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Synthesis and Characterization of Amino Acid-Based Ionic Liquids for Selective Absorption of CO₂ Using Liquid Foam Bed and Semi-Batch Reactors

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ABSTRACT

Carbon dioxide (CO₂) emissions are produced by the combustion of fossil fuels for power generation, automobiles, and other major industrial processes. Hence, there is a need for efficient CO₂ absorption using advanced materials and reactor systems within the framework of carbon capture and storage technologies. This work emphasizes the synthesis and characterization of amino acid-based ionic liquids (AAILs) for CO₂ (99.99% pure) absorption via a liquid foam-bed reactor (FBR) and semi-batch equilibrium studies (SBES). Seven different AAILs, namely [TETA][ALA], [TETA][LYS], [TETA][GLU], [TETA][ASP], [TETA][GLN], [TETA][MET], and [TETA][PHE], were newly synthesized. The AAILs were characterized using FTIR and Raman spectroscopy and ¹H and ¹³C NMR spectroscopy to confirm their formation. Physical properties such as viscosity and density were also presented. The highest and lowest densities of 1.1709 and 1.0646 g cm⁻³ were observed for [TETA][ASP] and [TETA][ALA], respectively, among the synthesized AAILs. The application of AAILs in FBR and SBES for CO₂ absorption was evaluated under different experimental conditions. Parameters such as initial concentration, surfactant, pH, and AAIL regeneration were studied. [TETA][LYS] gave the highest moles of CO₂ absorbed per mole of [TETA][LYS] used; the highest mole ratios of 0.442, 0.172, and 0.112 (dry basis) were observed at initial concentrations of 500, 300, and 100 mmol CO₂, respectively. Finally, the highest mole ratio of CO₂ per mole of [TETA][GLU] was 0.166, followed by 0.133 for [TETA][ALA], during desorption studies.

1. INTRODUCTION

The increasing concentration of greenhouse gases (GHGs), particularly carbon dioxide (CO₂), has become a critical global concern because of its significant contribution to climate change. Industrial activities and fossil fuel-based power generation are among the primary sources of CO₂ emissions. In this context, carbon capture and storage (CCS) has emerged as a vital technological strategy to mitigate CO₂ emissions and limit their release into the atmosphere. CCS involves three main stages: (i) capture of CO₂ from large point sources, (ii) transportation via pipelines or marine systems, and (iii) long-term storage in deep geological formations such as depleted oil and gas reservoirs or saline aquifers (IPCC 2005). This integrated approach enables substantial reductions in emissions while allowing continued use of existing energy infrastructure.

Despite its potential, conventional CO₂ absorption technologies face several limitations. Common absorption approaches, including pre-combustion,

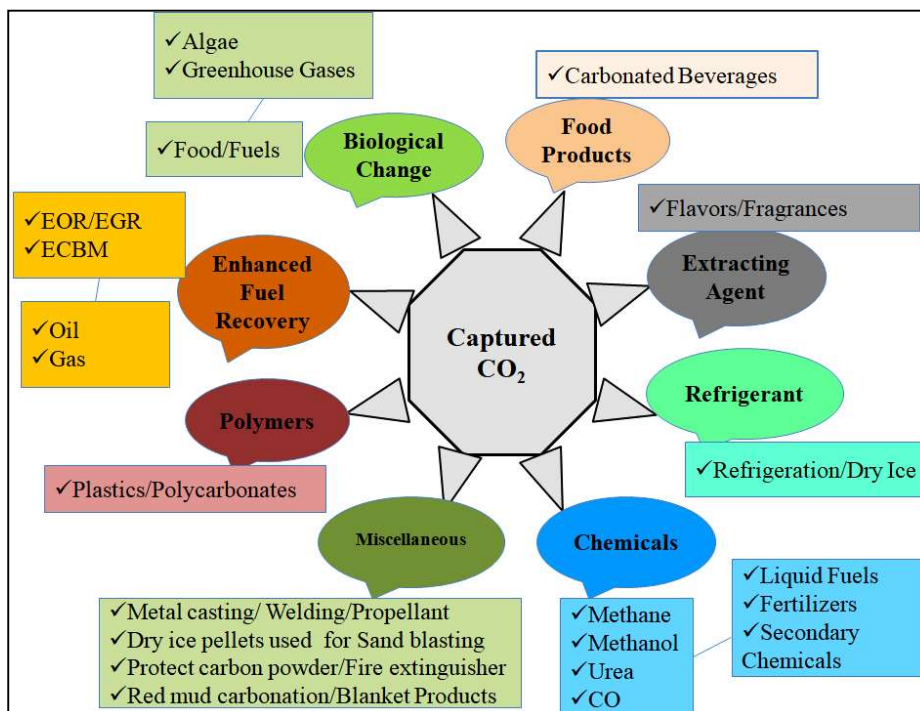


Fig. 1: Illustration of various products that can be obtained from CO₂-absorption technologies.

post-combustion, and oxy-fuel combustion, are often associated with high energy requirements, operational complexity, and high economic costs. In particular, solvent-based absorption systems using aqueous amines suffer from drawbacks such as solvent degradation, equipment corrosion, and high regeneration energy demand. Scaling these technologies to match large emission sources remains a major challenge, especially when considering the availability and suitability of storage sites. These limitations highlight the need for more efficient, cost-effective, and sustainable alternatives for CO₂ absorption. Fig. 1 illustrates the different products or compounds that can be obtained from CO₂ absorption technologies.

In recent years, ionic liquids (ILs) have gained considerable attention as promising materials for CO₂ absorption because of their unique physicochemical properties, including negligible vapor pressure, high thermal stability, and tunable chemical structures. Among them, amino acid-based ionic liquids (AAILs) have emerged as particularly attractive candidates because they combine the advantages of ILs with enhanced CO₂ affinity and environmental compatibility. AAILs exhibit strong chemical interactions with CO₂, leading to higher absorption capacities and selectivity compared with conventional solvents. Their design flexibility also allows optimization of performance characteristics such as viscosity, absorption kinetics, and regeneration efficiency. While ionic

liquids have also been explored in areas such as wastewater treatment and membrane separation, these applications are beyond the primary scope of this study and are therefore only briefly acknowledged.

To fully exploit the potential of advanced solvents such as AAILs, there is a growing need for innovative reactor systems that can enhance mass transfer and overall process efficiency. One such promising technology is the foam-bed reactor (FBR), which offers improved gas-liquid contact, higher interfacial area, and enhanced mixing characteristics compared to conventional packed or stirred reactors. The FBR system can significantly improve CO₂ absorption rates while potentially reducing solvent usage and energy consumption. Therefore, integrating AAILs with advanced reactor designs like FBR represents a forward-looking approach to overcoming the limitations of traditional CO₂ absorption systems.

In summary, the development of efficient CO₂ absorption technologies requires a synergistic combination of advanced materials and innovative process design. This study focuses on the application of amino acid-based ionic liquids in a foam-bed reactor system, aiming to provide an effective and sustainable solution for CO₂ absorption.

1.1. Background Research

Several carbon absorption technologies have been demonstrated at the laboratory scale, and some have also

been demonstrated industrially. These include physisorption or chemisorption processes (Zhang et al. 2011, Cahyonugroho et al. 2025, Mandal & Bandyopadhyay 2005), ionic liquid membranes and membrane separations (Makino & Kanakubo 2016, Ramdin et al. 2012, Jones 2011), and molecular sieves (Sarker et al. 2017, Wahby et al. 2012, Mohamedali et al. 2017). Combinations of ionic liquids (ILs) and composite materials have been used for CO₂ absorption (Pilli et al. 2014, Mulk et al. 2023), as have amines and amino acid ILs (Du et al. 2016, Qu et al. 2022, Yamini & Khanna 2014, Mumford et al. 2017). The use of novel materials, such as ionic liquids as electrolytes and nanostructured materials for cathodes, has the potential to enhance both the energy efficiency and lifespan of batteries (Padmanabhan et al. 2024).

Most of the literature is available on amine-based technologies. The most commonly used amines for CO₂ absorption are monoethanolamine, diethanolamine, and methyldiethanolamine, which absorb CO₂ via carbamate/carbonate formation. Although conventional CO₂ absorption technology of this type has been used industrially for a considerable period, several drawbacks remain. These include degradation of expensive solvents, limited thermal stability, corrosion, and high energy consumption during regeneration due partly to solvent loss by evaporation.

Reviews of ILs for CO₂ absorption have been reported in the literature (Cota & Martinez 2017). CO₂ adsorption, synthesis, and the use of metal-organic frameworks (MOFs) impregnated with ILs have also been reported (Lupa et al. 2024). Ionic liquids have also been used as membrane solvents or supported ionic liquid membranes (SILMs) for CO₂ absorption (Makino & Kanakubo 2006, Ramdin et al. 2012, Sarker et al. 2017, Elmobarak et al. 2023).

1.2. ILs in Wastewater Treatment and Membrane Filtration

A variety of polar and nonpolar materials, such as organic contaminants, heavy metals, and dyes, which are frequently found in industrial wastewater, can be dissolved by ILs (Zhang et al. 2011, Mandal & Bandyopadhyay 2011). This enables efficient removal of pollutants from wastewater. ILs have unique properties such as solubility, selectivity, and reusability, making them promising solvents for wastewater treatment, effective adsorbents for water treatment and contaminant extraction, and useful media for membrane filtration and CO₂ absorption and capture (Makino & Kanakubo 2006, Ramdin et al. 2012, Jones 2011, Sarker et al. 2017). To reduce costs, ensure environmental safety, and address stability and scalability, their use must be appropriately tailored.

Liquid-liquid extraction (LLE), in which pollutants are extracted from water and concentrated in the IL phase,

is one application of ILs (Mulk et al. 2023). The IL can then be recycled and reused. For the removal of organic contaminants such as petroleum hydrocarbons or chlorinated chemicals, this method can be highly effective. Targeting various pollutants, such as pesticides, dyes, and medicines, is made possible by fine-tuning the IL selectivity for specific contaminants through changes to the cation or anion (Sarker et al. 2017, Lupa et al. 2024). Ionic liquids can be added to membrane materials to enhance their filtration performance. These modified membranes are more efficient than conventional membranes at removing both organic and inorganic contaminants from wastewater, increasing the effectiveness of methods such as nanofiltration and reverse osmosis.

Although the primary focus of this study is CO₂ absorption, the discussion on wastewater treatment highlights the broader environmental applicability of ionic liquids. Their tunable physicochemical properties, low volatility, and potential for recyclability make them promising green solvents not only for pollutant removal but also for gas absorption processes. These characteristics support their selection as effective absorbents in CO₂ absorption systems.

1.3. Biodegradability, Toxicity, Reusability, and Recovery of ILs

Several ILs can be designed to be biodegradable and nontoxic, which are key factors for wastewater treatment (Babamohammadi et al. 2015). Conventional organic solvents or chemicals used for wastewater treatment may cause long-term environmental effects. However, ILs can be engineered to minimize these risks. Nonetheless, an IL's biodegradability depends on its chemical structure, and some ILs may still be toxic to aquatic organisms (Babamohammadi et al. 2015). Another significant advantage of ILs is recyclability. ILs can usually be regenerated using simple methods such as evaporation and distillation after wastewater treatment, thereby minimizing waste and lowering operating costs. Their reusability makes them a sustainable choice for large-scale applications.

