Fabrication of liquid-core fiber-optic structure for large-area \text{CO}_2\text{ sensing using} ionic liquids

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Abstract
Among the ionic liquids (ILs) that are known for their \text{CO}_2\text{} absorption properties, the optical properties of 1-ethyl-3-methylimidazolium acetate ([emim][Ac]) and 1-butyl-3-methylimidazolium acetate ([bmim][Ac]) have been investigated with the aim of assessing their suitability for use in a \text{CO}_2\text{} sensor with a liquid-core fiber-optic structure. Fiber-optic sensors offer multiple benefits, including a large-area sensing capability and immunity to electromagnetic interference. In these two ILs with their different cation alkyl chain extensions, similar levels of change in the refractive index were observed for both [emim][Ac] and [bmim][Ac]; this change was demonstrated to lead to a change in the numerical aperture of a waveguide equipped with an [emim][Ac] core with a maximum value of 0.017787. Waveguide samples were fabricated using both [emim][Ac] and [bmim][Ac] and the output spectra of these samples were compared in terms of their liquid absorption characteristics, which were measured before the samples were packed in a gas-permeable Teflon® AF cladding tube. The liquid-core waveguides demonstrated successful light transmission over a length of 10 cm that agreed with the absorption characteristics of each of the core liquids. The \text{CO}_2\text{} concentration level inside the core liquid was believed to cause the transparency of the waveguide to deteriorate as a result of bubble formation. The growth of the \text{CO}_2\text{} bubbles is irreversible and is assumed to be promoted by a kinetic stimulus and some other factors. The ILs comparison considered in this study will be useful for further development of the liquid-core waveguide-structured \text{CO}_2\text{} sensor. The transmission length of the sensor could be elongated by optimizing both the waveguide and the core IL.

Keywords: Ionic liquid (IL), Refractive index, Liquid-core waveguide, \text{CO}_2\text{ sensor}, Absorption

1. Introduction
Since the importance of reducing \text{CO}_2\text{ emissions has been widely recognized, a variety of \text{CO}_2\text{ sensors has been invented (Uemura and Matsui, 2012) (Tani et al., 2012). However, \text{CO}_2\text{ sensors with fiber-optic structures have not been reported to date. Fiber-optic sensors can realize large-area monitoring, are easy to install and demonstrate immunity to electromagnetic interference (Glisic and Inaudi, 2007). Ionic liquids (ILs) are now drawing considerable attention as potential new materials for \text{CO}_2\text{ collection (Besnard et al., 2012) (Brennecke and Maginn, 2001) (Carvalho et al., 2009) (Makino et al., 2013) (Makino et al., 2016) (Pinkert et al., 2011) (Shin and Lee, 2008). In particular, room temperature ILs are well known for their unique properties (Bowron et al., 2010) (Hallet and Welton, 2011) (Huang et al., 2005) (Makino et al., 2013) (Scovazzo et al., 2004). They are salts with melting points that are at or below room temperature. These salts show nonvolatility, incombustibility and high \text{CO}_2\text{ solubility. Room temperature ILs have numerous unique characteristics and they are good \text{CO}_2\text{ collection materials. However, effective use of ILs in devices has not been explored because of their liquid phase. Therefore, the utility of ILs for use in a fiber-optic \text{CO}_2\text{ sensor has been investigated in our research.

Figure 1 illustrates the principle of the fiber-optic sensor using a room temperature IL that is proposed in this research.
We constructed a waveguide-structured CO\textsubscript{2} sensor of this type using the IL as the core and attempted to detect the ambient CO\textsubscript{2} concentration using the changes in the optical properties of the IL in the waveguide. This waveguide-structured sensor is composed of the IL (i.e., the core) and an amorphous polymer called Teflon\textsuperscript{	extregistered}AF, which forms the cladding. When CO\textsubscript{2} enters from the atmosphere into the IL core, the optical properties of the IL will be changed. The spectrum of the injected light will then also change, and this will be accompanied by change in the properties of the IL. This will allow us to collect the CO\textsubscript{2} and detect the amount of CO\textsubscript{2} absorbed simultaneously. Furthermore, the cladding layer works as a container for the IL to form a solid device structure. The room temperature IL used in this research is 1-ethyl-3-methylimidazolium acetate ([emim][Ac]). [emim][Ac] forms chemical bonds with CO\textsubscript{2} and is known among ILs for its high capacity for CO\textsubscript{2} absorbance (Cadena et al., 2004). The purpose of this research is to characterize the changes in the optical properties of ILs after they absorb CO\textsubscript{2} to assess the possibility of implementation of a simple fiber-optic detection system using a liquid-core structure.

