




Article

CO₂ Absorption Mechanism by the Deep Eutectic Solvents Formed by Monoethanolamine-Based Protic Ionic Liquid and Ethylene Glycol

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Abstract: Deep eutectic solvents (DESs) have been widely used to capture CO₂ in recent years. Understanding CO₂ mechanisms by DESs is crucial to the design of efficient DESs for carbon capture. In this work, we studied the CO₂ absorption mechanism by DESs based on ethylene glycol (EG) and protic ionic liquid ([MEA][Im]), formed by monoethanolamine (MEA) with imidazole (Im). The interactions between CO₂ and DESs [MEA][Im]-EG (1:3) are investigated thoroughly by applying ¹H and ¹³C nuclear magnetic resonance (NMR), 2-D NMR, and Fourier-transform infrared (FTIR) techniques. Surprisingly, the results indicate that CO₂ not only binds to the amine group of MEA but also reacts with the deprotonated EG, yielding carbamate and carbonate species, respectively. The reaction mechanism between CO₂ and DESs is proposed, which includes two pathways. One pathway is the deprotonation of the [MEA]⁺ cation by the [Im]⁻ anion, resulting in the formation of neutral molecule MEA, which then reacts with CO₂ to form a carbamate species. In the other pathway, EG is deprotonated by the [Im]⁻, and then the deprotonated EG, HO-CH₂-CH₂-O⁻, binds with CO₂ to form a carbonate species. The absorption mechanism found by this work is different from those of other DESs formed by protic ionic liquids and EG, and we believe the new insights into the interactions between CO₂ and DESs will be beneficial to the design and applications of DESs for carbon capture in the future.

Keywords: capture; CO₂; deep eutectic solvents; ionic liquids; insights



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1. Introduction

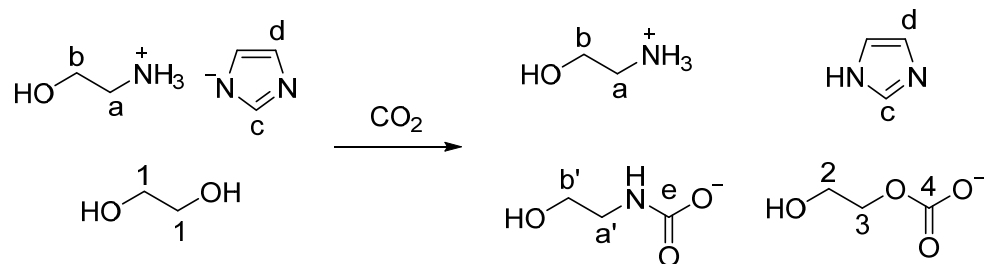
Recently, deep eutectic solvents have attracted much attention because of their unique properties, including a negligible vapor pressure, non-flammability, ease of synthesis, and tailorable polarity, which make them a promising alternative to traditional organic solvents [1,2]. Most of DESs are obtained by mixing appropriate hydrogen bond donors (HBDs) and hydrogen bond acceptors (HBAs). The applications of DESs in many fields have been investigated so far, such as organic synthesis, extractions processes, catalytic reactions and electrochemistry [3–6]. Moreover, DESs have been widely studied as absorbents for CO₂ capture [7–11]. Among them, functional DESs which could chemically absorb CO₂ have received much attention because of their high capacities. The chemical absorption of CO₂ by DESs depends on the chemical reactions between CO₂ and functional groups of DESs [12–16]. Therefore, understanding the reaction mechanisms between CO₂ and functional groups of DESs is important to the design of efficient DESs for CO₂ capture.

Trivedi et al. studied the CO₂ absorption mechanism by DESs employing ethylenediamine (EDA) as an HBD [17] and the results indicated that a carbamate species was formed between the amine group of EDA and CO₂. In our previous work, we found that CO₂ was attached to ethylene glycol (EG) with the formation of a carbonate species when CO₂