In this work, seven different ionic liquids (ILs) were newly synthesized using amino acids (AAs) as anions and triethylenetetramine (TETA) as the cation. The following sections describe ILs, amino acids, amines, their physical properties, and their applications in science and engineering.

1.4. Amines

Amines (aminoalkanes) are generally organic substances that have a basic nitrogen atom with a lone pair and are derived from ammonia by the substitution of one or more H⁺ atoms by organic groups such as alkyl or aryl groups. There are

four main types: primary, secondary, tertiary, and cyclic amines, with elevated melting and boiling points. Amines are widely used industrially in the preparation of dyes and

drugs. Amine-based compounds such as monoethanolamine (MEA), diisopropylamine (DIPA), diglycolamine (DGA), diethanolamine (DEA), and methyldiethanolamine (MDEA)

Table 1(a): List of amino acids used as anions in this study.

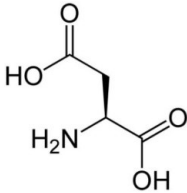
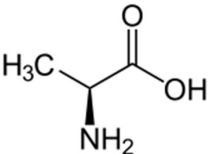
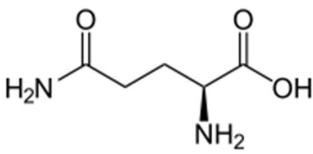
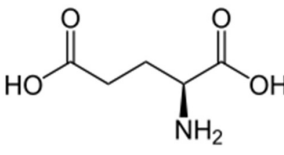
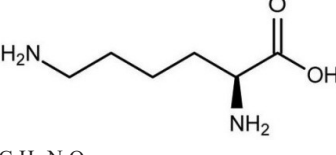
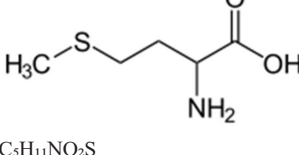
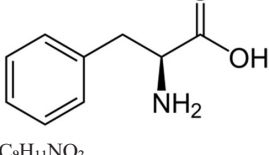
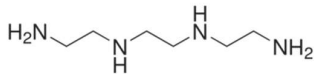
S. No.	Compound Name	Synonym/ IUPAC ID	Structure/Formula	CAS No.	Molecular Weight (g mol ⁻¹)
1.	L-Aspartic acid [ASP]	(S)-(+)-Aminosuccinic acid, (S)-Aminobutanedioic acid	 C ₄ H ₇ NO ₄	56-84-8	133.10
2.	L-Alanine [ALA]	(S)-2-Aminopropionic acid	 C ₃ H ₇ NO ₂	56-41-7	89.09
3.	Glutamine [GLN]	(S)-2,5-Diamino-5-oxopentanoic acid	 C ₅ H ₁₀ N ₂ O ₃	56-85-9	146.14
4.	Glutamic acid [GLU]	(S)-2-Aminopentanedioic acid	 C ₅ H ₉ NO ₄	56-86-0	147.13
5.	L-Lysine [LYS]	(S)-2,6-Diaminohexanoic acid monohydrochloride	 C ₆ H ₁₄ N ₂ O ₂	657-27-2	182.65
6.	L-Methionine [MET]	(S)-2-Amino-4-(methylmercapto)butyric acid	 C ₅ H ₁₁ NO ₂ S	63-68-3	149.21
7.	L-Phenylalanine [PHE]	(S)-2-Amino-3-phenylpropionic acid	 C ₉ H ₁₁ NO ₂	63-91-2	165.19

Table 1(b): List of amines used as cations in this study.

S. No.	Compound Name	Synonym/IUPAC ID	Structure/Formula	CAS No.	Molecular weight g mol ⁻¹	Density g cm ⁻³
1.	Triethylenetetramine [TETA]	N,N'-Bis(2-aminoethyl)-1,2-ethanediamine	 C ₆ H ₁₈ N ₄	112-24-3	146.24	0.982

are widely used in process industries for the removal of CO₂ and hydrogen sulfide (H₂S) from natural gas and petroleum-refining process gas streams. Amines have also been used for CO₂ absorption from combustion and waste gases and can contribute to the removal of greenhouse gas emissions (Babamohammadi et al. 2015, Fukumoto et al. 2005).

1.5. Amino Acids

AAs are simple organic compounds comprising a carboxyl (–COOH) and an amino (–NH₂) group. These carboxyl and amino functional groups are also useful for introducing other functional groups. It is well known that about 20% of the human body contains proteins. These macromolecules play significant roles in biological processes, and AAs are their building blocks. The advantages of AAs include low environmental impact, high biodegradability, negligible volatility, high resistance to oxidative degradation, and low volatility due to their ionic structure. Their physical characteristics include being crystalline solids with very high melting points (200–300°C), usually water-soluble and insoluble in nonpolar organic solvents such as hydrocarbons, possessing optical isomers because of asymmetric α-carbon atoms, and having a zwitterionic nature (Yamini & Khanna 2015). AAs can taste sweet, bland, or occasionally bitter. AAs are primarily used in cosmetics, active pharmaceutical ingredients (APIs), medical foods or medical nutrition, drinks, culture media, dietary supplements, functional foods, and polymer synthesis. More recently, these AAs have also been used in the synthesis and preparation of ILs (Walden 1914). The amino acids used in this study as anions and the amine used as the cation are listed in Tables 1(a) and 1(b), respectively.

1.6. Ionic Liquids as Green Solvents

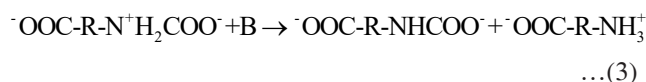
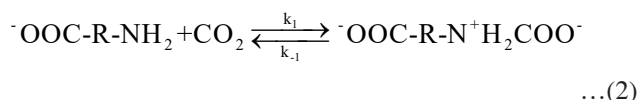
ILs are attractive materials that have gained considerable research attention in science and engineering, especially in modern physical chemistry, as green solvents. They are liquids at ambient temperature and are mainly used as substitutes for conventional volatile organic solvents such as tetrachloroethylene, formaldehyde, methylene chloride, benzene, ethylene glycol, toluene, and xylene. Applications of ILs in different fields of science and engineering are presented in Fig. 2. ILs are substances composed of organic

cations and organic or inorganic anions. ILs were first reported in 1914 by Paul Walden, a well-known Latvian chemist, in his work on stereochemical reactions (Walden 1914). ILs have attracted significant attention because of their notable and environmentally favorable properties, including low vapor pressure, reusability, broad chemical and thermal stability, nonflammability, high ion density, a wide electrochemical window, tunable viscosity, and superior electrical conductivity (Wilkes 2002). Chemical structures of frequently used cations and anions are presented in Table 1(c). Similarly, Table 2 compares various characteristics of volatile organic solvents, molten salts, and ILs.

Alternatively, amino acid-based ionic liquids (AAILs) can also be used for improved CO₂ absorption capacity. As mentioned above, AAs comprise both a carboxyl (–COOH) and an amino (–NH₂) functional group. The reaction of CO₂ with AAs has been reported to be similar to that of alkanolamines. However, the precise CO₂ reaction mechanism of AAs remains debated in the scientific community. In 2002, Kumar et al. described a mechanism different from the zwitterionic mechanism, proposing a direct 2:1 mechanism that produces carbamate and protonated amine, as indicated in Eq. (1) (Kumar et al. 2002).

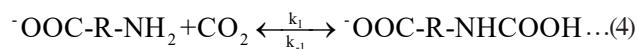


However, as described in the literature, a zwitterionic mechanism has been proposed for the reaction of CO₂ with potassium salts of glycine and taurine, as given in Eqs. (2) and (3) (Kumar et al. 2003).



where B is any alkali compound. In a nonaqueous medium, AAs behave as a base. In 2010, Gürkan et al. described that the reaction of CO₂ with ILs prepared from amino acids terminates with the formation of carbamic acid (Eq. 4) without forming carbamate species (Eq. 5) (Gürkan et al. 2010). Gürkan et al. examined CO₂ absorption in phosphonium-based cations combined with methionine

and proline amino acids as anions. They proposed a 1:1 stoichiometric reaction of CO₂ with AAILs, suggesting that the reaction ends with the formation of carbamic acid (Eq. 4).



The proposed mechanisms (Eqs. 1–5) are used to interpret experimental trends qualitatively, and detailed mechanistic validation is beyond the scope of the current study.

In previous work, Yamini and Khanna studied CO₂ solubility in different amino acid-based ILs synthesized with butylmethyl imidazolium ([BMIM]⁺) as the cation and amino acids as anions (Yamini & Khanna 2015). Goodrich et al. examined CO₂ absorption using various amino acid-based anions and phosphonium-based cations for preparing ILs and proposed reaction mechanisms (Goodrich et al. 2011). Kortunov et al. studied physical absorption techniques for CO₂ absorption in ionic liquids, including higher-pressure carbon absorption using imidazolium-based ionic liquids and amines mixed in ionic liquid solutions (Kortunov et al. 2015). As noted above, most reports support formation of a zwitterion intermediate (Eqs. 2 and 3) as the principal pathway rather than one-step carbamate formation (Eq. 1) or formation via carbamic acid (Eqs. 4 and 5) (Danckwerts 1979, Versteeg & van Swaaij 1988, van Holst et al. 2009, Xie et al. 2010).

The primary goal of this work is to examine the effect of amine functionalization on different amino acids used as anions. Another reason for selecting amino acids is that they are biodegradable, readily available, and comparatively inexpensive, although very few studies have been reported in the literature. In this work, seven different AAILs were synthesized, namely, [TETA][ALA], [TETA][ASP], [TETA][LYS], [TETA][GLU], [TETA][GLN], [TETA][MET], and [TETA][PHE]. CO₂ absorption studies were carried out in the FBR at room temperature.

Although amino acid-based ionic liquids (AAILs) have been widely reported, the originality of this work lies in the use of triethylenetetramine (TETA) as a polyamine-based cation combined with various amino acid anions, providing multiple active amine sites that enhance CO₂ interaction. A detailed comparative discussion has been incorporated to evaluate these TETA-based AAILs against commonly reported imidazolium- and phosphonium-based systems in terms of CO₂ absorption capacity, reaction mechanisms (chemisorption versus physisorption), availability of active sites, and environmental and economic aspects (Yamini & Khanna 2015). Furthermore, the application of these AAILs in a liquid foam-bed reactor (FBR) highlights their functional advantage through improved gas–liquid interfacial contact, reinforcing the distinct contribution of this study.

