ^{1}H NMR（400 MHz，DMSO-d_6）δ 5.80（s，1H），3.83（s，2H），3.59（s，2H），3.53（s，2H），3.26（s，6H）. ^{13}C NMR（101 MHz，DMSO-d_6）δ 69.65，64.33，55.08，49.89. Selected IR peaks（KBr，cm^{-1}）：σ 3457，1642，1466，1078，893，545.

IL3，brown viscous liquid. Yield：51%. ^{1}H NMR（400 MHz，DMSO-d_6）δ 3.91（s，1H），3.14（s，1H），2.76（s，1H）. ^{13}C NMR（101 MHz，DMSO-d_6）δ 69.59，64.24，55.05，49.85. Selected IR peaks（KBr，cm^{-1}）：σ 3329，3009，2955，1478，1097，970.

IL4，brown viscous liquid. Yield：38%. ^{1}H NMR（400 MHz，DMSO-d_6）δ 3.91（s，1H），3.14（s，1H），2.94（s，1H），2.76（s，1H）. ^{13}C NMR（101 MHz，DMSO-d_6）δ 65.23，63.55，55.34，51.49，19.60. Selected IR peaks（KBr，cm^{-1}）：σ 3413，2972，2725，1634，1474，1077.

IL5，brown viscous liquid. Yield：45%. ^{1}H NMR（400 MHz，DMSO-d_6）δ 5.48（s，1H），3.79（s，2H），3.45–3.38（m，4H），3.08（s，6H），1.70（s，2H），1.29（s，2H）. ^{13}C NMR（101 MHz，DMSO-d_6）δ 56.46，55.33，51.37，25.60，21.97，19.02. Selected IR peaks（KBr，cm^{-1}）：σ 3397，3139，2924，1405，1027，913.

IL6，brown viscous liquid. Yield：42%. ^{1}H NMR（400 MHz，DMSO-d_6）δ 5.25（s，1H），3.71（t，J = 5.2 Hz，2H），3.18（t，J = 5.3 Hz，3H），2.78（s，2H）. ^{13}C NMR（101 MHz，Deuterium Oxide）δ 57.33，55.17，40.59. Selected IR peaks（KBr，cm^{-1}）：σ 3373，2966，1637，1474，1079，955.

IL7，white solid. Yield：48%. ^{1}H NMR（400 MHz，DMSO-d_6）δ 5.27（s，1H），3.74（s，2H），3.37（s，1H），3.28（s，2H）. ^{13}C NMR（101 MHz，DMSO-d_6）δ 55.69，55.60，55.58. Selected IR peaks（KBr，cm^{-1}）：σ 3366，2940，1474，1341，1080，927.

2　Results and discussion

2.1　Effect of reaction parameters on the cycloaddition of CO_2 and epoxide

In order to evaluate the catalytic performance and acquire the optimal reaction conditions，various reaction parameters such as reaction temperature，CO_2 pressure，reaction time and catalyst loading were systematically studied，as shown in Tab1. The cycloaddition reaction of CO_2 and propylene oxide（PO）as a template reaction was catalyzed by **IL1**.

Temperature is an important reaction parameter in order to achieve optimal propylene carbonate yield and selectivity. As the temperature is lowered from 140 ℃ to 80 ℃, the yield of PC drops sharply to 78% (Tab.1, entries 1, 4). As the temperature is lowered from 140 ℃ to 100 ℃, the yield of PC is slightly lowered to 92% (Tab. 1, entries 1, 3). The results showed that the reaction was carried out at 100 ℃. Then, the dependence of PC yield on CO_2 pressure was investigated. The PC yield climbed slightly from 92% to 99% with increasing pressure (Tab.1, entries 3, 5). The results show that the reaction is carried out at 2 MPa. Finally, the amount of catalyst was discussed. As the catalyst loading is increasing from to 0.25% (molar ratio) to 0.5%, the yield of PC climbed slightly to 98% (Tab. 1, entries 3, 6). The results showed that the reaction was carried out at 0.25%.