![Diagram of CO\textsubscript{2} sensor](image)

Fig. 1 Structure of the proposed fiber-optic CO\textsubscript{2} sensor using a room temperature IL.

### 2. Experimental Method

#### 2.1 Materials

Two types of room temperature ILs were used in this work for comparison purposes. One is [emim][Ac], which is a typical material that is studied to determine the CO\textsubscript{2} absorption properties of ILs (Cadena et al., 2004) (Makino et al., 2013) (Makino et al., 2016) (Pinkert et al., 2011) (Shi et al., 2012). Because the alkyl chain length in the cation is known to have a major influence on its CO\textsubscript{2} absorption properties (Shin and Lee, 2008), 1-butyl-3-methylimidazolium acetate ([bmim][Ac]) was selected as the second test material. As the chemical structures of these ILs shown in Fig. 2 can confirm, the only difference between them is the length of the alkyl chain in the cation in each case. [emim][Ac] and [bmim][Ac] were purchased from Liquids Technologies and Sigma-Aldrich, respectively. Both products had purity of ≥95% and CO\textsubscript{2} absorption capacity of 30 mol% (Shiflett et al., 2012)(Makino et al., 2017).

The polymer tube used as the waveguide cladding was made from Teflon\textsuperscript{	extregistered}AF that was purchased from Biogeneral, Inc. This tube was selected because of its low refractive index (n = 1.29) and its high permeability to gases, including CO\textsubscript{2}. The inner and outer diameters of the Teflon\textsuperscript{	extregistered}AF tube were 1.0 and 1.6 mm, respectively. A 1.0-mm-diameter step-index polymer optical fiber (SI-POF) that was purchased from Mitsubishi Chemical Corporation (ESKA SK40) was used to plug the ends of the Teflon\textsuperscript{	extregistered}AF tube. The numerical aperture (NA) of the SI-POF was 0.5.

![Chemical structures of ILs](image)

Fig. 2 Chemical structures of [emim][Ac] (left) and [bmim][Ac] (right).
2.2 Sample Preparation and UV-Visible Spectroscopy Measurements

The eight samples that were prepared in our research were in eight different states. The criteria for distinction of these samples and the sample names are explained in Table 1.

| Sample name | Type of IL | CO₂ concentration | Appearance |
|-------------|------------|--------------------|------------|
| Sample 1    | [emim][Ac] | Pure               | In bottle  |
| Sample 2    | [bmim][Ac]| Pure               |            |
| Sample 3    | [emim][Ac] | Saturated          |            |
| Sample 4    | [bmim][Ac]| Saturated          |            |
| Device 1    | [emim][Ac]| Pure               |            |
| Device 2    | [bmim][Ac]| Saturated          |            |
| Device 3    | [emim][Ac]| Pure               |            |
| Device 4    | [bmim][Ac]| Saturated          |            |

The apparatus used for sample preparation is illustrated in Figure 3. First, the IL sample was loaded into a flask. Purified bottle samples (i.e., samples 1 and 3) were made by treating these samples in a vacuum of -0.1 MPa for more than 1 week. Each sample was stirred during the vacuum operation in an 80°C oil bath. This heating and vacuum treatment helped to remove impurities such as vapors and gases from the samples. Measurements of the purified samples were performed after these operations. CO₂-absorbed samples (i.e., samples 2 and 4) were then prepared by loading the samples with CO₂ gas at 0.1 MPa for more than 72 h at room temperature using the same apparatus. The time period settings for each procedure were determined to ensure that each required state was fully achieved. The absorbance characteristics of the bottle samples were measured using a spectrophotometer (V-630, Jasco Corp.) in a quartz glass cell (T-1-UV-10, Tosoh Quartz Corp.; 10 mm x 10 mm).

