

A screening study of alcohol solvents for alkanolamine-based CO₂ capture

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ABSTRACT

The present study was aimed at developing a non-aqueous absorbent system that does not form any precipitate/solid upon exposure to CO₂. More specifically, the non-aqueous absorbent system must be a homogeneous mixture (single phase) within the whole range of CO₂ loading. At the same time, sufficient amine regeneration must occur at a low temperature of 80–90 °C compared to reference non-aqueous monoethanolamine (MEA)-based absorbents. To achieve this target, 18 alcohols were investigated as solvents and 4 alkyl-linked alkanolamines were evaluated as absorbents. Several experiments mixing alkanolamines with alcohols were performed to evaluate their CO₂ capture performance. High-performance non-aqueous liquid absorbents (without solid/precipitate formation) with the advantages of higher absorption rates, higher cyclic capacities, higher regeneration efficiencies, lower specific heats, and lower heats of reaction compared to reference MEA-based non-aqueous absorbents were successfully developed herein. The favorable features of these absorbents will provide the basis for the discovery of potential non-aqueous amine-based absorbents.

1. Introduction

The carbon dioxide (CO₂) concentration in the atmosphere is increasing annually with the combustion of fossil fuels for power generation, which increases the emission of greenhouse gases that contribute to global warming and climate change (IPCC, 2005). CO₂ emissions resulting from the combustion of fossil fuels and industrial processes were the largest contributors (65%) to global greenhouse gas emissions in 2010 (IPCC, 2014). A drastic reduction in CO₂ emissions is required for the mitigation of global warming. In this context, carbon dioxide capture and storage (CCS) has been gaining attention as an innovative mitigation measure (Boot-Handford et al., 2014; Leung et al., 2014; Rochelle, 2009).

Many technologies are currently employed for the separation and capture of CO₂ from flue gas streams. These techniques are based on different processes, including absorption, adsorption, and membrane separation (MacDowell et al., 2010; Bernardo et al., 2009). Post-combustion CO₂ capture using an aqueous amine solution is the most mature and widely employed of the processes currently employed (Rochelle, 2009; Wang et al., 2017). There are commercial aqueous amine technologies, such as, KS-1, Econamine FG+, Oase Blue, and CANSOLV for CO₂ separation from flue gas sources that hold

considerable promise, with low heat energy consumptions of 2.3–2.4 GJ/t-CO₂. Previously, RITE also developed highly efficient single and mixed aqueous amine-based absorbents for targeting CO₂ generated from steel-making industries (Chowdhury et al., 2013; Onoda et al., 2016; Yamada et al., 2013). All of these CO₂ absorbents are capable of reducing the energy consumption associated with CO₂ separation by approximately 2.0 GJ/t-CO₂ (Onoda et al., 2016). However, the total separation cost still remains high.

Usually, during a chemical absorption process, CO₂ is absorbed into the amine solution at low temperatures (approximately 40 °C) and desorbed from the solution after heating (approximately 120 °C). The chemistry of this process is complex, but two main reactions take place, depending on the type of amine (Eqs. (1) and (2)) (Aboudheir et al., 2004; Barth et al., 1984; Vaidya and Kenig, 2007). The reaction between CO₂ and the unhindered (primary or secondary) amines forms a fairly stable carbamate, R¹R²NCOO[−] (Eq. (1)). On the other hand, the hindered amines, which form an unstable carbamate, and tertiary amines, go through an alternate reaction to form a bicarbonate ion, HCO₃[−] (Eq. (2)).

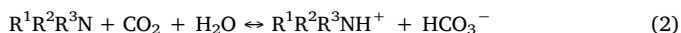


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The regeneration of amine from stable carbamate or bicarbonate is done by stripping with water vapor at 100–120 °C. Although aqueous amine solutions are promising to remove CO₂ from flue gas point sources, aqueous amine process often suffer from environment- and health-related concerns due to volatile-solvent losses, thermal and oxidative degradation of amines, corrosion problems, and high energy consumption for solvent regeneration (Reynolds et al., 2012; Gouedarda et al., 2012; Rao and Rubin, 2002). Therefore, there is a high demand for the development of a new alternative solvent that can overcome the aforementioned drawbacks and process larger quantities of CO₂ with a lower energy demand.

To identify cost-effective approaches for CO₂ capture, the past decade has witnessed the development of various new solvents, including concentrated non-aqueous/water-lean solvents. Non-aqueous amine solvents have potential advantages over aqueous amines, specifically lower heat capacity (approximately one-half), lower heat of vaporization of organic solvents, and higher boiling temperature compared to that of water. Many formulations of non-aqueous CO₂ selective solvents have been tested, including amine-based non-aqueous solvents (Aschenbrenner and Styring, 2010; Francesco et al., 2012, 2013, 2014; Lail et al., 2014; Phan et al., 2008; Vincenzo et al., 2013), CO₂-binding organic liquids (Feng et al., 2016; Heldebrant et al., 2008; Jian et al., 2013; Liu et al., 2006; Mathias et al., 2013; Privalova et al., 2012), aminosilicones (Perry and O'Brein, 2011), alkylimidazole blended with amine (Bara and Shannon, 2011), room temperature ionic liquids (RTILs) (Bara et al., 2010; Cadena et al., 2004; Dai et al., 2017; Shiflett and Yokozeki, 2007; Soriano et al., 2008; Yingying et al., 2016; Yujiao et al., 2014), amino-functionalized task-specific ionic liquids (TSILs) (Bates et al., 2002; Sánchez et al., 2011; Sharma et al., 2012; Zhang et al., 2009), and mixed RTILs with alkanolamines (Camper et al., 2008; Feng et al., 2013; Hasib-ur-Rahman et al., 2012; Shannon and Bara, 2011). All these non-aqueous absorbents possess several advantages over the aqueous absorbents namely, high-boiling-point, low vapor pressure, thermally stability with a lower heat capacity than that of water. Despite their potential for CO₂ capture, non-aqueous solvents possess some drawbacks: the non-linear increase in viscosity once CO₂ is absorbed, precipitate formation, or solvent gelation leading to a highly viscous gel or waxy solid.

