

# Arginine-Based Ionic Liquid in a Water–DMSO Binary Mixture for Highly Efficient CO<sub>2</sub> Capture from Open Air

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**ABSTRACT:** Carbon dioxide (CO<sub>2</sub>) absorption in the open air is essential for reducing the atmospheric CO<sub>2</sub> concentration and averting global climate change. The high CO<sub>2</sub> absorption capacity of amino acid-based ionic liquids (AAILs) makes them a more plausible carbon capture system than conventional amine solutions. We synthesized an ionic liquid derived from arginine, triethylmethylammonium arginate ([N<sub>2221</sub>][Arg]), and utilized it to capture pure CO<sub>2</sub> and CO<sub>2</sub> from the open air. Although AAILs have some notable advantages for CO<sub>2</sub> capture, like low toxicity, biocompatibility, and superior stability, they also have important drawbacks, such as high viscosity and low mass transfer. Now, in order to resolve these problems, we blended [N<sub>2221</sub>][Arg] with a binary mixture of water and dimethyl sulfoxide (DMSO) with a variable volume ratio. On increasing the volume of DMSO in water–DMSO binary mixtures, the CO<sub>2</sub> uptake gradually decreased, reaching a minimum in a pure DMSO medium and the maximum in an aqueous solution, which was obtained from the vapor–liquid equilibrium (VLE) experiment. We applied this [N<sub>2221</sub>][Arg] ionic liquid in the aqueous medium to absorb CO<sub>2</sub> from direct air. The open-air CO<sub>2</sub> uptake value was approximately 1.01 mol/mol in the aqueous solution of arginine-based ionic liquid; to the best of our knowledge, this is the first report of such an ionic liquid system having high CO<sub>2</sub> absorption efficiency under ambient air. We believe that our system will be advantageous for removing CO<sub>2</sub> from highly polluted air.

## 1. INTRODUCTION

One of the major issues of the 21st century is global warming and its consequences for the environment. Carbon dioxide (CO<sub>2</sub>) is the primary greenhouse gas in the atmosphere, and its emission from the combustion of fossil fuels, the burning of forests, and the refining of petroleum is the main culprit of global climate change.<sup>1–4</sup> Since the beginning of the Industrial Revolution, the average atmospheric concentration of carbon dioxide has increased by 50%, from 280 ppm in the middle of the 18th century to its current level of 421 ppm.<sup>5,6</sup> The conventional method for resolving the CO<sub>2</sub> crisis is the chemical absorption of CO<sub>2</sub> by aqueous amine solutions; however, this method has significant inherent drawbacks, such as energy-extensive consumption, the loss of solvent, and corrosion.<sup>7–11</sup> Because of this, it is essential to make use of novel solvents that are capable of absorbing CO<sub>2</sub> and overcoming the disadvantages associated with amine solutions.

Ionic liquids (ILs), also known as “designer solvents,” are synthesized by selecting sterically mismatched ions to oppose crystallization.<sup>12–20</sup> They display a variety of exceptional properties, including great solvating power, high ionic conductivity, substantial thermal stability, inflammability, and low volatility.<sup>21,22</sup> These properties, along with the tuning flexibility over different ion pairs, make them extremely useful in various fields of research like organic synthesis,<sup>23</sup> electrochemistry,<sup>24,25</sup> drug delivery,<sup>26</sup> pharmaceuticals,<sup>27</sup> catalysis,<sup>28–30</sup> etc. In addition to this, ILs have significant applications in the field of carbon capture processes.<sup>31–33</sup> After the groundbreaking discovery about the dissolution of CO<sub>2</sub> in 1-butyl-3-methylimidazolium hexafluorophosphate