![Schematic of sample preparation apparatus. The IL container was either connected to the vacuum pump or the CO₂ supply using a three-way adapter. The internal pressure was monitored using the pressure gauge.](image)

2.3 Measurement of Refractive Index

We measured the refractive index before and after absorption of the gas (samples 1-4) using a temperature-controllable Abbe refractometer (DR-M2, Atago Co., Ltd.). The measurement accuracy of this refractometer is ±0.0002. The temperature range and the wavelength were selected to be 15°C to 40°C and 589 nm, respectively. 589 nm is sodium D-ray. The light source of our experiment was halogen lamp, and we used optical filter was for extraction of target
wavelength.

### 2.4 Assembly of the Liquid-Core Waveguide and its Characterization

The prepared bottle samples (samples 1-4) were injected into Teflon® AF tubes and both ends of these tubes were then sealed using the SI-POF. These samples were then named Devices 1-4 (see Table 1). A schematic of a waveguide and the apparatus used to perform the measurements is shown in Fig. 4. In general, the refractive index of the ILs used in our research is higher than that of the Teflon® AF tube \((n = 1.29)\), which then enables light transmission. The IL waveguide was 10 cm long and the SI-POF connected to one end of the waveguide was 20 cm long. The intensity of the emitted light was measured using a spectrophotometer (FP8500, Jasco Corp.). A 10 m length of the SI-POF was connected to the waveguide to adjust the incident intensity.

![Schematic of a waveguide and apparatus](image)

**Fig. 4 Apparatus for waveguide performance measurement.**

### 3. Results and Discussion

#### 3.1 Appearance of the Prepared ILs

Photographs of samples 1–4 are shown in Fig. 5. The initial color of purified [emim][Ac] (Sample 1) was red-yellow and it became lighter in color after CO\(_2\) exposure (Sample 2). When the purified [emim][Ac] and [bmim][Ac] samples (Samples 1 and 3, respectively) were compared, the [bmim][Ac] (Sample 3) was darker in color than [emim][Ac] (Sample 1). In addition, the color of the [bmim][Ac] sample became darker after CO\(_2\) exposure (Sample 4).

![Photographs of samples](image)

**Fig. 5 Appearance of (a) purified [emim][Ac] (Sample 1), (b) [emim][Ac] after exposure to CO\(_2\) (Sample 2), (c) purified [bmim][Ac] (Sample 3) and (d) [bmim][Ac] after exposure to CO\(_2\) (Sample 4).**
3.2 Refractive Indices of the CO$_2$-Absorbed ILs

The results of the refractive index measurements are shown in Fig. 6. Due to the specification of refractometer, measurement uncertainty was assumed to be ±0.0002. The refractive indices of the samples after CO$_2$ exposure were lower than those of the pure samples for the cases of both [emim][Ac] and [bmim][Ac]. All samples showed a negative linear relationship with temperature. Among the pure samples, [emim][Ac] showed a higher refractive index than [bmim][Ac]. This difference is attributed to the higher density of [emim][Ac] because of the lack of an extra alkyl chain length.

Refractive index has positive linear relationship with density (Gladstone and Dale, 1863)(Larsen, 1909), and density of IL is linearly decreased by temperature rise (Shiflett and Yokozeki, 2009). Thus the results were assumed to be agreed with theory. However, sample 4 showed discontinuity at lower temperature. This result indicates the effect of alkyl chain length. The side chain including 4 or more carbons can cause tail aggregation in IL systems (Jose et al., 2006). This aggregation is important for interaction between cations and anions. Sample 4 contains CO$_2$ and alkyl chain consisting of 4 carbon atoms. Therefore, Sample 4 was assumed to have structural instability due to the synergistic effect of gas absorption and chain aggregation. The discontinuity in Fig. 6 indicated that this characteristic was promoted as density increased at lower temperature regime.