To overcome the aforementioned drawbacks, this work focuses on the development of a non-aqueous absorbent system that will reduce the viscosity of the CO₂ loaded solution and will not form any precipitate, viscous gel, or waxy solid upon exposure to CO₂. More specifically, the non-aqueous absorbent system must be a homogeneous mixture (single phase) within the whole CO₂ loading range. At the same time, sufficient amine regeneration at a low temperature range of 80–90 °C is desired. This makes it possible to use waste low temperature heat for regeneration at a low cost, resulting in a more cost-effective CO₂ absorption process. Another critical issue in the use of non-aqueous solvents is how to control the water, because water is omnipresent in the process, as it is introduced via the flue gas. The water tolerance of the non-aqueous solvent system is also briefly discussed.

2. Experimental

2.1. Materials

All amine absorbents and alcohol solvents were purchased from Sigma-Aldrich Chemical Co. (St. Louis, MO), Wako Pure Chemical Industries (Osaka, Japan), or Tokyo Chemical Industry Co. Ltd. (Tokyo, Japan) and used as received. The details and chemical structures of the investigated amine absorbents and alcohol solvents are shown in Table 1. Nitrogen (99.9999%) was purchased from Iwatani (Osaka, Japan). CO₂ (99.995%) and CO₂ (19.98%) with the balance being N₂, were supplied by Sumitomo Seika Chemicals (Osaka, Japan).

Table 1

Investigated amine absorbents and alcohol solvents.

Name and manufacturer	CAS number	Purity%	Chemical structure
2-Amino-1-propanol (2A1P), TCI	6168-72-5	98	
2-Amino-2-methyl-1-propanol (AMP), Wako	124-68-5	93	
2-(Ethylamino)ethanol (EAE), TCI	110-73-6	98	
2-(Isopropylamino)ethanol (IPAE), TCI	109-56-8	99	
2-Aminoethanol (MEA), TCI	141-43-5	99	
1-Butanol (Bu), TCI	71-36-3	99	
1-Hexanol (Hex), TCI	111-27-3	98	
1-Octanol (Oct), TCI	111-87-5	99	
3,3-Dimethyl-2-butanol (3,3-DM-2B), TCI	464-07-3	97	
Cyclohexanemethanol (CHM), TCI	100-49-2	98	
Diethylene glycol monoethyl ether (DEGME), TCI	111-90-0	99	
Ethylene glycol (EGL), Wako	107-21-1	99	
1,2-Propanediol (1, 2-PD), TCI	57-55-6	99	
2,3-Butanediol (2, 3-BD), Aldrich	513-85-9	98	
1,2-Butanediol (1,2-BD), Wako	584-03-2	98	
1,2-Hexanediol (1, 2-HEXD), TCI	6920-22-5	96	
1,2-Octanediol (1,2-OCTD), TCI	1117-86-8	96	
1,3-Propanediol (1,3-PD), TCI	504-63-2	98	
1,4-Butanediol (1,4-BD), TCI	110-63-4	99	
1,5-Pentanediol (1, 5-PED), TCI	111-29-5	97	
Triethylene glycol (TEG), TCI	112-27-6	99	
1,4-Cyclohexanedimethanol (1,4-CHDM), TCI	105-08-8	99	
1,2,6-Hexanetriol (1,2,6-HEXT), TCI	106-69-4	96	

2.2. Characterization of physical properties

Viscosity values of different CO₂-loaded solutions of EAE-TEG and reference MEA-TEG solutions were measured at 40 °C using an EMS-1000 programmable viscometer (Kyoto Electronics Mfg. Co. Ltd., Kyoto, Japan). Ten viscosity measurements were acquired, and the mean values were used. The specific heat capacity values for each alkanolamine-EGL, EAE-TEG, and MEA-EGL/MEA-TEG solution were measured at 40 °C; and the atmospheric pressure was measured using an SHA-500 (Kyoto Electronics Mfg. Co. Ltd., Kyoto, Japan). Three specific heat measurements were obtained, and the mean values were used.

2.3. Gas scrubbing test

The apparatus used for the gas scrubbing test was a 500-cm³ crystal glass cylindrical autoclave (Taiatsu Techno, Japan), as shown in Supplemental Information (Fig. S1). The autoclave was equipped with mass flow controllers for N₂ (max: 1 L/min) and CO₂ (max: 1 L/min), an electric heater, a mechanical stirrer, a condenser, a backpressure regulator, a pressure sensor, a thermocouple, and an ND-IR CO₂ detector.

The equipment can operate at temperatures from 30 to 140 °C and at a pressure ranging from ambient pressure to 1 MPa. Amine-containing non-aqueous solutions (mass fraction 30–80%) and reference MEA (mass fraction 30%) were used for gas scrubbing data collection. Experiments were conducted at 40, 80, and 90 °C according to the following procedure. First, 100 g of each amine-containing non-aqueous solution was fed into the autoclave. Then, N₂ gas was purged until all undesired gas was removed from the reaction vessel. Each solution was agitated using a mechanical stirrer at 700 rpm/min and the temperature remained constant during the absorption at 40 °C and regeneration at 80 or 90 °C, respectively. The concentration of the 20% CO₂ gas balanced with N₂ was controlled using mass flow controllers (SEC-E40, Horiba, Kyoto, Japan) and then supplied to the autoclave at a flow rate of 700 mL/min, which was maintained during both the absorption and desorption processes unless otherwise stated. This concentration was chosen based on the typical blast furnace flue gas composition. As will be described later, for regeneration, N₂ gas was supplied instead of CO₂/N₂, in a multi-cycle CO₂ absorption-regeneration test. During the experiments, the outlet gas concentration from the reactor was continuously monitored using a carbon dioxide analyzer (VA-3001, Horiba).