([BMIM][PF<sub>6</sub>]),<sup>34</sup> scientists have worked intensively to better understand and maximize the physical absorption of CO<sub>2</sub> in ILs. There are several reports of ionic liquids being able to capture CO<sub>2</sub><sup>35–37</sup> although the uptake values of CO<sub>2</sub> are not very high. Khanna and co-workers have reported that the absorption efficiency of CO<sub>2</sub> in amine-based ionic liquids improved 3-fold compared to nonfunctionalized ILs.<sup>38</sup> Now, to improve the solubility of CO<sub>2</sub>, researchers are developing a new class of ionic liquids known as amino acid-based ionic liquids (AAILs), with high carbon capture efficiency.<sup>39–44</sup> Along with their high CO<sub>2</sub> absorption efficiency, AAILs have a number of additional benefits, including high thermal stability, low toxicity, and biodegradability.<sup>38,45</sup> However, the high viscosity of AAILs makes it challenging to use them in CO<sub>2</sub> absorption studies with slow kinetics and low mass transfer.<sup>46</sup>

Open-air CO<sub>2</sub> capture<sup>47,48</sup> is crucial to mitigate the continuous increment of the CO<sub>2</sub> level in the global atmosphere. There are numerous studies of CO<sub>2</sub> absorption by ionic liquids using experimental setups in a bound condition; however, there is only one report of CO<sub>2</sub> capture from open air with CO<sub>2</sub> uptake value 0.5 mol/mol.<sup>49</sup> Now, the improvement of the direct air carbon capture efficiency utilizing ILs is an emerging field of research.

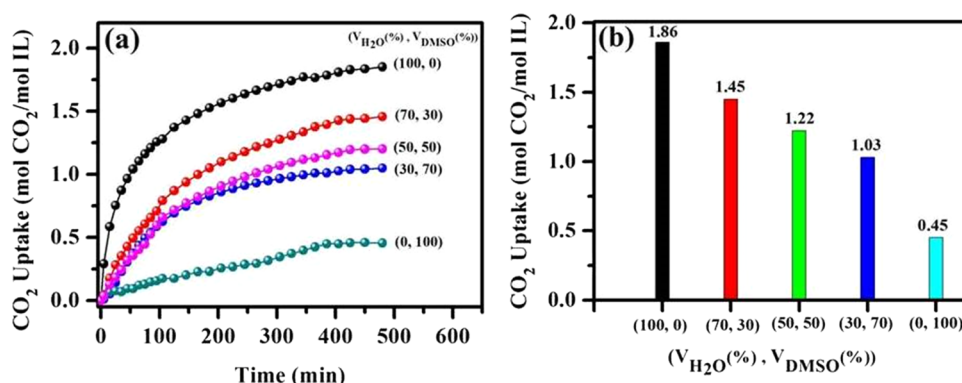
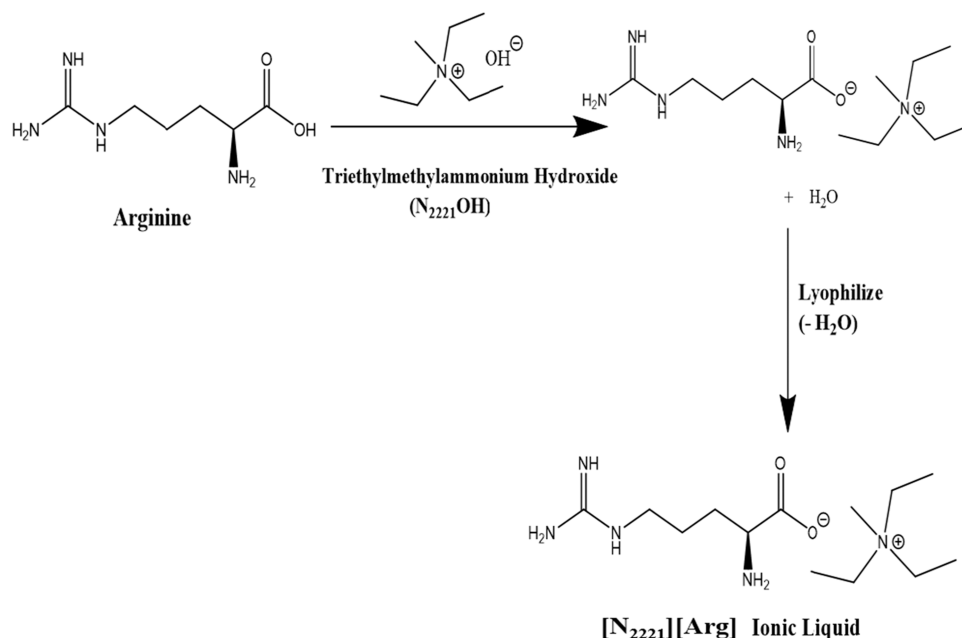
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Scheme 1. Schematic Representation of the  $[N_{2221}][Arg]$  Ionic Liquid Synthesis