Next, we assess the differences in the refractive index effects on the NA of the optical fiber. The NA represents the light-gathering capacity of an optical fiber and it is determined from Equation (1) using the refractive indices of the core $n_1$ and the cladding $n_2$ (Govind P. Agrawal, 2002).

$$\text{NA} = \sqrt{n_1^2 - n_2^2}$$  \hspace{1cm} (1)

Using the refractive indices of the bottle samples measured at 25°C and that of the Teflon$^\text{AF}$ cladding, the NAs of Devices 1–4 were calculated (Table 2). All four devices had shown a higher NA when compared with that of the SI-POF used for sealing. Therefore, coupling losses are expected at the output side of the IL waveguide, but these losses are not expected to be critical for the analysis because of the large core diameter of the system. The maximum difference in the NA that each IL can create by absorbing CO$_2$ gas was represented by $\Delta$NA and the $\Delta$NA values are listed in the right column of Table 2. For both IL types, the NA tended to drop after CO$_2$ absorption because of the drop in the density of the liquids, which leads to a reduction in the output intensity when using the fiber-optic device to detect environmental CO$_2$. Although $\Delta$NA was fairly similar for the two ILs studied in this work, $\Delta$NA was larger in [emim][Ac]. This difference is attributed directly to the $\Delta n_1$ value of each IL. It can be considered that the extension of the alkyl chain in the cation would not function to enhance the CO$_2$ absorption, and would instead work in a slightly negative manner. This
is considered to result from the smaller number of ions contained within a unit volume for [bmim][Ac] when compared with that of [emim][Ac] caused by the bulkiness of the cation.

Table 2 Refractive index values at 25°C and NA, where \( n_2 \) in Equation (1) was 1.29.

| Sample   | \( n_1 \) | \( \Delta n_1 \) | \( n_2 \)  | NA       | \( \Delta NA \) |
|----------|----------|----------------|----------|----------|----------------|
| Device 1 | 1.5001   | 0.0090         |          | 0.765637 | 0.017787       |
| Device 2 | 1.4911   |                | 1.29     | 0.747850 |                |
| Device 3 | 1.4960   | 0.0085         |          | 0.757572 | 0.016926       |
| Device 4 | 1.4875   |                |          | 0.740646 |                |

### 3.3 UV-Visible Spectroscopy of ILs and Liquid Core Waveguides

Figure 7 shows the transmitted spectra of the output light from the IL waveguides. Fig. 7(a) and (b) show that the intensity of the light emission decreased after CO\(_2\) exposure. This trend agrees with the magnitude relationship of the NA that was estimated earlier. After exposure to CO\(_2\) (Device 4), [bmim][Ac] lost most of its light transmission capability. According to the \( \Delta NA \) values shown in Table 2, \( \Delta NA \) was smaller for the [bmim][Ac] device than for the [emim][Ac] device. Therefore, any of the characteristic peaks in the [bmim][Ac] device will no longer be seen in Device 4, which means that the change in NA is clearly not the main reason for the spectrum change shown in Fig. 7(b). One possible reason for the loss of the waveguiding function after absorption of CO\(_2\) is scattering due to the CO\(_2\) bubbles. Because the CO\(_2\)-trapped liquid is under compression from the outer Teflon\(^\circledR\) cladding, the CO\(_2\) gas may no longer be dispersed uniformly inside the liquid and instead will aggregate in a size order that scatters the UV-visible waves.

Figure 8 shows the transmission spectra of Device 1, Device 3, and the SI-POF. The two devices have pure IL for each of their cores. The intensity is normalized with reference to the 765 nm peak for each waveguide. Most of the transmission band from 400 to 600 nm that is seen in SI-POF spectrum was lost from the Device spectra. This loss is attributed to the absorption characteristics of the ILs, which are discussed in detail later in this section. Device 3 showed larger losses in the short wavelength region than Device 1.

