Effect of Arginine-Based Deep Eutectic Solvents on Supported Porous Sorbent for CO_2 Capture Analysis

(Kesan Pelarut Eutektik Dalam Berasaskan Arginina pada Bahan Penjerap Poros Berpenyokong untuk Analisis Penangkapan CO_2)

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ABSTRACT

Carbon dioxide (CO_2) as one of the heat-trapping gases, has caused global warming. Being a greener and more economical material, amino acid-based deep eutectic solvents (AADES) have attracted interest in CO_2 capture applications. In this paper, the effect of L-arginine (Arg) in binary AADES of arginine-ethylene glycol (Arg-EG) and ternary AADES of choline chloride-ethylene glycol-arginine (ChCl-EG-Arg) on adsorption of CO_2 was studied. The solubility, basicity, and physicochemical characteristics were compared with the binary DES (ChCl-EG) before and after being impregnated into a silica gel (SG) via the wet impregnation method. The AADES/SG adsorbents were evaluated for CO_2 sorption performance using an automated gas sorption analyzer at 100% CO_2 loading and thermogravimetric analysis (TGA) at flue gas conditions (15% CO_2/85% N_2). Findings show the basicity and the nitrogen content (N%) of AADES/SG were increased as Arg was added and DES/AADES functional group peaks (amino, hydroxyl, alkyl groups) were observed after the impregnation. The CO_2 sorption of 16.0 mg/g at 25 °C and 1 atm was achieved by 30% Arg-EG(1:8)/SG followed by 30% ChCl-EG-Arg (1:2:0.1)/SG (14.8 mg/g) and 30% ChCl-EG/SG(1:2) (14.5 mg/g) using an Autosorb iQ$_2$ instruments with 100% CO_2 loading. The CO_2 uptake was increased almost linearly with increasing pressure and decreased with increasing temperature. The Arg-EG(1:8)/SG shows the highest selectivity toward CO_2 than other sorbents with 8.10 mg/g adsorption for 1 h at 15% CO_2 loading at 25 °C with higher thermal stability and surface area. Considering environmental, technological, and economic viewpoints, the Arg-EG(1:8)/SG can be explored more as a potential solid sorbent for CO_2 capture.

Keywords: Amino acid; carbon dioxide adsorption; deep eutectic solvent; silica sorbent; wet-impregnation

ABSTRAK

Karbon dioksida (CO_2) sebagai salah satu gas perangkap haba telah menyebabkan pemanasan global. Sebagai bahan yang lebih hijau dan lebih menjimatkan, pelarut eutektik dalam berasaskan asid amino (AADES) telah menarik minat dalam aplikasi penangkapan CO_2. Dalam kajian ini, kesan L-arginina (Arg) dalam AADES binari bagi arginina-etilena glikol (Arg-EG) dan ternari AADES bagi klorida kolina-etilena glikol arginina (ChCl-EG-Arg) terhadap penjerapan CO_2 telah dikaji. Kelarutan, kebesan dan ciri-ciri fizikokimia sebelum dan selepas diisi ke dalam gel silika (SG) melalui kaedah pengisitepuan basah telah dibandingkan dengan binari DES (ChCl-EG). Penjerap AADES/SG telah dinilai untuk prestasi penjerapan CO_2 menggunakan analisis penjerapan gas automatik pada 100% muatan CO_2 dan analisis termogravimetri (TGA) pada keadaan gas serombong (15% CO_2/85% N_2). Penemuan menunjukkan

bahawa kebesan dan kandungan nitrogen (N%) AADES telah meningkat apabila Arg ditambah dan puncak kumpulan berfungsi AADES (kumpulan amino, hidroksi dan alkil) telah diperhatikan selepas pengisitepuan. Penjerapan CO_2 sebanyak 16.0 mg/g pada 25 °C dan 1 atm telah dicapai oleh 30% Arg-EG(1:8)/SG diikuti oleh 30% ChCl-EG/SG(1:2) (14.5 mg/g) menggunakan instrument Autosorb iQ_2 dengan 100% muatan CO_2. Penjerapan CO_2 meningkat secara linear dengan peningkatan tekanan dan berkurang dengan peningkatan suhu. Arg-EG(1:8)/SG menunjukkan kepilihan tertinggi terhadap CO_2 berbanding dengan penjerap lain dengan penjerapan sebanyak 8.10 mg/g selama 1 jam pada 15% muatan CO_2 pada 25 °C dengan kestabilan termal dan kawasan permukaan yang lebih tinggi. Mengambil kira sudut alam sekitar, teknologi dan ekonomi, Arg-EG(1:8)/SG boleh diteroka lebih lanjut sebagai penjerap pepejal yang berpotensi untuk penangkapan CO_2.

Kata kunci: Asid amino; pelarut eutektik dalam; pengisitepuan basah; penjerapan karbon dioksida; penjerap silika

INTRODUCTION

The heat-trapping gases such as carbon dioxide (CO_2) increased by anthropogenic activities had caused global warming worldwide. Based on the data from Earth System Research Laboratory ESRL (2022), the trend of global atmospheric CO_2 concentration in May 2022, increased from 280 ppm in its pre-industrial value to 418.43 ppm, which is considered to be the leading contributor to the changes in global climate conditions. The CO_2 absorption by aqueous alkanolamines such as monoethanolamine (MEA) has been applied to post-combustion carbon capture for so many years and became one of the most effective commercialized technologies for CO_2 capture. However, the problem with this technique is that a considerable amount of energy is required to regenerate the solvent, corrosive and solvent degradation (Li et al. 2019; Mirza et al. 2017) and other major drawbacks of these organic solvents include high toxicity, volatility and not environmentally friendly (Wang et al. 2017). To overcome these drawbacks, it is critical to develop promising new sorbent materials for greener, more efficient, and cost-effective CO_2 capture.

Recently, researchers have started using deep eutectic solvent (DES) for CO_2 capture analysis and separation (Ariyanto et al. 2021; Ghazali et al. 2019). DES is a solvent composed of a mixture of two or more components that forms a eutectic with a lower melting point than individual components. DESs have more advantages in comparison to ionic liquids (ILs) because they are easily synthesized and much cheaper than ILs (Chemat et al. 2016a). Furthermore, DESs can be tailored according to desired applications. Depending on the compounds used to produce DES, CO_2 absorption in DESs can be physical, chemical, or mixed (Sarmad et al. 2017). Amine and amino acids (AAs) have been used as additional components for DES and ILs in recent study to improve their ability for CO_2 capture (Chemat et al. 2016b; Ghazali et al. 2020a; Haider et al. 2018; Li et al. 2019).