was captured by DESs consisting of azolide-based ILs and EG [18]. Zhang et al. studied the interactions between CO₂ and ternary DESs consisting of ionic liquid (IL) 1-butyl-3-methylimidazolium chloride ([Bmim][Cl]), imidazole (Im), and super base 1,5-diazabicyclo [4.3.0] non-5-ene (DBN) [19]. The results indicated that CO₂ was bonded to the C₂-position of the imidazolium ring of the cation [Bmim]⁺. Zeng et al. investigated the reaction pathway between CO₂ and DESs composed of protic IL 1,8-diazabicyclo-[0,4,5]undec-7-ene imidazolite ([DBUH][Im]) and EG [20] and it was found that CO₂ reacted both with the anion [Im]⁻ and EG. Another reaction mechanism between CO₂ and DESs consisting of ionic liquid 1-ethyl-3-methylimidazolium 2-cyanopyrrolide ([Emim][2-CNpyr]) and EG was reported by Gurkan and co-authors [21], and the results revealed that CO₂ was attached to the anion [2-CNpyr]⁻, the imidazolium cation and EG, by forming carbamate, carboxylate and carbonate species, respectively.

In a recent paper, Mukesh et al. reported the CO₂ capture by DESs based on EG and protic ILs [22]. The protic ILs they used were obtained by mixing polyamines or monoethanolamine (MEA) with imidazole (Im). These DESs showed a high absorption capacity for CO₂. The authors also investigated the reactions between CO₂ and the DESs used in their work. On the basis of the NMR and FTIR results, they believed that CO₂ was bonded to the amine group of polyamines or MEA, and EG or anion [Im]⁻ did not react with CO₂.

However, in our study, we found that CO₂ not only binds with the amine group but also reacts with EG in DESs (Scheme 1), suggesting the CO₂ absorption mechanism reported by Mukesh et al. is inaccurate [22]. The absorption mechanism found by this work is different from those of other DESs formed by protic ionic liquids and EG. For example, CO₂ reacted both with the anion [Im]⁻ and EG, but did not react with the cation [DBUH]⁺, when CO₂ was captured by [DBUH][Im]-EG DESs. The CO₂ absorption mechanism by [DBUH][MLU] (MLU: Methyl urea)-EG presented a similar phenomenon, i.e., CO₂ reacted with the anion [MLU]⁻ and EG rather than reacting with the cation [DBUH]⁺ [16]. However, the mechanism found by our work indicates that CO₂ binds to both the amino group on the cation [MEA]⁺ and EG, but CO₂ is not bonded to the anion [Im]⁻ in [MEA][Im]-EG (1:3) DESs. We believe that the findings of our work provide new insights about the interactions between CO₂ and DESs, which will be useful to design new, efficient DESs for carbon capture in the future.



Scheme 1. The reaction between CO₂ and [MEA][Im]-EG (1:3).

2. Results and Discussion

At first, we prepared the DESs [MEA][Im]-EG (1:3) following the procedures reported by Mukesh and co-authors [22]. The formation of [MEA][Im] was confirmed by the ¹H NMR results (Figure S1). As shown in Figure S1, the -NH₃⁺ peak can be observed at 5.54 ppm, suggesting the IL [MEA][Im] was obtained. The CO₂ capacity by the [MEA][Im]-EG (1:3) is 8.94 wt% (Figure S2), which is similar to that reported by Mukesh's paper, and the diagram of the CO₂ absorption apparatus is shown in Figure S3. The ¹H (Figure 1a) and ¹³C (Figure 1b) NMR spectra of [MEA][Im]-EG(1:3) before and after CO₂ capture were carefully studied. As shown in Figure 1a, the hydrogen peaks of -CH₂-group (H-a' and H-b') of MEA-based carbamate can be found after CO₂ absorption, [23] suggesting the CO₂ is bonded to the amine group. However, two other new peaks at 3.05 (H-2) and 3.35 (H-3) ppm can also be found in Figure 1a. As seen in Figure 1b,

the carbon signals of $-\text{CH}_2-$ group related to the MEA-based carbamate can be found at 42.7 (C-a') and 60.6 (C-b') ppm, and the carbonyl signal of MEA-based carbamate appears at 163.3 (C-e) ppm. However, there are three other new peaks at 60.7 (C-2), 65.9 (C-3), and 158.2 (C-4) ppm.

