A New Approach to CO₂ Capture and Conversion Using Imidazolium Based-Ionic Liquids as Sorbent and Catalyst

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Embora tecnologias de captura e armazenamento de carbono (CCS) estejam recebendo grande atenção para a mitigação do efeito estufa, ainda existem muitas desvantagens, tais como o aumento dos custos e gastos de energia associados à sua implementação. No entanto, o uso de CO₂ como bloco de construção C1 em síntese orgânica pode ser muito atraente para o desenho de processos ecológicos. Neste trabalho, foram estudados a sorção de CO₂ e atividades catalíticas de alguns líquidos iônicos (ILs) base imidazólio para síntese de carbonato cíclico. O trabalho demonstra que a presença de um grupo nucleófilo no sistema catalítico pode melhorar seu desempenho, através da utilização de um IL com ânion halogenado ou por mistura de co-catalisador halogenado com ILs. A última abordagem permitiu a obtenção de um sistema de captura de CO₂ eficaz, constituído por IL fluorado mais ZnBr₂ que realiza a síntese de carbonato cíclico com 90% de rendimento e 82% de seletividade.

Keywords: ionic liquids, CO₂ capture, cyclic carbonate

Introduction

The increase of CO₂ concentration in the atmosphere and, consequently, the intensification of global warming are posing major problems to the environment. Actions aiming to reduce this gas amounts in the atmosphere are imperative. Among the possibilities, are the CO₂ capture and geological storage and CO₂ capture and subsequent conversion in products with higher added value.

Although the carbon capture and storage (CCS) technologies are receiving great attention for mitigation of greenhouse gas effect, the increasing costs and energy penalties associated to its implementation are still major drawbacks. However, the use of CO₂ as a C1 building block in organic synthesis can be very attractive for the design of environmentally friendly processes. In this work, we have studied both the CO₂ sorption and catalytic activities of some imidazolium based ionic liquids (ILs) for cyclic carbonate synthesis. The work demonstrates that the presence of a nucleophilic group in the catalytic system can enhance its performance by the use of an IL with a halide anion or by mixing a halide co-catalyst with ILs. The latter approach allowed to obtain an effective system for CO₂ capture constituted by a fluorinated IL plus ZnBr₂ that performs cyclic carbonate synthesis with 90% yield and 82% of selectivity.

Keywords: ionic liquids, CO₂ capture, cyclic carbonate

The chemical transformation of CO₂ is one of the most interesting options for CO₂ mitigation. The synthesis of cyclic carbonates appears as an interesting and effective solution. These compounds are versatile, widely used in chemical industry as polar aprotic solvents and raw materials for the production of a broad range of products. However, CO₂ is a thermodynamically stable compound and the development of an effective and selective catalytic system is crucial. A catalytic system that could perform cyclic carbonates syntheses under mild conditions still remains as major challenge allied to the difficulties faced for catalyst separation and recycle.
In order to overcome these operational difficulties, various conversions of CO$_2$ to cyclic carbonates using ionic liquids (ILs) as catalysts have been reported in literature.$^{6,9-12}$ Ionic liquids are very versatile compounds. They are more environmentally friendly when compared to conventional organic solvents exhibiting the possibility of the catalyst separation and recycling.$^{6,13-16}$

Peng and Deng$^{10}$ reported the synthesis of propylene carbonate from CO$_2$, using the ILs [bpy][BF$_4$], [bmim][BF$_4$], [bmim][Cl] and [bmim][PF$_6$] and demonstrated that the catalytic activity of an ionic liquid depends on the nature of the cation and the anion. For the anions, the activity decreased in the order bmim$^+ >$ bpy$^+$ and BF$_4^− >$ Cl$^− >$ PF$_6^−$. The effect of the cation and the anion were also investigated by Yang et al.$^9$ using IL based on cations C$_6$DABCO$^+$, C$_6$DABCO$^+$, HDBU$^+$, HTBD$^+$ or HHMTA$^+$ and anions OH$^−$, Cl$^−$, Br$^−$, BF$_4^−$, PF$_6^−$, Tf$_2$N$^−$ and AcO$^−$. The results showed that the catalytic effect decreases in the order HDBU$^+ >$ HTBD$^+ >$ OMIM$^+ >$ C4DABCO$^−$ > C8DABCO$^+$ > BMIM$^+$ > HHMTA$^+$. The anion OH$^−$ presented the best result and Cl$^−$ and Br$^−$ showed good performance as well. The acetate anion bound to the cation HDBU also showed catalytic activity comparable to that of Cl$^−$, but its thermal stability was not ideal. The anions Tf$_2$N$^−$, PF$_6^−$, and BF$_4^−$ were not effective.

