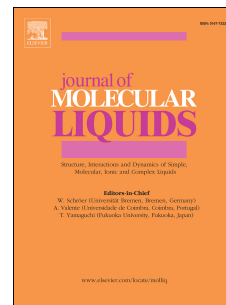


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Improving the CO₂ Solubility in Aqueous Mixture of MDEA and Different Polyamine Promoters: The Effects of Primary and Secondary Functional Groups

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Abstract

The main objective of this research is improving the CO₂ solubility in aqueous solution of MDEA by different polyamines including ethylenediamine (EDA), diethylenetriamine (DETA), triethylenetetramine (TETA), tetraethylenepentamine (TEPA) at low pressure conditions. Since the polyamines have more than one basic nitrogen groups, they could absorb CO₂ with higher rate and capacity compared to tertiary amines such as MDEA. In this regard, the performance of prepared solutions are experimented in an isothermal batch reactor in the temperature range of 293.15 to 313.15K, promoter fraction ranged from 0 to 15%, and pressure range 0 to 200 kPa. The prepared solvents are evaluated in terms of CO₂ absorption capacity, initial absorption rate and regeneration efficiency based on the number of ratio of secondary to primary functional groups in the promoter. The results show that increasing the promoter fraction in the prepared solution improves the absorption capacity and rate. Based on the experimental data, the higher CO₂ solubility, absorption rate, the boiling point and lower vapor pressure are the main advantages of the TEPA promoted MDEA solution in addition to higher absorption capacity and effective loading.

Keywords: CO₂ Capture; Amine solution; Polyamines; Promoter; Absorption Performance.

1. Introduction

Recently, the universal attention to global warming due to the emission of greenhouse gases has been attracted to inhibit the climate change and controlling the planet warm up.

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Typically, carbon dioxide is known as one of the main sources of greenhouse gases that is emitted because of increased consumption of fossil fuels [1, 2]. Pre-combustion and CO₂ capture from syngas, oxy-combustion and recovering exhausted gases and CO₂ capture from exhausted gases in post-combustion system are applied as the three main approaches for CO₂ capture and storage [3-5]. Post combustion capture of carbon dioxide based on chemical absorption is the most known and developed technology for CO₂ mitigation [6-8]. Increasing the efficiency of absorption in a gas-liquid system for CO₂ capture in the power sector along with other industrial zones with high emissions of carbon dioxide remains a technological challenge, which could be improved [9, 10]. During the past decades, alkanolamines are widely applied in gas sweetening and CO₂ separation units. Generally, alkanolamines are classified in three groups based on the number of hydrogen atom of ammonia substituted with other groups. The primary amines such as monoethanolamine (MEA) and secondary amines like diethanolamine (DEA) reacts faster with acidic gases such as CO₂ and need more energy for regeneration compared to tertiary amines such as methyldiethanolamine (MDEA) [11]. Nevertheless, the main advantage of tertiary amines are the higher absorption capacity, the lower energy for solvent regeneration, low volatility, and high thermal stability [12, 13]. In order to develop more efficient absorbents and to achieve the advantages of both primary-secondary and tertiary amine groups, the blend of primary-secondary and tertiary amines received more attention [3, 14-17]. Zhang et al. [14] studied the CO₂ solubility in binary mixture of MEA, MDEA and piperazine (PZ). The obtained results revealed that increasing the MDEA to PZ molar ratio in the blends not only led to the reduction in energy consumption, it also increased the cyclic capacity and the CO₂ desorption rate. Kao et al focused on the measuring CO₂ solubility in hindered amines [18]. Based on the obtained results, the high absorption capacity and low energy for solvent regeneration were the main advantages of hindered amines over conventional solvents. Singh et al. [19] studied the effect

of chain length on the CO₂ absorption performance of aqueous amines. The results showed that increasing the chain length of alkylamines causes a reduction in the initial absorption rate, while the total CO₂ loading is increased in furthermost of the amines. Hamidi et al. [20] studied the effect of DAMP on CO₂ solubility of binary mixture of MEA and MDEA. The obtained results revealed that increasing the DAMP concentration in the mixed amine solution could reduce the energy consumption due to the reduction in acquired circulation rate of solvent in industrial scale. Kim et al. [21] investigated the performance of different polyamines including MAPA, DETA, TETA, and TEPA for CO₂ capture in a semi-batch system. The obtained results revealed increasing absorption performance with the number of amino groups in the structure of applied amines. Muchan et al. [22] investigated the CO₂ absorption capacity, rates, solvent regeneration and desorption rate of pure aqueous solution of polyamines including EDA, DETA, TETA and TEPA. The obtained results showed that polyamines with the higher amine groups revealed more tendency for CO₂ absorption and desorption.

Most of the previous studies, investigated the polyamines as the main absorbing solvent for CO₂ separation. However, increasing the viscosity of these amines during the CO₂ absorption is the main drawback that prevents the application of these components in industrial scale. Thus, these amines could be applied as promoter of conventional amines such as MDEA to improve the absorption rate and capacity. Thus, the main goal of this study is to investigate the performance of aqueous mixture of MDEA and polyamines for CO₂ capture at low pressure. The MDEA is a commercial tertiary solvent, which is widely applied in industrial process to capture acid gases. The amines with more than one basic nitrogen group in its structure could absorb CO₂ with higher rates and capacities compared to tertiary amines such as MDEA. Thus, in this work, the absorption performance of MDEA solution promoted by

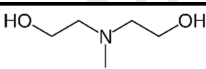
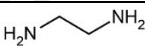
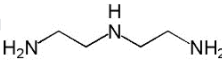
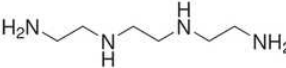
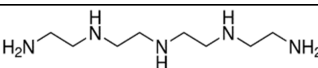
1 EDA, DETA, TETA and TEPA is investigated at different promoter concentration, operating
2 temperature and equilibrium pressure.

3 2. Material and Methods

4 2.1. Chemicals

5 Ethylene diamine, diethylenethriamine, triethylenetetramine, tetraethylenepentamine were
6 supplied from Merck Millipore company and used as received without further purification.
7 MDEA was purchased from Dae Jung Company with the purity of more than 99%. A
8 commercial grade of carbon dioxide gas was applied with purity of 99.9%. The physical
9 characteristics of all applied amines are listed in Table 1.

11 Table 1: Physical properties of MDEA, EDA, DETA, TETA and TEPA.

| Solvent | | Chemical Structure | Number of N:NH:NH ₂ groups | M.W. (g.mol ⁻¹) | Boiling temperature (K) | Partial pressure at 293.15K (Pa) |
|------------------------|------|---|---|--------------------------------|----------------------------|--|
| Methyl diethanolamine | MDEA |  | 1:0:0 | 119.16 | 520.15 | 1.0 |
| Ethylenediamine | EDA |  | 0:0:2 | 60.10 | 389.15 | 1333.2 |
| Diethylenethriamine | DETA |  | 0:1:2 | 103.17 | 480.15 | 10.7 |
| Triethylenetetramine | TETA |  | 0:2:2 | 146.24 | 539.75 | <1.0 |
| Tetraethylenepentamine | TEPA |  | 0:3:2 | 189.30 | 613.15 | <1.3 |

13 2.2. Test Method

14 CO₂ absorption experiments were implemented in a low pressure experimental setup. The
15 setup consists of the reactor vessel with the effective volume of 375ml stirred magnetically,
16 pressure transmitter, data logger, isothermal oil bath, computer and gas feeding system. In
17 each absorption test, after the temperature is set at desired value, the cell is evacuated with

vacuum pump. Subsequently, the CO₂ is fed to the vessel and when the pressure and temperature is reached to the desired set points, the solvent is injected using the injection pump and the stirrer is started to agitate the mixture with about 1500rpm. The data of pressure and temperature of vessel were measured and sent to a computer by means of the data logger. The experimental conditions including partial pressure of carbon dioxide, temperature, promoter type and weight percentage of it added to the MDEA solution are tabulated in Table 2. It should be noticed that for example 10 wt.% TETA promoted MDEA is composed of 10 wt.% of 2.5 molar TETA aqueous solution, balanced with 2.5 molar MDEA aqueous solution.