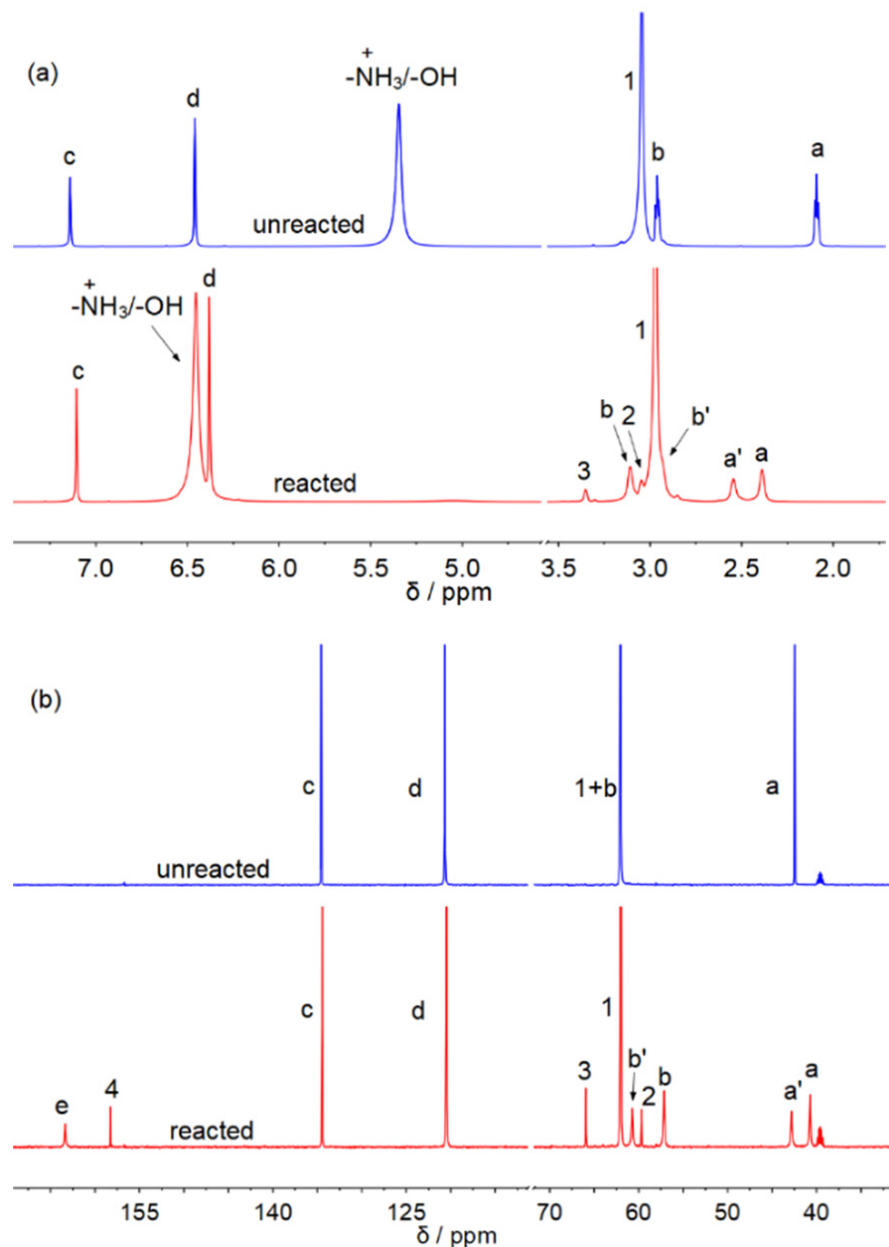


Figure 1. ^1H (a) and ^{13}C (b) NMR spectra of [MEA][Im]-EG (1:3) before and after CO_2 absorption.

In order to assign the new peaks in the ^1H and ^{13}C NMR spectra after CO_2 capture, the ^1H - ^{13}C HMBC NMR spectra of [MEA][Im]-EG (1:3) after CO_2 capture were investigated (Figure 2). As shown in Figure 2, C-e correlates with the H-a', suggesting the formation of the MEA-based carbamate. Moreover, the carbonyl carbon C-4 correlates with the H-3, and the correlation between C-4 and the hydrogens on the imidazole ring also could not be seen. The ^1H - ^{13}C HMBC suggested that the carbon C-4 should be the carbonyl carbon of the carbonate formed by CO_2 and EG. The carbons at 60.7 (C-2) and 65.9 (C-3) ppm are the $-\text{CH}_2-$ carbons of $\text{HO}-\text{CH}_2-\text{CH}_2-\text{OCOO}^-$ [20,21]. Similar carbon peaks could also be found in the Mukesh and co-authors' paper [22]. As shown in Figure S4 of Mukesh and co-authors' paper, the $-\text{CH}_2-$ carbons of $\text{HO}-\text{CH}_2-\text{CH}_2-\text{OCOO}^-$ could be clearly observed