2. MATERIALS AND METHODS

All amino acids, including L-aspartic acid, L-alanine,

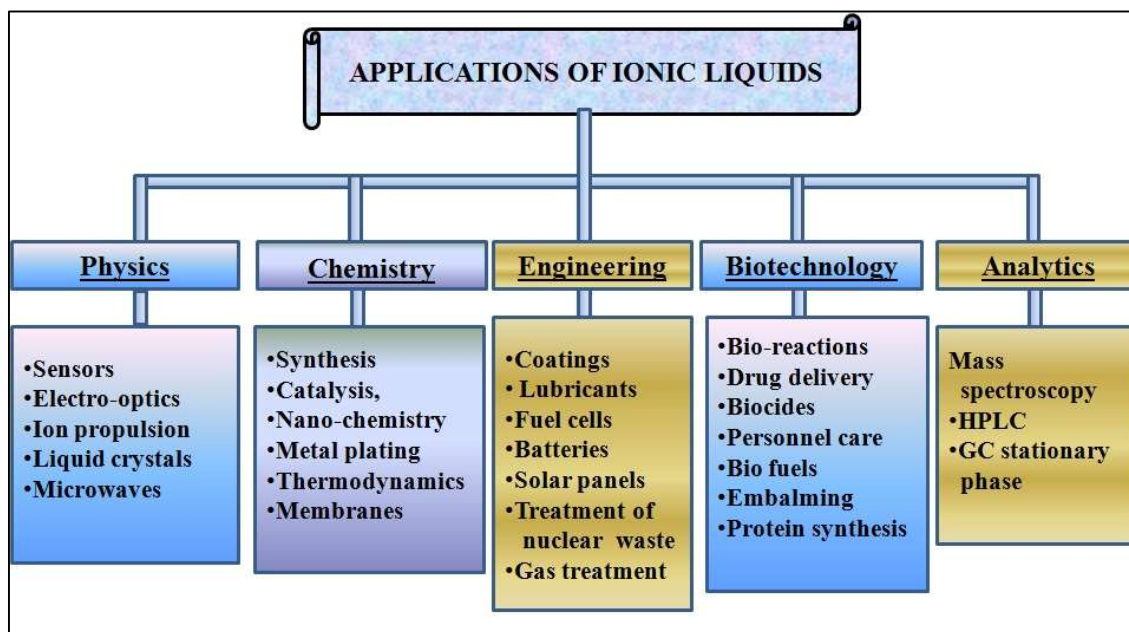
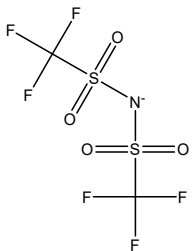
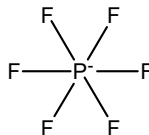
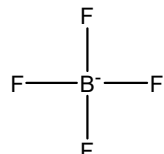
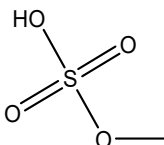
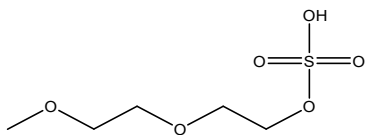
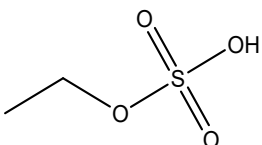
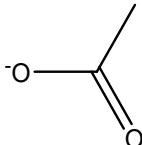
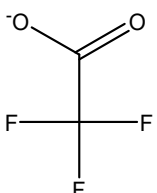
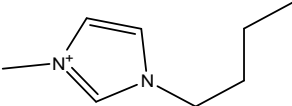
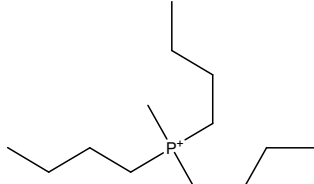
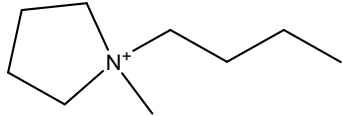
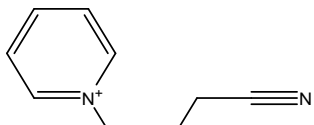
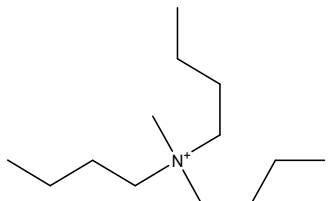
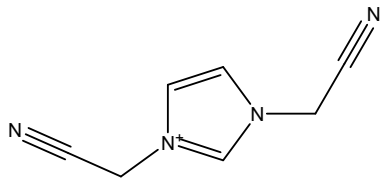


Fig. 2: Typical applications of ILs in different fields of science and engineering.

Table 1(c): Common anions and cations used in the preparation of ILs.

Anions		
		
bis(trifluoromethylsulfonyl) amide	Hexafluorophosphate	tetrafluoroborate
		
Methylsulfate	2-2 methoxy ethoxy ethyl sulfate	Ethylsulfate
		Br⁻ Bromide
acetate	trifluoroacetate	
Cations		
		
1-Butyl-3-methylimidazolium	Tributylmethylphosphonium	1-Butyl-1-methylpyrrolidinium
		
1-(3-Cyanopropyl)pyridinium	Tributylmethylammonium	1,3-Bis(cyanomethyl)imidazolium

L-lysine, L-methionine, L-phenylalanine, glutamine, and glutamic acid, were purchased from CDH, Delhi. Monoethanolamine and triethylenetetramine were also purchased from CDH, Delhi. Dichloromethane, dimethyl ether, sodium lauryl sulfate (SLS), cetrimonium bromide (CTAB), Triton X-100, Tween 80, amyl alcohol, methyl orange indicator, and methanol were procured from Fisher Scientific, India. All reagents were used as received;

no further purification was carried out unless stated otherwise.

2.1. Experimental Section

2.1.1. Synthesis and Purification of Ionic Liquids

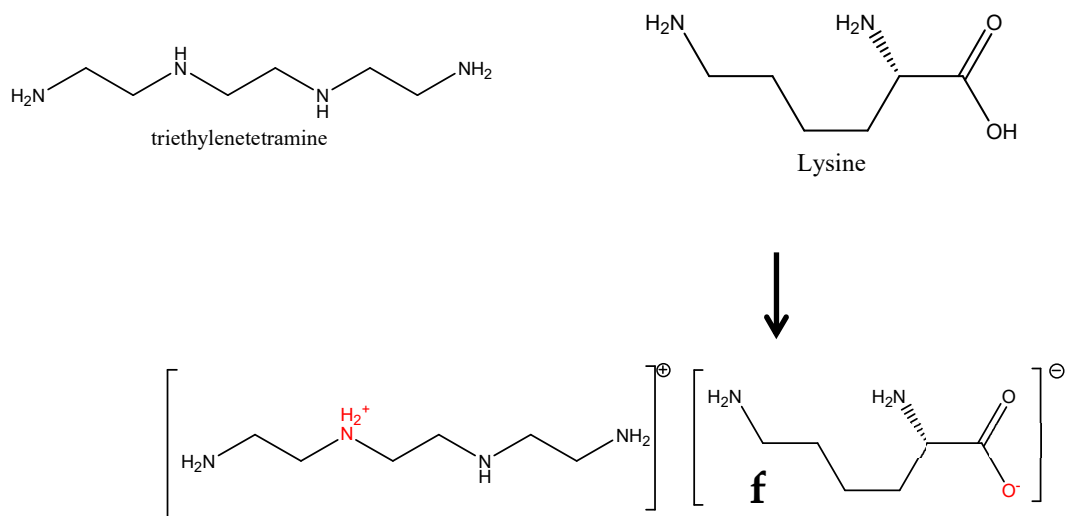
AAILs were prepared by H⁺ transfer between a Brønsted acid and a Brønsted base. Equimolar quantities of acid and base were reacted with each other. Dropwise addition of the

Table 2: Comparison of the various characteristics of volatile organic solvents, molten salts, and ILs.

S. No.	Property	Ionic Liquids	Volatile organic solvents	Molten salts
1	Basic constituents	Pair of ions	Molecules	Pair of ions
2	Application	Multifunction	Single function	Multifunction
3	Catalytic ability	Common and tunable	Rare	Common and tunable
4	Chirality	Common and tunable	Rare	Common and tunable
5	Vapor pressure	Negligible under ambient conditions	Obeys the Clausius-Clapeyron equation	Insignificant under ambient conditions
6	Flammability	Nonflammable	Highly flammable	Nonflammable
7	Solvation effect	Strong	Weak	Strong
8	Cost	Expensive (2–100 times the cost of organic solvents)	Cheap	Expensive
9	Recyclability	Economic imperative	Green imperative	Economic imperative
10	Corrosiveness	Low	Intermediate	High
11	Melting point	–20–150°C	< –20°C	> 200°C
12	Boiling point	< 400°C	30–100°C	> 400°C
13	Viscosity (cP)	Low (22–40,000)	Low (0.2–100)	High
14	Density (g cm ³)	High (0.8–3.3)	Medium (0.6–1.7)	High (0.8–3.3)
15	Conductivity	High	Low	High
16	Refractive index	High (1.5–2.2)	Medium (1.3–1.6)	High (1.5–2.2)

amine to the amino acid was performed in an ice bath because the reactions are highly exothermic; due care was taken during synthesis. At room temperature, the reaction mixture was agitated for one day. A small excess of amine was introduced and subsequently removed along with water by heating to 80–85°C to ensure complete reaction. To remove unreacted acid and water, the reacted mixture was further dried at 80–85°C under vacuum (about 300 mm Hg), and the

residue was subsequently extracted using diethyl ether (DE) or dichloromethane (DCM). Following evaporation of the solvent, the product was dried for at least 3–5 days at 80–85°C in a vacuum oven to remove any remaining moisture. The AAILs were then ready for further characterization and use. The synthesis reactions were conducted without solvent; when required, 10% methanol was used to dissolve the amino acid in deionized water (if it was in the solid state), stirred



Scheme 1: Proposed reaction mechanism of [TETA][LYS].

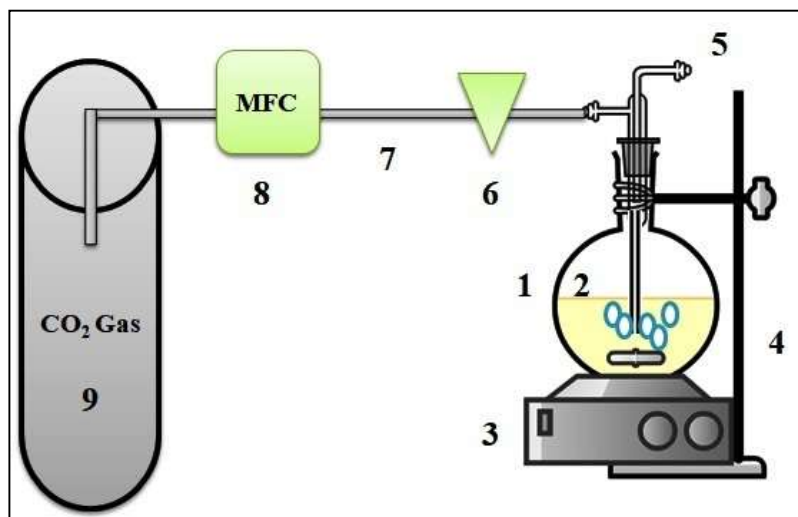


Fig. 3(a): Semi-batch equilibrium experimental setup used for CO₂ absorption. (Parts of the figure are as follows: 1. Round-bottom flask, 2. Ionic liquid, 3. Magnetic stirrer, 4. Stand, 5. Gas outlet, 6. Rotameter, 7. Gas inlet, 8. Mass flow controller with digital indication, and 9. CO₂ gas cylinder).

for 10–15 h, and then added dropwise to TETA. A similar procedure was used for the synthesis of all AAILs.