**Figure 1.** (a) CO<sub>2</sub> uptake by  $[N_{2221}][Arg]$  ionic liquid at different volume percentages of water and DMSO in a DMSO–water binary mixture with the variation of time. (b) Saturated CO<sub>2</sub> uptake by  $[N_{2221}][Arg]$  ionic liquid at different volume percentages of water and DMSO in a DMSO–water binary mixture (error in experimental results within  $\pm 7\%$ ).

In this paper, we have successfully synthesized an arginine-based ionic liquid and characterized it by NMR spectroscopy. To overcome the limitations caused by the high viscosity of these AAILs, we blended it with a dimethyl sulfoxide (DMSO)–water binary mixture and conducted CO<sub>2</sub> absorption studies. Also, we wanted to determine the effect of protic and aprotic solvents; therefore, we chose water and DMSO. In this study, we captured CO<sub>2</sub> in its pure form and also from ambient air and characterized it via <sup>13</sup>C NMR spectroscopy.

## 2. EXPERIMENTAL SECTION

**2.1. Chemicals.** The single amino acid L-arginine was obtained from TCI Chemicals (India). Triethylmethylammonium hydroxide solution (20% in water (w/w)) was purchased from Sigma-Aldrich and used as received. Spectroscopic-grade dimethyl sulfoxide (DMSO) was purchased from Spectrochem Pvt. Ltd., and double-distilled Milli Q water was used to prepare a DMSO–water binary mixture.

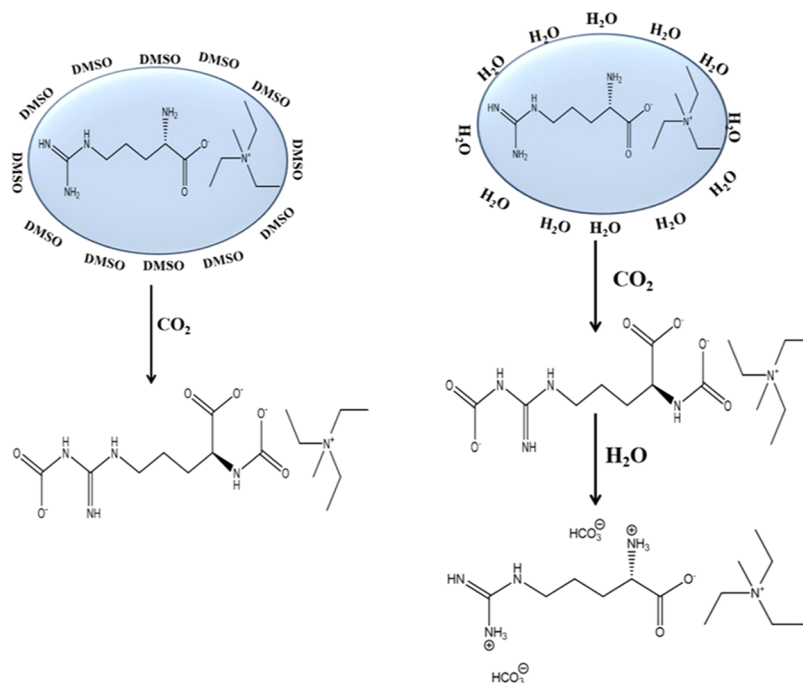
**2.2. Synthesis of the Arginine-Based Ionic Liquid.** We synthesized the arginine-based ionic liquid triethylmethylammonium arginate ( $[N_{2221}][Arg]$ ) via the neutralization reaction between triethylmethylammonium hydroxide ( $[N_{2221}][OH]$ ) and the single