AAs possess the characteristic and functional group similar to the amines and are expected to perform a similar task for CO_2 capture. Based on Song et al. (2012), due to the presence of identical amino functional groups ($-NH_2$) in their molecules, the reactivities of AA salts are similar to aqueous alkanolamines. AAs display several advantages in comparison to conventional amines, such as high biodegradability and surface tension, no formation of toxic intermediate, high resistance to thermal and oxidative degradation, low volatility, and fewer corrosion concerns (Mohamed Hatta et al. 2022). The addition of basic AAs such as L-arginine (Arg), which has an abundance of amine functional groups on the structure is thought to improve the CO_2 selectivity and thermal stability of eutectic solvents.

Several studies have been using amino acid-based DES (AADES) for the liquid absorption study of CO_2 gas. Chemat et al. (2016b) synthesized (ChCl + Glycerol) with Arg and they found out Arg improved the solubility of CO_2 in prepared mixtures. Ren et al. (2018) have synthesized a series of L-arginine/Glycerol from 1:4 to 1:8 molar ratio and discovered that L-Arg/Gly can be reused 5 times without an obvious decrease in their CO_2 absorption capacities. Mostly, DES solvents gave high viscosities that influence the rate of absorption, thus reducing their applications in industry. To overcome this problem, AADES was impregnated onto a porous silica gel (SG) for CO_2 capture technology. However, based on the record, there is very few studies have reported on amino acid-based DES impregnated on porous solid adsorbent for CO_2 capture analysis but mainly using liquid absorption studies.

There are several techniques to immobilize an organic amine onto porous surfaces such as; (i) impregnation (Ghazali et al. 2020a; Uehara, Karami & Mahinpey 2019) (ii) post-grafting (Mohd et al. 2021; Saha 2018) and (iii) direct co-condensation (Kim et al. 2008; Madden et al. 2016). Immobilization of amine functionalities onto porous support material with a high surface area will give an attractive option for great combination features from both solvent systems and solid sorbents. Furthermore, there are several advantages of the amino group's immobilization onto the solid supports such as their toxicity being reduced and thus reducing their corrosive nature (Saha 2018). In the present study, a porous silica gel material was used as an inexpensive support.

Jiang et al. (2013) are among the first researchers to carry out the development of amino acid and amino acid complex-based solid sorbents for CO_2 capture. They reported that Arg-impregnated PMMA sorbents solid sorbents had the highest CO_2 adsorption capacity. Recently, Lian et al. (2021) have utilized AADES and Pebax 1657 to develop a defect-free asymmetric composite membrane for an efficient CO_2 capture technology. Based on their findings, the CO_2/N_2 selectivity of the AADES-based composite membrane is increased by 17% as compared to the neat Pebax while less than 0.6% of the CO_2 permeability is reduced.

Therefore, in this paper, the effect of the addition of Arg on the DES ability to capture CO_2 will be evaluated after being impregnated onto porous silica gel sorbents. Besides that, the effect of using Arg as a hydrogen bond acceptor (HBA) in binary AADES was also compared with the binary DES and ternary AADES. The better composition of AADES-solid sorbent that shows good thermal stability, high porosity and better CO_2 capacity will be selected as promising sorbents. This study investigated and focus on non-aqueous solvents (deep eutectic solvents/amino acids) and solid adsorbents (silica gel) that can absorb/adsorb CO_2 in flue gas composition.

MATERIALS AND METHODS

PREPARATION OF SAMPLES

Wakogel C-300 HG (porous silica gel) was supplied by FUJIFILM Wako Pure Chemical Corporation, Osaka, Japan with particle size in the range of 0.200-0.063 mm. Amino acid-based DES solutions (AADES) have been prepared using the hydrogen bond acceptor (HBA) and hydrogen bond donor (HBD) at several molar ratios.

Choline chloride (ChCl) and ethylene glycol (EG) were purchased from Tokyo Chemical Industry (TCI) Co. LTD. Furthermore, L-arginine (Arg) was obtained from Sigma Aldrich Fine Chemicals (SAFC) Company and ethanol was acquired from FUJIFILM Wako Pure Chemical Corporation, Japan. The chemicals used to prepare AADES mixtures were shown in Table 1 and were used as received without further purification.

PREPARATION OF AMINO ACID BASED DEEP EUTECTIC SOLVENTS

In order to prepare AADESs, several combinations of HBA and HBD were prepared at several molar ratios as listed in Table 2. The HBA and HBD are selected based on references Chemat et al. (2016a) and Ren et al. (2018). The preparation procedure is made accordingly with slight modifications. Since Arg molecule contain both amino and carboxyl group in its structure, amino group can acts as HBD while carboxyl group can act as HBA depending on their ionization state and interacting group. For the preparation of binary AADES without choline chloride, the Arg will act as HBA and be mixed with HBD (ethylene glycol) at a 1:8 molar ratio. The mixture was heated at 70-80 °C and continuously stirred in a airtight vial until a clear-homogenous solution was formed. The 1:8 molar ratio was chosen after screening on their solubility and stability during preparation. The mixture was heated at 80 °C until a stable and homogeneous colourless liquid was formed.

In ternary AADESs, the mixture of ChCl-EG was prepared using the method reported by Abbott et al. (2003) with slight modification. The proper quantities of 1:2 molar ratio of the mixture were put in a well-sealed vial. The vial was then placed on a hot plate and stirred at around 80 °C for about 1 hour until a homogeneous colourless liquid was formed. The resulting DES was left to cool at room temperature before being mixed with several molar ratios of Arg (0.05, 0.10, and 0.15) in airtight vials which were immersed in an oil bath at 80 °C. In ternary AADES, since the ChCl-EG was mixed prior to the addition of Arg, Arg acts as HBD since it has an amino group in its side chain which can form hydrogen bonding with the ChCl-EG mixture. The mixtures were constantly stirred at 200-300 rpm with a magnetic stirrer until homogenous liquids were formed. All synthesized mixtures were left overnight and kept in sealed glass vials before being further used. All types of AADESs synthesized were labelled as ChCl-EG, Arg-EG, and ChCl-EG-Arg.