2.1.2. Proposed Reaction Mechanisms of Synthesized ILs

The reaction mechanism between triethylenetetramine and lysine is shown in Scheme 1. TETA consists of a long carbon chain with two primary ($-\text{NH}_2$) groups and two secondary ($-\text{NH}$) groups, whereas aspartic acid has two carboxylic groups ($-\text{COOH}$) and one amine group. When aspartic acid reacts with [TETA], it may donate protons to [TETA], forming [TETA][ASP]. It is proposed that all amino acids react with TETA to form AAILs. Reactions for other compounds, such as [TETA][ALA], [TETA][LYS], [TETA][GLU], [TETA][GLN], [TETA][MET], and [TETA][PHE], are provided in the supporting information, SI Schemes 1–4.

2.1.3. Procedure for Semi-Batch Equilibrium Studies

Equilibrium experiments were conducted in a 250 mL round-bottom flask (RBF) with a lid arrangement for continuous gas flow into and out of the RBF. A known amount of prepared AAIL was placed in the RBF. The desired amount of CO₂ gas was bubbled continuously into the AAIL in the RBF. Unreacted gas exited through the vent line of the RBF, as shown in Fig. 3(a). The entire setup was placed on a magnetic stirrer with the help of a stand. Semi-batch equilibrium experiments for CO₂ absorption were carried out for several hours until equilibrium was reached. Samples were collected at predefined time intervals and analyzed by titrimetry against 2 M HCl in the Chittick apparatus. For each sample, the amount of CO₂ absorbed over time and the moles of CO₂ absorbed per mole of AAIL were calculated. A similar procedure was used to conduct equilibrium experiments for different AAILs.

2.1.4. Regeneration of AAILs

Regeneration of used AAILs was carried out before further use for multiple cycles. Regeneration was performed by heating the AAIL at 80–90°C with continuous stirring. During regeneration, samples were collected for analysis at fixed time intervals. The rate of desorption was also studied over time. It was observed that regenerated AAILs could be used for a further cycle. The apparatus used for AAIL regeneration is presented in Fig. 3(b).

2.1.5. Experimental Procedure of the Liquid Foam-Bed Reactor

CO₂ absorption experiments were carried out in a semi-batch liquid foam-bed reactor (FBR). The experimental setup of the liquid foam-bed reactor used in this study is shown in Fig. 4(a). The FBR consists of two sections: the foam section and the storage section. It is a simple, transparent glass column (0.7 m high and 7.6×10^{-2} m in diameter) fitted with a distributor plate above the cone-shaped bottom (flow stabilization chamber), as shown in Fig. 4(a). The distributor plate contains several holes that allow the CO₂ gas/air mixture to enter the column during the experiment. The FBR also has a sample-charging port above the distributor plate and several sampling ports at different heights for collecting samples for analysis during the experiment.

During the FBR experiments, the regulator of the CO₂ gas cylinder was heated using infrared lamps. This heating prevented choking of the gas pipeline due to dry-ice formation. An air compressor was used to provide air and dilute the CO₂ gas. Precalibrated rotameters were used to measure CO₂ and air flow rates. As noted above, CO₂ gas was diluted by mixing it with air using a packed-bed setup

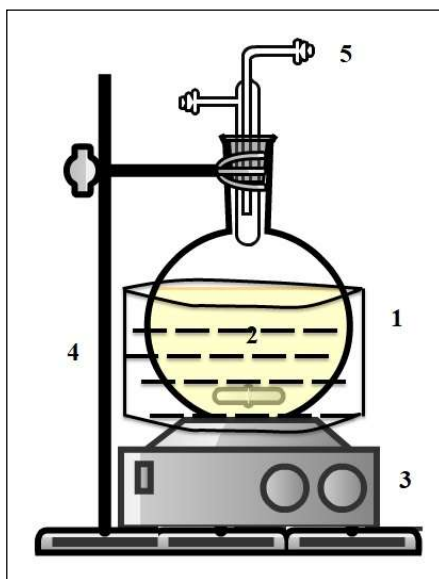


Fig. 3(b): Apparatus used for regeneration of AAILs. (Parts of the figure are as follows: 1. Round-bottom flask, 2. Ionic liquid, 3. Magnetic stirrer, 4. Stand, and 5. Gas outlet to storage.)

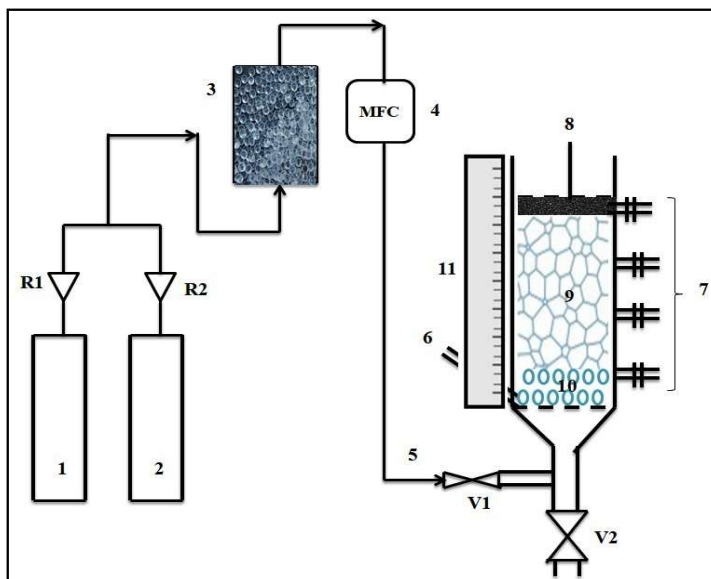


Fig. 4(a): Experimental setup of the liquid foam-bed reactor used in this study. (Parts are as follows: 1. Pure CO₂ gas supply, 2. Air supply, 3. Air and CO₂ mixture (packed bed with glass beads), 4. Mass flow controller with digital indication, 5. Gas inlet, 6. Liquid charging port, 7. Sampling ports, 8. Sieve plate for foam breaking, 9. Foam section, 10. Storage section, 11. Ruler, R1 and R2 gas rotameters, and V1 and V2 valves).

containing glass Raschig rings. This packed bed was used to mix CO₂ and air and to prevent dust or solid particles, if any, in the air from entering the FBR.

Initially, air at a known flow rate was released into the FBR. About 100 mL of aqueous AAIL of known concentration was mixed with a known amount of surfactant in a volumetric flask, stirred for 2–3 h, and then charged into the FBR through

the liquid inlet port. The foam height was then monitored. After the desired foam height was reached, CO₂ gas was released from the cylinder and passed through the FBR. The CO₂ gas was mixed with air before entering the FBR. Once CO₂ entered the FBR, the experiment was started and the time was recorded. During the experiment, CO₂ gas and air flow rates were monitored using a digital mass flow controller and

rotameter, respectively. The foam height was maintained using a circular foam breaker consisting of a 7.5×10^{-2} m-diameter sieve plate fitted with a circular sponge (foam, $\sim 7.8 \times 10^{-2}$ m) and connected to a thin iron rod about 0.8 m long, as shown in Fig. 4(a). A few drops of amyl alcohol were used in the foam-breaker sponge as an antifoaming agent. When foam bubbles reached the foam-breaker surface, they collapsed. Perforations in the sieve plate allowed unreacted CO_2 gas to escape. Through the Plateau borders, liquid in the foam films returned to the storage section. Using a stopwatch, liquid samples were collected at regular time intervals from the sampling ports near the storage section. The CO_2 experiments were continued for 30 min. The gas supply was then closed, and the solution was drained from the storage section.

CO_2 loading in a sample of AAIL solution was calculated using the following equation:

$$\alpha = \frac{(760 \text{ mmHg} - P_{\text{H}_2\text{O}})(V_{\text{CO}_2} - V_{\text{HCl}})}{(\text{sample vol. (mL)} \times R \times T)} \times 1000$$

Where:

α = CO_2 loading (mol CO_2 per mL of absorbent)

760 mm Hg = 1 atm (standard atmospheric pressure)

$P_{\text{H}_2\text{O}}$ = vapor pressure (mm Hg) at 302 K

V_{CO_2} = volume of solution displaced by CO_2 in the graduated tube (mL)

V_{HCl} = volume of HCl used for titration (mL)

Sample vol. = volume of sample drawn from the FBR (mL)

T = temperature (K)

R = universal gas constant ($\text{L} \cdot \text{torr} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$)

2.1.6. Analysis of Samples

After the samples were collected from the FBR at regular time intervals, the absorbed CO_2 was analyzed using the Chittick apparatus, as shown in Fig. 4(b). A magnetic stirrer, 25 mL conical flask, 50 mL burette, 10 mL pipette, glass stopper, graduated glass tube with a scale, glass reservoir, analytical balance (AUW-220D dual-range semi-microbalance), and methyl orange indicator were used for the analysis. A collected sample of known weight was placed in a 25 mL conical flask; one or two drops of methyl orange were added, and the sample was titrated against 2 N HCl while the entire setup was placed on a magnetic stirrer until the color changed from yellow to pale orange. The burette reading and the gas displaced in the glass reservoir were recorded for each sample, along with the time at which the sample was collected. Teflon tape or vacuum grease was used on the conical flask to prevent gas leakage. During the analysis, due care was taken to avoid leakage of CO_2 gas from the flask.

3. RESULTS AND DISCUSSION

3.1. Characterization of Ionic Liquids

3.1.1. FTIR Analysis

Fourier-transform infrared spectroscopy (FTIR) using a NICOLET iS50 FT-IR model was carried out to identify functional groups present in the prepared AAILs from

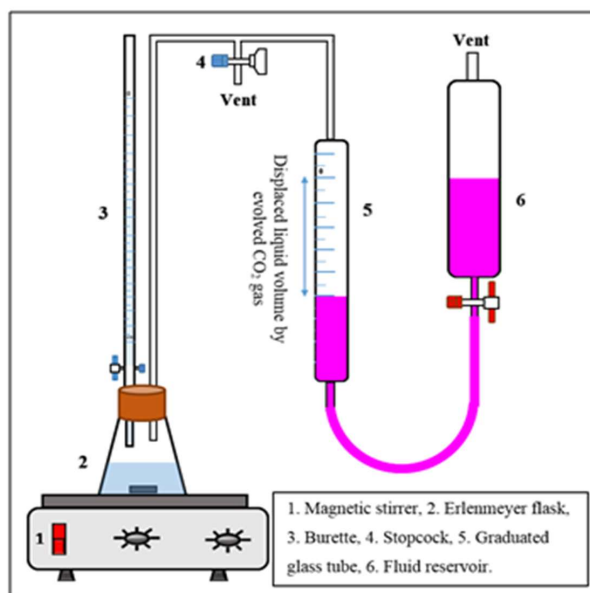


Fig. 4(b): Pictorial representation of the Chittick apparatus used for liquid sample analysis (Norouzbahari et al. 2016).

their infrared absorption spectra. The FTIR spectra were recorded over the wavenumber range of 500–4000 cm^{-1} . These infrared absorption bands identify molecular structural components. Figures 5(a)–5(f) show the FTIR spectra of pure [TETA] used as the cation, AAs used as anions, and the synthesized AAILs, namely [TETA][ALA], [TETA][ASP], [TETA][GLU], [TETA][LYS], [TETA][MET], and [TETA][PHE]. The FTIR spectra show all major functional groups present in the individual compounds and prepared AAILs. Distinct FTIR peaks representing C=O of $-\text{COO}^-$ in amino acids at 1700 and 1462 cm^{-1} and peaks corresponding to N–H stretching vibrations of $-\text{NH}_2$ in the 3600–3000 cm^{-1} range were identified for almost all AAILs (Barth 2000). The FTIR bands of the synthesized AAILs were very similar and showed the same bands at different wavenumbers. Figure 5(a) shows the FTIR spectra of pure anion [ALA] $^-$, pure cation [TETA] $^+$, and synthesized [TETA][ALA]. The characteristic FTIR peak corresponding to C=O of $-\text{COO}^-$ at 1647–1400 cm^{-1} and a peak corresponding to

strong N–H stretching of $-\text{NH}_2$ at 3559 cm^{-1} were observed for synthesized [TETA][ASP]. Near 3456 cm^{-1} , a strong N–H stretching vibration was observed, with sharp peaks at 1621 and 1395 cm^{-1} corresponding to C=O and carboxylic groups, respectively, as shown in Fig. 5(b). Similar peaks can be seen in Figs. 5(c)–5(f). Primary and secondary amines can generally be identified from absorption bands in the 3600–3000 cm^{-1} range corresponding to N–H stretching vibrations. As reported, N–H in-plane bending vibrations in NH_3^+ are estimated in the ranges around 3100, 1300–1550, and 1600–1750 cm^{-1} (Khanna & Moorie 1999).