Tab. 1 Catalytic performance of **IL1** under different reaction conditions

表1 **IL1**在不同反应条件下的催化性能

Entry	Catalyst	n (catalyst)/%	$p(CO_2)$/MPa	θ/℃	t/h	PC yield/%
1	**IL1**	0.25	2	140	3	99
2	**IL1**	0.25	2	120	3	99
3	**IL1**	0.25	2	100	3	92
4	**IL1**	0.25	2	80	3	78
5	**IL1**	0.25	3	100	3	99
6	**IL1**	0.50	2	100	3	98

Note: 1. Reaction conditions: PO (5 mL, 71.5 mmol). The selectivity of cyclic carbonate is more than 99%.
2. PC yield is determined by ^1H NMR spectra analysis using TMS as an internal standard.

2.2 Catalytic activity of various catalysts

With an optimized reaction condition (100 ℃, 2 MPa, 3 h, **IL1** loading 0.25%) in hand, various catalytic systems were utilized for the coupling reaction between CO_2 and PO under identically solvent- and additive-free conditions. As shown in Tab.2, all the bifunctional multi-hydroxyl (**ILs1-7**) showed a certain amount of yield and selectivity of PC, and catalytic activities of various catalysts were related to their structures (entries 1-7). The order of the activity of halogen anions was found to be $I^- > Br^- > Cl^-$ (entries 1, 2 and 3), which indicated that the rate-limiting step in the catalytic reaction is the PO C—O bond cleavage of epoxide initiated by the attack of halogen anion. The PC yield had no big difference with **IL1** and **IL4** (entries 1, 4) as catalyst under the same condition. As the synthesized **IL5** is solid, the solubility is poor, and the yield is only 25% lower (entry 5). It is suggested that the alkyl chain length of haloalkane has effect on the reaction that the longer the chain length, the greater the steric hindrance and the lower the reaction yield. It is also found that number of different hydroxyl groups catalytic systems is under the identical condition. As the number of hydroxyl groups increases, the more viscous the catalyst system, the lower the PC yield. Among them, PC yield of **IL7** has only 40%. Subsequently, the temperature was raised to 140 ℃, and the PC yield increased to 89%. (entries 1, 6, 7).

Tab. 2 Catalyst screening for the synthesis of propylene carbonate[a]

表2 合成碳酸亚丙酯的催化剂筛选

Entry	Catalyst	n (catalyst)/%	$p(CO_2)$/MPa	θ/℃	t/h	PC yield/%
1	**IL1**	0.25	2	100	3	92.0
2	**IL2**	0.25	2	100	3	58.9
3	**IL3**	0.25	2	100	3	72.0
4	**IL4**	0.25	2	100	3	88.5
5	**IL5**	0.25	2	100	3	28.0
6	**IL6**	0.25	2	100	3	87.0
7	**IL7**	0.25	2	100	3	40(89)

Note: 1. Reaction conditions: PO (5 mL, 71.5 mmol), temperature 100 ℃. The selectivity of cyclic carbonate is more than 99%.
2. PC yield is determined by ^1H NMR spectra analysis using TMS as an internal standard.

2.3 Cycloaddition of various epoxides and CO_2

In order to investigate the potential and universal applicability of these new bifunctional polyhydroxy **ILs**, the cycloaddition reactions of CO_2 with various epoxides were studied. As depicted in Fig. 1, the bifunctional polyhydroxy **IL1** was found to be effective for many epoxides to react with CO_2 to obtain the corresponding cyclic carbonates in excellent yields (Fig.2 2a-2k) no matter what kinds of substituentson the epoxide rings is (electron-withdrawing or electron-donating). Due to steric hindrance, internal epoxides, cyclohexene oxide (2l), 1, 2-epoxy-4-vinylcyclohexane (2m) and cyclopentene oxide (2n) exhibited much lower activity and the yields of corresponding cyclic carbonates obtained were very low even after longer time (24 h), and similar results have been well-reported in many literatures[15]. Present results also showed that our new multi-hydroxyl **IL1** catalyst could also catalyze the cycloaddition of CO_2 and diepoxide to produce bicyclic carbonate of PU raw material in excellent yield and selectivity (2i), which hinted the potential application in PU industry.