The effect of alkyl chain length was studied by Kawanami et al.$^6$ in the 1-alkyl-3-methylimidazolium through variations of the alkyl chain length from C$_2$ to C$_8$. The results indicated that the increase of the side alkyl chain in the cation has large effect on the catalyst performance since the yield has been improved with the increasing of the alkyl chain length. This result can be attributed to the increasing solubility of both the epoxide and the CO$_2$ in the ionic liquid. Further studies showed that the combination of ionic liquids and metallic compounds has proven to be effective, increasing the catalytic activity of ILs in the synthesis of cyclic.$^{11,15}$

Regarding CO$_2$ capture, the imidazolium cations allied to fluoroalkyl anions stand as very effective systems for CO$_2$ absorption.$^{17,18}$ Nevertheless, in relation to the reaction of CO$_2$ conversion in cyclic carbonates, the results reported in the literature are contradictory in respect to the activity of ionic liquids combined to fluoroalkyl anions.

This study aims to investigate the ionic liquids behavior in both catalytic activity in propylene carbonate syntheses as well as the CO$_2$ absorption capacity. The reaction conditions as temperature, pressure, reaction time, effect of IL cation and anion as well as the addition of metallic compounds to reaction media and the catalyst recycling were also investigated.

### Experimental

#### Chemicals

Acetone (Vetec, 99.5%), acetonitrile (Vetec, 99.5%), toluene (Merck, 99.9%), carbon dioxide (99.998%, Air Liquid), 1-chlorobutane (Acros Organics, 99%), dichloromethane (Vetec, 99.5%), 3,3-dimethylchlorobutane (Alfa Aesar, 97%), lithium(I) bis(trifluoromethanesulfonyl)imide - LiTf$_2$N (Aldrich, 99%), 1-methylimidazolide - C$_6$H$_5$N$_2$ (Aldrich, 99%), magnesium sulfate - MgSO$_4$ (Acros Organics, 97%), sodium tetrafluoroborate - NaBF$_4$ (Acros Organics, 98%), sodium hexafluorophosphate - NaPF$_6$ (Alfa Aesar, 99%), propylene oxide - C$_3$H$_6$O (Aldrich, 99%), zinc chloride - ZnCl$_2$ (Nuclear, 97%), zinc bromide - ZnBr$_2$ (Sigma-Aldrich, 98%).

#### Ionic liquids syntheses

The ionic liquids 1-butyl-3-methylimidazolium chloride [bmim][Cl], 1-butyl-3-methylimidazolium tetrafluoroborato [bmim][BF$_4$], 1-butyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide [bmim][Tf$_2$N] and 1-dimethylbutyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide [dmmbmim][Tf$_2$N] were synthesized following procedures well described in literature.$^{19,21}$

The ionic liquids were characterized by Fourier transform infrared spectroscopy (FTIR), using a Perkin-Elmer spectrophotometer model Spectrum 100 FT-IR with full attenuated reflectance accessory (ATR), as well as by proton nuclear magnetic resonance ($^1$H-NMR) on a Varian Spectrophotometer, model VNMR 300 MHz, using DMSO-$d_6$ as solvent and glass tubes of 5 mm in diameter and are in accordance with the literature.

#### Cycloaddition reaction

The syntheses of propylene carbonate (PC) from CO$_2$ and propylene oxide (PO) were carried out in the presence of imidazolium cation-based ILs combined to different anions Cl$^−$, BF$_4^−$ and Tf$_2$N$^−$. These compounds were tested in the presence of metallic halides (ZnCl$_2$ or ZnBr$_2$). The use of IL [dmmbmim][Tf$_2$N] was also evaluated.