2.4. Quantitative ¹³C NMR experiment

Alkanolamine-EGL-CO₂ mixtures were prepared according to our previous publication (Chowdhury et al., 2009) by flowing 20% CO₂ gas balanced with N₂ at a rate of 700 mL/min into 50 mL of a 20 wt% amine-containing non-aqueous solution in a glass scrubbing bottle controlled at 40 °C for a certain time at an appropriate CO₂ loading. The CO₂ loading of the prepared samples in the solution (mol-CO₂/mol-amine) was analyzed at room temperature using a total organic carbon analyzer (TOC-V_{CSH}, Shimadzu). An approximately 0.4-mL CO₂-equilibrated alkanolamine-EGL-CO₂ solution was taken into the 5-mm-outer-diameter NMR sample tube. Then, a capillary sealed with an internal standard [2 wt% 3-(trimethylsilyl)-propionic-2,2,3,3-d₄-acid sodium salt in D₂O] was placed into the NMR tube and it was closed using a rubber septum before measuring the ¹³C NMR spectra. The detailed NMR sample preparation is shown in Fig. S2. All NMR samples were analyzed using a Bruker Avance III 400 NMR spectrometer. Quantitative ¹³C spectra were recorded at 100 MHz using an inverse-gated decoupling technique with a delay of 60 s and a pulse width of 10 μs and 400 scans. It took approximately 8 h to obtain the quantitative ¹³C NMR spectrum of each sample.

2.5. Heats of reaction

The heats of reaction of the amine-containing non-aqueous solution (amine = 30–80 wt%) during CO₂ absorption were acquired using a differential reaction calorimeter (DRC; Evolution, Setaram, Pennsauken, NJ). An amine-containing solution was placed in two flasks, one for the CO₂ absorption reaction and the other for reference (150 mL each). CO₂ gas was provided in the reaction flask at a flow rate of 120 mL/min at 40 °C and atmospheric pressure. After reaching the desired CO₂ loading, a liquid sample (1 mL) was collected from the reaction mixture, and diluted with water to 1 L. The CO₂ concentration in the solution was determined using the TOC analyzer. The heat of reaction of the amine-containing solution [kJ/mol-CO₂] was calculated using the total heat generated during absorption [kJ] and the amount of CO₂ absorbed [mol].

2.6. Vapor liquid equilibrium (VLE)

VLE data were measured at 40 °C and 90 °C according to the following procedure (Chowdhury et al., 2013). Approximately 700 mL each aqueous amine solution was fed into an autoclave, followed by a N₂ purge. Each solution was agitated with a mechanical stirrer and the

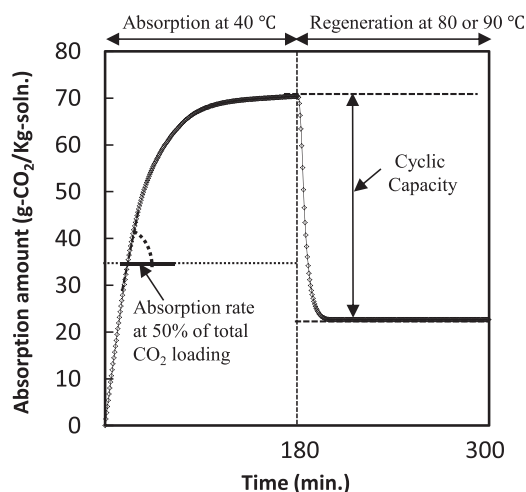


Fig. 1. Typical example of a gas scrubbing test.

temperature was held constant during the experiment. The concentration of the CO₂/N₂ gas mixture was controlled using mass flow controllers, and then the CO₂/N₂ gas mixture was supplied to the autoclave. The outlet gas was monitored using an infrared CO₂ analyzer (VA-3001, Horiba) after passing through a condenser. Equilibrium was obtained when the CO₂ analyzer indicated a constant CO₂ concentration ($\pm 0.01\%$). To analyze the equilibrium data, the total pressure in the gas phase was measured. The CO₂ loading in the liquid phase was measured by the TOC analyzer after collecting a sample from the autoclave.

3. Results and discussion

3.1. Gas scrubbing test

The goal of the gas scrubbing test was to clarify the overall reactivity of each absorbent with CO₂. Fig. 1 shows typical results obtained from the gas scrubbing test. The amount of absorbed CO₂ in the amine-containing non-aqueous solution (CO₂ loading) was calculated from the measured CO₂ concentration in the outlet gas flow. The CO₂ loadings were also checked by the TOC analyzer. The differences between values determined by these two methods are within 2%. As shown in Fig. 1, the CO₂ loading increased with time at 40 °C and then decreased at 80 or 90 °C. The gradient of the curve at 50% of the 180-min CO₂ loading was defined as the absorption rate. Because this was measured for a fresh solution, the initial gradient is not suitable for assessing the absorption rate in cyclic use. The gradient at 50% is not a mandatory selection but it is considered to represent the overall rate of absorption in the screening tests. This reference index was used to compare the behaviors of the non-aqueous amine solutions.

The difference between the maximum CO₂ loading at 40 °C and the minimum CO₂ loading at 80 or 90 °C was defined as the cyclic capacity. The reproducibility of the experiments was assessed and the error in all of the experimental measurements was less than 3%.

AMP is considered to be one of the alternative solvents to the conventional MEA due to its wide application as both an aqueous and a non-aqueous solvent (Francesco et al., 2013, 2018; Liu et al., 2018; Lv et al., 2020; Nwaoha et al., 2016; Patil and Vaidya, 2020; Svensson et al., 2014, 2016), reasonably inexpensive characteristic, and wide availability in bulk scale. Initially, all 18 alcohols were screened with AMP to select the best alcohol solvent in terms of absorption rate and cyclic CO₂ capacity. This time, the absorption experiments were conducted at 40 °C and the absorbed CO₂ was regenerated at 80 °C. From the absorption-regeneration curves it was possible to obtain the absorption rates, amount absorbed CO₂, and cyclic capacities for each absorbent. Table S1 summarizes the absorption rates, and the amounts

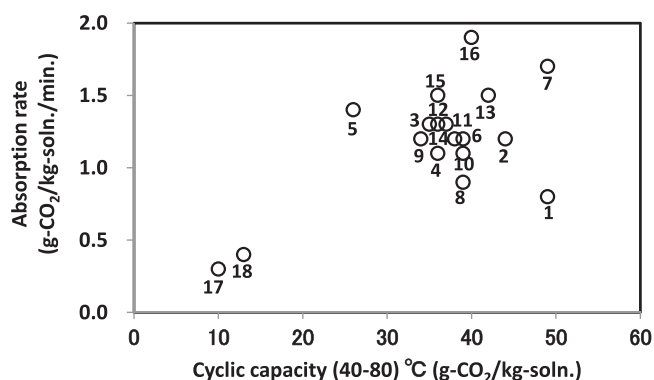


Fig. 2. CO₂ capture performance in terms of absorption rate versus cyclic capacity.