3.1.2. Raman Spectroscopy

The synthesized AAIL samples were analyzed using a benchtop Raman spectrometer (Renishaw plc, Model: Micro Raman Spectrometer, RA800-series, UK), equipped with an excitation laser wavelength of 785 nm and an excitation source operated at about 50% power. The accuracy of the measured wavenumber positions of the bands was ± 0.98 cm^{-1} , defined by the resolution of the diffraction grating,

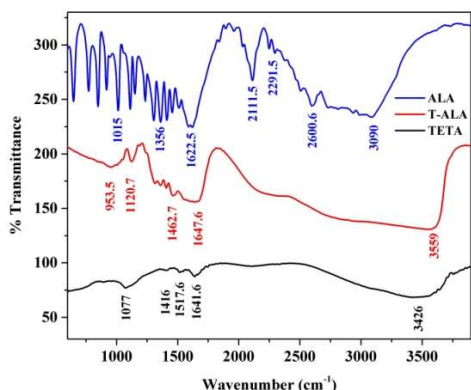


Fig. 5(a): FTIR spectra of pure anion [ALA] $^-$, pure cation [TETA] $^+$, and synthesized [TETA][ALA].

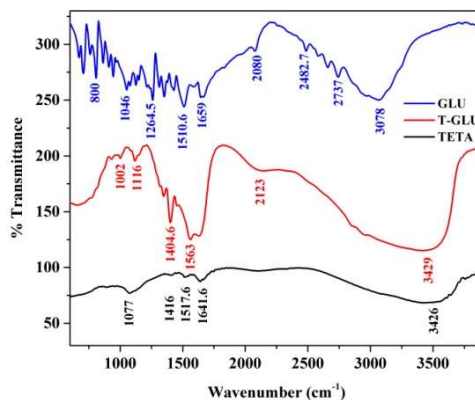


Fig. 5(c): FTIR spectra of anion [GLU] $^-$, cation [TETA] $^+$, and synthesized IL [TETA][GLU].

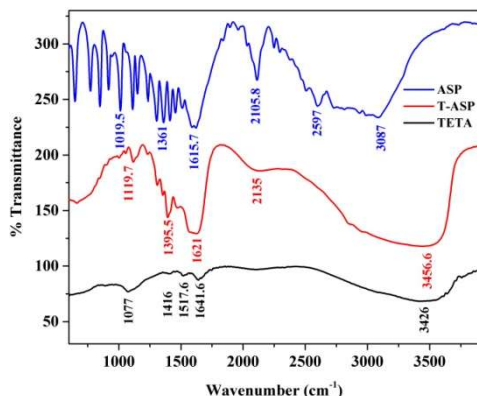


Fig. 5(b): FTIR spectra of pure anion [ASP] $^-$, pure cation [TETA] $^+$, and synthesized [TETA][ASP].

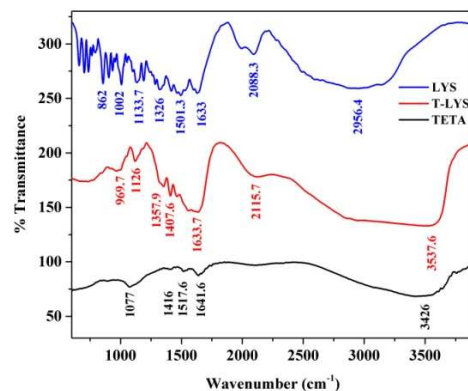


Fig. 5(d): FTIR spectra of anion [LYS] $^-$, cation [TETA] $^+$, and synthesized IL [TETA][LYS].

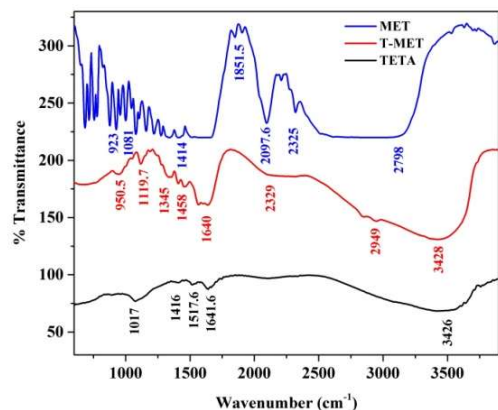


Fig. 5(e): FTIR spectra of anion $[MET]^-$, cation $[TETA]^+$, and synthesized IL $[TETA][MET]$.

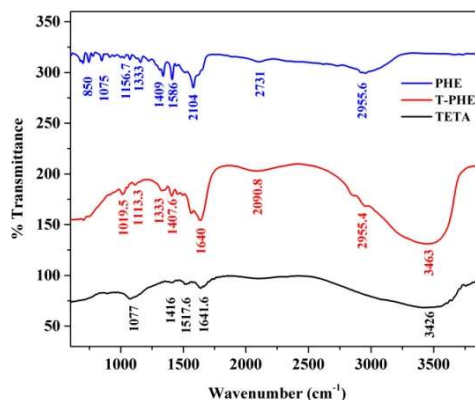


Fig. 5(f): FTIR spectra of anion $[PHE]^-$, cation $[TETA]^+$, and synthesized IL $[TETA][PHE]$.

while intensity measurements were typically recorded every 1.96 cm^{-1} . Fig. 6 shows the Raman spectra of the synthesized ILs $[TETA][LYS]$, $[TETA][GLU]$, $[TETA][ASP]$, $[TETA][ALA]$, $[TETA][MET]$, $[TETA][PHE]$, and cation $[TETA]^+$.

As shown in Fig. 6, bands above the 2500 cm^{-1} region are attributed to NH, OH, and CH stretching modes for all AAILs. The strongest symmetric CH_2 stretching band, $\nu_s(CH_2)$, in the Raman plot was detected in the $2900\text{--}3100\text{ cm}^{-1}$ range (at $2934, 2979, 3030, 3057, \text{ and } 3073\text{ cm}^{-1}$) for $[TETA][LYS]$, $[TETA][GLU]$, $[TETA][ASP]$, $[TETA][ALA]$, $[TETA][MET]$, and $[TETA][PHE]$. It was shifted in the solution-state Raman spectrum and was also extremely intense for $[TETA][LYS]$, $[TETA][ASP]$, $[TETA][ALA]$,

$[TETA][MET]$, and $[TETA][PHE]$. Similarly, pure cation $[TETA]$ showed very broad stretching. $C=C$ stretching was observed at $1012, 1033, 1063, \text{ and } 1076\text{ cm}^{-1}$; antisymmetric rocking of NH_3^+ was observed at $1143 \text{ and } 1183\text{ cm}^{-1}$ for all prepared AAILs. For $[TETA][PHE]$, vibrations associated with the aromatic ring were also detected at $848, 912, \text{ and } 949\text{ cm}^{-1}$. A characteristic band associated with $C-S$ stretching was observed at 659 cm^{-1} because $[TETA][MET]$ is composed of $C_5H_{11}NO_2S$ and $C_6H_{18}N_4$. Raman spectra of the pure amino acids and all synthesized AAILs are provided in the supporting information (SI Figs. 14–17).

3.1.3. NMR Spectroscopy

^{13}C NMR: The 1H and ^{13}C NMR experiments were carried

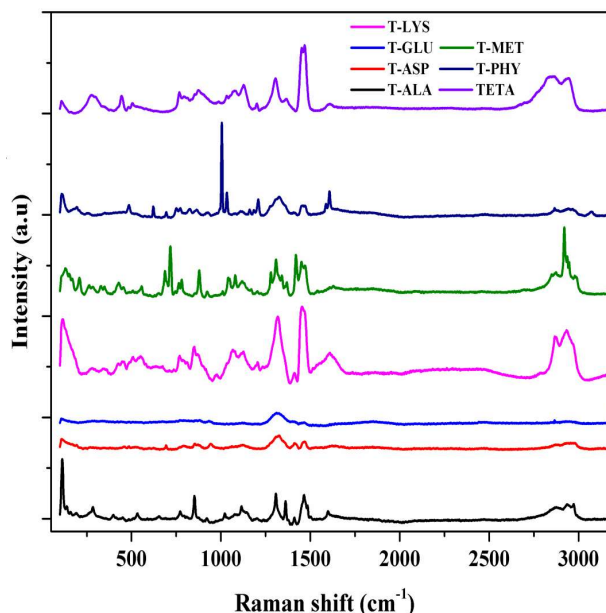


Fig. 6: Raman spectra of synthesized ILs $[TETA][LYS]$, $[TETA][GLU]$, $[TETA][ASP]$, $[TETA][ALA]$, $[TETA][MET]$, $[TETA][PHE]$, and cation $[TETA]^+$.

out at ambient temperature using a Bruker Avance 400 or 300 MHz spectrometer. NMR samples of different synthesized AAILs were prepared with DMSO- d_6 or D_2O . Precautions were taken while preparing the samples to avoid contamination; syringes or capillary tubes were used to transfer the NMR solvent or AAILs.

The peak assignments of ^{13}C NMR data for different synthesized AAILs are as follows (Fig. 7(a)):

Triethylenetetramine lysine [TETA][LYS]: δ_c (ppm) (300 MHz, DMSO- d_6), 177.96, 54.73, 52.34, 42.23, 36.58, 30.26, 24.66, 19.44.

Triethylenetetramine glutamine [TETA][GLN]: δ_c (ppm) (400 MHz, DMSO- d_6), 178.0, 56.13, 53.44, 44.90, 36.39, 27.51, 23.14.

Triethylenetetramine aspartic acid [TETA][ASP]: δ_c (ppm) (300 MHz, DMSO- d_6), 174.3, 53.95, 50.37, 44.53, 36.29, 34.05.

Triethylenetetramine glutamic acid [TETA][GLU]: δ_c (ppm) (300 MHz, DMSO- d_6), 177.45, 54.22, 50.08, 44.17, 36.08, 29.76, 24.15.