All cycloaddition reactions were performed in a stainless steel autoclave of 120 cm$^3$ equipped with magnetic stirring. For a typical reaction, it was used 100 mmol of propylene oxide, 0.625 mmol of metal salts (ZnCl$_2$ or ZnBr$_2$) and/or 2.5 mmol of ionic liquid. The syntheses were performed without any additional solvent. The autoclave was pressurized with CO$_2$ and heated to the desired working
temperature. After the reaction completion, the reactor was cooled to room temperature and slowly depressurized.

The separation of the catalyst from propylene carbonate was performed by a simple distillation under inert atmosphere (N₂). In the reactions carried out in the presence of metal salts, a filtration step was performed before the distillation process. The resulting liquid mixtures were analyzed using a gas-chromatograph Shimadzu GC-14B equipped with a flame ionization detector (FID) and a DB-5HT column (15 m × 0.32 mm × 0.10 µm) using acetophenone as internal standard and dimethyl ether as solvent.

### CO₂ absorption measurements

The CO₂ sorption of the samples were gravimetrically assessed in a magnetic suspension balance (MSB), (Rubotherm Prazisionsmesstechnik GmbH, 35 MPa and 673.15 K) equipped with a single sinker device for absorbate density determination and thermostatted with an oil bath (Julabo F25 ± 273.16 K). The apparatus details are well described in literature. When compared to other gravimetric sorption methods, the MSB device has the advantage of allowing high pressure sorption measurements since the sample can be potted into a closed chamber coupled to an external accurate balance (accuracy of ± 10 µg). The samples (0.06 to 0.09 g) were weighed and transferred to the MSB sample container and the system was subjected to a 10⁻⁷ MPa vacuum at the temperature of the sorption measurement, 298.15 K, for 24 h. For all tests, constant weight was achieved in this time. The CO₂ was admitted into the MSB pressure chamber till the desired pressure, 0.1-3 MPa in this study, pressure gauges with an accuracy of 0.01 bar. The solubility of CO₂ in the ILs pressure was measured 3 to 4 h until there was no more weight variation for CO₂ sorption. At this step of solubility of CO₂ in the ILs, the weight reading from the microbalance at pressure P and temperature T is recorded as \( w_s(P,T) \). After each sorption test, CO₂ desorption were performed and all the samples returned to its original weight at the end. The mass of dissolved CO₂ in the ILs \( (w_s) \), was calculated using the equation 1.

\[
W_s = W_s(P,T) - W_s(P,T) + \rho(P,T)(V_w(T) + V(T)) - W_s(vac,T)
\]

where \( W_s(P,T) \) is the weight of the sample container, \( \rho(P,T) \) CO₂ density, directly measured with the MSB coupled single-sinker device, being not necessary the application of any state equation to calculate the CO₂ sorption values. \( V_w(T) \) is the volume of the sample container, determined from a buoyancy experiment when no sample is charged into the sample container, \( V_w(T) \) is the original volume of the sample, and \( W_s(vac,T) \) is the weight of the sample under vacuum. The term \( \rho(P,T)(V_w(T) + V(T)) \) represents the buoyancy force. The results of CO₂ sorption were expressed in molar fraction.

### Results and Discussion

#### Effect of IL and co-catalyst

Aiming to evaluate the influence of the anion in the syntheses of propylene carbonate (PC) from CO₂ and propylene oxide (PO), cycloaddition reactions were carried out in the presence of ionic liquids (ILs) based on imidazolium cation combined to Cl⁻, BF₄⁻ and Tf⁻N⁺ anions.

The first choice for assessing the ILs catalytic activity for CO₂ conversion reactions were \([\text{bmim}][\text{Tf}_2\text{N}]\) and \([\text{bmim}][\text{BF}_4]\), since these compounds are reported in the literature as good solvents for carbon dioxide. The results depicted in Table 1 show that these ILs exhibited extremely low activities in the cyclic carbonates syntheses. Despite the good ability for CO₂ absorption presented by Tf⁻N⁺ and BF₄⁻ anions their catalytic activities were not satisfactory (entry 1 and 7).