amino acid L-arginine at a molar ratio of 1:1. The equimolar mixture of the aqueous solution of  $[N_{2221}][OH]$  and L-arginine was stirred for 24 h at room temperature, and then, the solution was lyophilized by liquid N<sub>2</sub>. A white, highly viscous, semisolid product was obtained, and the ionic liquid was characterized by <sup>1</sup>H and <sup>13</sup>C NMR spectroscopies (Figures S1 and S2). The ionic liquid was kept in a vacuum desiccator to remove a trace amount of water. The schematic representation of  $[N_{2221}][Arg]$  ionic liquid synthesis is shown in Scheme 1.

**2.3. Experimental Setup and Procedure.** Detailed descriptions of the vapor–liquid equilibrium (VLE) setup and the procedure of the CO<sub>2</sub> absorption experiment are included in the Supporting Information.

## 3. RESULTS AND DISCUSSION

**3.1. Pure CO<sub>2</sub> Absorption Study.** The  $[N_{2221}][Arg]$  ionic liquid is a highly viscous and semisolid product; therefore, we cannot use it directly in the CO<sub>2</sub> absorption experiment. It has been reported that the CO<sub>2</sub> uptakes of pure amino acid-based ionic liquids (AAILs) are considerably low due to their high viscosity, slow kinetics, and low mass transfer.<sup>46</sup> Now, in order to resolve this issue, we blended the  $[N_{2221}][Arg]$  ionic liquid

Scheme 2. Mechanism of CO<sub>2</sub> Absorption by [N<sub>2221</sub>][Arg] Ionic Liquid in DMSO and Aqueous Medium

with DMSO–water binary mixtures having different percentages of volume ratios. The pure CO<sub>2</sub> capture by the DMSO–water binary mixture blended ionic liquid (IL) was measured using the VLE setup at 298 K. In the aqueous solution of IL, the CO<sub>2</sub> uptake ( $\alpha = 1.86$  mol/mol) becomes maximum, whereas in the DMSO medium, the CO<sub>2</sub> uptake ( $\alpha = 0.45$  mol/mol) becomes minimum. With increasing water volume percentage of the DMSO–water binary mixture, the CO<sub>2</sub> uptake of IL gradually increases, as shown in Figure 1a,b. We calculated the numbers of moles of CO<sub>2</sub> absorbed by water and DMSO using time-dependent VLE studies (Figure S5), subtracted them from the number of moles of CO<sub>2</sub> absorbed by IL solutions at different times, and finally determined the uptake  $\alpha$  (mol<sub>CO<sub>2</sub></sub>/mol<sub>IL</sub>) values.