1H NMR: The peak assignments of 1H NMR data for different synthesized AAILs are as follows (Fig. 7(b)):

Triethylenetetramine lysine [TETA][LYS]: δ_H (ppm) (400 MHz, D_2O ; solvent peak observed at ~ 4.7): 3.4 (s, J

= 2.8 Hz, 1H), 2.8, 2.7 (s, J = 2.8 Hz, 1H), 1.59, 1.3 (s, J = 2.8 Hz, 1H).

Triethylenetetramine glutamine [TETA][GLN]: δ_H (ppm) (400 MHz, D_2O ; solvent peak observed at ~ 4.7): 4.0 (s, J = 2.8 Hz, 2H), 3.5 (s, J = 2.8 Hz, 2H), 2.73 (d, J = 2.8 Hz, 2H).

Triethylenetetramine alanine [TETA][ALA]: δ_H (ppm) (400 MHz, D_2O ; solvent peak observed at ~ 4.7): 3.4 (s, J = 2.8 Hz, 2H), 3.28 (s, J = 2.8 Hz, 2H), 2.60 (s, J = 2.8 Hz, 1H), 2.54 (s, J = 2.8 Hz, 1H).

Triethylenetetramine aspartic acid [TETA][ASP]: δ_H (ppm) (400 MHz, D_2O ; solvent peak observed at ~ 4.7): 3.6 (s, J = 2.78 Hz, 2H), 2.71 (s, J = 2.8 Hz, 2H), 2.44 (s, J = 2.8 Hz, 1H).

Triethylenetetramine glutamic acid [TETA][GLU]: δ_H (ppm) (400 MHz, D_2O ; solvent peak observed at ~ 4.7): 3.1 (s, J = 2.8 Hz, 2H), 2.82 (s, J = 2.8 Hz, 2H), 2.65 (s, J = 2.8 Hz, 1H).

The remaining ^{13}C NMR and 1H NMR spectra are provided in the supporting information (SI Figs. 2–13).

The FTIR, Raman, and 1H NMR analyses provide evidence of successful formation of TETA-based AAILs. Characteristic shifts and disappearance of peaks corresponding to free amino acids and unreacted amine

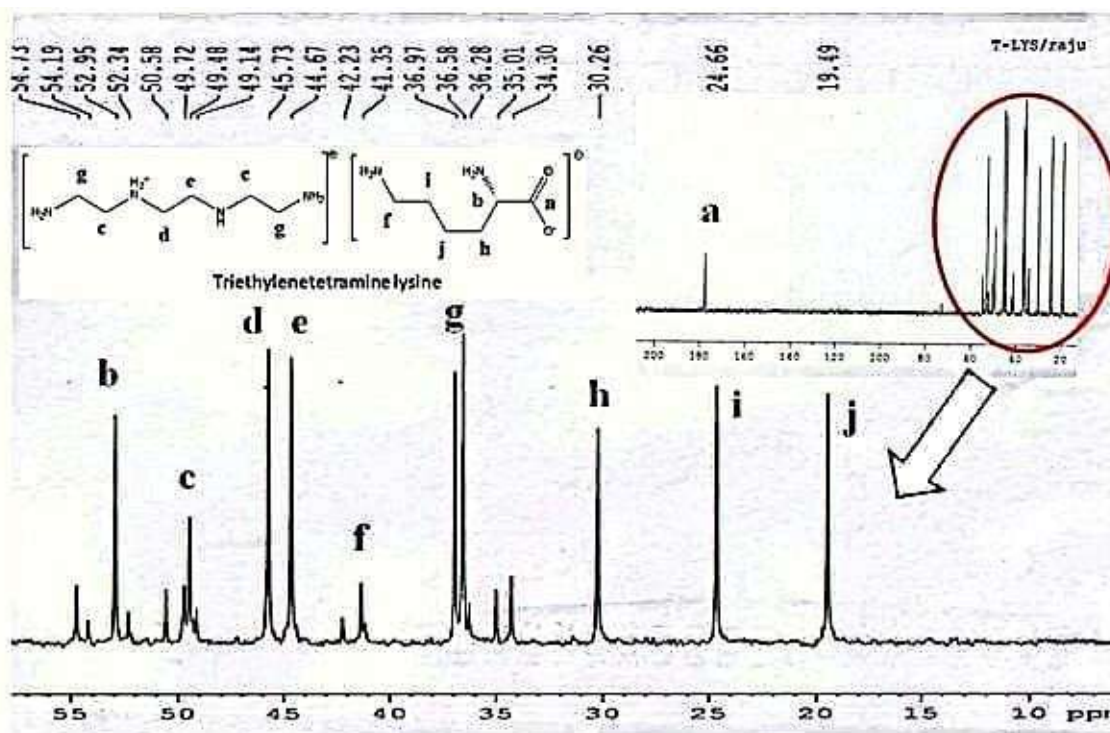


Fig. 7(a): Stack plot of ^{13}C NMR spectra of [TETA][LYS] with peak assignment.

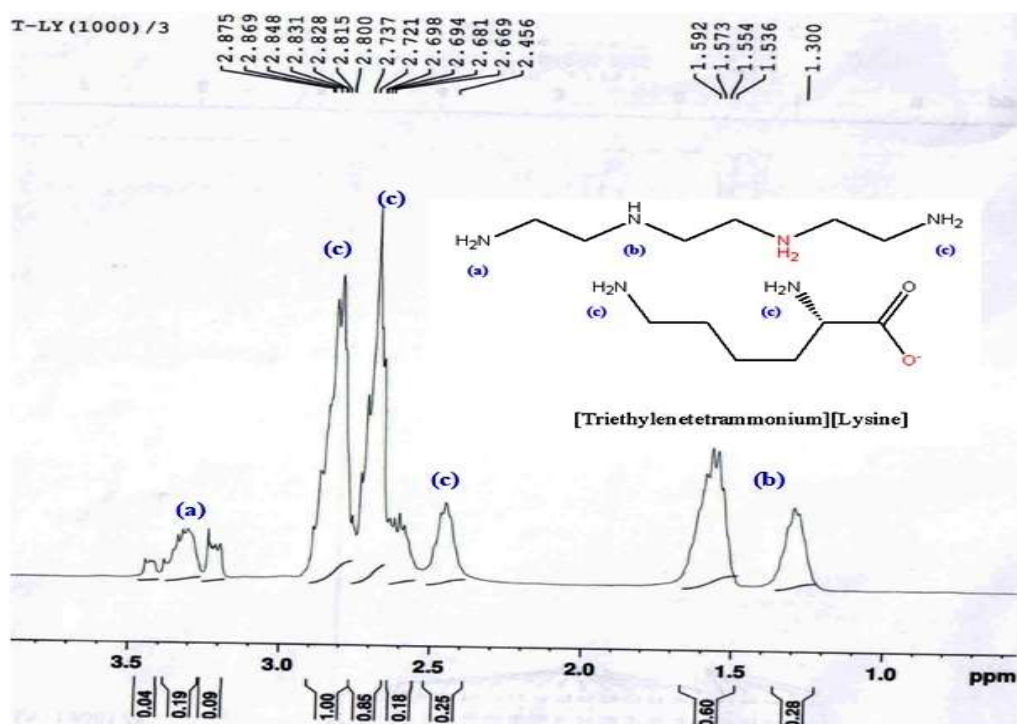


Fig. 7(b): Stack plot of ^1H NMR spectra of [TETA][LYS] with peak assignment.

groups indicate effective acid-base interaction and conversion. NMR peak assignments have been linked to the expected ionic structures, supporting the proposed formation of the AAILs. While quantitative ^1H NMR integration and 2D NMR were not performed, the combined spectroscopic data provide consistent evidence supporting ionic liquid formation, in line with previous reports on similar systems.

3.1.4. Density

Density measurements were performed using a 25 mL specific-gravity bottle. A known volume of AAIL was placed in the specific-gravity bottle and weighed on an analytical balance at room temperature, and the weights were recorded. The precision of the analytical balance was ± 0.0001 g. Density is equal to mass per unit volume; densities were calculated accordingly. Each sample was measured four times, and reproducibility was of a much better order of magnitude. The highest and lowest densities of 1.170 and 1.0646 g cm^{-3} were observed for [TETA][ASP] and [TETA][ALA], respectively, among the synthesized AAILs. Table 3 presents the experimentally measured densities of different AAILs at ambient conditions and atmospheric pressure.

3.1.5. Viscosity Measurements

Dynamic viscosity measurements were carried out for all synthesized ILs as a function of temperature (25, 35,

and 50°C) using a modular compact rheometer (Anton Paar MCR302 SN81193479) fitted with a Peltier plate at different temperatures and a 40 mm, 2° stainless-steel cone. Measurements were performed at angular velocities of 0.01 – 10 rad s^{-1} and shear rates of 0 – 200 s^{-1} under ambient conditions. The viscosity of the ILs decreased as temperature increased. An error of 5% was indicated by the manufacturer's data and analysis of standard liquids; however, reproducibility was better. For the ILs studied, viscosity remained moderately constant with increasing shear rate. This linearity between shear stress and shear rate corresponds to Newtonian behavior, as described by Huddleston et al. and Seddon et al. (Huddleston et al. 2001, Seddon et al. 2004).

Table 3: Experimentally measured densities of AAILs at room temperature and atmospheric pressure.

S. No.	Name of IL	Density (g cm^{-3})	Error
1	[TETA][GLU]	1.1523	0.00153
2	[TETA][ALA]	1.0646	0.00111
3	[TETA][PHE]	1.1468	0.00143
4	[TETA][ASP]	1.1709	0.00066
5	[TETA][LYS]	1.0702	0.00071
6	[TETA][GLN]	-	-
7	[TETA][MET]	-	-

Table 4: Experimentally measured viscosities of AAILs as a function of temperature and atmospheric pressure.

S. No.	Name of IL	Viscosities (Pa·s)		
		25°C	35°C	50°C
1	[TETA][ASP]	9.8	8.6	4.18
2	[TETA][GLN]	8.53	5.33	3.73
3	[TETA][GLU]	31.6	8.38	1.38
4	[TETA][ALA]	38.3	18.2	7.91
5	[TETA][LYS]	-	-	14.3
6	[TETA][MET]	-	-	-
7	[TETA][PHE]	-	-	-

The observed decrease in viscosity with increasing temperature can be attributed to weakening of hydrogen bonding and other intermolecular interactions within the TETA-based AAILs. ILs such as [TETA][GLU] and [TETA][ALA] exhibit higher viscosities at 25°C because of more extensive hydrogen-bonding networks, which are partially disrupted as temperature rises, resulting in a sharp decrease in viscosity. Compared with benchmark imidazolium- and phosphonium-based ILs and conventional aqueous amine solutions, the TETA-based AAILs maintain moderate viscosities at elevated temperatures, supporting efficient mass transfer and CO₂ absorption in FBR systems (Huddleston et al. 2001, Seddon et al. 2004).

However, density values are missing from Table 3 for [TETA][GLN] and [TETA][MET]. These measurements could not be obtained reliably because these AAILs were insufficiently stable for reproducible density measurements; difficulties in handling small sample volumes and the temperature sensitivity of these particular AAILs also contributed. Similarly, some viscosity data are missing from Table 4 for [TETA][LYS] (at 25 and 35°C), [TETA][MET], and [TETA][PHE], as these AAILs did not exhibit stable or consistent behavior under the measurement conditions. These issues will be addressed in future work.