On the other hand, the IL \([\text{bmim}][\text{Cl}]\) presented an interesting catalytic performance presenting an yield of 84% and 90% of selectivity in PC and a turnover frequency (TOF) of 5 h¹ (entry 6) as seen in Table 1. This behavior is probably due to its good nucleophilic character. These results demonstrate that the activity of ILs is strongly influenced by the nature of the anion and are in accordance with the results described by Yang et al. in relation to the anions behavior. The low catalytic activity for Tf⁻N⁺ and BF₄⁻ anions can be directly linked to the structural volume of the anions and their low nucleophilicity indicating that the best ILs for CO₂ capture are not necessarily the best catalysts for CO₂ transformation. However, the combination of these ILs with metallic compounds contributed to an increase on the catalytic activity.

The results of the combination of metal compounds to \([\text{bmim}][\text{Tf}_2\text{N}]\) and \([\text{bmim}][\text{BF}_4]\) revealed that the addition of ZnBr₂ contributed to an increase in catalytic activity for both ILs (entry 3-4; 9, Table1), indicating that the presence of Lewis acidic compounds as cocatalysts greatly enhances the activity of ionic liquid for the cyclic carbonate syntheses. With the exception of \([\text{bmim}][\text{Cl}]\), the catalytic activities of ionic liquids were quite influenced by the nature of halide bonded to zinc atoms. The reactivity was found to be in the order ZnBr₂ > ZnCl₂ as seen in Table 1, suggesting the importance of the nucleophilicity of halide counterions.
Aiming to increase the catalytic activity, an equimolar mixture of the catalytic ILs [bmim][Tf₂N] + [bmim][Cl] and the metallic compound ZnBr₂ has been evaluated (entry 10). However, the yield of this system was lower than that attained separately for each IL in the presence of ZnBr₂ (entry 4-8).

One of the acceptable mechanisms for the cycloaddition of CO₂ to epoxide catalysed by IL involves a nucleophilic attack of the IL anion to the less hindered carbon atom of the epoxide ring. In the next step, an oxy anion species is formed. The carbon atom of the CO₂ interacts with the anion species producing an anion alkylcarbonate converted to the cyclic carbonate by intermolecular cyclic elimination.15

In our work, we concluded that the Tf₂N⁻ and BF₄⁻ containing ILs need a metal halide, a Lewis acid to interact with the oxygen of the epoxide atom allowing the IL basic anion to perform the attack to the less hindered atom of the epoxide ring, see Scheme 1a. This could be assigned to the non-nucleophilic natures of the Tf₂N⁻ and BF₄⁻ anions.27 When [bmim][Cl] is used as catalyst, probably the attack of the epoxide is performed by the Cl⁻ in the less hindered carbon atom, in the first step of the reaction, see Scheme 1b.

As we can see in Table 1, the IL [bmim][Tf₂N] combined with ZnBr₂ showed the best yield (90%) (entry 4). Thus, it has been selected for the evaluation of temperature; pressure, reaction time as well as the behavior of this system in recycle.

**Effect of temperature**

The selectivity, yield and activities values obtained at different temperatures are shown in Table 2. It is evidenced that the increase in temperature from 373.15 K to 383.15 K resulted in a significant increase in the yield (entry 13-4), and after 383.15 K the increasing of the temperature decreases the yield (entry 4-16).
It is possible that the yield decreasing is associated with the reduction in the PC selectivity in higher temperature, resulting in side reactions such as PO isomerization and PC polymerization.

Influence of CO$_2$ pressure

Pressure affects cyclic carbonate synthesis, and the optimal pressure occurs at around the CO$_2$ critical pressure.$^{12}$ From the results shown in Table 3, it can be seen that our results showed a different behavior. The results shown in Table 3 are in accordance to the literature, evidencing that an insufficient or excessive amount of CO$_2$ results in relatively low conversions, i.e., there is an ideal molar ratio of propylene oxide and CO$_2$ to obtain a high conversion. A higher CO$_2$ pressure can retard the interaction between epoxide and the catalyst. On the other side, it can be highlighted a significantly improve in the carbonate selectivity with the pressure augmentation.$^{9,10}$

Influence of the reaction time

The influence of the reaction time on the propylene carbonate synthesis is presented in the Table 4. It was observed that the reaction time reduction from 6 up to 2 h (entry 4-19), provided a decreases in yield, curiously the increase in reaction time from 6 to 8 h (entry 4-21) decreases the yield. The reaction time of 6 h was ideal for this the system.