in the ^{13}C NMR spectra of $[\text{TEPA}]_2[\text{Im}]:\text{EG}-4$ after CO_2 absorption, which disappeared after CO_2 desorption. In Figure S10a of their paper, the $-\text{CH}_2-$ carbons of $\text{HO}-\text{CH}_2-\text{CH}_2-\text{OCOO}^-$ could also be found in the ^{13}C NMR spectra of TPEA-EG solution after CO_2 capture, and the carbonyl carbon of EG-based carbonate appeared at 159.67 ppm. In the ^{13}C NMR spectra of $[\text{TEPA}]_2[\text{Im}]:\text{EG}-4$ after CO_2 absorption (Figure S10c of their paper), the carbonyl carbon of EG-based carbonate appeared at 159.79 ppm. Therefore, in our opinion, the ^{13}C NMR results of $[\text{TEPA}]_2[\text{Im}]:\text{EG}-4$ after carbon capture indicated that CO_2 reacted with EG by forming carbonate species. Interestingly, as seen in Figure 1a, the peaks H-c, H-d, and H-1 shift slightly to upfield, and the H-a peak shift to downfield after carbon capture. These shifts may be due to the change in the hydrogen bonds network in the DESs because of the formations of carbamate and carbonate species after capture.

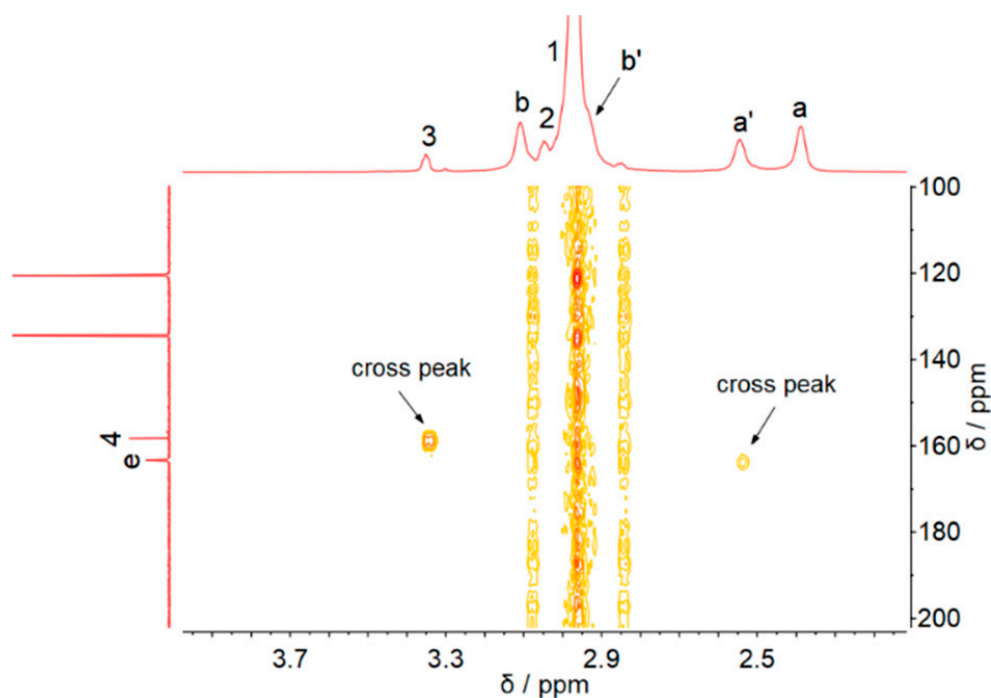


Figure 2. The $^1\text{H}-^{13}\text{C}$ HMBC spectra of $[\text{MEA}][\text{Im}]-\text{EG}$ (1:3) after CO_2 capture.