Table 2: Experimental condition applied in the present work.

| Parameter | Range |
|--|-----------------------|
| CO ₂ partial pressure (kPa) | 20-220 |
| Temperature (K) | 293.15-313.15 |
| Promoter type | EDA, DETA, TETA, TEPA |
| Promoter weight percentage | 0-15 |

In addition to equilibrium tests, search for finding the reaction rate and reaction rate parameters could significantly help us for initial investigation of different promoted amine-based solutions with conventional solvents and is vital for better investigation and scale up of such solvents. The reaction of amine and CO₂ could be considered based on zwitterion mechanism. This mechanism could be useful for the modeling of CO₂ absorption in the aqueous solutions of various alkanolamines [23, 24]. With the steady-state assumption for the concentration of zwitterion ion, the reaction rate can be calculated by means of the initial slope of pressure or loading versus time. Previous studies exhibited that the overall reaction rate of amine-based absorbers follow first order reaction rate [25-27].

3. Results and Discussions

3.1. Theoretical Results

Generally, mixing polyamine and monoamine compounds is a practical solution to overcome the disadvantages of conventional amines to absorb CO_2 and achieving the desired properties of poly and mono amines simultaneously. The nitrogen functional groups in the structure of amines could be found in three different positions including primary, secondary and tertiary. Generally, the primary and secondary amines present different performance when compared with the tertiary amines for CO_2 absorption. The primary and secondary amines are very reactive and react with CO_2 through direct and indirect reactions. The carbamate is produced through direct reaction of primary and secondary amines and CO_2 , while bicarbonate ion may be produced through hydrolysis of carbamate [28]. The tertiary amines only form bicarbonate and protonated amine through indirect hydrolysis reaction [29, 30]. As the first difference, since direct reaction is faster compared to the hydrolysis, the primary and secondary amines represent higher CO_2 absorption rate compared to the tertiary amines. As the second difference, since two moles of amine react with one mole of CO_2 based on the absorption mechanism of secondary and primary amines, those amines present lower absorption capacity compared to the tertiary amine. As the third difference, the produced carbamate is more stable compared to the bicarbonate, and thus the required energy for regeneration of primary and secondary amines is higher than required regeneration energy of tertiary amines. Since the linear polyamines are members of primary and secondary amine family, they could increase the rate of CO_2 absorption in the base tertiary amine solution. On the other hand, the presence of two or more functional groups in the structure of selected polyamines results in a high CO_2 solubility compared to the mono amines. It is expected that the considered polyamines present the higher absorption capacity and rate compared to the MDEA at the same condition and could promote the performance of base solution

considerably. However, the main disadvantage of polyamines is higher regeneration energy compared to the MDEA.

3.2. Set up Reliability

In order to prove the reliability of the absorption system, the solubility of CO₂ in aqueous solution of MDEA (2.5M) is experimented at 313.15K. Fig. 1 shows the comparison of measured solubility in term of absorption capacity with the presented data in literature at different equilibrium pressures [31]. It revealed the good agreement between the obtained data in this research and presented data in the literature, which shows the reliability of the testing apparatus.

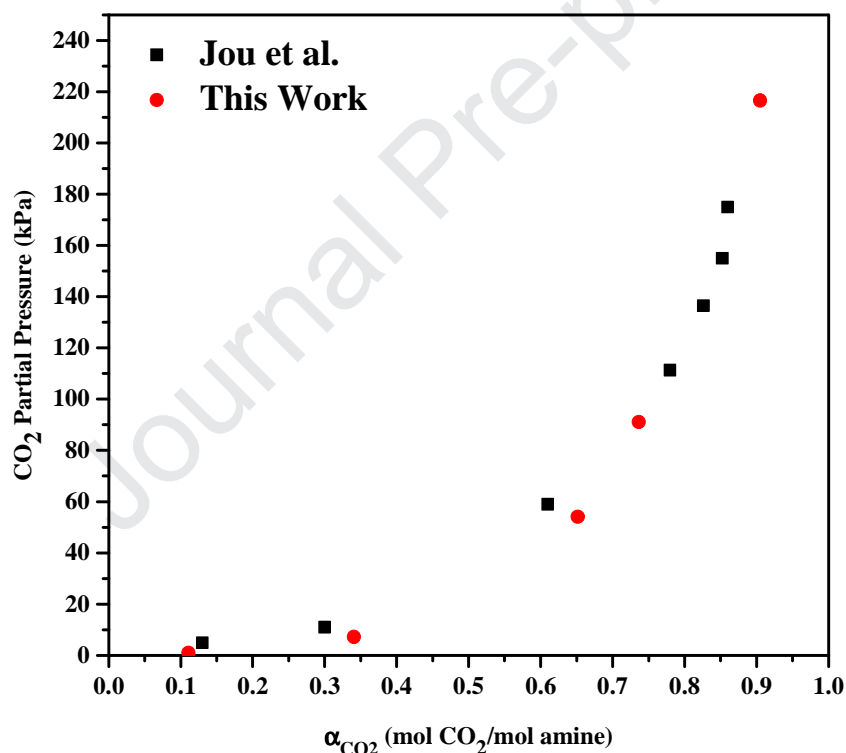


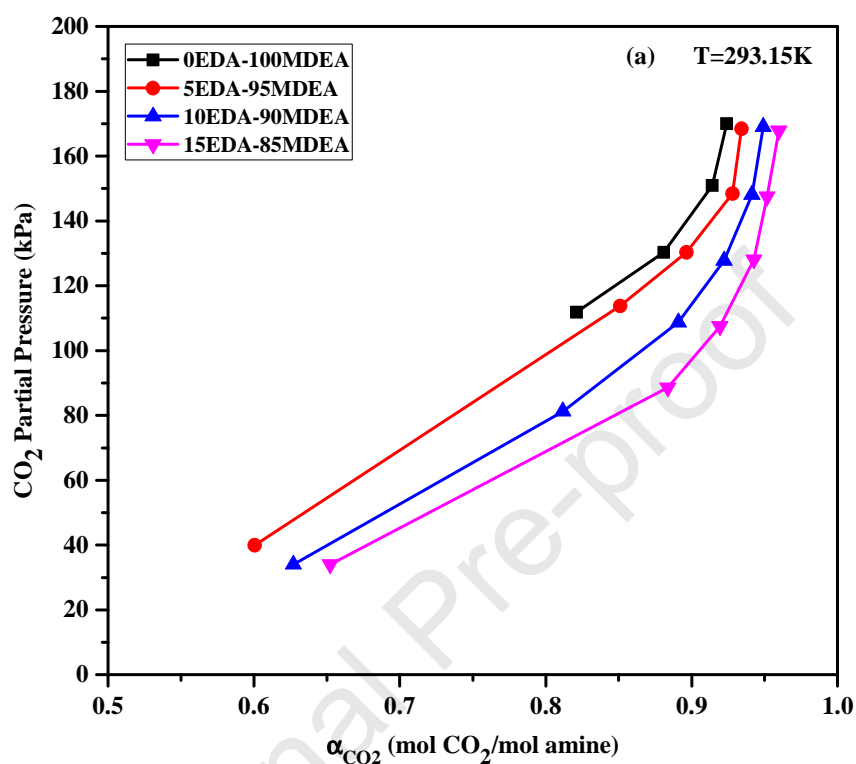
Fig. 1: The comparison of CO₂ solubility of MDEA and presented data in literature.