Figure 9 shows the absorbance spectra of the bottled samples. The absorption peak at 355 nm of the pure [emim][Ac] (Sample 1) had increased in magnitude after CO\(_2\) absorption (Sample 2). The same trend was also confirmed for the 360 nm peak in [bmim][Ac]. The absorption band of [bmim][Ac] is in a higher wavelength region than that of [emim][Ac]. This is the main cause of the loss of the 400–600 nm band seen in Device 3 in Fig. 8. The 427 nm peak of [bmim][Ac] had also increased in magnitude after CO\(_2\) absorption, but had also shown a red shift (Samples 3 and 4). Although [bmim][Ac] did not show much of a difference in terms of refractive index change when compared with that of [emim][Ac] after CO\(_2\) absorption, it did show a much more pronounced change in its absorption property. Both [emim][Ac] and [bmim][Ac] are believed to form chemical bonds with CO\(_2\) (Shi et al., 2012) (E.J. Maginn, 2004). As indicated by the refractive index measurement results that were discussed earlier, the density of [emim][Ac] is higher than that of [bmim][Ac], which is in good agreement with other reports (Shiflett and Yokozeki, 2009) (Shiflett et al., 2008). The trend seen in Fig. 9 suggests that [bmim][Ac] has a high CO\(_2\) capacity, which is the opposite trend to that described above. Another report has suggested that the free volume created by the presence of the extra alkyl chain in [bmim][Ac] helps it to have a larger CO\(_2\) absorption capacity when compared with that of [emim][Ac] (Shin and Lee, 2008). Because the 427 nm peak that is only present in [bmim][Ac] is not seen in the [emim][Ac] spectra, it is believed that [bmim][Ac] has another bonding site for CO\(_2\) in addition to the site that corresponds to the 360 nm peak.
Fig. 7 Output UV-visible transmittance of liquid core devices fabricated using (a) [emim][Ac] and (b) [bmim][Ac]. before and after CO₂ exposure.

Fig. 8 Transmittance of the waveguide samples. The intensity was normalized with reference to the 765 nm peak of each spectrum.
The changes in the absorbance of the core liquid and the transmittance of the waveguide after CO$_2$ absorption for each type of IL are shown in Fig. 10. These figures are identical to those presented in Fig. 7 and Fig. 9 and each waveform was normalized at its peak intensity. A simple comparison of the waveforms of Devices 1 and 2 indicates that [emim][Ac] shows reduced transmittance in the 500–685 nm region (Fig. 10, left). This difference is not believed to be essential because of the vast difference from the original intensity (Fig. 7(a)). The increase in the 355 nm peak after CO$_2$ absorption (see the waveforms for Samples 1 and 2 in Fig. 9(b) and Fig. 10) is not demonstrated in the waveguide output (from Devices 1 and 2), in which the transmittance around 400 nm is very low in general. This trend is even worse for the [bmim][Ac] devices, in which almost no characteristic peaks are seen from the gas-absorbed device (Device 4). The reason for this result is the CO$_2$ bubbles that are contained inside the core, as mentioned in the discussion earlier. This indicates that the proposed sensor structure may not be applicable to the fully saturated state. Otherwise, the pronounced absorption change observed in the [bmim][Ac] liquids (see Samples 3 and 4 in Fig. 10, right) is a favorable characteristic for CO$_2$ sensing using spectroscopy.

Fig. 9 Absorbance of bottled samples. (a) and (b) show the same waveforms on different scales.

Fig. 10 Absorbance of core IL at 298 K and transmittance of liquid-core waveguides for [emim][Ac] (left) and [bmim][Ac] (right).
4. Conclusions

The optical properties of ILs with different cation alkyl chain extensions were investigated with the aim of estimating their ability to form a liquid-core waveguide-structured CO\textsubscript{2} sensor. Both liquids under test, i.e., [emim][Ac] and [bmim][Ac], were observed to show changes after CO\textsubscript{2} exposure in their refractive index, which changes the NA by a maximum of 0.017787 when using [emim][Ac]. [bmim][Ac] showed a significant change in the absorption spectrum after CO\textsubscript{2} absorption; this is represented by the increase in magnitude and red shift of the original 427 nm peak. Devices using gas-permeable Teflon\textsuperscript{®}AF cladding with a low refractive index demonstrated successful transmission of light through their liquid cores. The transmission characteristic showed an agreement with the absorption of each of the core liquids, in which short wavelength region is lost. Although the waveguides showed poor transmittance after the liquids became fully saturated, the transmittance is assumed to be improvable at lower CO\textsubscript{2} concentrations when the effect of the CO\textsubscript{2} that cause scattering in the UV-visible region is reduced. The comparison performed for the two ILs considered in this study will be useful for further development of the liquid-core waveguide-structured CO\textsubscript{2} sensor. The transmission length of 10 cm that was demonstrated in this work could be elongated by optimizing both the waveguide and the core IL.

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