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Table 2. Temperature effect in the PC synthesis using [bmim][Tf$_2$N] and ZnBr$_2$ as catalyst

<table>
<thead>
<tr>
<th>entry</th>
<th>Temperature / K</th>
<th>Yield / %</th>
<th>Selectivity / %</th>
<th>TON</th>
<th>TOF / h$^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>13</td>
<td>373.15</td>
<td>65</td>
<td>96</td>
<td>24.9</td>
<td>4.16</td>
</tr>
<tr>
<td>4</td>
<td>383.15</td>
<td>90</td>
<td>82</td>
<td>29.5</td>
<td>4.92</td>
</tr>
<tr>
<td>14</td>
<td>393.15</td>
<td>75</td>
<td>68</td>
<td>20.4</td>
<td>3.40</td>
</tr>
<tr>
<td>15</td>
<td>403.15</td>
<td>68</td>
<td>94</td>
<td>25.6</td>
<td>4.26</td>
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<tr>
<td>16</td>
<td>413.15</td>
<td>64</td>
<td>86</td>
<td>22.0</td>
<td>3.67</td>
</tr>
</tbody>
</table>

Reaction conditions: PO 100 mmol, catalyst 2.5 mmol, 0.625 mmol of ZnBr$_2$, initial CO$_2$ pressure 4.0 MPa, t = 6 h. TON = mmol of products/mmol of catalyst. TOF = TON/time.

Table 3. CO$_2$ effect pressure in the PC synthesis using bmim[Tf$_2$N +ZnBr$_2$ as catalyst

<table>
<thead>
<tr>
<th>entry</th>
<th>CO$_2$ pressure / MPa</th>
<th>Yield / %</th>
<th>Selectivity / %</th>
<th>TON</th>
<th>TOF / h$^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>17</td>
<td>2.5</td>
<td>84</td>
<td>53</td>
<td>17.8</td>
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<tr>
<td>4</td>
<td>4.0</td>
<td>90</td>
<td>82</td>
<td>29.5</td>
<td>4.92</td>
</tr>
<tr>
<td>18</td>
<td>5.0</td>
<td>52</td>
<td>95</td>
<td>19.8</td>
<td>3.30</td>
</tr>
</tbody>
</table>

Reaction conditions: PO 100 mmol, catalyst 2.5 mmol, 0.625 mmol of ZnBr$_2$, T = 383.15 K, t = 6 h. TON = mmol of products/mmol of catalyst. TOF = TON/time.
Recycling

To investigate the reuse effect in the catalyst system ([bmim][Tf$_2$N] and ZnBr$_2$) the recycling experiment was conducted at the best conditions achieved (4.0 MPa, 383.15 K and 6 h). The procedure adopted for the separation of the catalytic system and propylene carbonate was the same used for all syntheses and described in experimental section. This procedure was repeated four times. The catalytic activity was not maintained even for only four recycle reactions. This behavior is probably due to the loss of the metallic compound (ZnBr$_2$) in the PC separation operation, decreasing the catalytic activity.

Aiming to study the ZnBr$_2$ loss in PC recycle, reactions were carried out in the same conditions of the recycle reactions presented before. After the PC separation step the ZnBr$_2$ was re-added (0.625 mmol) and the reaction performed. With the addition of the ZnBr$_2$ after each reaction the yield increased 17% when compared to the reaction without the addition of the metal halide and remained almost constant. These results evidenced that the loss of the ZnBr$_2$ is probably occurring as described in literature.