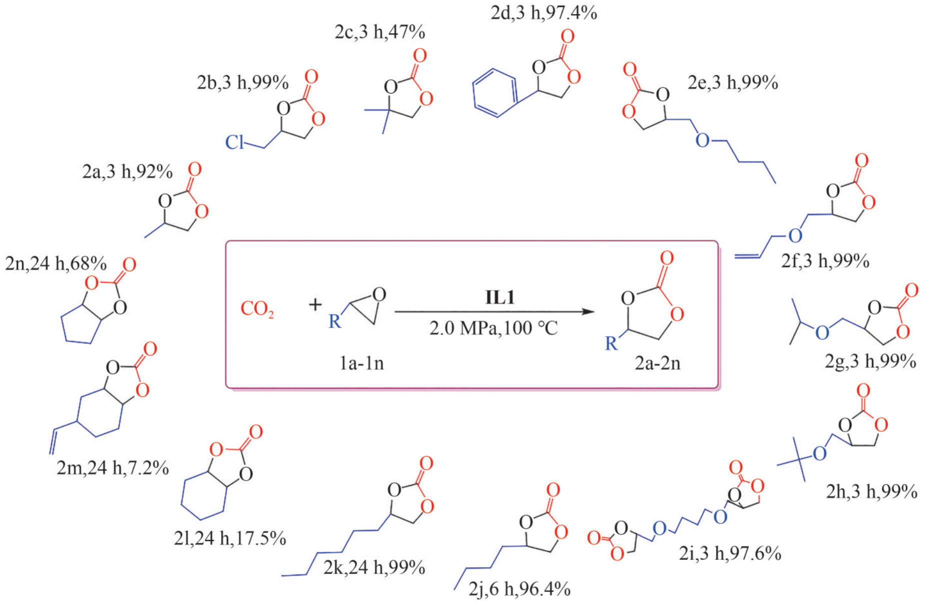


Fig. 2　Various cyclic carbonates synthesis catalyzed by **IL1** under high pressure (all the selectivities are more than 99%)

图2　**IL1** 在高压下催化合成各种环状碳酸酯(选择性均大于99%)

Up to now, running the reaction under atmospheric pressure (0.1 MPa) is still a great challenge and most-wanted in industry. So the catalytic performance of **IL1** for the economic coupling reaction of CO_2 with various epoxides under 0.1 MPa is also evaluated. In order to keep the reaction temperature below the boiling point of the substrates, 80 ℃ was chosen as the reaction temperature. The **IL1** catalyst can catalyze the coupling reaction in moderate to excellent yield at 0.1 MPa within 24 h (Fig.3 4a-4h). As a result, diepoxides, 1, 2-ethanediol diglycidyl ether and 1, 4-butydiol diglycidyl ether could also produce their bicyclic carbonates satisfyingly under as mild as atmospheric CO_2 pressure. However, for internal epoxides, cyclohexene oxide and 1,2-epoxy-4-vinyl cyclohexane, there were no reaction happened because of the large steric hindrance (4i,4j).