Although Mukesh and co-authors investigated the HMBC spectra of $[\text{TEPA}]_2[\text{Im}]:\text{EG}-4$ after CO_2 capture (Figure S11 of their paper) and they believed that CO_2 did not react with EG on the basis of the HMBC results, we think their HMBC results are not accurate because the ^{13}C peaks in the HMBC spectra are not consistent with those in the ^{13}C NMR spectra of Figure S10c of their paper. In the ^{13}C NMR spectra of Figure S10c, the carbamate signals were in the range of 164 to 165 ppm and the strong carbonate signal was at 159.79 ppm. In their HMBC spectra, the carbamate signals can be found near 165 ppm, but the peak near 159 ppm could not be observed, so the HMBC spectra they obtained may be incorrect.

Our findings were further confirmed by the FTIR spectra. The FTIR spectra of $[\text{MEA}][\text{Im}]-\text{EG}$ (1:3) before and after CO_2 capture were shown in Figure 3. The peak at 1635 cm^{-1} is associated with the combined $\text{C}=\text{O}$ stretching band of carbonate and carbamate species, and the peak at 1573 cm^{-1} corresponds to COO^- asymmetric stretching of carbamate $-\text{NHCOO}^-$. The new peak at 1296 cm^{-1} is ascribed to the stretching band of $\text{RO}-\text{COO}^-$ [21]. The FTIR results suggested again that both carbonate and carbamate species are formed after CO_2 capture.

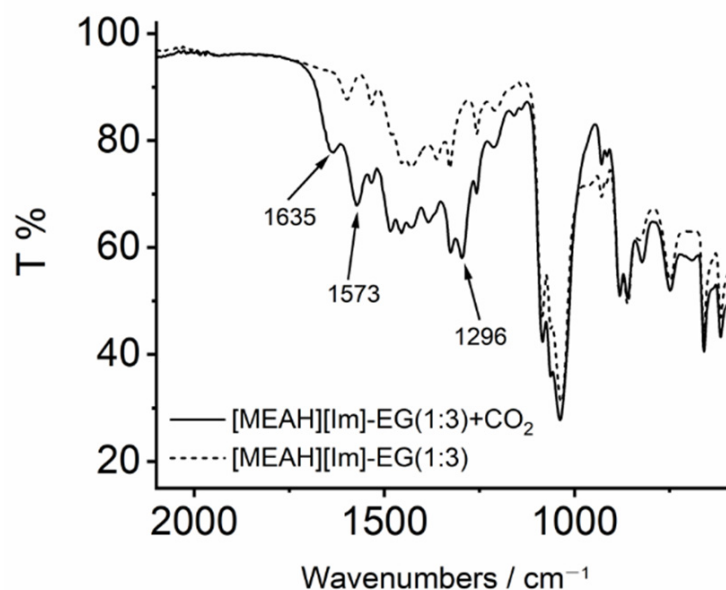
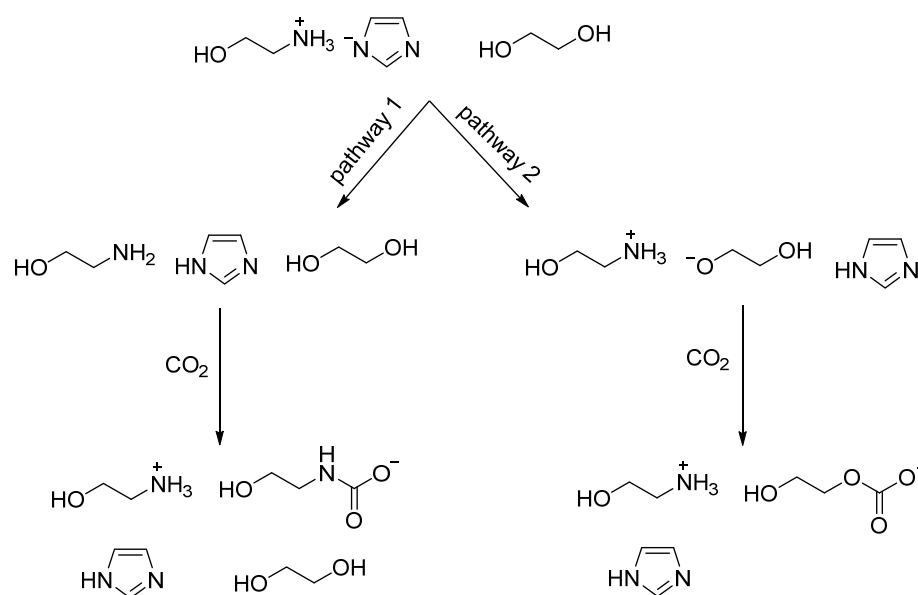


Figure 3. The FTIR spectra of [MEA][Im]-EG (1:3) before and after CO₂ capture.