and cyclic capacity values for all AMP-containing alcohol solutions. The relative performances of all tested alcohol absorbents in terms of absorption rate versus cyclic capacity were compared as shown in Fig. 2. Most of the (AMP + alcohol) solutions were precipitated with increased CO₂ loading, except EGL, 1,3-PD, 1,4-BD, and TEG showed a single phase. EGL and TEG were selected as organic solvents because of their higher absorption rate (Fig. 2 #16, AMP + TEG) and higher cyclic capacity (Fig. 2, #7, AMP + EGL) compared to those of the other tested alcohols.

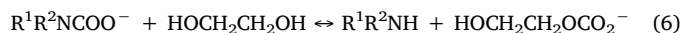
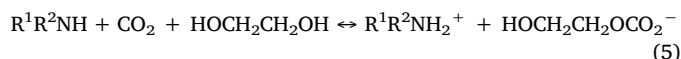
3.2. Analysis of the quantitative ¹³C NMR spectrum of the CO₂-loaded alkanolamine solutions

Quantitative ¹³C NMR spectroscopy was used to study the CO₂-absorbed solutions of the MEA, 1A2P, AMP, EAE, and IPAE to determine the variations in the levels of the different chemical species in solution. Two major pathways that contribute to CO₂ absorption in alkanolamine-CO₂-EGL solutions include the formation of amine-carbamate and hydroxyethyl carbonate anion (Eqs. (3)–(6)) (Francesco et al., 2013; Heldebrant et al., 2008; Jimil et al., 2009; Kim et al., 1999; Kortunov et al., 2015). The carbamate reaction path under anhydrous conditions proceeds via an unstable zwitterion/carbamic acid species, which can further react with excess amine to form stable amine carbamate anion and protonated cation (Eqs. (3) and (4)).



The other reaction path is the formation of hydroxyethyl carbonate anion, where the reaction directly forms hydroxyethyl carbonate by the involvement of one EGL molecule via Eq. (5) or the unstable carbamate formed in Eq. (4) further reacts with EGL to form hydroxyethyl

carbonate (Eq. (6)).



Figures S3–S7 show the quantitative ¹³C NMR spectrum obtained for CO₂-absorbed solutions of the MEA, 1A2P, AMP, EAE, and IPAE, respectively. In amine-CO₂-EGL solutions, dissolved CO₂ molecules are present as amine-carbamate and hydroxyethyl carbonate anions. The peaks were identified and the peak areas enabled the species to be quantitated as shown in Figs. S3–S7. In the spectrum, the methyl carbons (–CH₃), methine carbons (–CH–), and methylene bridge carbons (–CH₂–) appeared at 15.8–69.3 ppm. The peaks at 165.7–166.5 ppm and 159.4–160.1 ppm in the lower field were assigned to carbons of amine-carbamate and hydroxyethyl-carbonate species, respectively. The physically absorbed CO₂ was not observed in this work, indicating its minor contribution, although it cannot be fully excluded in the scrubbing condition. The quantitative ¹³C NMR species analyses of CO₂-loaded solutions (for example, MEA 0.21, 0.51, and 0.59 mol–CO₂/mol-MEA) of each Amine-CO₂-EGL absorbent system are summarized in Fig. 3.

Gas scrubbing tests were also performed, and the results are presented with the collected reaction heat and specific heat in Table 2. The intent was to compare the results of the ¹³C NMR analysis to the data obtained from the CO₂-capture experiments. From Fig. 3 it can be seen that all five amine absorbent carbonate ratios linearly increase when CO₂ loading increases; the carbamate showed the opposite trend. Sterically hindered AMP and IPAE carbonate formation predominates even at a lower CO₂ loading compared to that of less-hindered 2A1P and EAE. Gas scrubbing test results (Table 2) show that the measured loading capacities were much greater than the theoretical maximum of 0.5 mol-CO₂/mol-amine, clearly indicating that the carbonates were competing with carbamates during the absorption step. Quantitative NMR analysis clearly explained this phenomenon. The maximum CO₂ loading capacity of all alkanolamines in Fig. 3 is within the range of 0.59–0.82 mol-CO₂/mol-amine, which exceeds the theoretical maximum of 0.50 mol-CO₂/mol-amine for carbamate formation, indicating that CO₂ capture occurs in the form of hydroxyethyl carbonate.

From Table 2, alkanolamine that efficiently absorbs CO₂ in the form of carbonate showed lower heat of reaction, lower specific heat, and higher cyclic capacity values. Amine steric hindrance has also been identified as an important factor in reducing carbamate stability (Gangarapu et al., 2013; Yamada et al., 2010, 2011) and hence favors the formation of the hydroxyethyl carbonate derivative of EGL. The trend of carbonate formation was AMP > IPAE > EAE > 2A1P > MEA.

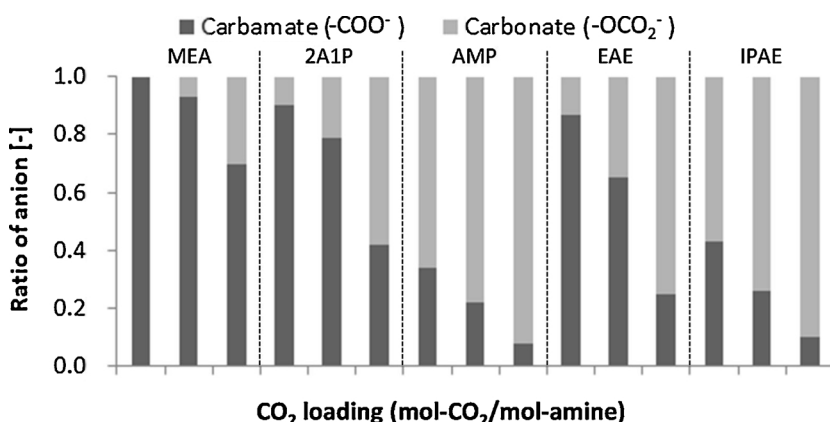


Fig. 3. CO₂-loaded solution carbamate and carbonate ratios obtained from quantitative ¹³C NMR. MEA CO₂ loading = 0.21, 0.51, and 0.59 mol-CO₂/mol-MEA; 2A1P CO₂ loading = 0.20, 0.50, and 0.70 mol-CO₂/mol-2A1P; AMP CO₂ loading = 0.24, 0.48, and 0.82 mol-CO₂/mol-AMP; EAE CO₂ loading = 0.20, 0.51, 0.80 mol-CO₂/mol-EAE; and IPAE CO₂ loading = 0.19, 0.50, and 0.77 mol-CO₂/mol-IPAE.

