



## Study on microporous supported ionic liquid membranes for carbon dioxide capture



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### ABSTRACT

To capture carbon dioxide directly from ambient air, the fabrication of supported ionic liquid membranes (SILMs) on asymmetric and symmetric microporous poly(vinylidene fluoride) (PVDF) membranes were studied. The effect of support membrane structure on the IL loading, the stability of fabricated SILMs, and the carbon dioxide (CO<sub>2</sub>) capturing abilities of both SILMs were evaluated. The evaluations include the gas permeation of pure CO<sub>2</sub> and nitrogen (N<sub>2</sub>), the mixed CO<sub>2</sub>-N<sub>2</sub> gases containing 50%, 10% and 1% CO<sub>2</sub>, and the directly compressed ambient air. The results demonstrated that pure CO<sub>2</sub> showed the highest permeance, followed by CO<sub>2</sub>-air and then N<sub>2</sub>. Compared with asymmetric SILMs which had higher gas permeance, the symmetric SILMs had better selectivity for CO<sub>2</sub>/N<sub>2</sub>, to more than 20 of CO<sub>2</sub>/N<sub>2</sub> selectivity, for both single and mixed gases. The SILMs also were found to be capable of capturing CO<sub>2</sub> from air from 2 to more than 5 of CO<sub>2</sub>/air selectivity for separation of air enriched with low CO<sub>2</sub> concentrations (10%, 1% and zero) as the transmembrane pressure increased to 0.25 MPa. Thus, this work might provide a potential method for sequestering CO<sub>2</sub> directly from atmosphere to avoid the need for extensive CO<sub>2</sub>-transportation.

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## 1. Introduction

Since the pre-industrial age, the CO<sub>2</sub> concentration in atmosphere has increased from 280 ppm to current level of about 400 ppm (Bacocchi et al., 2006; Yu et al., 2008). Unlike existing carbon capture and storage (CCS) mainly fixes CO<sub>2</sub> of 5–15% concentrations from fixed point sources, the CO<sub>2</sub> capture from ambient air is a process that can be used to capture lower concentration CO<sub>2</sub> (less than 400 ppm) directly from the ambient air (Keith, 2009; Mahmoudkhani and Keith, 2009; Power et al., 2013) at different locations and time, the ideal capture unit of which could be run at an optimal sequestration site, thus to avoid the need for extensive CO<sub>2</sub>-transportation infrastructures (Nikulshina et al., 2006).

The necessity to capture CO<sub>2</sub> from ambient air comes from the ability of CO<sub>2</sub> to remain in the atmosphere for extended periods. Using the Bern carbon cycle model as shown in Eq. (1), where  $t$  represents years, it has been predicted that CO<sub>2</sub> levels could remain

at approximately 33% of initial CO<sub>2</sub> concentrations after 100 years, and approximately 20% even after 1000 years (Hansen et al., 2007).

$$\text{CO}_2(\%) = 18 + 14e^{-t/420} + 18e^{-t/70} + 24e^{-t/21} + 26e^{-t/3.4} \quad (1)$$

However, the capture of CO<sub>2</sub> directly from ambient air is difficult because of the high thermodynamic barrier caused by the lower concentration of CO<sub>2</sub> in atmosphere (Keith, 2009). Such a high degree of CO<sub>2</sub> dilution limits the practical capture options of sorption approaches. Chemical and physical absorption appears to be one of the best commercial processes for atmospheric air separation on a small scale, and alternative methods utilizing the alkali salts of various weak acids such as sodium, potassium, phosphate, ammonia and phenolate have been reported to adsorb CO<sub>2</sub> from air (Lu and Hu, 2009). The separation of CO<sub>2</sub> using chemical absorption involves four main steps: absorption, chemical production, regeneration and hydrolysis (Rahaman et al., 2011). Although chemical absorption using NaOH is known, the energy required for NaOH to complete this reaction is approximately 150 kJ per molecule of CO<sub>2</sub> captured (Mahmoudkhani and Keith, 2009). Mérel et al. (2006) stated that the stronger the affinity or absorbency, the more difficult it is to desorb the gas, which

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increases the heat energy required for recycling and reusing the adsorbent in the next cycle. The use of a liquid absorbent, such as monoethanolamine (MEA), shows a higher absorption capacity for CO<sub>2</sub> (a mole of CO<sub>2</sub> absorbed per two moles of MEA). However, this absorption process is generally uneconomic, as it can consume up to 70% of the total operating costs (Idem et al., 2006).

The CO<sub>2</sub> capture from ambient air using membrane separation has recently emerged as a promising technology, exhibiting high efficiency, high energy savings, and no pollution as shown in our recent review (Rahaman et al., 2011). The immobilization of stable chemicals, such as ionic liquid (IL), can increase the performance of membrane (Kim et al., 2011). This type of membrane is classified as supported ionic liquid membrane (SILM), which avoids chemical volatility problems compared with supported liquid membranes with traditional liquid absorbents, such as MEA or NaOH. According to Hasib-ur-Rahman et al. (2010), IL also shows the potential for capturing CO<sub>2</sub> from ambient air. Other works (Myers et al., 2008; Carlisle et al., 2010) also state that CO<sub>2</sub> has an extraordinary solubility into ILs. For instance, Myers et al. (2008) used SILMs for separating CO<sub>2</sub> from hydrogen (H<sub>2</sub>) at elevated temperature, and found the selectivity was maximum at 85 °C. Carlisle et al. (2010) found that the selectivity of CO<sub>2</sub>/N<sub>2</sub> was comparable with all studies. The imidazolium cations contained SILM can increase CO<sub>2</sub>/N<sub>2</sub> selectivity (Bara et al., 2009). For example, 1-butyl-3-methylimidazolium tetrafluoroborate ([Bmim]BF<sub>4</sub>) has been used to separate CO<sub>2</sub> (Neves et al., 2009; Sanchez et al., 2007; Cserjési et al., 2010), and it is found that CO<sub>2</sub> is more likely to be dissolved in alkylimidazolium-based ILs than in other types of ILs (Hasib-ur-Rahman et al., 2010).

