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Short Communication

# Microwave-assisted, rapid cycloaddition of allyl glycidyl ether and CO<sub>2</sub> by employing pyridinium-based ionic liquid catalysts



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## ABSTRACT

This study investigated the use of pyridinium-based ionic liquids (ILs) as an efficient catalyst for the rapid solvent-free microwave-assisted cycloaddition of allyl glycidyl ether (AGE) and  $CO_2$  to yield allyl glycidyl carbonate (AGC) under moderate reaction conditions. The cycloaddition reaction occurred over a short reaction time of 30 s, resulting in a high turnover frequency (TOF) ranging from 200 to 7000 h<sup>-1</sup>. The effects of alkyl chain length and anion of pyridinium-based catalysts on the cycloaddition reactivity were studied. The effects of reaction parameters such as the amount of catalyst, microwave power,  $CO_2$  pressure, and reaction time were also investigated.

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## 1. Introduction

Conventional methods in chemical synthesis are orders of magnitude too slow to satisfy the current demand for the manufacture of new compounds. Chemists have been under growing pressure to develop new methods that are rapid and environmentally friendly. The introduction of microwave flash heating for organic synthesis in recent years has resulted in dramatically reduced reaction times and cleaner reactions with lesser side products, thereby increasing product yields and simplifying the course of reactions for combinatorial chemistry [1–4]. For the past two decades, great efforts have been made towards the fixation of carbon dioxide due to the growing concern over the deleterious effects of greenhouse gases on the environment [5,6]. The synthesis of five-membered ring carbonates from epoxide and CO<sub>2</sub> is a promising methodology from a resource utilization standpoint because of the 100% atom economy of the reaction and the wide variety of applications of cyclic carbonates--such as polar aprotic solvents, precursors of polymeric materials, and intermediates in the syntheses of pharmaceuticals [6,7].

Until now, a wide variety of catalysts such as Lewis acids, transitionmetal complexes, and organometallic compounds under high  $CO_2$  pressure have been employed for the synthesis of cyclic carbonates [8–10]. The most important criteria for the synthesis of cyclic carbonates from  $CO_2$  and epoxide is the presence of a strong nucleophilic anion group, which helps in the ring opening of epoxides for effective  $CO_2$  cycloaddition. Hence, ionic liquids (ILs) possessing strong nucleophilic anions would be the most appropriate choice as the catalyst for this process [11]. ILs have advantages such as negligible vapor pressure, excellent thermal stability, and other special characteristics compared to conventional organic and inorganic solvents. ILs are composed of cation/anion (Lewis acid and Lewis base sites) combinations, which activate  $CO_2$ molecules and simultaneously interact with other substrates [11–14].

Over the past decades, numerous strategies have been proposed for the chemical absorption of CO<sub>2</sub> by employing ILs as a catalyst. Despite their high catalytic activity, ILs still fall short owing to their inherent drawbacks such as extensive energy consumption for CO<sub>2</sub> desorption, low capture efficiency, and slow sorption kinetics. On the other hand, microwave-heating technology relies on an ionic conduction mechanism. Molecules possessing a high dipole moment can be easily heated by microwave radiation [1–4]. There exist limited studies on the microwave-assisted synthesis of cyclic carbonates from CO<sub>2</sub> and epoxides [15–18]. Similarly, among the various epoxides employed for the cycloaddition reactions, epoxides bearing ether oxygen in the side chain showed higher activity in IL associated catalytic systems [15, 19–27]. Allyl glycidyl ether (AGE) can be copolymerized with  $CO_2$  to form biodegradable aliphatic polycarbonate bearing pendant allyl functionality [28]. And also, studies on pyridinium-based ILs are few compared to those on other types of ILs [19]. Thus, this paper presents a detailed investigation of solvent-free microwave-assisted cycloaddition of AGE and CO<sub>2</sub> for the synthesis of allyl glycidyl carbonate (AGC) by



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employing pyridinium-based ILs as the catalyst (Scheme S1, Supplementary information).