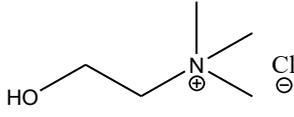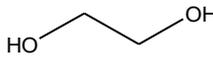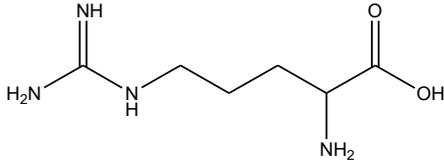TABLE 1. DES and AADES's composition and their chemical structure

Choline chloride (ChCl)	139.62 g/mol	
Ethylene glycol (EG)	62.07 g/mol	
L-Arginine (Arg)	174.2 g/mol	

TABLE 2. List of prepared DES solvents with their molar ratio and respective hydrogen bond donor and acceptor

DES	Hydrogen bond acceptor (HBA)	Hydrogen bond donor (HBD)		Molar ratio (HBA:HBD)		
Arg-EG	Arg	EG	-	1:8		
ChCl-EG	ChCl	EG	-	1:2		
ChCl-EG-Arg	ChCl	EG	Arg	1:2:0.05	1:2:0.10	1:2:0.15

PREPARATION OF ADSORBENT

Porous silica gel (SG) was dried prior to usage. The DES and AADES functionalized SG was prepared by the wet-impregnation method with several modifications. About 0.77 g of AADES solvent was mixed with 3.60 g of ethanol for 15 min. Next 1.80 g of SG was added to the mixed solution. The AADES/SG mixtures were mixed and sonicated for five minutes and continuously stirred for 2 h in a closed system. The resulting slurry mixture was left to dry. The mass ratio of SG/ethanol was maintained constant at 1:2 for each sample while AADES/ethanol ratio was varied for each loading. The resultant adsorbents were kept in a sealed bottle and stored in a desiccator before further use.

CHARACTERIZATION

Before the impregnation, the appearance and solubility of the prepared DES and AADES mixtures were observed. Their basicity was analyzed using the AS600 pH meter. The characterizations of impregnated silica gel with DES and AADES are done to determine the additional effect of Arg functionalities and different DES compositions on their surface morphology, thermal stability, and CO_2 adsorption capacity. The Perkin Elmer Attenuated Total Reflectance-Fourier Transform Infrared (ATR-FTIR) spectrometer was used for recording the FTIR spectra of the DES and AADES before and after impregnation at wavenumber range: 450-4000 cm^{-1}. The elemental analyzer MICRO CORDER JM10 (J-science Lab

Co., Ltd., Kyoto, Japan) is used to analyze the C, H, and N content in the samples.

Thermal stabilities of all the samples were analyzed under air atmosphere using SII EXSTAR 6000 TG/DTA 6300 thermogravimetric analyzer (TGA). The samples were heated at the temperature range from 30 °C to 500 °C with a heating rate of 10 °C/min at 100 mL/min flow rate. An automated gas sorption analyzer (Autosorb iQ_2 Quantachrome Instruments, Germany) was used to measure the surface areas, pore volume and pore size distribution of the prepared sorbents by the Brunauer-Emmet-Teller (BET) and Barrett-Joynes-Halenda (BJH) methods, respectively. Prior to each N_2 adsorption-desorption analysis, the samples were dried overnight using a vacuum oven at 80 °C and degassed at 100 °C for 300 min to remove excess moisture and adsorbed gas.

THE CO_2 ADSORPTION ISOTHERM

To analyze the CO_2 adsorption isotherm of the prepared sorbents, an automated gas sorption analyzer (Autosorb iQ_2 Quantachrome Instruments) was used with the same degas condition as mentioned in the surface area characterization. About 0.06-0.08 g of sorbents were used for the evaluation. The liquid N_2 was replaced with the pure 100% CO_2 gas and the temperature was set at 25 °C and atmospheric pressure for the adsorption process analysis. Adsorption capacity in (mg/g) was calculated as equation (1);

$$\text{Adsorption capacity} = \frac{\text{Adsorption volume (STP)mL}}{g} \times \frac{1\,l}{1000\,mL} \times$$

$$\frac{1\,mol}{22.4\,l} \times \frac{44\,g}{1\,mol} \times \frac{1000\,mg}{1\,g} \qquad (1)$$

THE CO_2 SORPTION CAPACITY USING TGA (15% CO_2 ANALYSIS)

The CO_2 selectivity of the sorbents over N_2 was evaluated using TGA under 15% CO_2/85% N_2 loading. The mixed gas was used to stimulate real flue gases condition emitted from coal and gas-fired power plants. About 12 mg of dried 30% AADES/SG adsorbent was placed into the TGA aluminium pan in the thermobalance and was pre-treated to 120 °C under 100% N_2 atmosphere at a flow rate of 100 mL/min for 180 min. The temperature was then reduced to 25 °C and held for about 60 min before the feed gas was changed to 15% CO_2/85% N_2 mixed gases. The CO_2 capture analyses at 25 °C was then conducted in the mixed gases which were supplied at a

100 mL/min flow rate for about 60 min. The adsorption capacity was calculated from the weight gained after CO_2 exposure and was expressed in mg of CO_2 per g of adsorbent. Afterwards, the CO_2 desorption was performed by introducing 100% N_2 gas into the TGA sample cell at 100 °C for 60 min.

RESULTS AND DISCUSSION

THE PROPERTIES OF THE AADES

According to Abbot et al. (2003), the eutectic solvents must be a homogeneous, clear and liquid phase at room temperature. For the preparation, the solvents must be mixed less than 120 min, heated to less than 100 °C and no solid precipitation after cooled at room temperature overnight. As shown in Table 3, the colours of the ternary ChCl-EG-Arg mixture changed from colourless to transparent orangish yellow after the addition of Arg into binary ChCl-EG and the eutectic mixture is stable up to a 0.10 molar ratio. However, the ChCl-EG-Arg cannot form a eutectic mixture at a (1:2:0.15) molar ratio. The inhomogeneous liquid formed at 0.15 molar ratio of Arg might be due to the saturation as the chloride ion (Cl⁻) is unable to form hydrogen bonding with excess HBD. Therefore the selected molar ratio of Arg used in the system is up to 0.10 for this study. Besides, the 1:2 molar ratio composition for Arg-EG has been prepared for comparison with ChCl-EG, however the mixture not forming a eutectic mixture might be due to insufficient HBD to form hydrogen bonding with Arg. Thus the 1:8 molar ratio composition is chosen after screening on their solubility and pH value. The eutectic point can be achieved by mixing the components in a specific ratio that is determined by their individual melting points and solubilities. The interactions of hydrogen bonding and van der Waals prevent the starting materials from crystallizing, resulting in the mixtures remaining in a liquid state (Francisco, van den Bruinhorst & Kroon 2013).

The pH of the amino acid-based DES is shown in Table 4. The pH value of the amino acid-based DES is important because both the chemical forms and distribution of amino and carboxylic groups in AA depend on it (Uehara, Karami & Mahinpey 2017). In this study, the pH for ChCl-EG is 8.10, while the AADESs's pH values were found to be more alkaline (11.84) for ternary ChCl-EG-Arg and 11.63 for binary Arg-EG, respectively. The increase in pH value is probably caused by the presence of the basic side chain from the Arg. As CO_2

is a Lewis acid, the introduction of Lewis bases might increase the material's affinity toward CO_2 (Petrovic, Gorbounov & Masoudi Soltani 2021) and contributes to better CO_2 sorption by acid-base interaction.

