Performance Test Membrane Contactor for CO_2 Desorption from DEA

Yeni Rahmawati[1*], Salasa Ariq Sungkono[1], Zulfahmi Hawali[1], Fadlilatul Taufany[1], Susianto[1], Siti Nurkhamidah[1], Ali Altway[1]

Abstract—**Membrane-based carbon dioxide (CO2) desorption using a membrane contactor is considered a novel process for separating CO_2 from solvents. It can be carried out using temperature regeneration, sweep gas, and vacuum method. This work applies the vacuum regeneration method in the CO_2 desorption process from 30% wt of diethanolamine (DEA) solution. This study investigates the effect of operating parameters such as liquid flowrate, vacuum pressure, and CO_2 loading on the mass transfer rate and desorption efficiency of CO_2. The highest mass transfer rate of 2.2013×10^{-7} mol/m²s is achieved at a liquid flowrate of 500 mL/min, CO_2 loading of 0.27 mol CO_2/mol DEA, and vacuum pressure of 50 cmHg. In contrast, the maximum desorption efficiency of 71.45% is achieved at a lower liquid flowrate of 100 mL/min, CO_2 loading of 0.27 mol CO_2/mol DEA, and vacuum pressure of 50 cmHg. The result demonstrates that membrane contactor is a promising method for the CO_2 desorption process which requires further investigation.**

Keywords—**Carbon dioxide, Desorption, Desorption efficiency, Diethanolamine, Vacuum regeneration**

I. INTRODUCTION

Natural gas is a source of energy widely used in industry and for daily needs. Its use as an energy source continues to increase by 2% per year. This growth rate is the highest compared to those of other fossil fuels. Most of the natural gas content is a short-chain hydrocarbon compounds such as methane, ethane, and propane.

Several contaminant compounds are also found in natural gas such as sulfide acid (H_2S), CO_2, carbon disulfide (CS_2), nitrogen (N_2), sodium dioxide (NO_2), and water (H_2O). One of the dominating contaminants is CO_2 gas, whose concentration can be as high as 71% [1].

The high CO_2 concentration cause several disadvantages such as lowering heating values and low temperatures resulting in freezing and clogging in the piping systems. When mixed with water, CO_2 forms a corrosive acid that damages the piping system and equipment, increasing compression costs, and potentially poisoning the catalysts in the downstream process [2,3]. Therefore, CO_2 removal is essential to eliminate this problem [1].

The CO_2 separation method using a membrane contactor is a hybrid process that combines solvent absorption/desorption and membrane technology. A porous membrane is utilized as a contacting medium between gas and solvent in this process. The porous membranes can be either ultrafiltration or microfiltration, but microfiltration is preferred. The advantages of membrane contactors include a higher gas-liquid contact area, independent control of gas and liquid flowrates which reduces flooding, foaming, entraining, and channeling, and ease of scale-up due to membrane contactors is modular [4].

The report of CO_2 absorption–desorption using membrane contactor in separate system [5,6] or simultaneous [7-9, 3] processes can be found in several literatures. Feron and Jansen found that, compared to the conventional packed column, the hybrid process which combined membrane-based absorption and conventional desorption process significantly reduced plant footprint up to 10 times smaller [10].

Further development of membrane-based CO_2 desorption process using steam [5] or nitrogen sweep gas [11, 12] significantly reduced energy consumption because membrane-based CO_2 desorption process can be carried out at lower temperatures. In addition, lower energy consumption was achieved by the vacuum regeneration method [3, 13-15]. This method reduced solvent temperature decreasing energy requirement [16].

Rahbari-Sisakht et al. [17] investigated CO_2 desorption using membrane contactors in DEA solvents and polyvinylidene fluoride (PVDF) membrane. The desorption efficiency reached 82% by increasing the flow rate to 200 mL/minute from 50 mL/min. Listiyana et al. [3] studied the desorption process by vacuum regeneration using DEA solvents activated by arginine, piperazine (PZ), and potassium carbonate (K_2CO_3). The results showed that desorption efficiency reached 77.8% in DEA solvents with K_2CO_3.