3.3. Equilibrium CO₂ Solubility

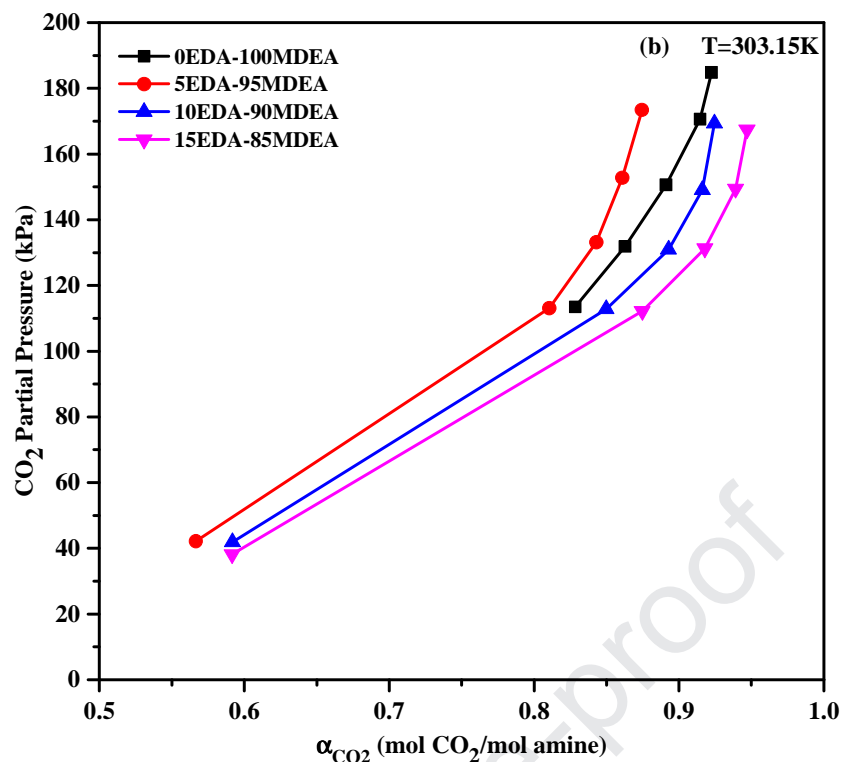
The CO₂ solubility data presents a quantitative criteria to compare the performance of absorbents and helps the process designer to select the adequate solvent for industrial process.

1 In this regard, the physical property, absorption rate, capacity and desorption characteristics
2 of solvents are measured experimentally and recorded in literatures. In this research, the CO₂
3 solubility in aqueous solution of MDEA promoted by EDA, DETA, TETA and TEPA is
4 experimented in a batch reactor at different promoter concentration, and operating conditions.
5 Since the low temperature is the favorable condition in the CO₂ absorption process by
6 amines, the tests are designed in temperature range 293-313K. Figure 2 (a-c) presents the
7 equilibrium CO₂ solubility in the aqueous mixture of MDEA and EDA at 293.15, 303.15 and
8 313.15K, respectively. Diethylenediamine as is the lowest member of ethylene polyamines, is
9 a linear compound containing two primary nitrogen groups. Although diamines presents the
10 higher the CO₂ absorption capacity compared to mono amines at the same operating
11 condition, steric hindrance influence the reaction between CO₂ and amine groups in diamine
12 compounds and could decrease CO₂ solubility. Even though EDA is a strongly base diamine,
13 it is expected that the low distance between amine groups in the chemical structure of EDA
14 results in the lower absorption capacity compared to other high molecular weight diamines. It
15 appears that increasing EDA concentration in base solvent increases CO₂ solubility.
16 Increasing the absorption capacity by mixing high performance promoters and conventional
17 amines reduces the solvent circulating rate in the industrial purification processes. Based on
18 the experimental data increasing temperature decreases gas solubility in the liquids, and
19 increasing diamine concentration in the solution could overcome the appeared decay in the
20 solubility. Increasing temperature from 293.15 to 313.15K decreases CO₂ solubility in
21 aqueous solution of MDEA from 0.925 to 0.86 at 170 kPa. In addition, increasing
22 temperature at the considered range decreases CO₂ absorption capacity from 0.965 to 0.875
23 in the binary mixture of MDEA and EDA (15%) at 170 kPa. Based on the experimental
24 increasing temperature has a more negative effect on the CO₂ solubility in binary mixture of
25 MDEA and EDA compared to the MDEA solution in the considered pressure range. In

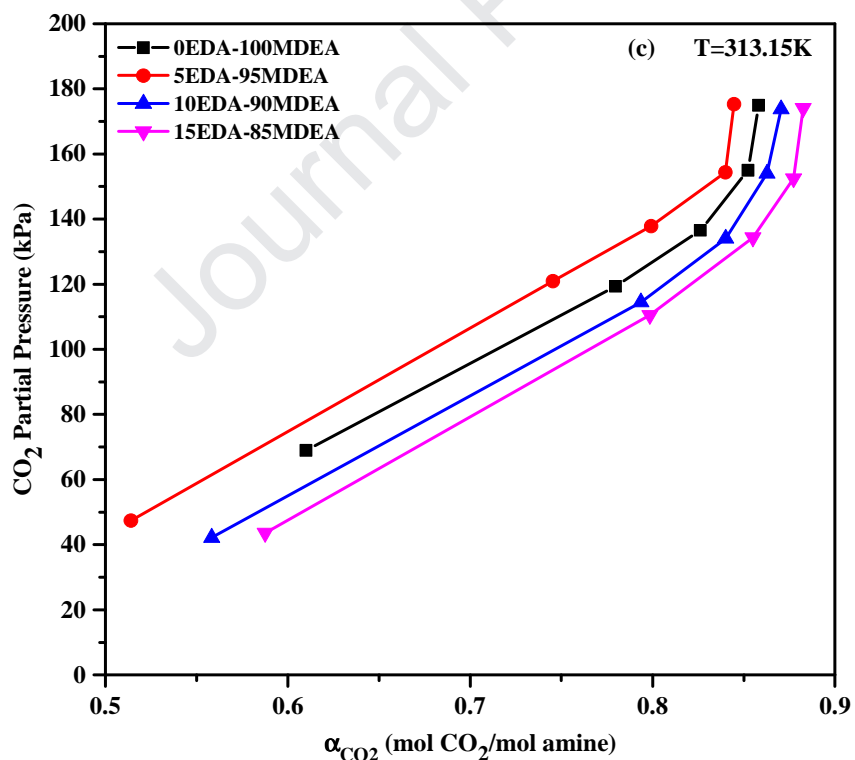
1 addition, increasing the CO₂ partial pressure has positive effect on the solubility of carbon
 2 dioxide for all absorption temperatures and concentrations according to the nature of
 3 absorption reactions [32, 33].