The FTIR spectra of Arg, ChCl, prepared DES and AADES are shown in Figure 1. The hydrogen bonding between the HBA with HBD is the main factor for the formation of DESs. Based on the spectrums, the broad IR band at 3330 cm^{-1} and 3300 cm^{-1} can be assigned to the O-H stretching from ChCl and EG, respectively (Delgado-Mellado et al. 2018). In the binary AADES (Arg-EG), the existence of Arg gives the N-H stretching vibration peak at 3170 cm^{-1} overlapped with the O-H peak resulting in the broadening of the peak. A similar trend has also been observed by Zhu et al. (2020). The slight broadening effect happened due to the hydrogen bonding interaction between -OH and –NH for Arg-EG. While for ternary AADES (ChCl-EG-Arg), the amount of Arg added is not significant for the -NH peak to appear. Peaks at 2940-2870 cm^{-1} are shown in all samples that corresponded to the CH_2 and CH_3 stretching modes while the peak at 1210 cm^{-1} refers to C-O-H bending (Delgado-Mellado et al. 2018). Additional peaks around 1669-1571 cm^{-1} attributed to the bending of primary and secondary amine peaks from Arg. The band at 1480 cm^{-1} and 1080 cm^{-1} are associated with CH_2 bending peak (Ghazali et al. 2020a) and CN vibration from ChCl (Sivrikaya 2019). In addition, carboxyl group stretching vibration can be seen in 1410 cm^{-1} for the Arg-EG mixture (Lian et al. 2021). In comparison with the raw materials (Arg, EG and ChCl), the presence of those characteristic peaks in the AADES spectrum indicates that the desired AADES was obtained.

TABLE 3. Effect of Arg's molarity on the appearance of prepared ternary AADES

DES	ChCl:EG	ChCl:EG:Arg		
Arginine molar ratio	0	0.05	0.10	0.15
Appearance of DES				

TABLE 4. The pH value of prepared DES and AADES solvents

DES	Molar ratio	pH
Arg-EG	1:8	11.6
ChCl-EG	1:2	8.1
ChCl-EG-Arg	1:2:0.10	11.8

CHARACTERIZATION OF IMPREGNATED SG

The chemical compositions in terms of carbon (C), hydrogen (H) and nitrogen (N) contents in the samples are shown in Table 5. Based on this CHN data, the contents of N in the samples were increased from 0 to 2.25% of N after the wet impregnation with DES and AADES mixture. Amongst all the prepared sorbents, Arg-EG/SG shows the highest amount of N% (2.25%) followed by ChCl-EG-Arg/SG (1.97%) and ChCl-EG/SG (1.62%). This highest value might be attributed to the higher molar ratio of Arg used in the (Arg-EG) mixture compared to the (ChCl-EG-Arg) and (ChCl-EG) mixture which contain 0 and 0.1 molar ratios of Arg, respectively. According to Mohamed Hatta et al. (2022), the chemisorption process of CO_2 could be enhanced by the presence of heteroatom-containing groups. The use of heteroatoms like nitrogen and oxygen has been shown to improve surface property, selectivity, and adsorption capacity due to improved acid-base reaction, quadrupolar and hydrogen bonding interactions (Shao et al. 2022; Yu et al. 2022). Since Arg-EG/SG has higher nitrogen content than others, the CO_2 adsorption capacity of prepared sorbents could be better than other sorbents.

According to the FT-IR spectra in Figure 2, the strong and broad adsorption bands at 1055 cm⁻¹ are due to the Si-O-Si asymmetric stretching vibration (Yusof et al. 2021), while the peak at 796 and 465 cm⁻¹ for all samples are assigned to Si-O-Si and Si-O symmetric stretching and bending, respectively (Zhang, Zhao & Xu 2017). The Si-OH peak has also been observed at 970 cm⁻¹ for pure SG (Ghazali et al. 2020a). After impregnation of silica gel with the Arg-EG, ChCl-EG, and ChCl-EG-Arg, new peaks appeared and can be assigned as follow; the N-H stretching vibration (3790-3563 cm⁻¹), O-H stretching
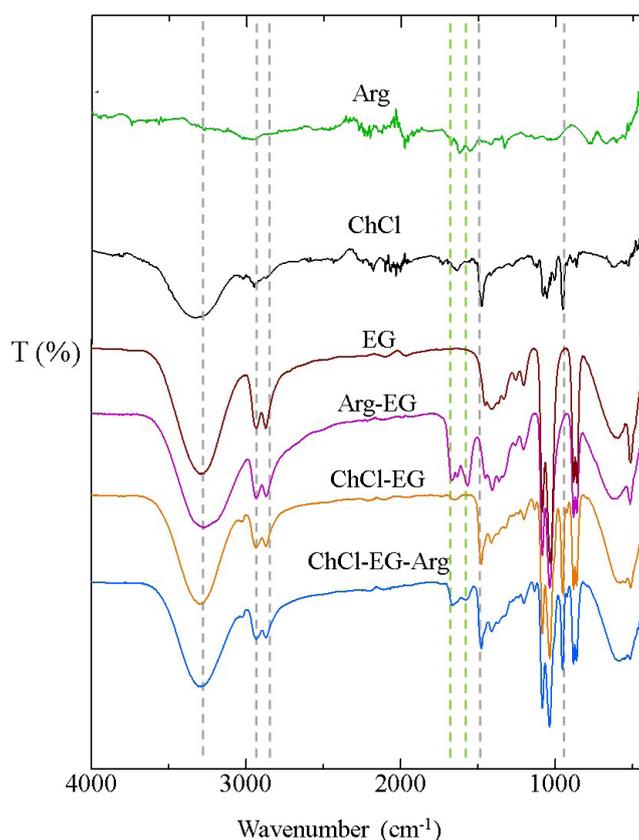

FIGURE 1. The FT-IR spectra of Arg, ChCl, EG, DES and AADES's solvents

vibration (3677 cm^{-1}), C-H stretching vibration (2950 cm^{-1}) (Al-Absi et al. 2023), N-H bending vibration of secondary amine (1715-1671 cm^{-1}) and primary amine (1594-1424 cm^{-1}) for Arg, and C-N vibration peaks (1080-1050 cm^{-1}) (Sivrikaya 2019). Physical interaction through hydrogen bonding between DES components (-NH, Cl$^-$, -OH) with Si-OH of the silica gel can be observed at 951 cm^{-1}, where the peak slightly shifted to a lower adsorption band. Besides, the intensity of the peak at 1055 cm^{-1} has increased due to the overlapping of C=O carbonyl stretching and Si-O-Si asymmetric stretching vibration after the addition of Arg. All of these

observations showed that the DES/SG and AADES/SG sorbents have been prepared successfully.