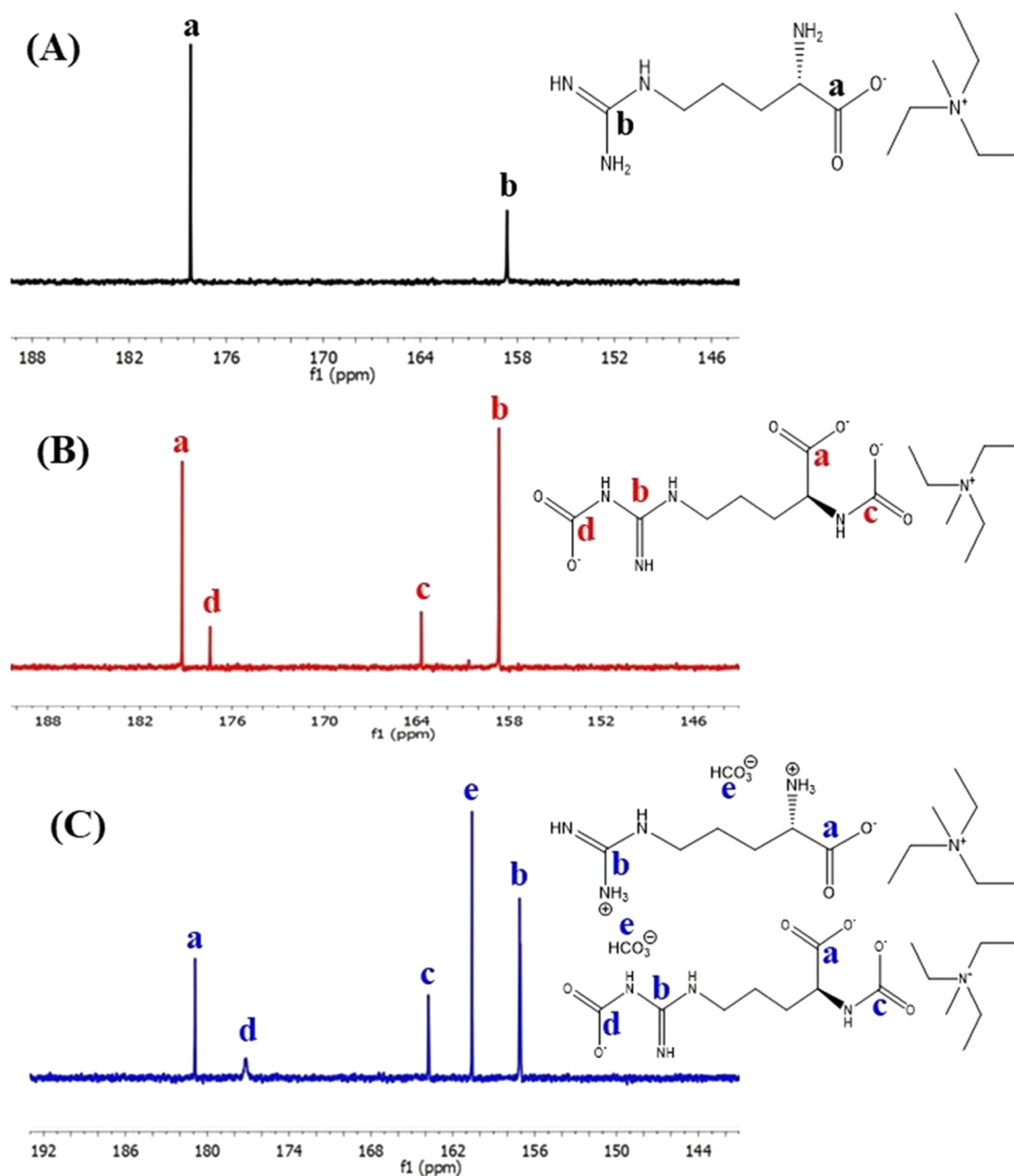
The mechanism of CO<sub>2</sub> absorption by arginine-based ionic liquids in various solvent media is key to understanding these findings. Here, CO<sub>2</sub> is absorbed through a chemisorption process. In the DMSO medium, the CO<sub>2</sub> molecules react with the amine groups of the arginine moiety of the [N<sub>2221</sub>][Arg] ionic liquid and form the carbamate groups. However, in the aqueous medium, the reaction mechanism is very different; at first, carbamates are formed after a reaction between CO<sub>2</sub> and amine groups, and then, those carbamates react with water and form ammonium and bicarbonate ions (Scheme 2). Those ammonium groups again react with CO<sub>2</sub> to form bicarbonate ions and are saturated in the solution. In the aqueous medium, water molecules participate in the CO<sub>2</sub> absorption process by forming bicarbonate ions, whereas the aprotic solvent DMSO is inert. Consequently, the CO<sub>2</sub> uptake of IL in the aqueous solution is greater than that in the DMSO medium, and this CO<sub>2</sub> uptake value gradually decreases with increasing volume percentage of DMSO in the DMSO–water binary mixture.