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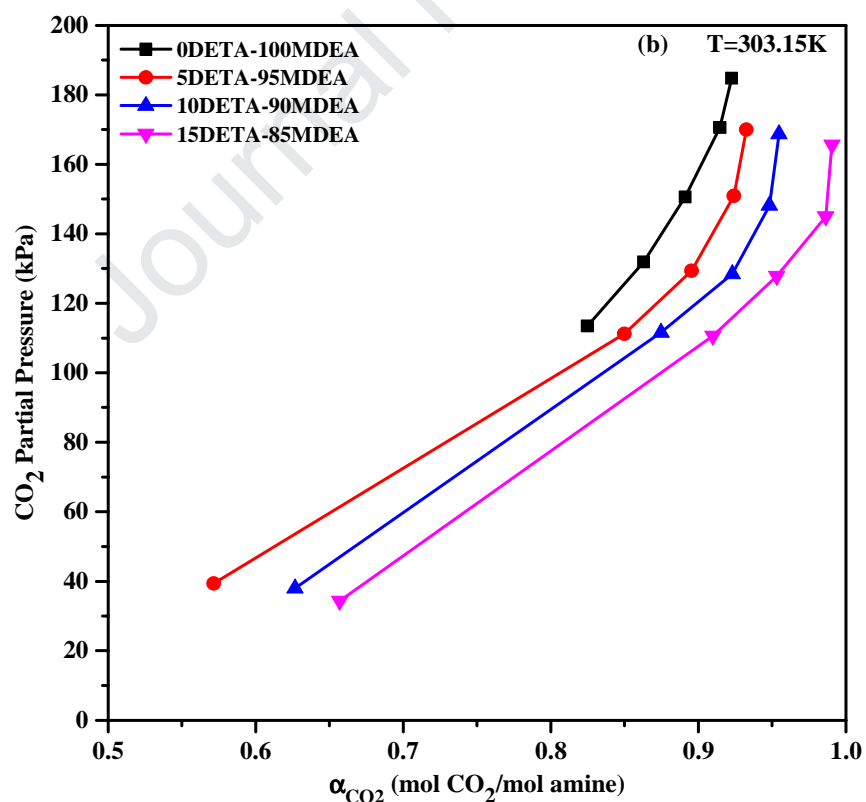
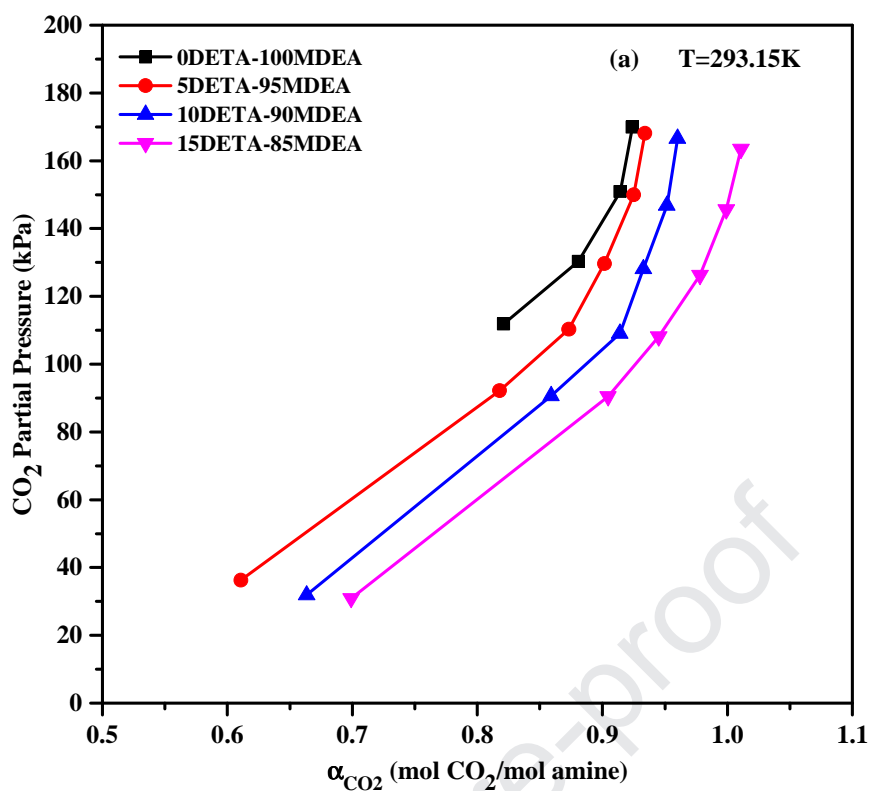
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3 Fig. 2: The effect of EDA weight percentage (0-15wt.%) on CO₂ solubility at different
 4 equilibrium temperatures of (a) 293.15K, (b) 303.15K and (c) 313.15K.

Figure 3 (a-c) shows the equilibrium solubility of CO₂ in the binary mixture of MDEA and DETA at 293.15, 303.15 and 313.15K, respectively. Diethylenetriamine as a symmetrical hydrocarbon is a linear ethylene polyamine containing one secondary and two primary amine groups. Since DETA has two primary and one secondary amine functional groups simultaneously, those groups could increase the CO₂ absorption rate and capacity in the base solution. Typically, the primary functional groups influence the absorption rate, while the secondary group enhances the CO₂ absorption capacity. Thus, it is expected that the DETA presents the higher absorption capacity and rate compared to EDA and MDEA. In addition, the chemical linearity increases absorption capacity in the prepared aqueous solution due to lower steric hindrance of nitrogen groups in the DETA. Since the EDETA is a tri-amine compound, increasing DETA concentration increases equilibrium CO₂ solubility in the prepared mixture. Based on the experimental data increasing promoter concentration in the base solution up to 15% increases absorption capacity from 0.91 to 0.99 at 303.15K and 148 kPa. In addition, increasing test temperature from 293.15 to 313.15K decreases the appeared solubility improvement in the binary mixture of MDEA and DETA from 11.1% to 9.6% at equilibrium pressure 140kPa. As well as higher CO₂ absorption capacity, the higher boiling point and lower vapor pressure are another advantages of the DETA over the EDA that decrease solvent loss in the industrial absorption and regeneration. It is confirmed that boiling points of EDA, DETA and MDEA are 389.15K and 480.15 and 520.15, respectively.



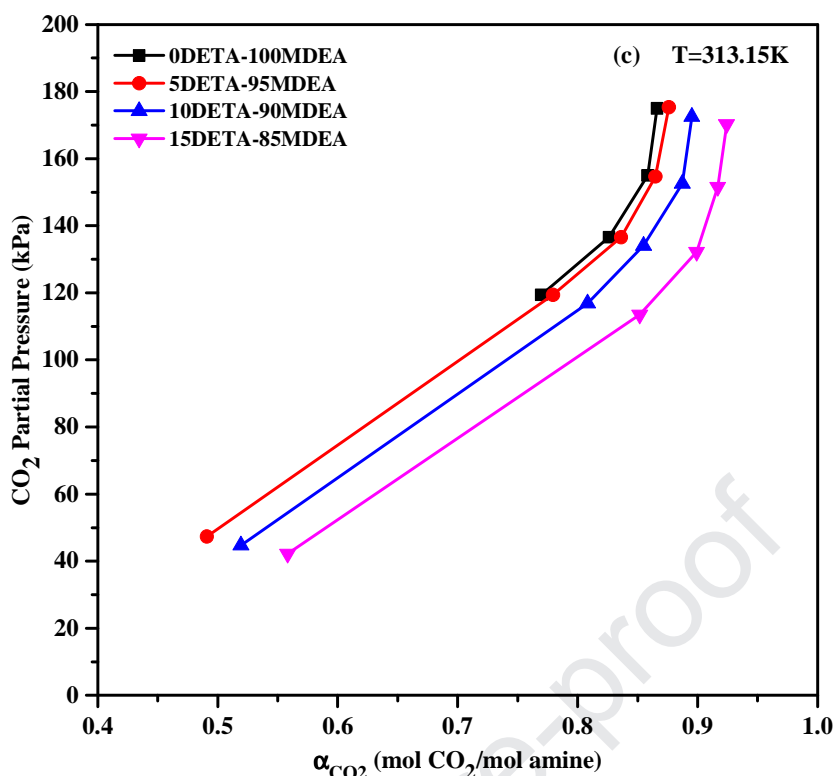
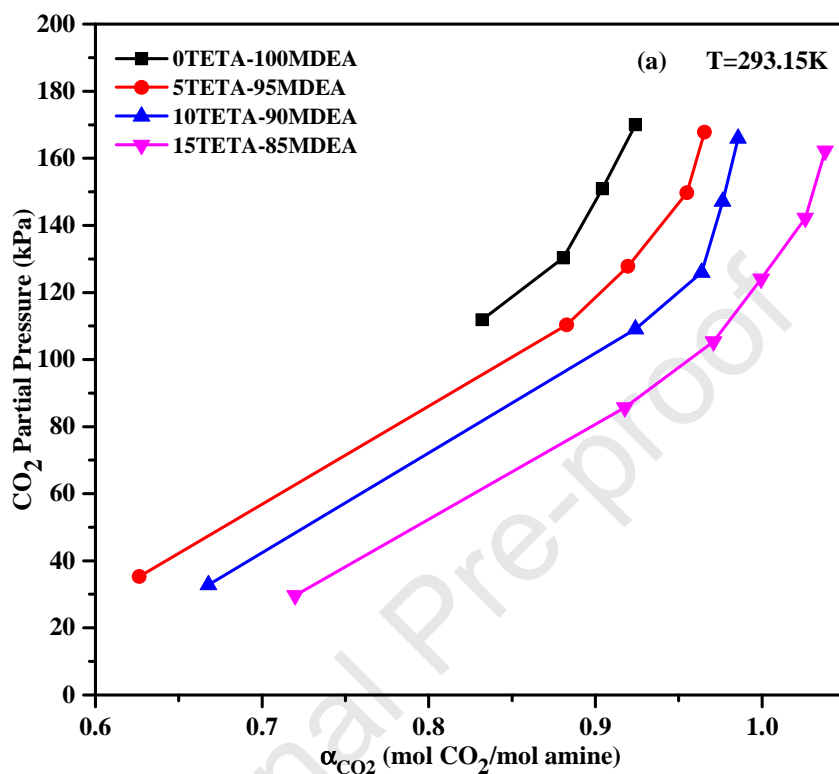