THERMOGRAVIMETRIC ANALYSIS (TGA)

Thermal stabilities of all the samples under the air atmosphere, including pure silica gel were analyzed and shown in Figure 3. From TG curves in Figure 3(a), the first weight loss can be observed at a temperature below 100 °C that indicates the removal of physisorbed water and residual organic solvent like ethanol from the impregnation method for all samples. TG curve for the pure silica gel support was maintained above 100 °C

TABLE 5. Elemental analysis data

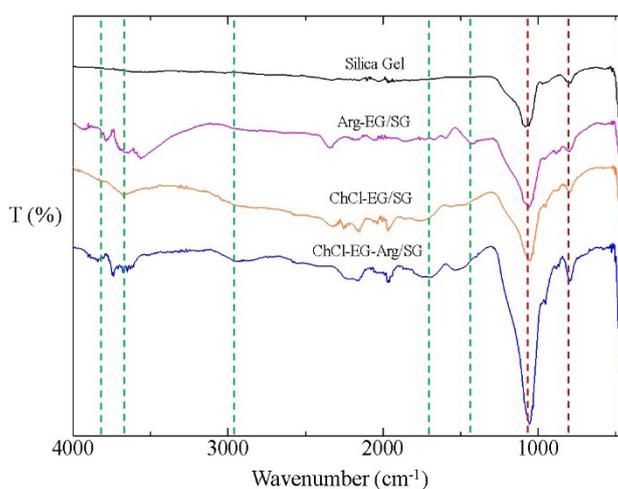| Sample	DES molar ratio	C (%)	H (%)	N (%)
SG	-	0.21	0.93	0.00
Arg-EG/SG	1:8	9.72	2.83	2.25
ChCl-EG/SG	1:2	10.98	3.51	1.62
ChCl-EG-Arg/SG	1:2:0.10	10.29	3.12	1.97

FIGURE 2. FTIR spectra of pure silica gel and impregnated SG with DES and AADES sorbents

which indicated the decomposition temperature for SiO_2 was beyond 500 °C. After the impregnation with DES and AADES, there are additional weight loss curves were observed. Thermograms for Arg-EG/SG show additional two weight loss curves while ChCl-EG/SG and ternary ChCl-EG-Arg show three curves of weight loss. This indicated the weight loss and decomposition temperature of HBD and HBA in the eutectic mixture on SG thus proving the impregnations of the eutectic mixtures were successful.

The differential thermal gravimetric analysis (DTG) peaks indicated the maximum decomposition temperature (T_{peak}) of samples and were shown in Figure 3(b). For ChCl-based DES, the second weight loss with T_{peak} at 132.5 °C and 141.7 °C for ChCl-EG-Arg/SG and ChCl-EG/SG indicated the evaporation of EG below its boiling point, respectively. The third weight loss with the T_{peak} at around 232-235 °C can be attributed to the evaporation of EG once it reaches and exceeds the boiling point and Arg (Delgado-Mellado et al. 2018; Weiss et al. 2018).

The thermal stability of DESs is significantly influenced by the hydrogen bond and other intermolecular interaction. As for Arg-EG/SG, the second weight loss with the T_{peak} at around 192.1 °C was attributed to the maximum decomposition of Arg-EG. This result shows the use of Arg as HBA gives better thermal stability compared to ChCl-EG/SG and ChCl-EG-Arg/SG which would related to stronger intermolecular interaction between Arg-EG components. Francisco, van den Bruinhorst and Kroon (2013) have stated that the DES that formed by strong intermolecular interactions demonstrated high stability upon heating without noticeable weight loss, but for weaker hydrogen-bonding interactions the strength of the bonds diminished with increasing temperature. Since Arg-EG mixture contain more molar ratio of Arg that contained the most hydrogen bonding groups, their intermolecular force interaction is stronger and more energy is needed to break the hydrogen bond interaction.

The peaks at around 314-347 °C attributed to the maximum decomposition of ChCl and Arg residues. The results were in agreement with the works in previous literature (Abbas & Binder 2010; Ghazali et al. 2020b; González et al. 2022). Among all prepared sorbents, Arg-EG/SG shows better thermal stability with higher decomposition temperatures. It should be noted that the thermal stability studies of samples were analysed in air environments, therefore the influence of oxygen contents might affect the degradation temperature of the samples

compared to other reported literature that uses N_2.

SURFACE AREA AND POROSITY CHARACTERIZATION

An automated gas sorption analyzer was used to evaluate the surface area and pore structure of prepared sorbents. Based on Table 6, the surface area and pore volume of the pure silica gel is 360 m^2/g and 0.68 cm^3/g, respectively. After wet impregnation with 30% loading of ChCl-EG, ChCl-EG-Arg and Arg-EG, there are significant decreased in the surface area (222-138 m^2/g) and pore volume (0.51-0.33 cm^3/g). The reduction in the surface area might be due to some pore blocking and pore filling of the DES and AADES layer onto SG at high loading and this indicates that DES and AADES have been successfully impregnated on silica pores (Ghazali et al. 2019; Marliza et al. 2022). This can be proven by the N_2 adsorption-desorption isotherm in Figure 4(a), where the isothermal curves types are preserved as type IV with H2(b) hysteresis in the IUPAC classification for all the impregnated sorbents. This type of hysteresis loop is related to pore blocking with the larger size distribution of neck widths (Thommes et al. 2015). The isotherm also indicates monolayer-multilayer adsorption and capillary condensation (Khdary & Abdelsalam 2020; Rameli et al. 2022) and this shows that DES and AADES-loaded SG are still classified as the mesopores type material with mesopores (ranging from 2-50 nm in diameter) (Sotomayor, Cychosz & Thommes 2018).

As can be seen in Figure 4(b), the BJH pore size distributions are centered around 13.03 nm for SG, 8.10 nm, 8.96 nm and 8.98 nm for ChCl-EG/SG, Arg-EG/SG and ChCl-EG-Arg/SG, respectively, confirming the presence of mesopores. The broad pore size distribution shape of SG was changed and shifted to the left after 30% loading of DES and AADES solvents where all sorbents have sharper and smaller pores size distribution than SG. This may indicate that the lining of the pores did occur and pore size was reduced but still in the range of mesoporous even after the impregnation step.