In this study, the IL of [Bmim]BF<sub>4</sub> was deposited into PVDF membranes, which have an extremely high ability to prevent [Bmim]BF<sub>4</sub> solvent loss (Hasib-ur-Rahman et al., 2010). The effect of support membrane structure is then investigated on the IL loading, the obtained SILMs stability, and the CO<sub>2</sub> capturing abilities of both SILMs, including the gas permeations of pure CO<sub>2</sub> and N<sub>2</sub>, the mixed gases containing 50%, 10% and 1% CO<sub>2</sub>, and the directly compressed ambient air. Since the process of capturing CO<sub>2</sub> directly from ambient air using membrane separation is a rather significant proposal for compensating chemical absorption processes (Power et al., 2013; Rahaman et al., 2011; Cheng et al., 2008), our aim here is to prepare SILMs for capturing CO<sub>2</sub> directly from ambient air, which to the best of our knowledge, has not been reported in literature.

## 2. Experimental

### 2.1. Membrane preparation

[Bmim]BF<sub>4</sub> was purchased from Sigma–Aldrich. The supporting membranes including both asymmetric and symmetric PVDF were supplied by the Jiangsu Dafu Membrane Technology, China. Prior to SILM preparation, the membrane was treated by drying a PVDF membrane under vacuum conditions for 1 h at ambient temperature in order to remove air and moisture trapped in the membrane.

The SILMs were fabricated by pouring [Bmim]BF<sub>4</sub> IL directly onto the membrane and then allow the membrane for 30 min in the ambient condition to let the IL flows through the pores freely prior to vacuum operations. Then, the nascent membranes were heated to 60 °C under vacuum conditions for 12 h, followed for further 12 h at 25 °C to remove the residual air and moisture. Finally, the excessive IL on the surface was swept by tissue, and the fabrication process of SILMs was completed.

### 2.2. Membrane characterization

The asymmetric and symmetric SILMs were characterized using both a conventional camera and a scanning electron microscope (SEM) (Carl Zeiss D, Germany). The morphological structure of the resultant membranes was investigated using an Ultra 55 emission scanning electron microscope. The membranes were frozen in liquid nitrogen, broken to obtain the cross section and coated with gold prior to SEM observation.

### 2.3. Membrane stability test

Prior to the gas separation test, the stability of both SILMs, based on symmetric and asymmetric membranes, were evaluated by measuring the weight differences at different transmembrane pressures and the variation of N<sub>2</sub> permeation, respectively. The stability were measured based on minimal weight difference after providing a pressure and N<sub>2</sub> permeances alteration.

### 2.4. Gas separation test

Gas permeation tests were carried out using the traditional volumetric technique. Fig. 1 shows the schematic of experimental set-up for capturing CO<sub>2</sub> using SILMs. The performance of membranes was tested using both single and mixed gas permeation processes. The gas inlet pressure was maintained above atmospheric pressure. Feed gas regulators were used to vary feed pressure from 0.1 MPa to 0.6 MPa. The permeation of single gases (CO<sub>2</sub>, and N<sub>2</sub>) and the ambient air were investigated. The performance of mixed gases were also measured using different CO<sub>2</sub> concentrations, including CO<sub>2</sub>/N<sub>2</sub> (50%, v/v), CO<sub>2</sub>/air (10%, v/v), CO<sub>2</sub>/air (1%, v/v) and CO<sub>2</sub> of the ambient air (directly compressed by a pump).

The flow rate of permeate was measured using a bubble flow meter. In case of the mixed gas permeation, a valve controlled the flow rate of the retentate stream. The compositions of all gas streams were analyzed by a gas chromatograph (Agilent Model 1790, Agilent Technologies) equipped with a packed column and a thermal conductivity detector. The feed gas stream consisted of a mixture of air enriched with CO<sub>2</sub>. An air compressor (Hangzhou Hangkong Compressor Co., Ltd.) was adapted to obtain the desired CO<sub>2</sub> concentration. The performance of the fabricated membranes were evaluated using the gas permeance which is flux of component *i* ( $Q_i$ ), and selectivity ( $S_{i/j}$ ) as shown in Eqs. (2) and (3), respectively.

$$Q_i = \frac{JX_i}{A_{\text{eff}}\Delta P_i} \quad (2)$$

$$S_{i/j} = \frac{Q_i}{Q_j} \quad (3)$$

where  $J$  (mol/m<sup>2</sup> s) indicates the gas permeance at steady state,  $X_i$  signifies the volume fraction of permeated component *i*,  $A_{\text{eff}}$  (m<sup>2</sup>) denotes the effective membrane area of 136.56 cm<sup>2</sup>, and  $\Delta P_i$  (Pa) is the transmembrane partial pressure difference of component *i*.  $Q_i$  and  $Q_j$  (mol/m<sup>2</sup> s Pa) correspond to the permeances of components *i* and *j*, respectively.

## 3. Results

### 3.1. Membrane characterization

#### 3.1.1. SEM and visual characterization

Fig. 2 shows the morphology of pristine asymmetric and symmetric PVDF membranes (a and b: membrane surface; c and d: cross-section). Asymmetric membrane is the membrane that has

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