Table 2

Summary results for each alkanolamine solution.

30 wt% amine in EGL	Absorption rate ^a (g-CO ₂ /kg-soln./min.)	Absorption amount ^b (g-CO ₂ /kg-soln.)	Absorption amount (mol-CO ₂ /mol-amine)	Cyclic capacity ^c (g-CO ₂ /kg-soln.)	Specific heat ^d (J/g K)	Heats of reaction ^e (kJ/mol-CO ₂)	CO ₂ loading (mol-CO ₂ /mol-amine)
2A1P	2.61	91	0.52	30	2.50	89.2	0–0.45
AMP	1.80	90	0.61	54	2.40	71.5	0–0.48
EAE	2.67	84	0.57	39	2.48	74.4	0–0.57
IPAE	2.27	83	0.65	51	2.46	69.5	0–0.53
MEA	2.66	108	0.50	22	2.45	91.2	0–0.48

^a CO₂ absorption rates were calculated at 50% of the 120-min CO₂ loading at 40 °C.^b 120-min CO₂ loading at 40 °C.^c Difference between CO₂ loadings at 40 °C and 80 °C.^d Specific heat of all (alkanolamine + EGL) solutions were measured at 40 °C and atmospheric pressure.^e Heats of reaction were measured at 40 °C and atmospheric pressure within a CO₂ loading range of approximately 0.5 mol-CO₂/mol-amine.

3.3. Optimization of the EAE + TEG solvent system

Table 2 shows that the CO₂ solubility was measured at a low amine concentration (30 wt%) in EGL, which may not be suitable for industrial application. Therefore, several solutions of alkanolamines were studied while varying the concentrations of 2A1P, AMP, EAE and IPAE in EGL or TEG to increase alkanolamine concentrations with respect to the organic solvents EGL or TEG, and it was found that only the (EAE + TEG) solution will not result in precipitate/solid formation up to ~80 wt% absorbent concentration with the maximum CO₂ loading.

The experimental CO₂ absorption–regeneration profiles obtained in this study for the six different sets of (EAE + TEG) absorbents were compared to the (MEA + TEG) absorbent shown in Fig. S8. From the absorption–regeneration curves shown in Fig. S8 it is possible to calculate the absorption rates, amounts of CO₂ absorbed, cyclic capacities, and regeneration efficiencies for all tested absorbents. Table 3 summarizes the absorption rates, amounts of CO₂ absorbed, cyclic capacities, and regeneration efficiencies for all the screened absorbents shown in Fig. S8. From Table 3, it can be seen that the CO₂ absorption rate, CO₂ loading capacity and cyclic capacity of the (EAE + TEG) solution linearly increased (Table 3, Entries 1–6) with increasing EAE concentration. At the same time, it maintained excellent mol/mol basis CO₂ loading and reasonable CO₂ regeneration efficiency at 90 °C. All the (EAE + TEG) solutions showed higher cyclic capacity and regeneration efficiency compared to the reference (MEA + TEG) solution. Of the six tested (EAE + TEG) solutions, only two (EAE + TEG) solutions showed higher absorption rates when compared to the reference (MEA + TEG) solution (Table 3, Entries 5–6).

Typically, at higher absorbent concentrations, the CO₂ absorption rate or solubility decreases because of the high viscosity of the formed carbamated/carbonated derivative. Fig. 4 shows the unique property of (EAE + TEG) solutions in maintaining a high absorption rate and high CO₂-loading capacity even at a high absorbent concentration. It can be

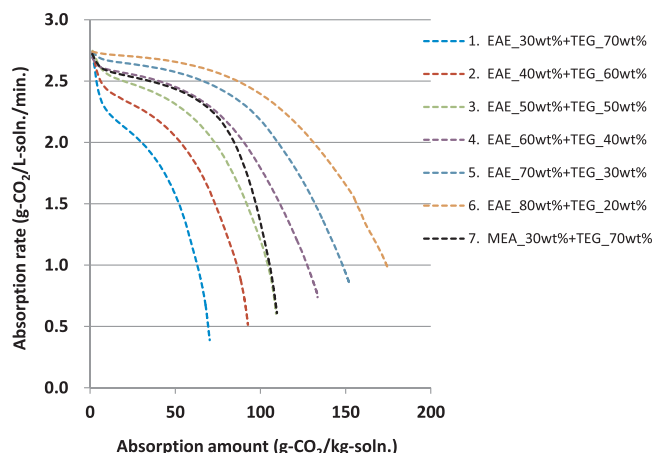


Fig. 4. Apparent absorption rate profiles for six different sets of (EAE + TEG, 1–6) solutions compared to that of the reference (MEA + TEG, 7) solution.

seen from Fig. 4 that the rate profiles #4, #5, and #6 show a higher absorption rate and higher CO₂ solubility in contrast to that of the reference (MEA + TEG) solution.

3.4. Viscosity as a function of CO₂ loading

Low viscosity is another desirable solvent property during CO₂ capture. CO₂ solubility is closely related to solution viscosity, because viscosity significantly affects the liquid film coefficient for mass transfer (Fu et al., 2012; Zhao et al., 2010). High viscosity in a solution leads to a lower gas diffusion coefficient, hindering absorption. Viscosities were measured at 40 °C as a function of CO₂ loading for (EAE_30 wt% + TEG_70 wt%) and the reference (MEA_30 wt% + TEG_70 wt%)

Table 3CO₂ capture performances of different (EAE + TEG) concentration solutions.