Fig. 3: The effect of DETA weight percentage (0-15wt.%) on CO₂ solubility at different equilibrium temperatures of (a) 293.15K, (b) 303.15K and (c) 313.15K.

Figure 4 (a-c) shows the equilibrium solubility of CO₂ in the binary mixture of MDEA, TETA and TEPA at 293.15, 303.15 and 313.15K, respectively. Typically, TETA has two primary and two secondary amine functional groups, while there are two primary and three secondary functional groups in the structure of TEPA. It appears that TEPA with highest number of amine groups, and secondary to primary ratio $\frac{3}{2}$ has a high affinity for CO₂ absorption. Typically, increasing concentration of polyamines in all samples increases equilibrium CO₂ solubility at the same operating condition. Based on the experimental data, increasing the TETA and TEPA fraction in the base solution up to 15% increases absorption capacity about 13.9% and 19.5% compared to the base case at 140kPa and 293.15k, respectively. It concludes that the higher CO₂ solubility and lower solvent loss through absorption and regeneration are the main advantages of TEPA compared to the DEA, DETA

1 and TETA, which make TEPA as an attractive promoter to enhance the absorption property
 2 of MDEA solution in industrial scale. The boiling points of DETA, TETA and TEPA are
 3 480.15K and 539.15K and 613.15K, respectively.



4

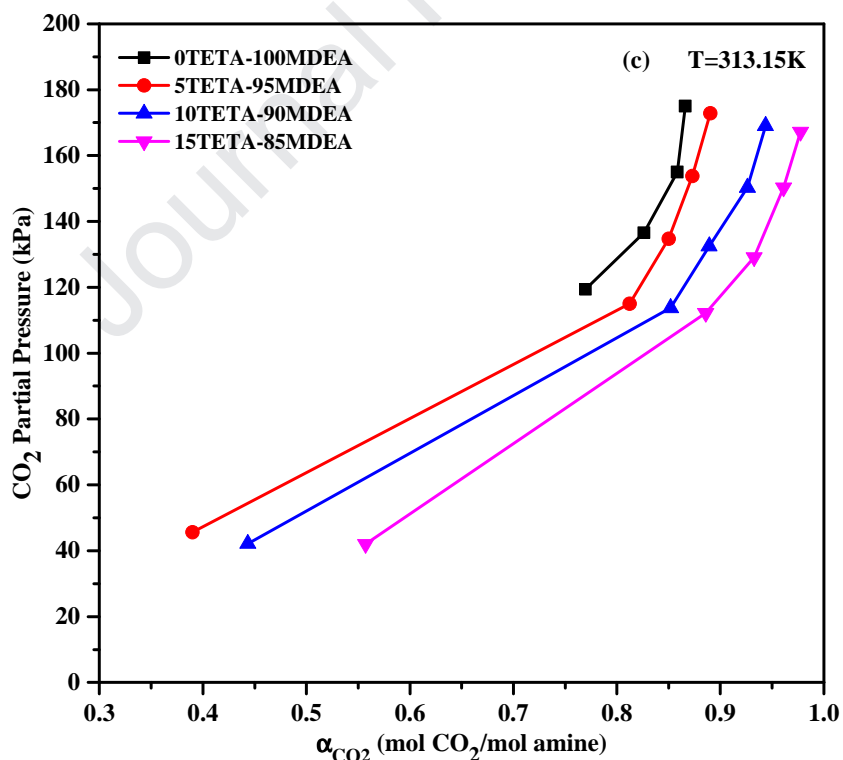
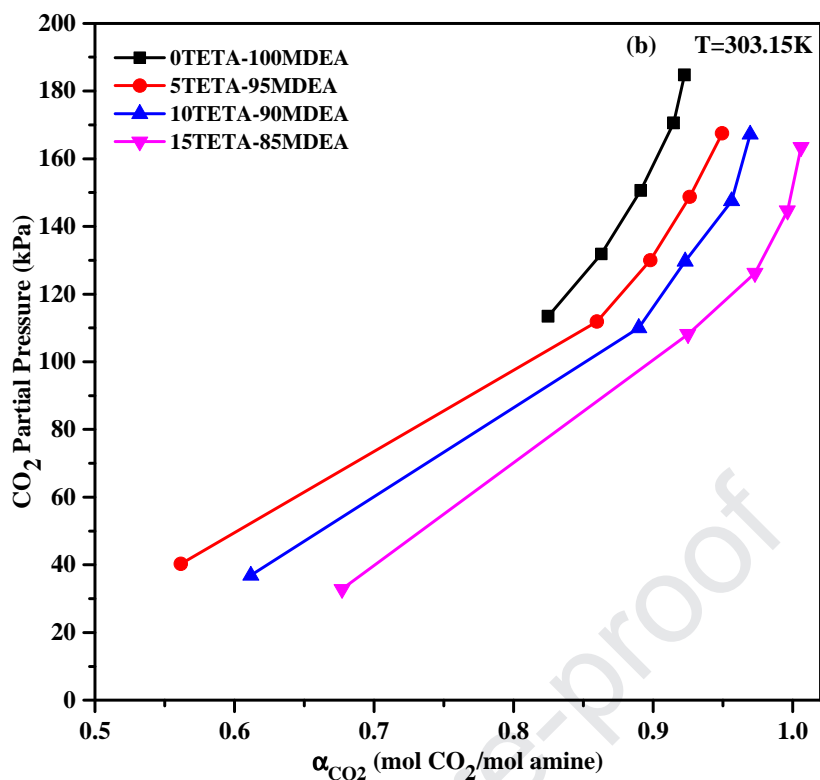
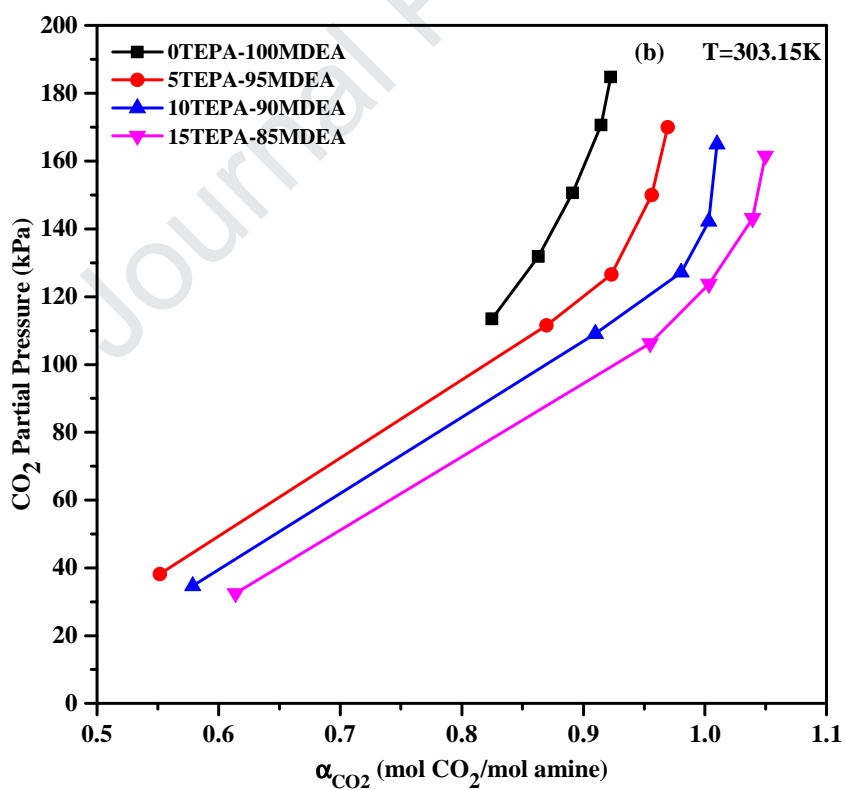
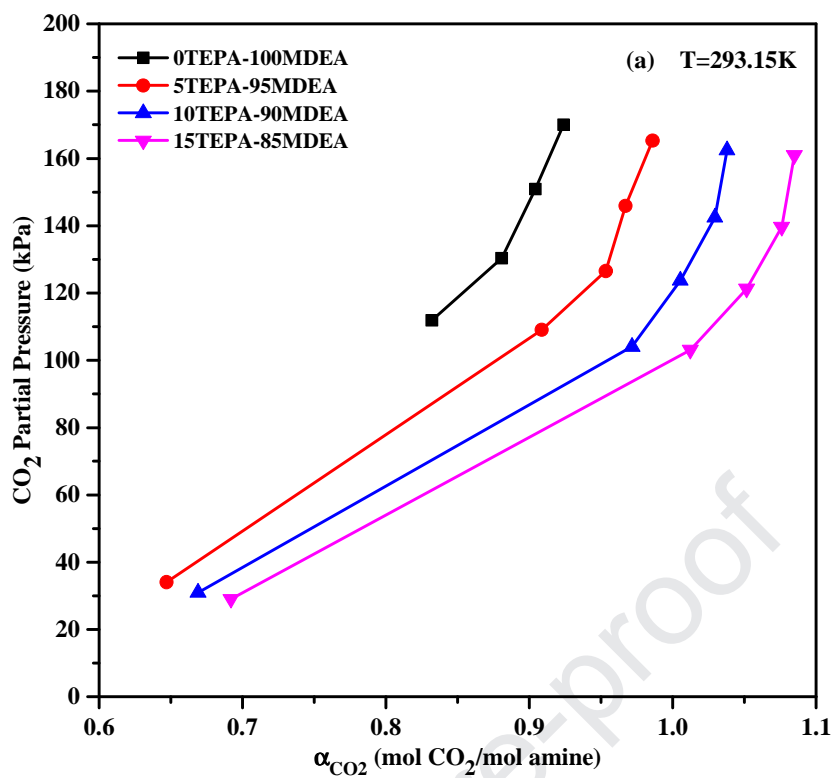