THE CO_2 ADSORPTION ANALYSIS

The CO_2 adsorption capacity of pure SG, Arg-EG/SG, ChCl-EG/SG, and ChCl-EG-Arg/SG sorbents were evaluated at 25 °C with pure loading of CO_2. An equilibrium data from adsorption isotherms for single and multicomponent systems define the relative amounts a specific material can adsorb at a given pressure (Fauth et al. 2012). The isotherms are presented in Figure 5. The CO_2 sorption at 1 atm of 16.0 mg/g was achieved

by Arg-EG(1:8)/SG followed by ChCl-EG-Arg(1:2:0.1)/SG (14.8 mg/g) and ChCl-EG/SG(1:2)(14.5 mg/g). The CO$_2$ uptake of all sorbents increased almost linearly with increasing pressure, as also observed by Raja Shahrom et al. (2019) using activated carbon impregnated with amino acid ionic liquid (AAILs). The higher capacity of the CO$_2$

isotherm at low pressure on Arg-EG/SG might cause by the chemical reaction between CO$_2$ and the primary amine groups (–NH$_2$) (Sánchez-Zambrano et al. 2018; Wang & Yang 2011). This shows that, CO$_2$ adsorption is more favorable for the Arg-EG/SG at a lower pressure which has a higher percentage of nitrogen than others.

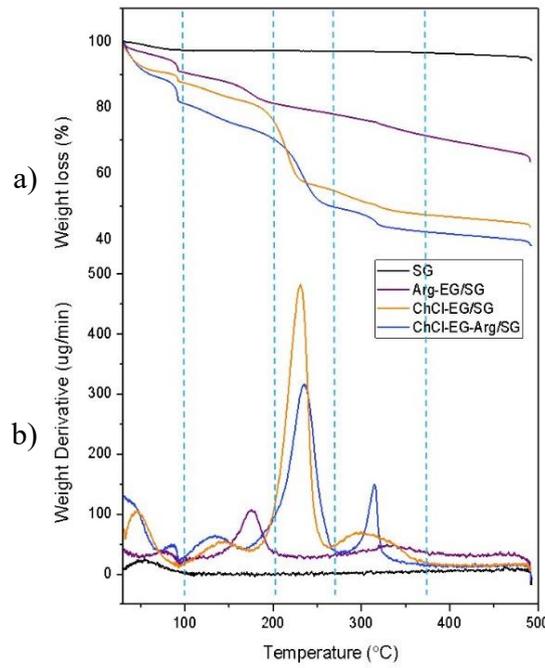TABLE 6. Physical properties and CO$_2$ adsorption isotherms

FIGURE 3. TGA a) and DTG b) thermograms of pure SG and impregnated SG sorbents
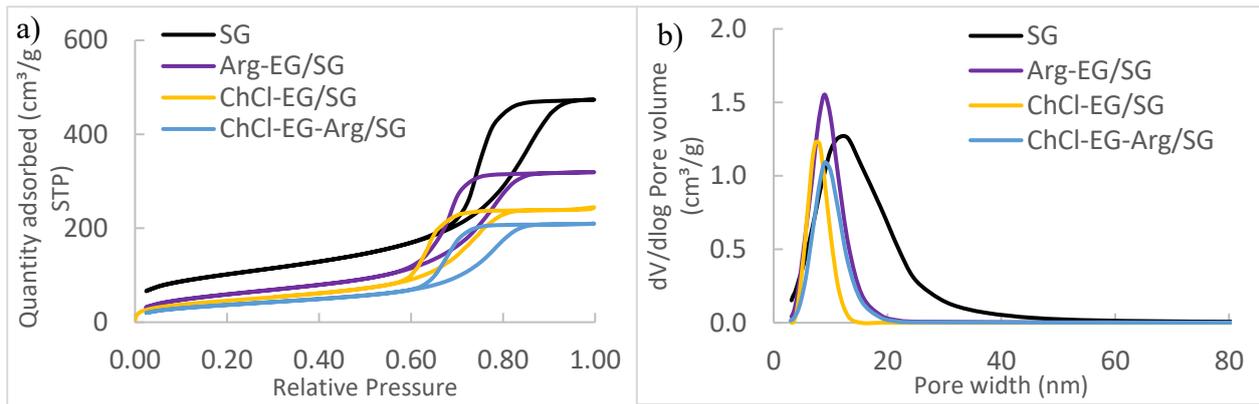

FIGURE 4. a) N$_2$ adsorption and desorption isotherm, b) BJH pore distribution of pure SG and 30% loading of ChCl-EG/SG, ChCl-EG-Arg/SG and Arg-EG/SG sorbents

of SG and impregnated SG with Arg-EG, ChCl-EG and ChCl-EG-Arg at 100% CO_2 loading

Sample	Surface area (S_{BET}) (m²/g)	BJH adsorption cumulative pore volume (cm³/g)	Adsorption capacity (mg/g) at 1 atm
SG	360	0.68	19.4
Arg-EG/SG	222	0.51	16.0
ChCl-EG/SG	147	0.39	14.5
ChCl-EG-Arg/SG	138	0.33	14.8

Porosity plays an important role to determine the adsorption capacity (Yusof et al. 2021). CO_2 is

physically being adsorbed onto the mesoporous silicas and further increased from 0.1 to 1.0 atm, attributed to the physisorption of CO_2 on the DES/SG and AADES/SG material (Sánchez-Zambrano et al. 2018). Besides, the isotherm of AADES sorbents is not very steep, which would be good for the regeneration step, but the adsorption capacity was rather low compared to SG as the pressure increased. Based on the BET (N_2) and CO_2 adsorption analysis results in Table 6, the trend of adsorption capacities is similar to the reducing trend of DES and AADES's surface area. This phenomenon has also been observed in impregnated ChCl-Urea onto nanoporous silica (NS) sorbents conducted by Ghazali et al. (2020b). After impregnation, the -OH from the silica gel surface formed hydrogen bonding with the –N group from AADES solvents that are predicted to decrease the physical adsorption of CO_2 (Ghazali et al. 2020a). This indicated that the reduction in the surface area and pore volume of the impregnated sorbents gives an impact on the low physical adsorption capacities of CO_2 at 1 atm.

The CO_2 adsorption comparison between binary ChCl-EG/SG and ternary ChCl-EG-Arg/SG shows that the adsorption was influenced by the presence of Arg by a slight increase in adsorption. This is because ternary ChCl-EG-Arg/SG contains a slightly higher amount of N and is more basic. However, the increase is not that significant due to the small amount of molar ratio of Arg added. Amongst all the impregnated samples, binary Arg-EG/SG that uses Arg as HBA gives a higher surface area and better CO_2 adsorption (16.0 mg/g) than the other impregnated SG. Besides, the -OH from the EG also contributes to the adsorption of CO_2.