Absorbent concentration	Absorption rate ^a (g-CO ₂ /kg-soln./min.)	Absorption amount ^b		Cyclic capacity ^c (g-CO ₂ /kg-soln.)	CO ₂ regeneration efficiency ^d (%)
		(g-CO ₂ /kg-soln.)	(mol-CO ₂ /mol-amine)		
1. EAE_30 wt% + TEG_70 wt%	1.91	70	0.47	56	80
2. EAE_40 wt% + TEG_60 wt%	2.10	93	0.47	74	80
3. EAE_50 wt% + TEG_50 wt%	2.28	109	0.44	92	84
4. EAE_60 wt% + TEG_40 wt%	2.33	134	0.45	104	79
5. EAE_70 wt% + TEG_30 wt%	2.43	153	0.44	122	80
6. EAE_80 wt% + TEG_20 wt%	2.50	175	0.44	139	80
7. MEA_30 wt% + TEG_70 wt% (reference)	2.40	110	0.50	40	36

^a CO₂ absorption rates were calculated at 50% of the 180-min CO₂ loading at 40 °C.^b 180-min CO₂ loading at 40 °C.^c Difference between CO₂ loadings at 40 and 90 °C.^d (c/b) × 100%.

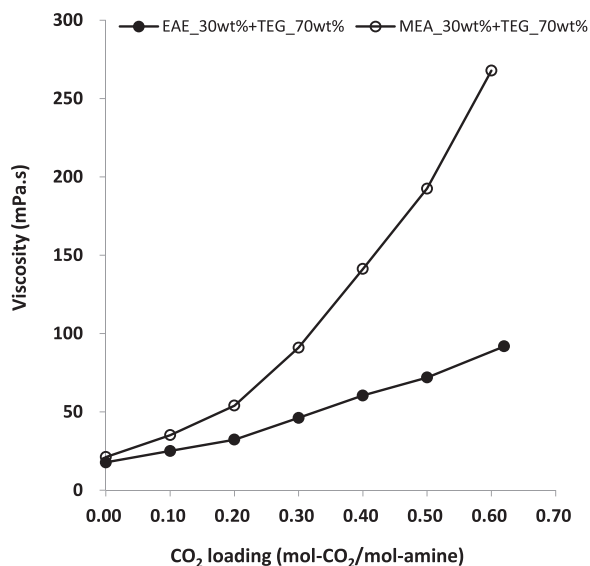


Fig. 5. Viscosity as a function of CO₂ loading for (EAE + TEG) and (MEA + TEG) solutions at 40 °C.

solutions, as shown in Fig. 5. As expected, the viscosity increases with increasing CO₂-loading. For any measured CO₂-loading solution, a relatively lower viscosity value was associated with the (EAE + TEG) solutions in contrast to that of all (MEA + TEG) solutions. For example, from Fig. 5 it can be seen that the viscosity of the (EAE + TEG) solution (71.9 mPa.s) was decreased by 62.6% in comparison to that of the reference (MEA + TEG) solution (192.5 mPa.s), when the CO₂ loading was considered at 0.5 mol-CO₂/mol-amine.

For rational viscosity comparison with conventional aqueous amine solution, the viscosity of 30 wt% aqueous MEA solutions at different CO₂ loadings carried out at 40 °C was also measured as shown in Table S2 and the obtained results are compared with those of 30 wt% EAE in TEG. The viscosity of 30 wt% aqueous MEA solutions under the same conditions is approximately one order of magnitude lower than that of 30 wt% EAE in TEG. Although the EAE in TEG showed high absorption and desorption kinetics in this work, such relatively high viscosities would pose some challenges for practical operations.

3.5. CO₂ regenerability

The apparatus used for the determination of CO₂ regenerability is the same as that reported in this work for CO₂ absorption and desorption (Fig. S1). Nine cycles were conducted to test the regeneration ability of the CO₂-saturated (EAE_15 wt% + TEG_85 wt%) solution as shown in Fig. 6. For each cycle the absorption of CO₂ was completed at 40 °C for 1 h and the CO₂ regeneration from the vessel was completed at 80 °C for 1 h with N₂ gas purging. The regenerated solution was subsequently directly used for the next cycle of CO₂ absorption-desorption. The adsorption capacity after each cycle is shown in Fig. 6.

After nine cycles, the absorption capacity of the (EAE_15 wt% + TEG_85wt%) solution decreased from 0.50 mol-CO₂/mol-EAE (highest) to 0.49 mol-CO₂/mol-EAE (average nine cycles), a decrease of only 2%. The following nine absorption-desorption cycles indicate a fairly high regeneration efficiency and excellent absorbent reversibility. The high regeneration efficiency meets the requirement of green chemistry. However, further research is needed on the long term stability and the regeneration strategies for the absorbent.

3.6. Specific heat and heats of reaction

The gas scrubbing test results suggested that several (EAE + TEG) solutions had better absorption characteristics compared to those of the

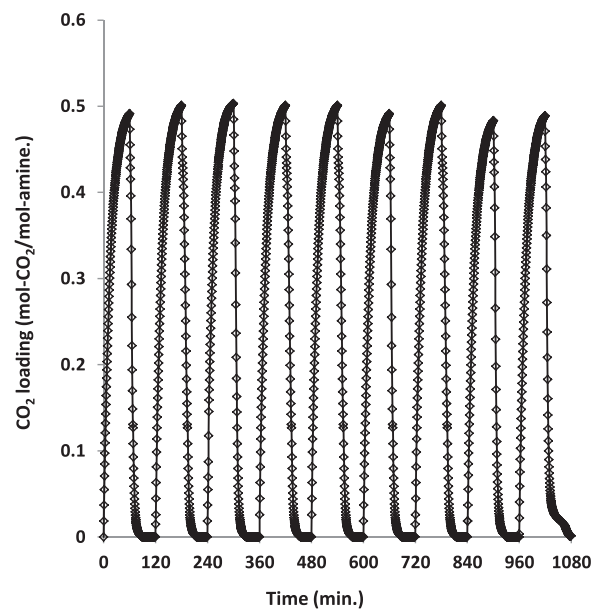


Fig. 6. Multi-cycle CO₂ absorption-regeneration profiles for the EAE_15 wt% + TEG_85 wt% solution.

reference (MEA + TEG) solution. These solutions showed relatively high absorption rates, high cyclic capacities, and high regeneration efficiencies for CO₂ compared to those of the (MEA + TEG) solution. Therefore, the specific heat capacities and heats of reaction (ΔH_r) were measured for these four solutions at 40 °C and atmospheric pressure within a range of CO₂ loading (0–0.45) mol-CO₂/mol-amine. Our results and reported data are summarized in Table 4.