Fig. 4: The effect of TETA weight percentage (0-15wt.%) on CO₂ solubility at different equilibrium temperatures of (a) 293.15K, (b) 303.15K and (c) 313.15K.



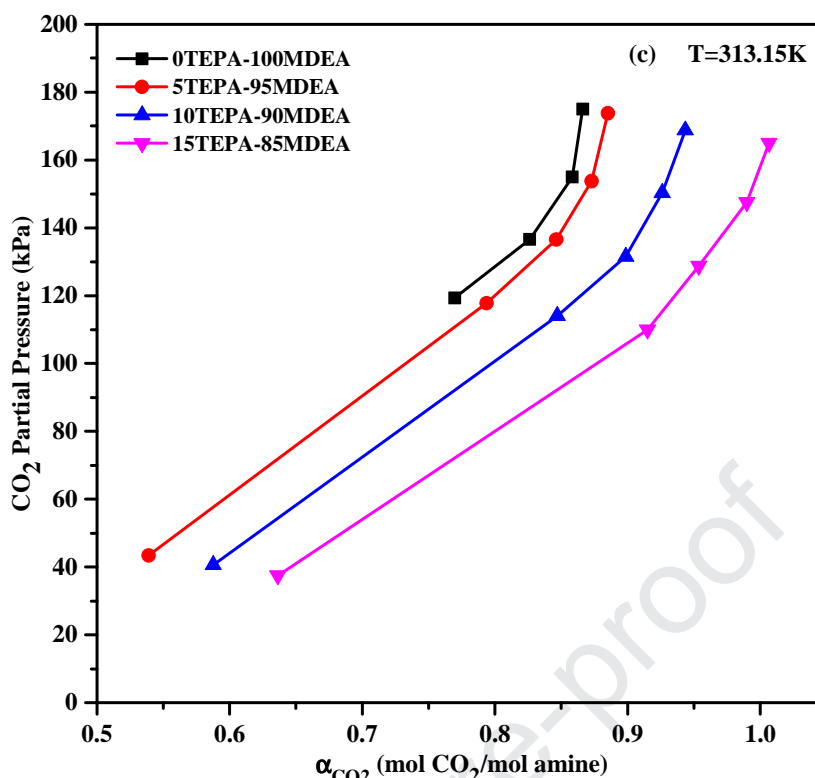
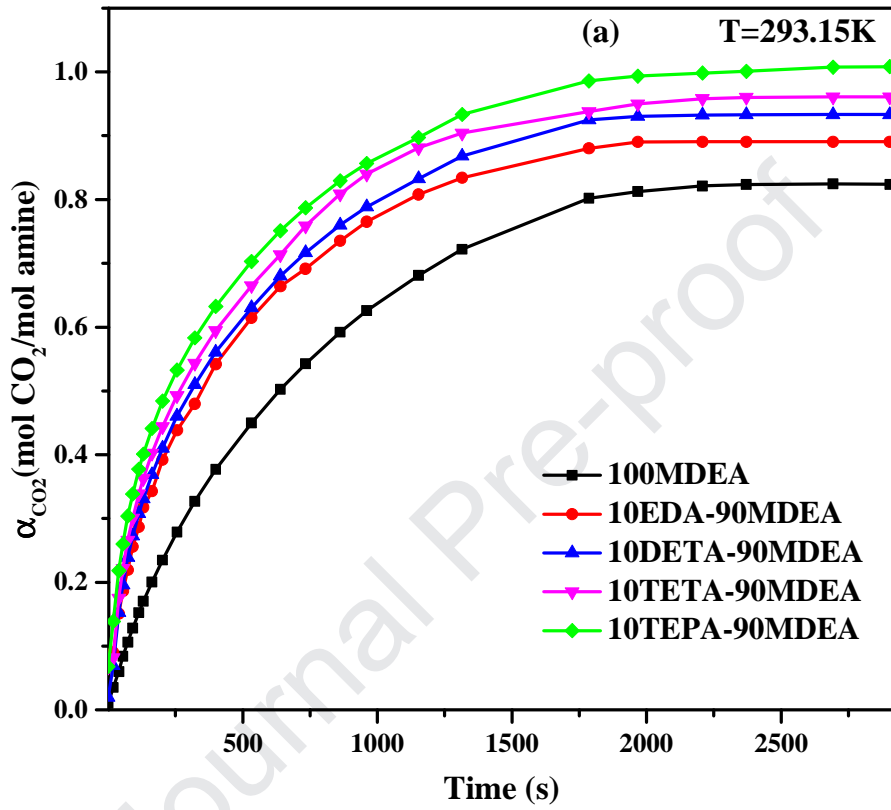