## 2. Experimental

## 2.1. Materials

1-Ethylpyridinium chloride (>98%) (EPyCl), 1-propylpyridinium chloride (>98%) (PPyCl), 1-butylpyridinium chloride (>98%) (BPyCl), 1-hexylpyridinium chloride (>98%) (HPyCl), 1-butylpyridinium bromide (>98%) (BPyBr), and 1-butylpyridinium hexafluorophosphate (>98%) (BPyPF<sub>6</sub>) were purchased from TCI chemicals whereas 1-butyl-4-methylpyridinium iodide (>99%) (BMPyI), 1-butyl-3-methyl-imidazolium bromide (>97%) (BMImBr), and tetrabutylammonium bromide (>98%) (TBAB) were obtained from Aldrich and used without further purification. Allyl glycidyl ether (AGE) was purchased from Aldrich and used as received. Carbon dioxide of 99.999% purity was used without further purification. CH<sub>2</sub>Cl<sub>2</sub> was obtained from SK Chemicals, Korea, and used as received.

#### 2.2. Cycloaddition of epoxides and CO<sub>2</sub>

For the reaction, a multi-mode microwave reactor (KMIC-2KW) containing a source with a continuously adjustable power setting from 0 kW to 2 kW (adjusted via a 3-stub tuner) as well as a temperature controller unit operating at a frequency of 2.450 GHz was used. The surface temperature of the reactor was measured using an IR temperature detector. The cut off temperature was kept at 175 °C considering the subtle stability of Pyrex glass microwave reactor. The synthesis of AGC from AGE and CO<sub>2</sub> (Scheme S1) using pyridinium based ILs as a catalyst was performed with the microwave reactor in a 40 mL Pyrex glass reactor equipped with a magnetic stirrer. For each typical reaction, ILs (0.1-2.5 mmol) and AGE (18.6 mmol) were charged into the reactor without a solvent and then purged several times with CO<sub>2</sub>. The reactor was then pressurized with CO<sub>2</sub> and heated to a desired temperature by stirring the reaction mixture at 600 rpm. After the completion of reaction time, cycloaddition was stopped by cooling the reaction mixture to room temperature and venting the remaining CO<sub>2</sub>. The product was dissolved in dichloromethane and an analysis was carried out using a gas chromatography/mass spectrometry (GC-MS, Micromass, UK) analysis. The conversion of epoxides was obtained from gas chromatography (GC, HP-5 capillary column, HP 6890, Agilent Technologies, Santa Clara, CA, USA) data.

## 3. Results and discussion

## 3.1. Catalytic activity

The catalytic activity of pyridinium-based ILs was tested using a microwave-assisted solvent-free cycloaddition reaction using AGE as the epoxide substrate. The observed results are tabulated in Table 1. Entry 1 shows the need for an appropriate catalyst because no product was formed in the absence of a catalyst. When the size of the cations in the pyridinium IL was increased from ethyl to butyl (entries 2-4), catalytic activity also increased reaching a maximum of 86% conversion for BPyCl with a high turn-over frequency (TOF) of 1927 h<sup>-1</sup> under a moderate reaction condition of 0.96 MPa of CO<sub>2</sub> pressure, 200 W of microwave power, and reaction time of 30 s. The rate-determining step of the epoxide-CO<sub>2</sub> cycloaddition reaction involved a nucleophilic attack of anions on the epoxide ring. Therefore, bulky ILs, having longer distances between cations and anions, have a higher anion activation capacity [13]. However, a further increase in cationic size (entry 5) resulted in a slight reduction of catalytic activity. This could be explained by the steric hindrance of the bulky ILs, which compensated for the increase of the anion activation ability by increasing the alkyl chain length [29].

#### Table 1

Catalyst screening for the cycloaddition of AGE and CO<sub>2</sub>.