The formation of carbamates and bicarbonate ions is proven by <sup>13</sup>C NMR spectra of the [N<sub>2221</sub>][Arg] ionic liquid before and after CO<sub>2</sub> absorption. As the chemical shifts of C=O groups are our primary concern, we have limited our focus in Figure 2 to the region between 140 and 190 ppm. Before CO<sub>2</sub>

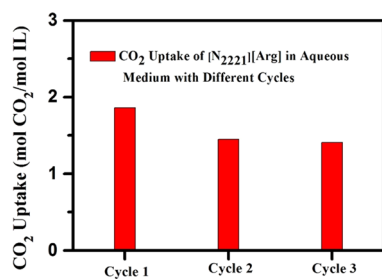
absorption, we obtained two peaks within the 140–195 ppm region: the first peak at 158.7 ppm for the guanidinium group, and the second at 179.3 ppm corresponding to the carboxylate group of the arginate anion (Figure 2A). In the DMSO medium, two new peaks appear at 177.4 and 164 ppm after CO<sub>2</sub> absorption, indicating carbamate formation, as shown in Figure 2B. In the aqueous medium, along with two carbamate peaks, one new peak was generated at 160.6 ppm after CO<sub>2</sub> capture, indicating the formation of bicarbonate ions (Figure 2C). Due to different chemical environments, the guanidinium and carboxylate peaks are slightly shifted from their positions in the aqueous and DMSO mediums. From the Fourier transform infrared (FTIR) spectra (Figure S3) of the [N<sub>2221</sub>][Arg] ionic liquid in the aqueous medium, we can conclude that because of the formation of HCO<sub>3</sub><sup>-</sup> after reaction with CO<sub>2</sub>, we obtained a new peak at 1360 cm<sup>-1</sup> due to the symmetric stretching of CO<sub>2</sub>.

**3.2. Regeneration and Recycle of [N<sub>2221</sub>][Arg] IL.** To regenerate the sample, we heated the solution of CO<sub>2</sub> absorbed ionic liquid in an aqueous medium at 70 °C for 4 h. Here, it is essential to mention that the solution of ionic liquid is heated for 30 min and then cooled down at room temperature for 15 min under a closed condition; then, again, it is heated for 30 min, and this process is repeated for 4 h to avoid any degradation of the ionic liquid. The loss of water due to vaporization is compensated for by adding it externally. The status of degradation is checked by an NMR study. Subsequently, we performed the VLE study to reabsorb the sample. The saturated CO<sub>2</sub> uptake values up to the third cycle are given in Figure 3. Here, the uptake value of the regenerated sample is slightly less due to the loss of the ionic liquid during the transfer of the sample from the equilibrium cell to our desired container.

**3.3. Open-Air CO<sub>2</sub> Absorption Study.** From the pure CO<sub>2</sub> absorption study via VLE measurements, we observed that the CO<sub>2</sub> capture efficiency of the [N<sub>2221</sub>][Arg] ionic liquid is at a maximum in the aqueous medium; therefore, we



**Figure 2.**  $^{13}\text{C}$  NMR spectra of (A)  $[\text{N}_{2221}][\text{Arg}]$  ionic liquid in DMSO, (B) DMSO solution of  $[\text{N}_{2221}][\text{Arg}]$  ionic liquid in the presence of  $\text{CO}_2$ , and (C) aqueous solution of  $[\text{N}_{2221}][\text{Arg}]$  ionic liquid in the presence of  $\text{CO}_2$  (500 MHz instrument).



**Figure 3.**  $\text{CO}_2$  uptake by the  $[\text{N}_{2221}][\text{Arg}]$  ionic liquid in an aqueous medium up to the 3rd cycle of regeneration (experimental error  $\pm 13\%$ ).

performed the open-air  $\text{CO}_2$  absorption of the  $[\text{N}_{2221}][\text{Arg}]$  ionic liquid in an aqueous medium. The as-synthesized 7.5