Fig. 5: The effect of TEPA on CO₂ solubility at different equilibrium temperatures of (a) 293.15K, (b) 303.15K and (c) 313.15K.

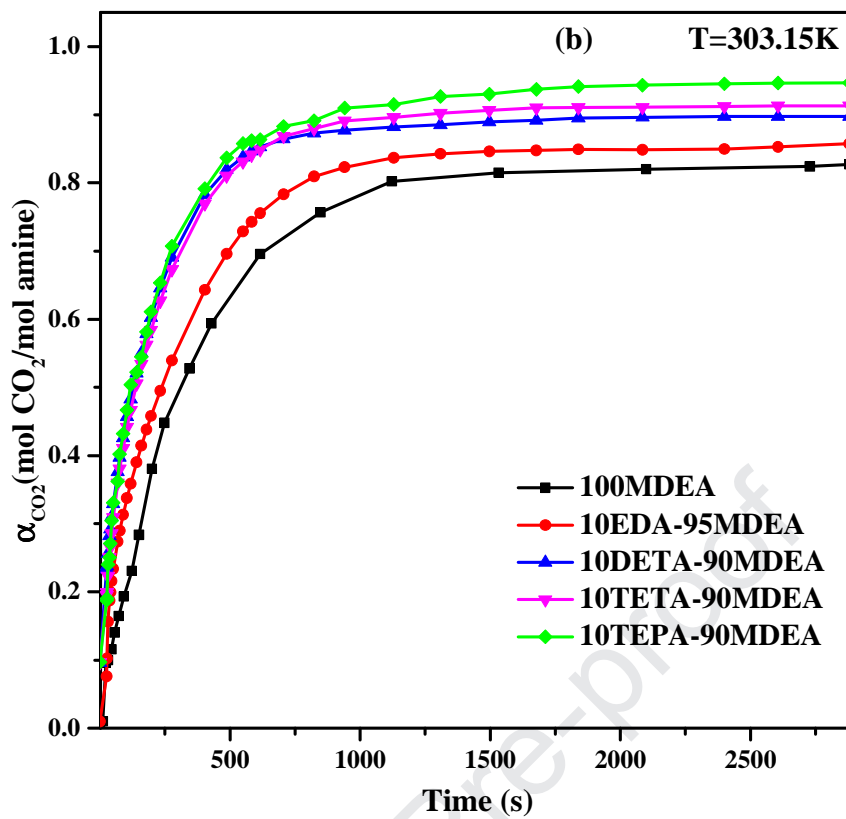
3.3. CO₂ Absorption Rate

In this section, the rate of CO₂ absorption in the prepared solution contains 10% promoter is presented at different temperatures. Figure 6 (a-c) and Figure S2 and S3 (Supplementary information) show the absorption rate of MDEA, and binary mixture of MDEA, EDA, DETA, TETA and TEPA at different operating temperatures. As mentioned, the direct reaction has faster rate compared to the hydrolysis, and thus the primary and secondary amines presents the higher CO₂ absorption rate compared to the tertiary amines. TEPA has five primary and secondary amine functional groups and the aqueous mixture of TEPA and MDEA presents the highest absorption rate between selected promoters. Since the CO₂ absorption in the aqueous solution of mono and poly amines is chemically, the temperature reveals a considerable effect on the absorption rate. It appears that increasing temperature increases absorption rate in all sample, while the absorption capacity decreases gradually.

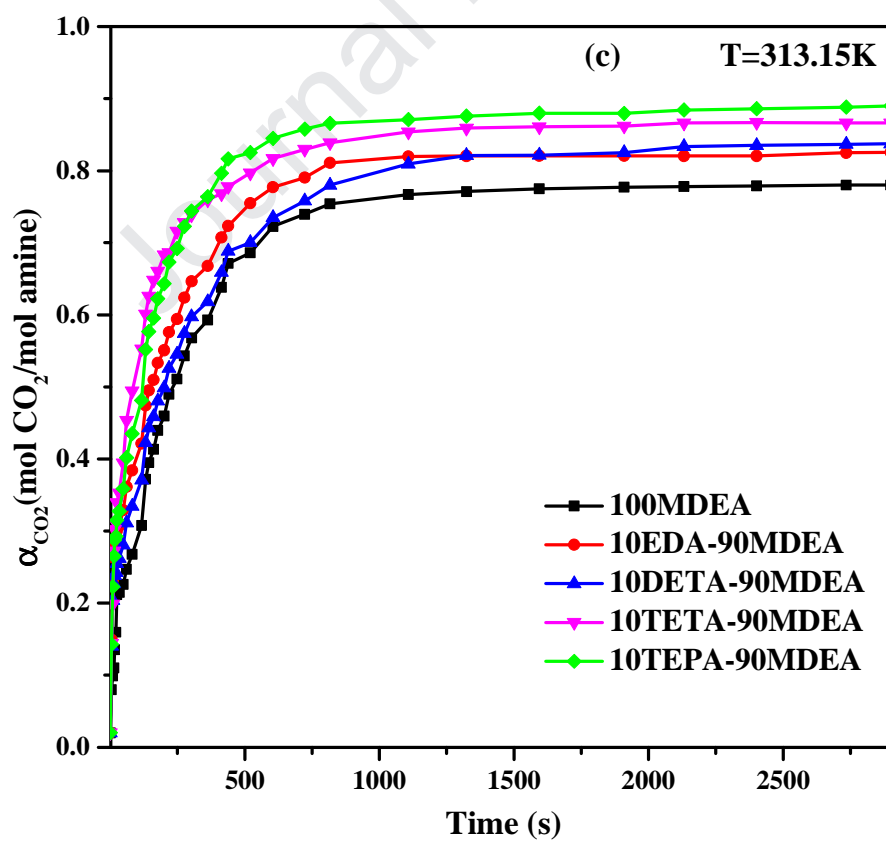
Typically, absorption rate determines the required contact time between amine and gas in the contactors and influence the tower efficiency and required number of stages. Consequently, owing to high absorption capacity and rate, the MDEA promoted by TEPA is a good candidate to improve the absorption performance CO₂ separation units.



5



1



2

Fig. 6: CO₂ loading rate of 10wt.% promoted solvents at (a) 293.15K, (b) 303.15K and (c) 313.15K.

According to Fig. 6, the rate of CO₂ absorption could be measured by means of the initial slope of each diagram at different temperatures and promoter type with 10 wt.% of promoter for $t_{0.4}$ ($t_{0.4}$ is the absorption time that 40% of the final loading is attained) [26]. The comparison of absorption rates represented in Table 3 represents that increasing the temperature and amine group in promoter structure increases the absorption rate.