2.4　Effect of reaction parameters on the cycloaddition of CO_2 and 1-propyl-2-phenylaziridine

In order to investigate the catalytic activity of the bifunctional catalyst **IL1** for the cycloaddition reaction of aziridine with CO_2, the cycloaddition reaction of 1-propyl-2-phenylaziridine (AZ-Pr) with CO_2 was chosen as a model reaction. The yield of the resulting 5-substituted oxazolidinone and 4-substituted oxazolidinone was selected to demonstrate the catalytic activity, and the results are shown in Tab. 3. The temperature has important effect on the conversion and the selectivity of products. When the reaction temperature was lowered from 130 ℃ to 25 ℃, rapid increase in yield and excellent selectivity for **a2** (5-substituted oxazolidinone) were achieved under 2 MPa CO_2 pressure within in 9 h (Tab.3, entries 1-4). However, there is no big differences were found for the yield and selectivity when CO_2 pressure was reduced from 2 MPa to 1 MPa. In general, the cycloaddition reaction of 1-propyl-2-phenylaziridine with CO_2 mainly produces a 5-substituted oxazolidinone product 3-propyl-5-phenyloxazolidin-2-one because of the steric hindrance requirement. Otherwise, although the yield rapidly decreases to 33.5% under 0.1 MPa at 25 ℃, it is still encouraged to note that high selectivity for 4-substituted oxazolidinone **a3** was achived surprisingly as (n(**a2**)∶n(**a3**)=1∶99). That is to say that our new catalyst **IL1** possesses the capability of adjusting the selectivity for the 5-substituted oxazolidinone and 4-substituted oxazolidinone only via modifying the reaction parameters.

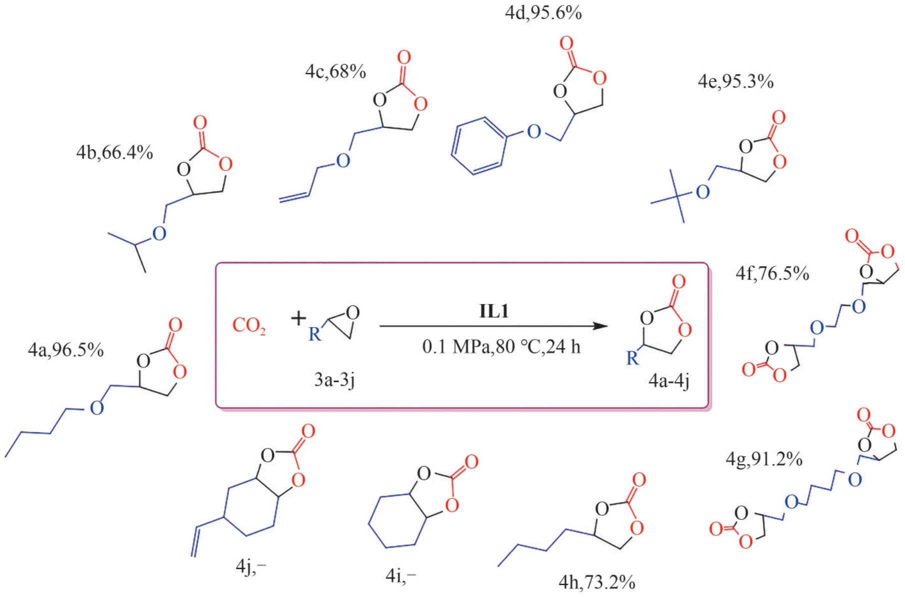
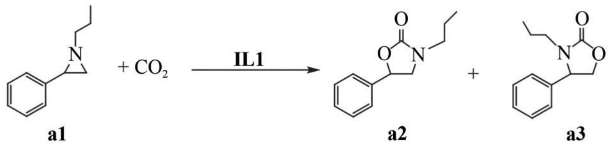


Fig. 3 Various cyclic carbonates synthesis catalyzed by **IL1** under atmospheric pressure (all the selectivities are more than 99%)

图3 常压下**IL1**催化合成各种环状碳酸酯（选择性均大于99%）

Tab. 3 Cycloaddition reaction of CO_2 with 1-propyl-2-phenylaziridine under different reaction conditions[a]

表3 不同反应条件下CO_2与1-丙基-2-苯基氮吡啶的环加成反应



Entry	θ/℃	$p(CO_2)$/MPa	t/h	n(catalyst)/%	Yield/%①	Regio-sel②
1	130	2.0	9	1	45.7	97∶3
2	110	2.0	9	1	61.4	98∶2
3	90	2.0	9	1	72.4	98∶2
4	25	2.0	9	1	94.4	98∶2
5	25	1.0	9	1	94.5	97∶3
6	25	0.1	24	1	33..5	1∶99

Note：Reaction conditions：Substrate AZ-Pr (1 mmol).