3.2. CO₂ Absorption Experiments Using a Liquid Foam-Bed Reactor

The absorption studies were also conducted in a semi-batch liquid foam-bed reactor (FBR) to evaluate the effect of gas-liquid (G-L) interfacial contact area on the CO₂ absorption rate. The experimental data presented below were obtained at ambient temperature with a bed height of 30 cm. SLS was used with AAILs to generate foam in the FBR. The FBR experiments were carried out for about 30 min at a CO₂ flow rate of $2.5 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ and an air flow rate of $8.33 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$. Moles of CO₂ absorbed per mole of AAIL used were plotted against time. Several parameters, including pH,

initial concentration, surfactant effects, regeneration, and use of AAILs after regeneration for multiple cycles, were studied and reported.

Figs. 8–11 show the effect of different initial concentrations (500, 300, and 100 mmol) of [TETA][LYS], [TETA][GLU], [TETA][GLN], and [TETA][ASP] on CO₂ absorption, as well as the desorption performance of different AAILs and the use of regenerated AAILs for multiple cycles in the FBR. CO₂ uptake increased with increasing AAIL concentration up to a certain point because of the increased CO₂ absorption rate.

Fig. 8 shows the moles of CO₂ absorbed per mole of [TETA][LYS] used, with the highest mole ratios of 0.442, 0.172, and 0.112 observed at initial concentrations of 500, 300, and 100 mmol, respectively. After 30 min of experimentation, the FBR was saturated and reached equilibrium. Figs. 8–11 show that the highest mole ratio, 0.442, was observed for [TETA][LYS], followed by [TETA][GLN] (0.209) and [TETA][GLU] (0.208), among all AAILs used in this study. The highest mole ratio of CO₂ absorbed per mole of [TETA][LYS] used may be attributable to differences in the chemical and physical properties of AAILs based on their structures. In [TETA][LYS], the [LYS][−] anion contains two primary amine groups. Moreover, the higher CO₂ uptake in [TETA][LYS] is primarily attributed to the presence of multiple accessible amine groups and higher basicity, which enhance chemisorption capacity. The molecular structure also facilitates better accessibility of reactive sites, improving interaction with CO₂ molecules. Similarly, [TETA][GLN] contains two primary amine groups and gave the second-highest mole ratio of CO₂ absorbed per mole of [TETA][GLN]. A 500 mmol concentration of [TETA][LYS] gave higher CO₂ uptake because more [TETA][LYS] ions were available to react with CO₂. After 30 min, the available [TETA][LYS] ions were saturated because of the continuous passage of CO₂ and air through the FBR. As CO₂ uptake increased over time, the viscosity of [TETA][LYS] increased, which in turn decreased CO₂ diffusivity. During CO₂ absorption by the [TETA][LYS] solution, the initial carbamate changed to an intermediate form of carbonic acid, i.e., bicarbonate, which may contribute to the increased viscosity of [TETA][LYS]. Similar results were observed for [TETA][GLU], [TETA][GLN], and [TETA][ASP], as shown in Figs. 9–11.

In 2013, Xing et al. reported molecular simulation studies of CO₂ absorption that examined differences between amine-based cation ILs and AAILs in terms of the accessibility of amine groups in IL ions. These differences affect cation-anion interactions and their predicted behavior (Xing et al. 2013). Xing et al. also reported that stronger

interactions between amine-functionalized cations and anions result in smaller interfacial spaces, thereby reducing amine-group accessibility. In contrast, for AAILs, weaker anion-cation interactions make the amine group in the anion more accessible for CO₂ interaction (Xing et al. 2013). However, the influence of anion-cation interactions on amine accessibility is discussed based on literature reports and has not been directly validated in this study. Therefore, this effect is presented as a reasonable interpretation, and further spectroscopic or computational investigations are required for confirmation.

In this study, the new integrated approach combines the design of TETA-based amino acid ionic liquids (AAILs) with reactor engineering. The presence of multiple active amine sites in TETA enhances CO₂ absorption via chemisorption, while the liquid foam-bed reactor (FBR) provides a high gas-liquid interfacial area, enabling improved mass transfer. This combined strategy aims to maximize CO₂ absorption efficiency by leveraging both material properties and reactor performance.

Amines such as TETA used as cations in ILs absorb CO₂ through both chemical and physical absorption. The strength of physical absorption depends primarily on the type of anion used; in this case, the anions are AAs. Although [LYS] as an anion absorbed CO₂ appreciably by physisorption, [TETA][LYS] could attain the stoichiometric amount of CO₂ under the given experimental conditions because of limited accessibility of amine groups caused by anion-cation interactions. Figs. 8–11 indicate that chemisorption is the

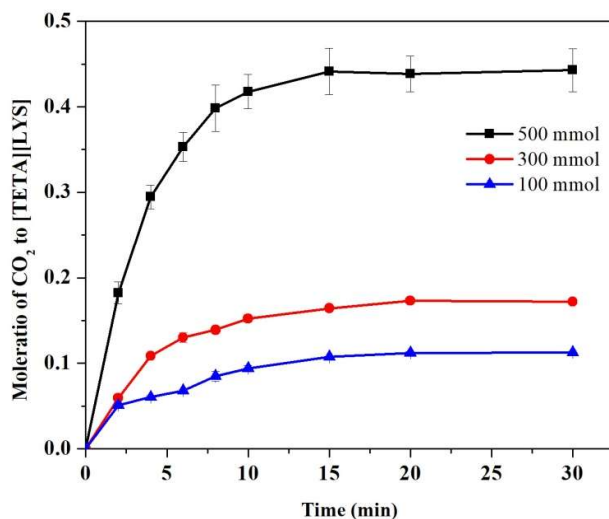


Fig. 8: Effect of initial concentration of [TETA][LYS] on CO₂ absorption. [Conditions: sample volume = 100 mL, SLS added = 0.1 g, initial pH = 9.6 (for 100 mmol), 9.46 (for 300 mmol), 9.63 (for 500 mmol), bed height = 30 cm, CO₂ flow rate = $2.5 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ (1.5 LPM), air flow rate = $8.33 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ (5 LPM), and temperature = $\sim 25^\circ\text{C}$].

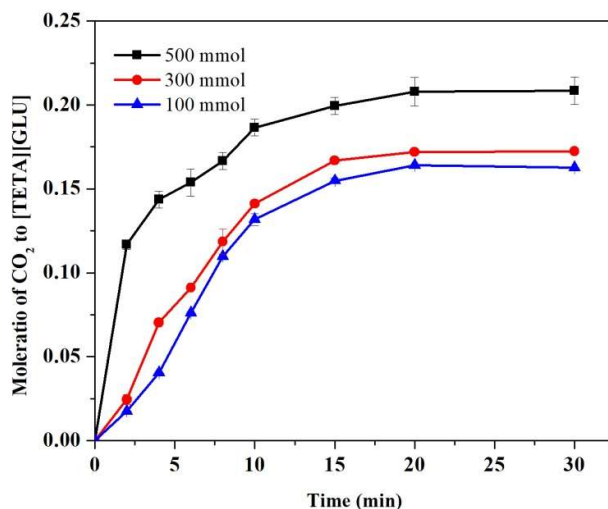


Fig. 9: Effect of initial concentration of [TETA][GLU] on CO₂ absorption. [Conditions: sample volume = 100 mL, SLS added = 0.1 g, initial pH = 7.4 (for 100 mmol), 7.46 (for 300 mmol), 7.55 (for 500 mmol), bed height = 30 cm, CO₂ flow rate = $2.5 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ (1.5 LPM), air flow rate = $8.33 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ (5 LPM), and temperature = $\sim 25^\circ\text{C}$].

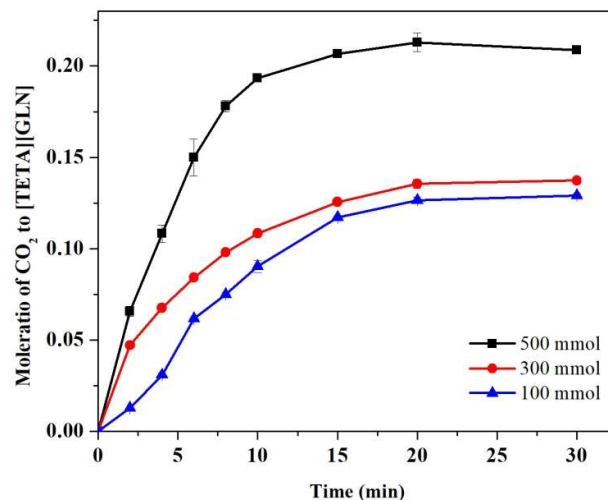


Fig. 10: Effect of initial concentration of [TETA][GLN] on CO₂ absorption. [Conditions: sample volume = 100 mL, SLS added = 0.1 g, initial pH = 8.04 (for 100 mmol), 8.3 (for 300 mmol), 9.35 (for 500 mmol), bed height = 30 cm, CO₂ flow rate = $2.5 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ (1.5 LPM), air flow rate = $8.33 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ (5 LPM), and temperature = $\sim 25^\circ\text{C}$].

predominant mechanism for the AAILs. As reported in the literature, nonfunctionalized ILs such as [bmim][BF₄] show lower CO₂ molar uptake through physical absorption (Yamini & Khanna 2015).

The results of CO₂ absorption in the FBR were compared with those obtained in a bubble-column reactor in which no surfactant was used. In both cases, equal liquid volumes were used for the experiments. CO₂ absorption through chemical

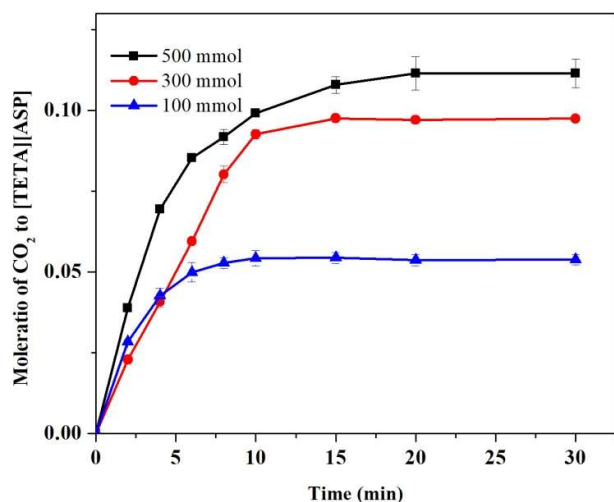


Fig. 11: Effect of initial concentration of [TETA][ASP] on CO₂ absorption. [Conditions: sample volume = 100 mL, SLS added = 0.1 g, initial pH = 7.4 (for 100 mmol), 7.46 (for 300 mmol), 7.55 (for 500 mmol), bed height = 30 cm, CO₂ flow rate = $2.5 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ (1.5 LPM), air flow rate = $8.33 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ (5 LPM), and temperature = $\sim 25^\circ\text{C}$].

reactions was described by Bhaskarwar and Kumar, who reported that the FBR provides a larger contact area than a bubble-column reactor (Bhaskarwar & Kumar 1984). In the present study, the average CO₂ absorption rate in the FBR was 20% greater than that in the bubble column.