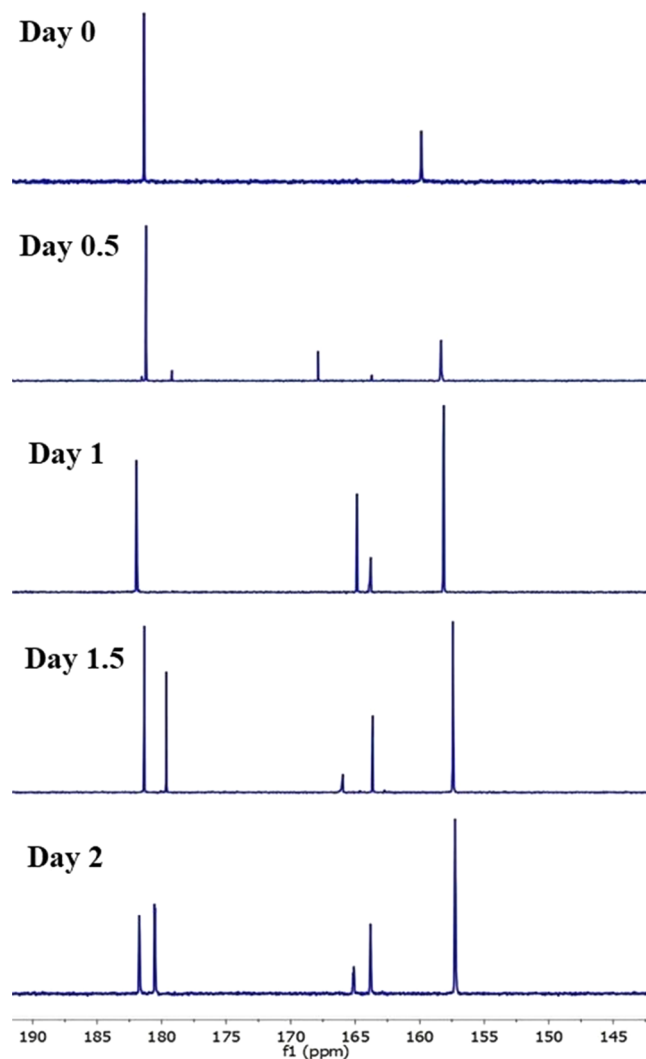
mmol of  $[\text{N}_{2221}][\text{Arg}]$  ionic liquid was first dissolved in 10 mL of Milli Q water, and then, the total solution was placed in a clean Petri dish and kept in the open air in our laboratory. After that, every 12 h, we took 300  $\mu\text{L}$  of this solution over 3 days and performed  $^{13}\text{C}$  NMR measurements of these solutions in a 50%  $\text{D}_2\text{O}$  medium.

During the open-air  $\text{CO}_2$  absorption study, we compensated for the water loss due to evaporation by externally adding it at each 3 h interval. For this, we measured the initial and final (after every 3 h) volumes of the solution, and the loss of volume was compensated for by adding water. The volumes of water added at different times are listed in Table S1. The initial volume of the solution was 10 mL.

Before the  $\text{CO}_2$  absorption, only two peaks observed between 140 and 190 ppm correspond to the guanidinium



(157 ppm) and carboxylate (181.5 ppm) groups. After 12 h (day 0.5), three new peaks appeared around 163, 165, and 180 ppm, which indicates the formation of carbamates and bicarbonate species as depicted in Figure 4. With increasing



**Figure 4.**  $^{13}\text{C}$  MNR spectra of  $[\text{N}_{2221}][\text{Arg}]$  ionic liquid in an aqueous medium at different time intervals of  $\text{CO}_2$  exposure to open air (500 MHz instrument).

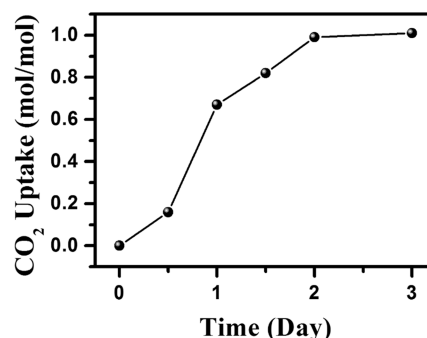
exposure time to the open air, the intensities of these newly appearing peaks significantly increase, corroborating the successful capture of  $\text{CO}_2$  from open air. The mechanism of  $\text{CO}_2$  capture is similar to that mentioned in the pure  $\text{CO}_2$  absorption study (Scheme 2). Although  $^{13}\text{C}$  NMR is inadequate for quantitative measurements, this can be resolved by increasing the number of scans and the relaxation time of the measurements.<sup>50–52</sup> Woerpel and co-workers integrated the  $^{13}\text{C}$  NMR peaks to determine the compound ratio quantitatively using the standard broad-band decoupled (BBD) mode with relaxation time (2 s).<sup>53</sup> In our experiment, all  $^{13}\text{C}$  NMR spectra were acquired with 3000 scans and 2 s of relaxation time using the BBD mode. We calculated the open-air  $\text{CO}_2$  uptake (mol/mol) of the  $[\text{N}_{2221}][\text{Arg}]$  ionic liquid after integrating the  $^{13}\text{C}$  NMR peaks and have tabulated it in Table 1. A detailed description of the determination of  $\text{CO}_2$  uptake (mol/mol) is described in the Supporting Information.