Based on the above discussion, the possible absorption mechanism by [MEA][Im]-EG (1:3) is presented in Scheme 2. Pathway 1 is the deprotonation of the [MEA]⁺ cation by [Im][−] anion, resulting in the formation of the neutral molecule MEA, which then reacts with CO₂ to form a carbamate species. Pathway 2 is that EG is deprotonated by [Im][−], forming the HO-CH₂-CH₂-O[−] anion, which then binds with CO₂ to form a carbonate species. The absorption mechanism by [MEA][Im]-EG (1:3) is different from that of [DBU][Im]-EG, which may be due to that DBU is a much stronger base than MEA. In other words, [DBU]⁺ is a much weaker acid than [MEA]⁺, rendering the deprotonation of [DBU]⁺ by [Im][−] extremely difficult, so CO₂ is not found to be bonded to DBU in the [DBU][Im]-EG DESs after CO₂ capture [20]. In our previous work, the steric hindrance of functional groups is also found to be a factor that affects the CO₂ absorption mechanism. [24,25]. Therefore, we suggest that the factors, which may include the basicity, acidity, and the steric hindrance of functional groups of DESs, should be considered to modulate the CO₂ absorption mechanism by DESs.



Scheme 2. The proposed reaction mechanism between CO₂ and [MEA][Im]-EG (1:3).

3. Materials and Methods

3.1. Materials and Characterizations

Imidazole (Im, 98%), ethylene glycol (EG, 99.5%), and monoethanolamine (MEA, 99%) were obtained from J&K Scientific Ltd. (Beijing, China). CO₂ (≥99.99%) was supplied by Beijing ZG Special Gases Sci. and Tech. Co. Ltd. The ¹H NMR (600 MHz) and ¹³C NMR (151 MHz) spectra were recorded on a Bruker spectrometer using DMSO-d₆ as an external solvent. FTIR spectra were recorded on a PerkinElmer Frontier spectrometer with an attenuated total reflection (ATR) accessory. The water content of the DESs was determined using Karl Fisher titration by V20 Volumetric KF titrator (Mettler Toledo, Zaventem, Belgium).

3.2. Synthesis of [MEA][Im] and DESs

The procedure to prepare [MEA][Im] followed the methods reported by Mukesh et al. MEA and Im were mixed in a flask at a 1:1 molar ratio, and then the mixtures were stirred at 80 °C for 2 h. The obtained [MEA][Im] was stored in a glass vial.

The DESs [MEA][Im]-EG (1:3) was obtained by mixing [MEA][Im] and EG in a glass vial at a 1:3 molar ratio at room temperature. The water content of [MEA][Im]-EG (1:3) is 0.23 wt%.

3.3. CO₂ Absorption

[MEA][Im]-EG (1:3) (2.0 g) was added into a glass tube with a diameter of 10 mm. The tube was equipped with a rubber lid, which contained two needles. The tube was partially immersed in a water bath (25 ± 0.3 °C), and CO₂ was bubbled into the solvent through one needle at a flow rate of ~50 mL/min, and the other needle was for a CO₂ outlet. The weight difference of the tube before and after CO₂ absorption was determined by an electronic balance (±0.1 mg).

4. Conclusions

The reaction mechanism between CO₂ and DES [MEA][Im]-EG (1:3) is studied carefully by using NMR and FTIR spectroscopy. The CO₂ capacity of [MEA][Im]-EG (1:3) is 8.94 wt% at 25 °C and 1.0 atm. The results indicate that CO₂ not only binds with the amino group of MEA but also reacts with the deprotonated EG in the solvent. There are two possible reaction pathways that form carbamate and carbonate species. One is the deprotonation of the [MEA]⁺ cation by [Im][−] anion, forming the neutral molecule MEA, which then reacts with CO₂ to form a carbamate species. Another one is the reaction between EG and [Im][−], forming the HO-CH₂-CH₂-O[−] anion, which then binds with CO₂ to form a carbonate species. We believe the new insights about the interactions between CO₂ and DESs will bring benefits to the design and applications of DESs for carbon capture in the future.

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