Table 3: Reaction rates of 10 wt.% promoted MDEA with CO₂ for promoter types at three absorption temperatures ($P_{\text{initial}}=100$ kPa).

| T (K) | Reaction rate: $-r_{\text{CO}_2} \times 10^3$ (mole CO ₂ /l.s) | | | | |
|--------|---|------|------|------|------|
| | Promoter type (10 wt. %) | | | | |
| | Pure MDEA | EDA | DETA | TETA | TEPA |
| 293.15 | 0.45 | 0.75 | 0.75 | 0.77 | 0.78 |
| 303.15 | 2.25 | 3.00 | 3.80 | 3.80 | 3.83 |
| 313.15 | 6.25 | 6.57 | 6.45 | 6.89 | 6.90 |

3.4. Cyclic Performance

In this section, the cyclic performance of prepared solutions are compared through continuous absorption and desorption processes. The previous studies proved the formation of primary carbamate, primary–primary dicarbamate, and primary–secondary dicarbamate ions in aqueous solutions of DETA, TETA and TEPA in CO₂ absorption process [21, 34].

The stability of formed ions is ordered as primary carbamate, primary–primary dicarbamate and primary–secondary dicarbamate, respectively. This phenomenon results in the harder regeneration of polyamines compared to the tertiary amines. The cyclic performance could be helpful to investigate the effective loading and deactivation of absorbents during the continuous cycles. In this research, the loading capacity of each sample is investigated through the saturation of solvent with carbon dioxide at 303.15K followed by the regeneration at 353.15K during three cycles. Figure 7 shows the effective loading of aqueous solution of MDEA and binary mixture of MDEA, EDA, DETA, TETA and TEPA, when the weight percent of promoter in the solution is 10%. Due to the low regeneration temperature, the difference between first and second cycles are higher than that of second and third cycles. The reduction percentage of absorption capacity between second and third cycles is reduced by the addition of promoter to MDEA solvent. Although MDEA presents the lower CO₂ absorption capacity, it is easily regenerated at low temperature condition compared to EDA and DETA promoted solvents. In addition, the polyamines have higher absorption capacity and present a higher resistance to regeneration. Increasing the number of secondary to primary amine groups is the main reason for increasing the effective loading of solvents. Furthermore, the deactivation of solvent during the cycles (reduction of cyclic absorption between second and third cycles) is reduced by increasing the secondary to primary amine groups from about 3.97% for EDA promoted MDEA to 1.77% for TEPA promoted solvent. Based on the presented data, the binary mixture of MDEA and TEPA has the maximum effective loading due to higher absorption capacity, in spite of lower regeneration rate.

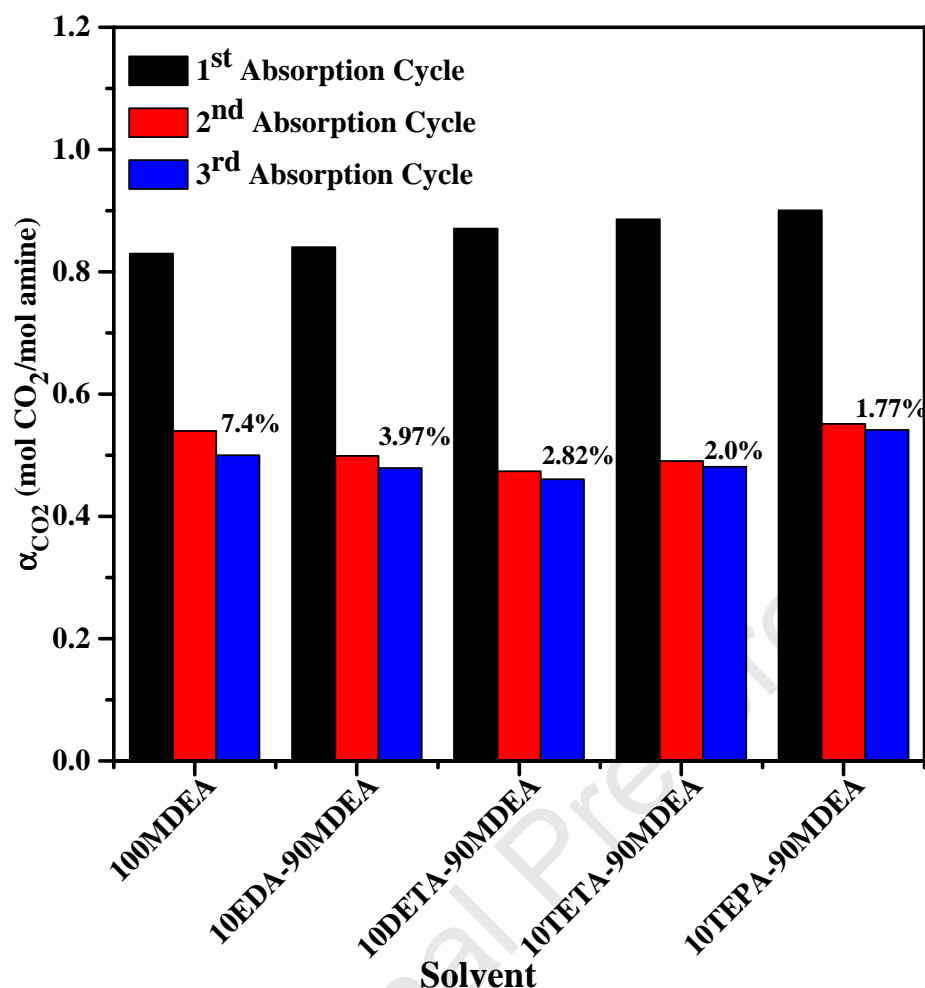


Fig. 7: Cyclic absorption/desorption performance of different solvent promoted with 10% EDA, DETA, TETA and TEPA at 303.15K.

4. Conclusion

Different polyamines with two primary and several secondary amine groups including EDA, DETA, TETA and TEPA are added as promoter to MDEA aqueous solution. The promoter type and weight percentage (5-15wt.%), and absorption temperature (293.15-313.15K) were investigated in different CO₂ partial pressures. Increasing the amine groups improved the equilibrium CO₂ capacity and absorption rate, which is the consequence of more reactive amine sites. The cyclic absorption desorption of different promoted solutions at 303.15K showed that the difference between cyclic capacities were decreased with increasing the

primary to secondary amine groups, which shows the better regeneration of solutions. The desorption results showed better and more complete regeneration of amine blends with higher secondary to primary amine groups that might be due to the formation of more secondary carbamate and dicarbamate ions compared to primary carbamate.

15wt. % TEPA promoted MDEA aqueous solution with the highest secondary to primary amine groups was found to have the highest absorption capacity and rate in all absorption temperatures. For instance, the absorption capacity is increased from about 0.91 for pure MDEA, 0.94, 0.99 and 1.01 for 15wt. % EDA, DETA and TETA in MDEA, respectively and up to 1.05 for 15wt. % TEPA promoted solvent. Considering the obtained results, TETA and TEPA promoted MDEA aqueous solutions investigated in this study can be selected as good solvents, achieving high CO₂ loading capacity and high absorption rate with good regeneration efficiency. It could lead to reduce the absorber circulating rate and regeneration energy in the absorption process, along with increase of tower processing gas or decreasing the number of stages.

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Research Highlight:

- Polyamines were examined as a potential promoter of MDEA solution in CO₂ absorption
- Cyclic CO₂ capacity of promoted amines was studied at various operating conditions
- Absorption/desorption performance is increased with secondary/primary amine ratio
- MDEA/TEPA solution is selected as the best solvent from different viewpoints