**Table 1.** Open-Air  $\text{CO}_2$  Uptake by an Aqueous Solution of  $[\text{N}_{2221}][\text{Arg}]$  Ionic Liquid after Different Exposure Times

duration of $\text{CO}_2$ exposure to open air (day)	$^{13}\text{C}$ NMR experiment duration (h)	$\text{CO}_2$ uptake (mol/mol) <sup>a</sup>	$\text{CO}_2$ uptake (mg/g)
0	20.46	0	0
0.5	23.56	0.16	24.32
1	23.46	0.67	101.85
1.5	20.24	0.82	124.66
2	20.15	0.99	150.50
3	20.07	1.01	153.54

<sup>a</sup>The experimental error  $\pm 11\%$ .

From Table 1 and Figure 5, we can conclude that with increasing time the  $\text{CO}_2$  uptake of the IL gradually increased



**Figure 5.** Open-air  $\text{CO}_2$  uptake by an aqueous solution of  $[\text{N}_{2221}][\text{Arg}]$  ionic liquid.

and finally saturated. Here, it is essential to mention that the average concentration of  $\text{CO}_2$  in our laboratory was 408 ppm. The uptake value may increase if we conduct this experiment in highly polluted areas like factories, power plants, and populated regions. In our subsequent work, we extend this experiment in this area.

#### 4. CONCLUSIONS

In summary, we have successfully synthesized an arginine-based ionic liquid and characterized it by NMR spectroscopy. We blended this ionic liquid (IL) with a DMSO–water binary mixture to overcome the high viscosity issue. In the pure  $\text{CO}_2$  absorption study using the vapor–liquid equilibrium (VLE) setup, the saturated  $\text{CO}_2$  uptake gradually decreases with increasing the volume percentage of DMSO. The  $\text{CO}_2$  uptake of the IL is the maximum in the aqueous medium, and it is minimum in the DMSO medium.  $^{13}\text{C}$  NMR spectra support the mechanisms of absorption of  $\text{CO}_2$  by IL in aqueous and DMSO mediums. Finally, we performed the open-air  $\text{CO}_2$  absorption study utilizing an aqueous solution of IL. With increasing time of exposure to the open air, the  $\text{CO}_2$  uptake gradually increased. After observing the  $\text{CO}_2$  capture experiment carefully, we came to the conclusion that our system is able to absorb  $\text{CO}_2$  from ambient air.

#### ■ ASSOCIATED CONTENT

##### Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.energyfuels.3c03647>.

Chemicals; instrumentation and procedure for pure  $\text{CO}_2$  absorption; calculation of open-air  $\text{CO}_2$  uptake by  $^{13}\text{C}$

NMR;  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of the  $[\text{N}_{2221}][\text{Arg}]$  ionic liquid; FTIR spectra of the  $[\text{N}_{2221}][\text{Arg}]$  ionic liquid in an aqueous medium before and after  $\text{CO}_2$  absorption;  $\text{CO}_2$  uptake by water and DMSO via VLE experiment; supporting tables of volumes of water added at different times in the open-air  $\text{CO}_2$  capture experiment and viscosity values before and after  $\text{CO}_2$  capture (PDF)

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### Notes

The authors declare no competing financial interest.

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