Entry	Catalyst	Conversion (%)	Selectivity (%)	$TOF(h^{-1})$
1	None	0	0	0
2	EPyCl	79	90	1770
3	PPyCl	82	91	1838
4	BPyCl	86	96	1927
5	HPyCl	78	93	1748
6	BPyBr	95	95	2129
7	BMPyI	93	87	2084
8	BPyPF <sub>6</sub>	10	94	224
9	BMImBr	92	96	2062
10	TBAB	73	89	1636

Reaction conditions: AGE = 18.6 mmol, catalyst amount = 1 mmol,  $P_{CO2} = 0.96 \text{ MPa}$ , microwave power = 200 W, time = 30 s. TOF: moles of AGE converted per mol of ionic liquid/h.

The anion effect of pyridinium-based ILs was investigated using anions possessing Cl, Br, I, and PF<sub>6</sub> (Table 1). Comparing BPyCl, BPyBr, and BMPyI (entries 4. 6, 7), catalysts containing I<sup>-</sup> or Br<sup>-</sup> anions showed high catalytic activities of 93% and 95% with excellent TOF of 2084 and 2129  $h^{-1}$  (respectively) under moderate reaction conditions (0.96 MPa, 200 W, 30 s). This was most likely due to the high nucleophilicity and leaving ability of these anions. Compared to I<sup>-</sup> and Br<sup>-</sup> systems, less nucleophilic anions  $Cl^-$  and  $PF_6^-$  (entries 4 and 8) showed lower catalytic activity [24,30]. Various other ILs, such as imidazoliumbased BMImBr and ammonium salt-based TBAB, were also tested (entries 9 and 10) using solvent-free microwave cycloaddition and they resulted in good activity. The high activity observed for BMImBr and TBAB confirms the most important criteria for obtaining high catalytic activity for cyclic carbonate synthesis from CO<sub>2</sub> and epoxides, namely, the presence of strong nucleophilic anions with appropriate alkyl chain length cations. From the above studies and various others [8,24,29,30], bromide or iodide ions with butyl group-containing cations could serve as the best catalyst for solvent-free cycloaddition of epoxide and CO<sub>2</sub> under moderate reaction conditions. In most cases, the TOFs for IL-based cycloaddition of epoxides and CO<sub>2</sub> were lower than 50  $h^{-1}$  [30–34]. To date, the highest TOF obtained for an ILbased system (HETBA) is in the range of 3000–14,000  $h^{-1}$  for the synthesis of propylene carbonate from propylene oxide and CO<sub>2</sub> in a micro-reactor (3.5 MPa, 180 °C, 14 s) [35]. Pyridinium-based ILs have been employed earlier for the cycloaddition of butyl glycidyl ether and CO<sub>2</sub> to yield butyl glycidyl carbonate, but it resulted in a very low TOF of 15  $h^{-1}$  under conventional reaction conditions of 0.82 MPa at 140 °C for 1 h [19]. Utilizing microwave-assisted cycloaddition of AGE and CO<sub>2</sub> to yield AGC in the presence of pyridinium-based ILs, high TOFs ranging from 200 to 7000  $h^{-1}$  were achieved under moderate reaction conditions of 0.96 MPa, 200 W, and 30 s. In this study, we achieved a 450-fold increase in the TOF by using microwaves, which is much larger than the TOF achieved using conventional synthesis techniques [19].

## 3.2. Effect of catalyst amount

Since BPyBr showed good catalytic activity, it was used to examine the effects of reaction parameters (catalyst amount, time, microwave power, and CO<sub>2</sub> pressure) on the cycloaddition reaction of AGE with CO<sub>2</sub>. Table 2 shows the relationship between the AGE conversion and the amount of catalyst. The conversion increased from 31% to 95% when the amount of catalyst increased from 0.1 mmol to 1.0 mmol, whereas the TOF decreased from 6947 h<sup>-1</sup> to 2129 h<sup>-1</sup> (entries 1–3). This can be explained by the effect of the catalytically active sites in AGE. The conversion efficiency of the substrate is mainly determined by the collision frequency between the catalytically active sites and the substrate. As the number of catalytically active sites increases with the increase of catalyst concentration under a constant substrate volume, the collision frequency based on the catalytically active sites

100 95

Table 2
Effect of catalyst amount on the reactivity of BPyBr for the synthesis of AGC.