Generally, ΔH_r values depend on CO₂ loadings. Consequently, this work expressed the ΔH_r values as the differential (average) enthalpies within the absorbent loading range. The specific heat and ΔH_r values for the (MEA + TEG) solution were also measured for comparison. From Table 4 it can be clearly seen that the ΔH_r values decreased in all (EAE + TEG) solutions in comparison to those of the (MEA + TEG) solution. All heat capacity values of the (EAE + TEG, Table 4, Entries 1–4) solutions are nearer the reference (MEA + TEG) solution. In fact, the heat required to regenerate the solvent in the desorber column of the CO₂ capture process can be approximated as the sum of three terms: the heat necessary to desorb CO₂ from the solution (heat of absorption), the sensible heat to increase the temperature of the solvent from the absorber to the desorber temperature, and the heat of evaporation required to produce the stripping steam in the reboiler (Oexmann and Kather, 2010). The same capacity among two solvents shows that a greater temperature swing between the absorber and the desorber, can result in a higher heat of absorption. Solvents with low heats of absorption might benefit from regeneration below atmospheric pressure and at low temperatures (Oyeneke and Rochelle, 2007). EAE-TEG solutions may also offer significant advantages over conventional aqueous amine solutions, particularly with regard to the energy required to process CO₂. EAE-based TEG solutions have a lower heat capacity of water (~2.54 J/g K vs. 4.18 J/g K) (Shimizu et al., 2006; Valkenburg et al., 2005).

3.7. VLE CO₂ solubility

The VLE solubilities of CO₂ in different CO₂ partial pressures and temperature obtained for the (EAE_70 wt% + TEG_30 wt%) and reference (MEA_30 wt% + TEG_70 wt%) solutions were shown in Fig. 7. The (EAE_70 wt% + TEG_30 wt%) absorbs much CO₂ at a given CO₂ partial pressure compared to the reference (MEA_30 wt% + TEG_70 wt%) solvent at 40 °C. As a result, for instance, the (EAE_70 wt

Table 4
Specific heat and heats of reaction of CO₂ in amine-containing TEG solutions.

Absorbent concentration	Specific Heat ^a J/g K	Heats of reaction ^b kJ/mol-CO ₂	CO ₂ loading (mol-CO ₂ /mol-amine)
1. EAE_50 wt% + TEG_50 wt%	2.53	87.4	0–0.49
2. EAE_60 wt% + TEG_40 wt%	2.54	86.9	0–0.47
3. EAE_70 wt% + TEG_30 wt%	2.54	85.4	0–0.43
4. EAE_80 wt% + TEG_20 wt%	2.59	85.3	0–0.42
5. MEA_30 wt% + TEG_70wt% (reference)	2.41	96.8	0–0.44

^a Specific heats of all (EAE + TEG) and (MEA + TEG) solutions were measured at 40 °C and atmospheric pressure.

^b Heats of reaction were measured at 40 °C and atmospheric pressure within the range of CO₂ loading (0–0.50) mol CO₂/mol amine.

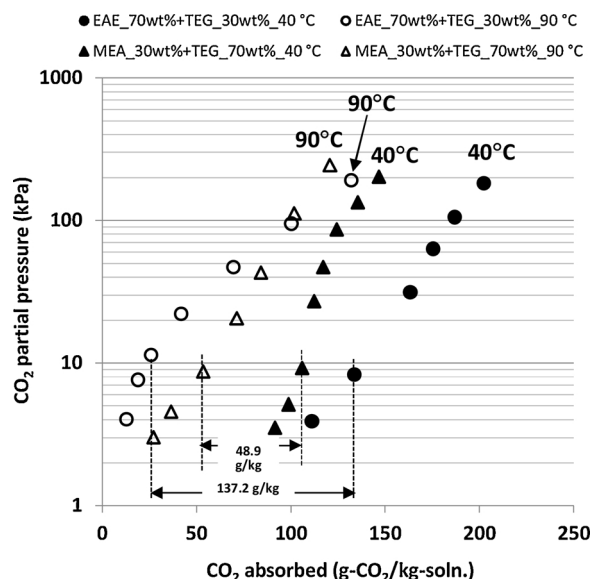


Fig. 7. The effect of CO₂ partial pressure and temperature on CO₂ solubility.

% + TEG_30 wt%) solution shown has an effective cyclic capacity 137.2 g/kg under 10 kPa CO₂ partial pressure, which is 2.8 times larger than that of (MEA_30 wt% + TEG_70 wt%) as shown in Fig. 7. Therefore, it can be concluded that the (EAE_70 wt% + TEG_30 wt%) has a potential to achieve larger working capacities with smaller changes in temperature, which allows this solvent to be regenerated at lower temperatures, providing the possibility to use low-quality steam.

3.8. Proposal of suitable (EAE + TEG) absorbents

For the selection of new (EAE + TEG) absorbents, this work focused on absorbents that had lower heats of reaction, higher absorption rates, higher cyclic capacities, and higher regeneration efficiencies compared with the reference (MEA + TEG) absorbent. Absorbents with the aforementioned characteristics are expected to capture CO₂ from gas streams using less energy. Fig. 8 shows the relationships in terms of the reaction heat versus absorption rate and cyclic capacity for four different EAE based-TEG absorbents (1–4) with the reference (MEA + TEG) absorbent 5. Fig. 8 shows the absorption rate and cyclic capacity values taken from Table 3 and heats of reaction acquired from Table 4. Although aqueous MEA has been considered a benchmark absorbent for post-combustion CO₂ removal, in this work both MEA-based TEG/EGL absorbents show a lower cyclic capacity, lower absorption rate and higher heats of reaction compared to the absorbents in Table 2 and 3. All four (EAE + TEG) absorbents in Fig. 8 show a lower heat of reaction, excellent cyclic capacity, and decent absorption rate compared to the reference MEA-based TEG absorbent 5. Particularly, absorbents (3–4) are the most suitable absorbents for CO₂ separation using less energy.