① Total yield of **a2** and **a3** is determined by ^1H NMR spectra analysis using TMS as an internal standard.

② Molar ratio of **a2** to **a3**.

2.5 Cycloaddition reactions of different substituted aziridines with CO_2 catalyzed by IL1

In order to further explore and prove catalytic potential of **IL1** for the synthesis of valuable oxazolidinones, several typical aziridines were prepared according to the literatures and employed as cycloaddition reaction substrates[16-17]. Under the catalytic conditions of (25 ℃, 1 MPa, 9 h, n(ILs) = 1%), the substrate adaptation of CO_2 to various aziridine in cycloaddition reaction was investigated (Fig.4). As shown in Fig.3, the bifunctional catalyst **IL1** can catalyze the reaction of various aziridine substrates with CO_2, and most of the products obtained are 5-substituted oxazolidinone with good to excellent yield except for those aziridines (5f–5h) with larger steric hindrance.

2.6 Reaction mechanism

Based on the experimental results and referring to the reported literatures[18-20], an anion-cation synergetic catalysis mechanism for the bifunctional multi-hydroxyl **ILs** was shown in Fig.5. Firstly, the epoxide was activated by hydrogen bond interaction between hydroxyl group of **ILs** and oxygen of epoxide, which would accelerate the ring opening step. Next, the nucleophilic X^- attacked the less hindered carbon atom of epoxide to open the ring opening, and then CO_2 inserted, following by an intramolecular ring-closure step to produce cyclic carbonate and regenerate the IL catalyst simultaneously. The hydrogen bond in this process helped to stabilize the intermediates, which would facilitate the conversion[21-23].

Similar to the reaction of CO_2 and epoxide, the first step is the activation of aziridine's ring via the formation of hydrogen bond between the hydrogen atom on the hydroxyl group in catalyst **IL1** and the nitrogen atom in aziridine molecule. Meanwhile, ring-opening is followed by the nucleophilic attack of the free iodine ion on the ***a*** or ***b*** sites of the ternary ring (Fig.6). Then, the carbon dioxide insertion forms two ionic intermediates, respectively, followed by the nucleophilic

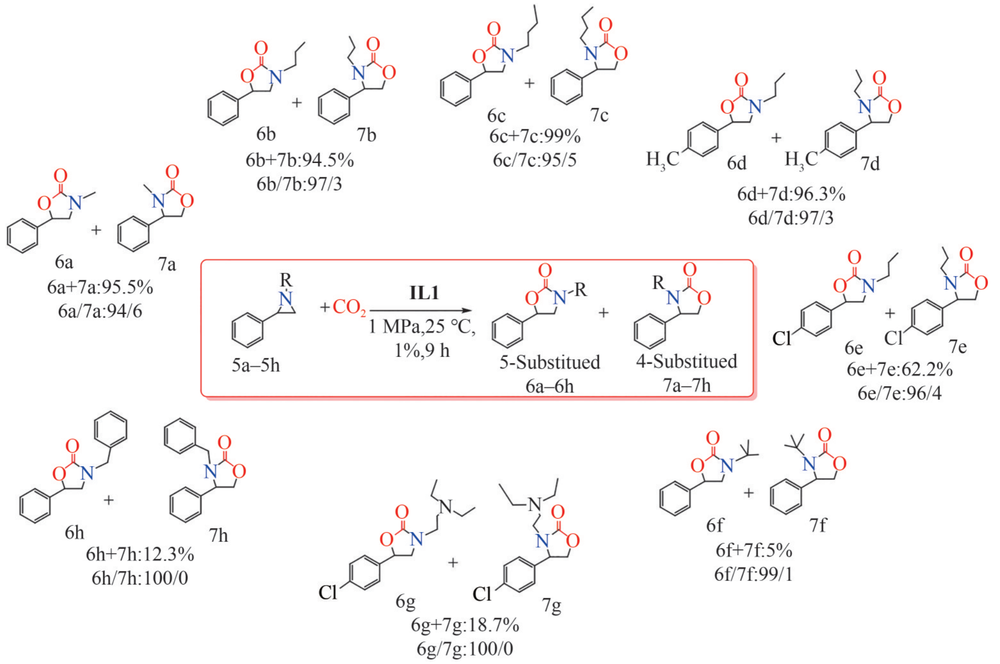


Fig. 4　Synthesis of various oxazolidone from CO_2 and aziridine catalyzed by **IL1** under room temperature