CO$_2$ SORPTION ANALYSIS USING TGA (15% CO_2/85% N_2)

The CO_2 capture performance of sorbents was measured by a TGA analyzer at 15% CO_2/85% N_2 mixed gases concentration to stimulate the real flue gases emitted by coal and gas-fired plants (Uehara, Karami & Mahinpey 2019). As shown in Figure 6, the 3 h of pre-treatment time at 120 °C gives the highest adsorption of CO_2 compared to the others. The longer the pre-treatment (degasification) time, the more adsorbed moisture and gas were desorbed. Therefore, more available surfaces for adsorption to take place. Besides that, as the temperature increased from 25 °C to 30 °C the adsorption capacity of Arg-EG/SG was decreased. This finding was in agreement with the study conducted by Wang et al. (2013), where the CO_2 capture capacity of [EMIM][Lys]-PMMA decreased as the temperature increased from 25 °C to 100 °C due to a sorbent–sorbate's interaction was weakened as temperature increases. This shows that CO_2 adsorption of AADES is exothermic in nature (Sang Sefdi & Luuis 2019). Therefore, these AADES sorbents were more suitable for room temperature (25 °C) CO_2 capture technology.

In Figure 7, the CO_2 adsorption by AADES-supported sorbents occurred in a two-stage process, similar to supported amino acid ionic liquid and amine sorbents (Uehara, Karami & Mahinpey 2019). The first stage is a rapid reaction with CO_2 that results in a significant weight gain on the surface of sorbents (surface reaction). In the second stage, adsorption occurred much slower with the smaller weight change. As seen in Figures 7 and 8, the CO_2 capture capacity's order of prepared sorbents was Arg-EG/SG (8.1 mg/g) > SG (4.2 mg/g) > ChCl-EG-Arg/SG (2.8 mg/g) > ChCl-EG/SG (2.2 mg/g). Amongst impregnated SG, the Arg-EG/SG shows the highest CO_2 adsorption in 15% CO_2/85% N_2 loading and significantly, doubles the adsorption by the SG. This indicated that

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the CO_2 adsorption at flue gas compositions is more favorable for the Arg-EG/SG, which might be attributed by its highest percentage of nitrogen (N%) compared to other sorbents. This result shows that the higher amount of Arg enhanced the CO_2 adsorption capacity. The result correlated with the study conducted by Chemat et al. (2016b), where Arg gives a pronounced effect on the CO_2 solubility of prepared DES. This might happen since Arg has basic side chains with more amino species to react with CO_2. Based on the basicity of prepared Arg-EG and the acidic role of CO_2, it is expected that the impregnation of Arg-EG on SG improved the CO_2 capture performance. However, the CO_2 adsorption capacities of ChCl-EG/SG and ChCl-EG-Arg/SG were lower than SG. This is due to the comparatively weak attraction of the ChCl-based DES to porous SG by wet impregnation and DES may leach to the gas stream (Wang et al. 2013). This is then contributing to the loss of amine which affects the sorption of CO_2. Besides, the low adsorption capacity for ternary ChCl-EG-Arg/SG could be due to low amount of Arg added (0.1 mol ratio) as compared to Arg-EG/SG sorbents, thus reducing the acid base and intermolecular interaction with CO_2.

Based on both CO_2 adsorption test results at 100% and 15% CO_2 loading, the physisorption and chemisorption of CO_2 might occur for the Arg-EG/SG. The proposed mechanism of CO_2 sorption by Arg-EG/SG sorbents was illustrated in Figure 9. Physical adsorption of CO_2 occurred with the –OH group from silica gel, EG, and Arg. Physical adsorption usually involves physical forces such as Van der Waals, dipole-dipole, polar, electrostatic, or hydrophobic association

of physisorbed CO_2 (Lahuri & Yarmo 2022). There are four amino functional groups at the Arg structure, including two primary and two secondary amino groups that can be active sites for chemical reaction with CO_2. Due to the similarity of the amino acid functional group with alkanolamines, their reaction mechanism with CO_2 is expected to be similar to those of alkanolamines (Mahmud et al. 2019; Sang Sefidi & Luis 2019). The amino group from Arg has a strong affinity towards CO_2 and binds via chemisorption (Mohamed Hatta et al. 2022) where 1 mol of –NH$_2$ group from Arg-EG will react with 1 mol of CO_2 and form an intermediate zwitterion. Subsequently, the reaction proceeds with the protonation reaction between 1 mol zwitterion with 1 mol NH$_2$ to form a carbamate. In conclusion, 2 mols of NH$_2$ groups are needed to react with 1 mol of CO_2.

In an absence of ChCl, the structure of Arg-EG is less complex and contain more available active sites that contributes to better reaction between the amino group and CO_2 and lead to higher adsorption capacity. Since the adsorption can only take place on the surface, by increasing the sorbent porosity makes more available space for adsorption to occur. Therefore, higher loading of DES and AADES in this study may prevent adsorbate molecules from entering the pore by clogging the surface layer of SG upon impregnation. Therefore, improvement on the AADES's loading on the porous sorbents should be optimized and utilizing a porous material with higher surface area are important in order to get better CO_2 adsorption performance.
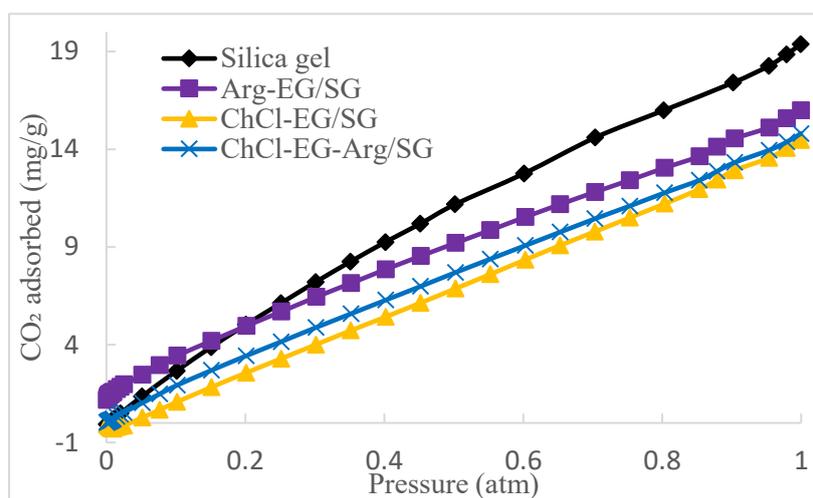
CONCLUSIONS