In order to improve the separation process and also study the influence of the increase of IL concentration in the reaction medium, a reaction with 100 mmol of PO, 28 mmol of [bmim][Tf$_2$N], 0.625 mmol of ZnBr$_2$, in the same reaction conditions (383.15 K, 6 h and 4 MPa of CO$_2$) was performed. The yield, selectivity and activity were the same obtained with 2.5 mmol of IL. The increment of IL content in the reaction medium, in this case, does not facilitate the PC separation. These results evidenced that the catalytic conversion of propylene oxide and CO$_2$ in PC works with catalytic quantities of IL but for integration of CO$_2$ capture and transformations process it is possible to use a higher IL volume and maintain the same yield.

### Table 4. Influence of the reaction time in PC synthesis using bmimTf$_2$N+ZnBr$_2$ as catalyst

<table>
<thead>
<tr>
<th>entry</th>
<th>Reaction time / h</th>
<th>Yield / %</th>
<th>Selectivity / %</th>
<th>TON</th>
<th>TOF / h$^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>19</td>
<td>2</td>
<td>31</td>
<td>89</td>
<td>11.0</td>
<td>5.52</td>
</tr>
<tr>
<td>20</td>
<td>4</td>
<td>55</td>
<td>96</td>
<td>21.1</td>
<td>5.28</td>
</tr>
<tr>
<td>4</td>
<td>6</td>
<td>90</td>
<td>82</td>
<td>29.5</td>
<td>4.92</td>
</tr>
<tr>
<td>21</td>
<td>8</td>
<td>74</td>
<td>77</td>
<td>22.8</td>
<td>2.85</td>
</tr>
</tbody>
</table>

Reaction conditions: PO 100 mmol, catalyst 2.5 mmol, 0.625 mmol ZnBr$_2$, initial CO$_2$ pressure 4.0 MPa, T = 383.15 K. TON = mmol of products/mmol of catalyst. TOF = TON/time.

#### CO$_2$ solubility

The results shown for PC syntheses using ILs as catalysts evidenced the importance of the anion. For CO$_2$ capture also the anion plays a major role. In order to combine the CO$_2$ capture and conversion, it is important to find a system that can act both as a solvent for CO$_2$ separation and as a catalyst for CO$_2$ transformation. In Figure 1 it can be seen the different CO$_2$ absorption capacity for the ILs that proved to perform also the role as a catalyst for propylene carbonate syntheses.

As it can be seen from Figure 1, the use of [bmim][Cl]$^-$ for CO$_2$ capture is not a good option, despite of this IL act as a good catalyst for PC synthesis. The CO$_2$ sorption at 0.5 MPa is near 0 mol% steady for the combination of [bmim][Tf$_2$N] and ZnBr$_2$, it is approximately 14 mol%. The fluoroalkyl anions allied to imidazolium cations present a relatively high CO$_2$ solubility and have been described in the literature for CO$_2$ absorption process. The use of these compounds for PC syntheses needs the addition of a metallic compound in order to achieve an acceptable yield. It must be mentioned that the best conditions for CO$_2$ capture and chemical conversion are distinct. Nevertheless, the results presented in Figure 1 evidenced that the CO$_2$ solubility in a solvent combining [bmim][Tf$_2$N] and ZnBr$_2$ can be a good option for CO$_2$ capture and conversion of this molecule in a PC adding value to this chemical.

#### Conclusions

The results showed that the best ILs for capturing CO$_2$ are not necessarily the best catalysts for CO$_2$ conversion. The activities of the ILs in both processes are strongly influenced by the nature of the anion. While the solubility...
of CO₂ is improved by fluorinated anions in catalysis for CO₂ conversion, the activity is favored by a higher nucleophilicity of the anion. Our results evidenced that the combination of imidazolium-based ILs/fluoroalkyl anions and a metal halide compound is a good approach for combining the CO₂ capture and conversion. In catalytic conversion of CO₂ to carbonates, the temperature, pressure and reaction time showed to play an important role in the conversion and selectivity. The best result was obtained with the catalytic system [bmim][Tf₂N]/ZnBr₂ at 4.0 MPa of pressure, 383.15 K of temperature and 6 h of reaction time.

Acknowledgements

The authors would like to thank Petrobras for financial support and S. E. and R. L. thank CNPq for technological development research scholarship.

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Submitted on: May 23, 2014
Published online: July 29, 2014