图4　用**IL1**催化CO_2和氮丙啶在室温下合成各种噁唑烷酮

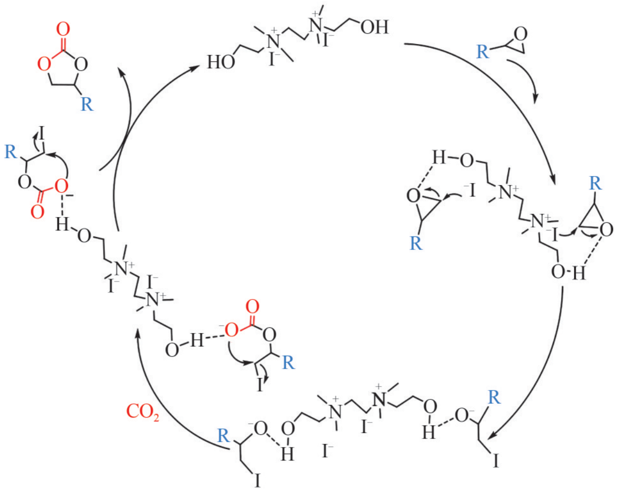


Fig. 5　Proposed mechanism for the coupling reaction of CO_2 with epoxide catalyzed by **IL1**

图5　**IL1**催化CO_2与环氧化物耦合反应的机理

attack of the negative oxygen ion on the iodine-linked carbon atom and the departure of the iodide ion to close the ring to obtain the 5-substituted or 4-substituted oxazolidinones.

3　Conclusions

In summary, a series of new multi-hydroxyl **ILs** was designed and synthesized via simple procedure to be cheap organo catalysts for the chemical fixation of CO_2 to cyclic carbonates and oxazolidinones without any co-catalyst or solvent under mild conditions down to room temperature and atmospheric pressure. The effects of catalyst structure and reaction parameters on the catalytic activity were investigated systematically. This series of catalysts were proved to be efficient catalysts for wide scope of epoxides and aziridines to synthesize cyclic carbonates and oxazolidinone with high selectivity. Finally, possible reaction mechanisms were proposed. The development of this type of **ILs** as

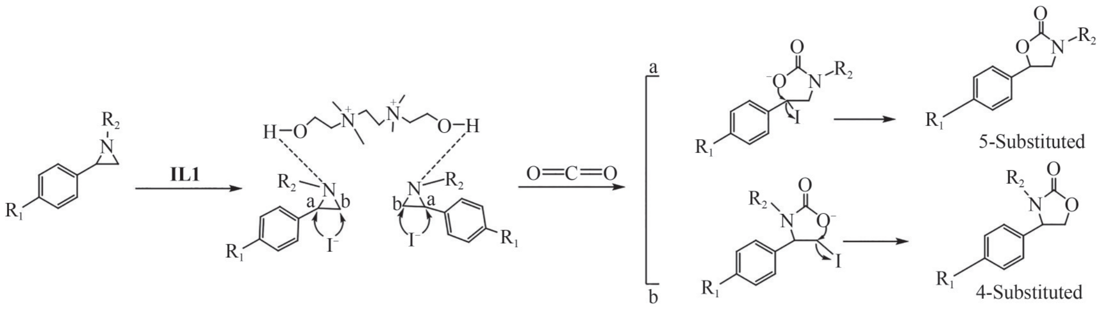


Fig. 6　Reaction mechanism of CO_2 with aziridines

图6　CO_2与氮丙啶的反应机理

organ-catalysts represents an easy-to-handle, low-cost and metal-free process of CO_2 conversion to high value-added products.

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