FIGURE 5. The CO_2 adsorption isotherms of pure SG, Arg-EG/SG, ChCl-EG/SG and ChCl-EG-Arg/SG at 100% CO_2 loading
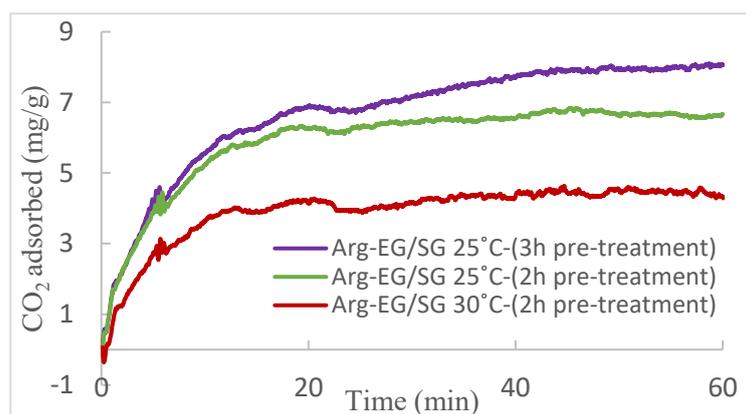
FIGURE 6. Effect of adsorption temperature (25-30 °C) and pre-treatment time (2h;3h) on the CO_2 capture of Arg-EG/SG using TGA
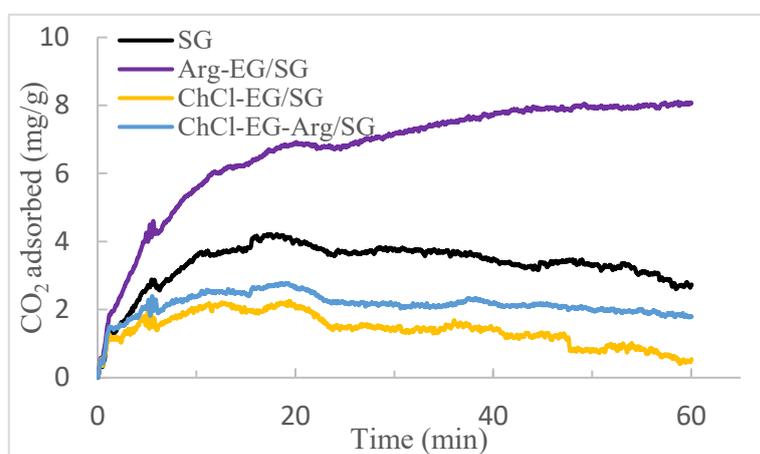

FIGURE 7. CO_2 adsorption performance of all samples at flue gas condition (15% CO_2 /85% N_2) and 25 °C using TGA analysis
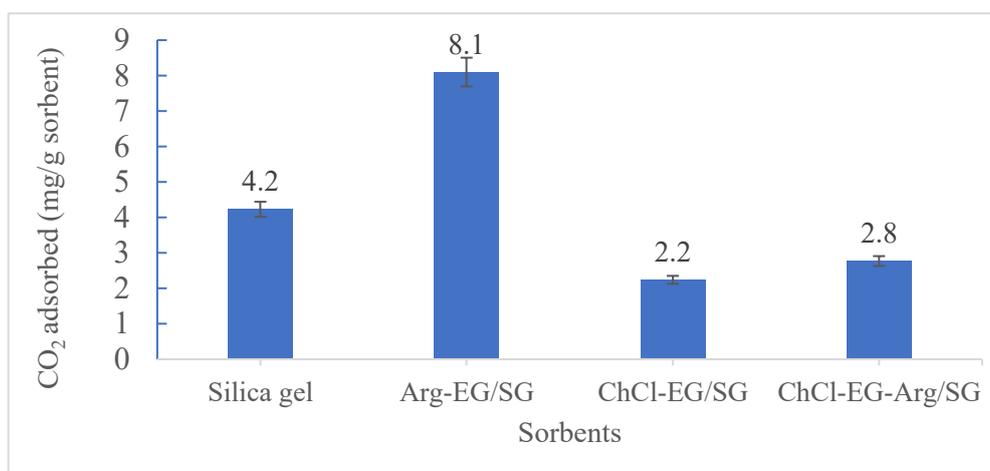

FIGURE 8. Maximum CO_2 adsorption capacity for all samples using TGA at 25 °C and 15% CO_2 loading
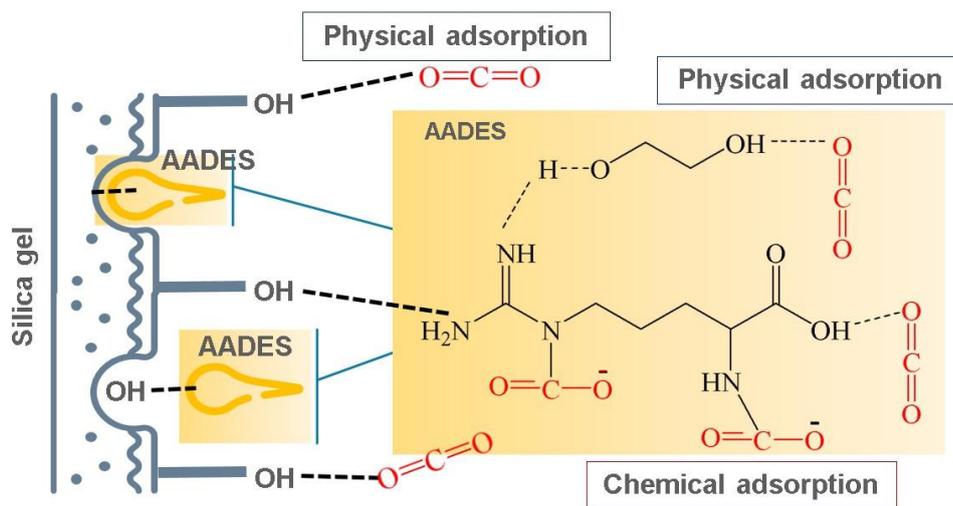
FIGURE 9. Proposed mechanism of CO_2 sorption by Arg-EG/SG sorbents

In this study, binary and ternary AADES (Arg-EG; ChCl-EG-Arg) were prepared and impregnated into the porous silica gel supports and compared with the binary ChCl-EG for their physical characteristics. The FTIR, CHN and N_2 adsorption analyses conducted have proved the successfulness of wet impregnation of DES and AADES on SG. The CO_2 adsorption isotherm and CO_2 adsorption test studies showed that the binary and ternary AADES gives better CO_2 adsorption capacity than (ChCl-EG/SG) sorbents. The increasing temperature decreased the CO_2 adsorption while increasing pressure increased the CO_2 adsorption for all impregnated SG. Among impregnated SG, Arg-EG/SG shows better CO_2 capture due to higher surface area, higher percentage of nitrogen (N%) and better reaction between amino group and CO_2. The presence of primary and secondary amine in Arg provides more active sites and enhances the CO_2 adsorption on the Arg-EG/SG. Overall, the Arg-EG/SG sorbents can be utilised as a potential green sorbent for CO_2 capture as it shows good CO_2 adsorption capacity at flue gas composition.

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