The enhanced CO₂ absorption observed in the FBR compared to the semi-batch equilibrium system (SBES) is attributed to the synergistic effect of material properties and reactor hydrodynamics. The intrinsic characteristics of TETA-based AAILs, including amine functionality, viscosity, and reactivity, significantly influence their performance under different reactor conditions. The FBR system effectively utilizes these properties by increasing interfacial contact, leading to superior absorption performance.

3.3. Effect of Surfactant Concentration

In this work, different surfactants, including sodium lauryl sulfate (anionic), cetrimonium bromide (cationic), Triton X-100 (nonionic), and Tween 80 (nonionic), were used in the FBR to generate foam. The most stable foam and highest CO₂ uptake were observed with sodium lauryl sulfate. Triton X-100 and Tween 80 produced very poor and unstable foam when mixed with AAILs during FBR experiments. All surfactants were used at concentrations selected on the basis of their critical micelle concentration (CMC) values. The experiments showed that adding more surfactant decreased the absorption rate. Below the CMC value (8.2 mM) of SLS, however, the high-concentration resistance decreases and the diffusion flux of CO₂ uptake increases. CO₂ absorption increases with surfactant concentration up to the CMC, after

which further addition may reduce mass-transfer efficiency because of excessive foam stabilization. Quantitative measurements of interfacial area and foam stability were not performed. This limitation is noted, and quantitative characterization remains a subject for future investigation.

Previously, Roy et al. (1995) explained that when surfactant concentration was increased, the solubility of CO₂ in the surfactant solution increased because of micellar solubilization (Roy et al. 1995). Nonetheless, the amount of dissolved and unreacted CO₂ in the foam film is extremely low in a gas-liquid reacting system. This could increase the potential for CO₂ solubility. However, as the surfactant concentration increased, more surfactant molecules surrounded the gas-film interface, resulting in the formation of several layers. This left a smaller interfacial surface available for the foam liquid to absorb CO₂. Nevertheless, at surfactant concentrations greater than the CMC, the micelles adopted cylindrical shapes, which provided some free space for CO₂ absorption, and the absorption rate marginally improved.

3.4. Semi-Batch Equilibrium Experiments

Semi-batch equilibrium experiments on CO₂ absorption were carried out at room temperature for more than 5 h. [TETA][GLU], [TETA][GLU], and [TETA][ASP] AAILs at a concentration of 100 mmol and a volume of 200 mL were selected for the equilibrium experiments. A continuous supply of pure CO₂ was bubbled through the solutions with stirring. After the equilibrium experiment, the AAILs were regenerated and used for further cycles.

Figs. 12–14 describe the equilibrium experiments on CO₂ absorption and desorption for [TETA][GLU], [TETA][ALA], and [TETA][ASP]. These studies indicated that the reaction involving deprotonation by the water molecule is slow. The experiments showed that the moles of CO₂ absorbed per mole of AAIL used were lower than in the FBR experiments because of the smaller interfacial area and the slow reaction involving deprotonation by the water molecule. As shown in Figs. 12–14, the moles of CO₂ absorbed per mole of AAIL increased slightly with time and reached equilibrium after about 4–5 h. The ionic liquid was saturated with CO₂ after completion of the absorption procedure. As explained in the experimental section, desorption investigations were also conducted after the experiment. The IL weight was recorded before the absorption cycle to ensure proper desorption of CO₂ from the IL. When all CO₂ in the CO₂-IL system had decomposed, the IL weight remained constant. CO₂ was first absorbed into the IL until absorption equilibrium was reached and was then desorbed from the CO₂-rich IL system. This constituted an absorption-desorption cycle.

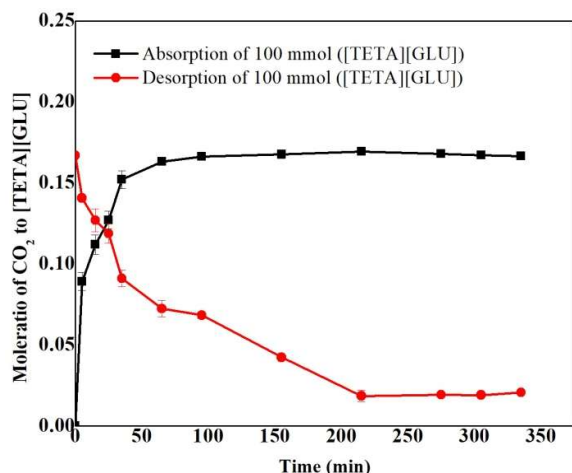


Fig. 12: Plot of absorption and desorption of [TETA][GLU] during CO₂ absorption. [Conditions: sample volume = 200 mL, RBF volume = 500 mL, initial pH = 7.4 (for 100 mmol), CO₂ flow rate = $2.5 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ (1.5 LPM), stirring speed = 80 rpm, and temperature = $\sim 25^\circ\text{C}$].

During desorption, the moles of CO₂ absorbed per mole of AAIL decreased with time and reached equilibrium after 4 h. As shown in Fig. 12, the highest mole ratio of CO₂ per mole of [TETA][GLU] was 0.166, followed by 0.133 for [TETA][ALA], as shown in Fig. 14. Desorption studies were plotted on the same figures; the mole ratio of CO₂ per mole of [TETA][GLU] decreased from 0.124 to 0.025, while that for [TETA][ALA] decreased from 0.12 to 0.013, as shown in Figs. 12 and 14, respectively. The lowest CO₂ uptake, 0.07, was observed for [TETA][ASP], as shown in Fig. 13. The used [TETA][GLU] was subjected to another experimental cycle, as shown in Fig. 15. Fig. 15 shows that [TETA][GLU] in the first cycle gave the highest CO₂ mole

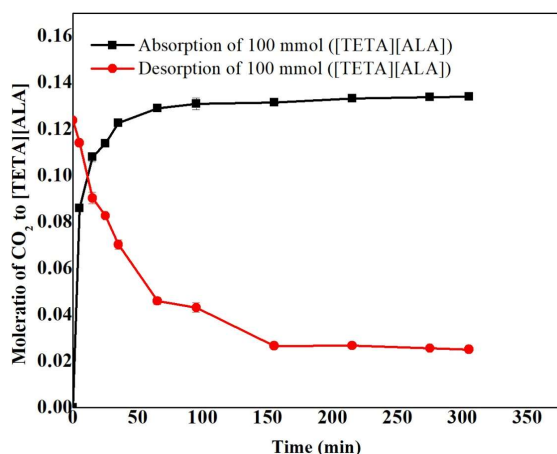


Fig. 14: Plot of absorption and desorption of [TETA][ALA] during CO₂ absorption. [Conditions: sample volume = 200 mL, RBF volume = 500 mL, initial pH = 7.9 (for 100 mmol), CO₂ flow rate = $2.5 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ (1.5 LPM), stirring speed = 80 rpm, and temperature = $\sim 25^\circ\text{C}$].

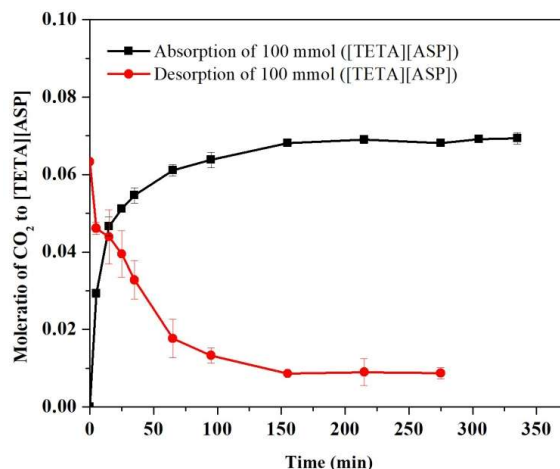


Fig. 13: Plot of absorption and desorption of [TETA][ASP] during CO₂ absorption. [Conditions: sample volume = 200 mL, RBF volume = 500 mL, initial pH = 7.5 (for 100 mmol), CO₂ flow rate = $2.5 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ (1.5 LPM), stirring speed = 80 rpm, and temperature = $\sim 25^\circ\text{C}$].

ratio of 0.166 per mole of AAIL used; in the second cycle, the CO₂ mole ratio was 0.121 per mole of [TETA][GLU]. This experiment shows that AAILs can be used for multiple cycles after regeneration.

4. CONCLUSIONS

This work describes the synthesis and characterization of AAILs for CO₂ absorption in an FBR. About seven new ILs, namely [TETA][ALA], [TETA][LYS], [TETA][GLU], [TETA][GLN], [TETA][MET], and [TETA][PHE], were synthesized. The prepared AAILs were characterized

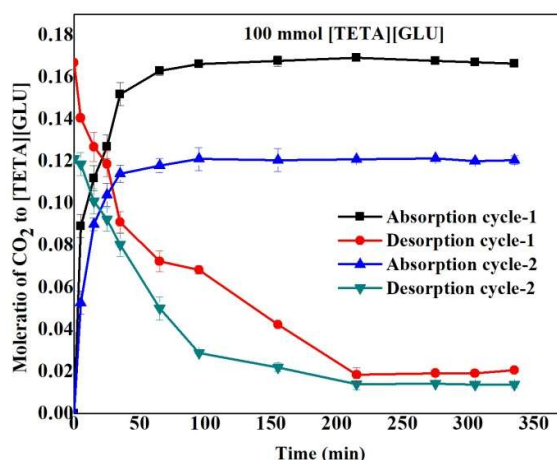


Fig. 15: Plot of absorption and desorption of [TETA][GLU] for two different cycles during CO₂ absorption. [Conditions: sample volume = 200 mL, RBF volume = 500 mL, initial pH = 7.4 (for 100 mmol), CO₂ flow rate = $2.5 \times 10^{-5} \text{ m}^3 \text{ s}^{-1}$ (1.5 LPM), stirring speed = 80 rpm, and temperature = $\sim 25^\circ\text{C}$].

using FTIR, Raman spectroscopy, and ^1H and ^{13}C NMR spectroscopy to confirm their formation. Parameters such as initial concentration, surfactant effects, and pH were studied. SLS was the most suitable surfactant for the FBR experiments. Viscosity and density measurements were carried out to characterize the physical properties of the AAILs. The application of AAILs in the FBR for CO_2 absorption was evaluated under different experimental conditions. The highest and lowest densities of 1.170 and 1.0646 g cm^{-3} were observed for [TETA][ASP] and [TETA][ALA], respectively, among the synthesized AAILs. Viscosity measurements showed that AAIL viscosity decreased with increasing temperature. [TETA][LYS] gave the highest moles of CO_2 absorbed per mole of [TETA][LYS] used; the highest mole ratios of 0.442, 0.172, and 0.112 were observed at initial concentrations of 500, 300, and 100 mmol, respectively, among the AAILs used. Regeneration studies were also carried out to evaluate multiple uses of AAILs for CO_2 absorption. The highest mole ratio of CO_2 per mole of [TETA][GLU] was 0.166, followed by 0.133 for [TETA][ALA], during desorption studies. Data from the FBR and semi-batch equilibrium studies suggest that amino acids combined with amines are suitable candidates for CO_2 absorption compared with other ionic liquids. The prepared AAILs are biodegradable, and the synthesis procedure was straightforward and can be conducted at low cost compared with conventional commercial ionic liquids.

5. ACKNOWLEDGEMENT

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