Entry	Catalyst amount (mmol)	Conversion (%)	Selectivity (%)	$TOF(h^{-1})$
1	0.1	31	99	6947
2	0.5	79	97	3541
3	1	95	95	2129
4	1.5	96	95	1434
5	2	94	92	1053
6	2.5	93	90	834

Reaction conditions: AGE = 18.6 mmol, catalyst = BPyBr,  $P_{CO2} = 0.96 \text{ MPa}$ , microwave power = 200 W, time = 30 s. TOF: moles of AGE converted per mol of ionic liquid/h.

decreases. As a result, the number of substrate molecules which need to be activated per unit time by each active site, is reduced [30]. Further increase in the amount of catalyst did not produce any significant increase in the conversion most likely owing to the hindrance of the mass transfer between the active site and reagent caused by the low dispersity of the excess catalyst in the reaction mixture [35].

## 3.3. Effect of reaction time

The effect of reaction time on the microwave-assisted, solvent-free cycloaddition of AGE and CO<sub>2</sub> to yield AGC is illustrated in Table 3. Experiments were performed at a microwave power of 200 W, 0.96 MPa of CO<sub>2</sub> pressure, and 1 mmol of BPyBr. The results indicated that the reaction proceeded rapidly for 30 s, with 95% conversion and 95% selectivity. After this period, the reaction rate did not vary significantly. Thus, a reaction time of 30 s was determined to be the maximum time required for AGE conversion. Remarkably, the selectivity remained greater than 90% over the entire course of the reaction. The only byproduct obtained during the course of the reaction was 3-allyloxy-1,2-propanediol.

## 3.4. Effect of microwave power

Fig. 1 shows the effects of microwave power on the BPyBr-catalyzed cycloaddition of AGE and  $CO_2$  at 0.96 MPa of  $CO_2$  pressure over a power range of 50-500 W for 30 s. The heat generated by microwave irradiation greatly depends on the concentration of the reaction mixture and the dielectric properties of the material [1–4]. The high catalytic activity of BPyBr observed over a relatively short reaction time of 30 s arises from the effective polarization of the catalyst by microwave radiation. There was a steady increase in AGE conversion as the microwave power increased from 50 W to 200 W. However, a further increase in microwave power from 200 W to 500 W resulted in a slight decrease in the selectivity. It appears that the higher temperatures attained with higher microwave power suppressed the dissolution of CO<sub>2</sub>, resulting in an insufficient supply of CO<sub>2</sub> molecules available for cycloaddition to epoxides [15-18]. In order to understand the heat generated during the microwave assisted BPyBr-catalyzed cycloaddition of AGE and CO<sub>2</sub>, a graph is plotted (Fig. S1, Supplementary information) showing the temperature profiles of the reactor under different microwave power. The temperature increased rapidly until 30 s and then it increased slowly until a constant value was reached.

Table 3

Effect of reaction time on	the reactivity of	of BPyBr for	the synthesis	of AGC.
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Entry	Time (sec)	Conversion (%)	Selectivity (%)	$TOF(h^{-1})$
1	10	77	94	5115
2	20	83	95	2757
3	30	95	95	2129
4	60	98	94	1091
5	120	99	96	553

Reaction conditions: AGE = 18.6 mmol, BPyBr = 1 mmol,  $P_{CO2} = 0.96 \text{ MPa}$ , microwave power = 200 W. TOF: moles of AGE converted per mol of ionic liquid/h.

Conversion, Selectivity (%) 90 85 80 75 70 Conversion (%) Selectivity (%) 65 60 0 100 200 300 400 500 600 Microwave power (W)

**Fig. 1.** Effect of microwave power on the reactivity of BPyBr for the synthesis of AGC. Reaction conditions: AGE = 18.6 mmol, catalyst amount = 1 mmol,  $P_{CO_2} = 0.96$  MPa, time = 30 s.