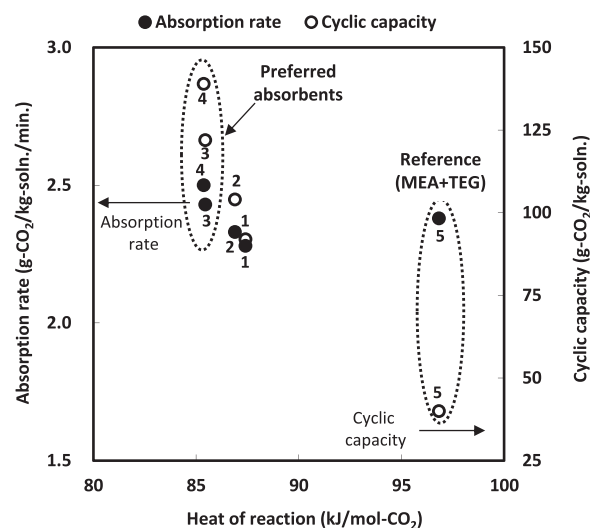


Fig. 8. Comparison of CO₂ capture performances of (EAE + TEG)-based absorbents with reference to (MEA + TEG) absorbents. The absorbents used in this study are as follows: 1. (EAE_50 wt% + TEG_50 wt%), 2. (EAE_60 wt% + TEG_40 wt%), 3. (EAE_70 wt% + TEG_30 wt%), 4. (EAE_80 wt% + TEG_20 wt%), and 5. (MEA_30 wt% + TEG_70wt%) as a reference.

3.9. Water tolerance of non-aqueous solvent

Finally, general considerations about the water tolerance of the non-aqueous solvent system are briefly addressed. To develop non-aqueous solvents for post-combustion CO₂ capture, a sound understanding of water tolerance is required because water vapor is a component of flue gas and ubiquitous in the process. Typical power plant flue gas contains roughly 10% water by volume after the SO₂ scrubber and particulate baghouse. Many formulated non-aqueous/water-lean solvents exhibited critical issues in the presence of water, for example, precipitation, forming a second phase, indicating formation of bicarbonate and formation of a solid. Water control is very important to non-aqueous/water-lean solvent performance because lower water content is linked to reduced reboiler heat duty (Mathias et al., 2013; Lail et al., 2014; Koech et al., 2011). However, lack of water management strategies will be limiting their advantages compared to their aqueous counterparts. Recently, there have been considerable works in non-aqueous solvent development and some of the notable solvents have shown acceptable water tolerance using conventional infrastructure. For example, Heldebrant et al. (2010) demonstrated water tolerance for concentrated primary, secondary, and diamine systems; Perry et al. (2011, 2012) demonstrated that aminosilicones have a significant water tolerance of over 10 wt%; Koech et al. (2013) showed their alkanolguanidine could dissolve 50% bicarbonate in solution without any observed precipitation, making a vast improvement in water tolerance. Lail et al. (2014) demonstrated not only water tolerance but also water management, by separating their CO₂-rich phase from the water phase. All these results indicate that costly dehumidification systems such as the Drizo process may not be required for water control. At least new configurations and

Table 5
Performances of (EAE + TEG) solution in CO₂ capture with or without water.

Absorbent concentration	Absorption rate ^a (g-CO ₂ /kg-soln./min.)	Absorption amount ^b		Cyclic capacity ^c (g-CO ₂ /kg-soln.)	CO ₂ regeneration efficiency ^d (%)
		(g-CO ₂ /kg-soln.)	(mol-CO ₂ /mol-amine)		
1. EAE_70wt% + TEG_20 wt% + H ₂ O_10 wt%	2.43	166	0.48	117	70
2. EAE_70 wt% + TEG_15 wt% + H ₂ O_15 wt%	2.45	169	0.49	114	67
3. EAE_70 wt% + TEG_30 wt%	2.43	153	0.44	122	80

^a CO₂ absorption rates were calculated at 50% of the 180-min CO₂ loading at 40 °C.^b 180-min CO₂ loading at 40 °C.^c Difference between CO₂ loadings at 40 and 90 °C.^d (c/b) × 100%.

process designs will further improve their water tolerance efficiency.

In this work, the CO₂ absorption and regeneration experiments in the presence of 10 wt% and 15 wt% of water to the (EAE + TEG) solvent system were done and their CO₂ capture performances are compared in Table 5. The (EAE + TEG) solution in presence of H₂O showed good water tolerance and did not exhibit any of the aforementioned critical issues and maintained a similar performance when compared to the non-aqueous solvent.

4. Conclusions

This work investigated 18 alcohols as solvents and 4 alkyl-linked alkanolamines as absorbents. Several fundamental experiments mixing alkanolamines with alcohols were performed in the laboratory to evaluate their CO₂ capture performance. This work succeeded in developing high-performance non-aqueous liquid absorbents (without solid/precipitate formation) with the advantages of higher absorption rates, higher cyclic capacities, higher regeneration efficiencies, lower specific heats, and lower heats of reaction compared to reference MEA-based non-aqueous absorbents. The new absorbents have another favorable feature; they can easily release CO₂ at a lower temperature (80–90 °C) than that of the conventional regenerative process (120–140 °C). This provides the possibility of using wasted low temperature heat at a low cost for regeneration, making the CO₂ absorption process more cost effective. All these favorable features of the new absorbents will provide basic guidelines for the discovery of potential non-aqueous amine-based absorbents.

CRedit authorship contribution statement

Firoz Alam Chowdhury: Writing - original draft. **Kazuya Goto:** Supervision. **Hidetaka Yamada:** Writing - review & editing. **Yoichi Matsuzaki:** Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:<https://doi.org/10.1016/j.ijggc.2020.103081>.

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