## 3.5. Effect of CO<sub>2</sub> pressure

Fig. 2 shows the effect of  $CO_2$  pressure on the microwave-assisted cycloaddition of AGE and  $CO_2$  in the presence of BPyBr at 200 W for 30 s. As seen in Fig. 2, the AGE conversion increased from 10% to 74% when the  $CO_2$  pressure increased from 0.34 MPa to 0.82 MPa. Upon a further increase in the pressure (0.96 MPa), the conversion increased to a maximum of 95%. During these catalytic reactions, higher  $CO_2$  pressure can effectively increase the solubility of  $CO_2$  in AGE, enabling the reaction equilibrium to shift towards carbonate formation. However, it has been observed that a too high pressure decreases the conversion owing to the effect of dilution by excessive quantities of  $CO_2$  [8].

## 3.6. Cycloaddition of CO<sub>2</sub> with various epoxides

Various epoxide substrates were subjected to the cycloaddition reaction using BPyBr under the optimized conditions determined as explained above. The results are summarized in Table 4. Among the terminal epoxides, AGE (entry 2) showed the highest conversion. It is also observed in our previous works, that the epoxide substrates



Fig. 2. Effect of  $CO_2$  pressure on the reactivity of BPyBr for the synthesis of AGC. Reaction conditions: AGE = 18.6 mmol, catalyst amount = 1 mmol, microwave power = 200 W, time = 30 s.



Reaction conditions: epoxide = 18.6 mmol, BPyBr = 1 mmol,  $P_{CO_2} = 0.96 \text{ MPa}$ , microwave power = 200 W, time = 30 s.

possessing an additional ether oxygen linkage in the side chain (AGE), showed higher catalytic activity compared to other epoxide substrates [18,21]. Propylene oxide (entry 1) showing the lowest activity among the terminal epoxides may be due to the absence of an effective catalyst-substrate contact as a result of its low boiling point. The selectivity of the cyclic carbonates was high (>90%) indicating negligible formation of byproducts in the cycloaddition reaction. The disubstituted epoxide, cyclohexene oxide (entry 4), exhibited the lowest activity for the production of the corresponding cyclic carbonate, probably because of the high steric hindrance produced by the cyclohexene ring [24]. A plausible mechanism (Scheme S2, Supplementary information) for this reaction is the ring opening of the epoxide by means of a nucleophilic attack by the halide anion, which leads to an oxy anion species affording the corresponding cyclic carbonate after reaction with carbon dioxide [11].

## 4. Conclusions

In summary, pyridinium-based ILs were employed as an efficient catalyst for the solvent-free microwave-assisted cycloaddition of AGE and CO<sub>2</sub> to yield AGC under moderate reaction conditions. The reaction progressed rapidly within 30 s to produce AGC in high yields with a TOF ranging from 200 to 7000  $h^{-1}$ . Among the various pyridinium-based ILs, BPyBr showed the best conversion and selectivity with a high TOF of 2129 h<sup>-1</sup>. IL with bulkier alkyl chain length and more nucleophilic anion showed better catalytic activity. Higher microwave power was favorable for high AGE conversion, but the AGC selectivity decreased at too high microwave power. The AGE conversion increased with increasing CO<sub>2</sub> pressure due to the increase of CO<sub>2</sub> solubility in the solution of AGE and catalyst. 1 mmol of BPyBr at a microwave power of 200 W with 0.96 MPa of CO<sub>2</sub> pressure for 30 s was the optimal condition to obtain the highest conversion of 95% for AGE. Employing microwave irradiation for the synthesis of cyclic carbonates has advantages such as shorter reaction time, better energy efficiency, simplicity, environmentfriendliness, and cost-effectiveness.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at http://dx. doi.org/10.1016/j.catcom.2014.05.016.

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