Studies have been conducted to investigate the application of membrane contactor technology for CO_2 desorption process either experimentally [11, 12, 17, 19, 20, 21] or numerically [6, 18, 22, 23, 24]. A long-term stability test had been evaluated by Kumazawa [7] using sterically hindered 2-Amino-2- Methyl-1-Propanol (AMP) and Shimada et al. [8] using sterically hindered methyl amino ethanol (MAE) for 22 hours of operation. The results demonstrated that absorption–desorption process was stable for long-term operation. Kosaraju et al. [6] used a polypropylene membrane contactor with polyamidoamine (PAMAM) solvent simultaneously in continuous absorption and desorption processes for 55

[1] Department of Chemical Engineering, Institut Teknologi Sepuluh Nopember, Kampus ITS Sukolilo, Surabaya 60111 Indonesia. E-mail: rifqah_18des@chem-eng.its.ac.id

days using a novel solvent and helium gas as stripping gas. The results of this study indicated that the performance of the membrane contractor was stable and the material of the polypropylene membrane was not wetted by solvents.

The operating conditions of the CO_2 desorption process affect the desorption process. The desorption efficiency increases with increasing DEA concentration, liquid flowrate, and temperature [17]. The type of solvent used also affect the performance of the CO_2 desorption process using a membrane contactor. Rahim et al. [2] compared several additions of different amino acid salt solutions as solvents and observed the performance of the CO_2 absorption-desorption process. The results showed that increases liquid flowrate will increasing the desorption flux for variations in the addition of amino acid solutions to the solvent. The gas flowrate gave opposite results, it does not have a significant effect on the desorption flux. The addition of amino acids shows an insignificant effect on either temperature changes or the absorption-desorption performance [2]. Other studies on the effect of activators were also found in the literature. In the process of CO_2 absorption, it showed that the highest CO_2 removal is obtained when using PZ-DEA solution, while the highest desorption rate in the desorption process is achieved when using arginine-DEA solution. The performance stability test exhibits the stability of performance after running for 60 minutes [3].

In the CO_2 desorption process, the important factors affecting CO_2 separation are temperatures, CO_2 solubilities, sweeping gas species, and vacuum pressures. Vacuum pressures play a role in the desorption process, which have the advantage of lowering the regeneration temperatures so that, on a larger scale, low regeneration temperatures will increase economic value by reducing energy consumption. To our best knowledge, availability of reports on the effect of combined vacuum pressures and liquid flowrates on CO_2 desorption process is still limited. This study aimed to determine the effect of DEA solution flowrates, vacuum pressures, and CO_2 loadings on CO_2 desorption process performance based on membrane contactor flux and CO_2 desorption efficiency.

II. METHOD

A. Materials

The materials used in this study were hollow fiber polypropylene (PP) membrane (GDP Filter-Indonesia), 40% vol. CO_2 (balanced with N_2) from PT Samator, and 99% wt of diethanolamine (DEA) from Merck KgaA as solvent. Hydrochloric acid (HCl) 37%vol, sodium chloride 99% (NaCl), and methyl orange (MO) indicator from Merck KgaA, and aquadest were used as solutions in chittick titration. The membrane specifications are presented in Table 1.

TABEL 1.
SPECIFICATION OF POLYPROPYLENE HOLLOW FIBER
MEMBRANE CONTACTOR

Parameter	Specification
Outside diameter	0.5 mm
Inside diameter	0.35 mm
Pore size	0.1 μm
Effective fiber length	14 cm
Number of fibers	300
Porosity	0.65

B. Desorption Experiment

The performance test of membrane contactors for CO_2 desorption was carried out to determine the ability of the membrane in the desorption process by observing variations in operating parameter variables such as DEA solution flowrates, vacuum pressures, and CO_2 loadings on mass transfer rates and % CO_2 desorption efficiency. The operating conditions used were operating temperature of 70 °C, DEA solvent concentration of 30 % wt, variations in liquid flowrates from 100 mL/min to 500 mL/min, CO_2 loading variations of 0.20; 0.24; and 0.27 mol CO_2/mol DEA and a vacuum pressure between 10 - 50 cmHg. Fig. 1 shows a series of CO_2 desorption process equipment using a membrane contactor, where a DEA solution containing CO_2 with a temperature of 70°C enters the membrane contactor tube side, in the shell side it is connected to a vacuum pump for the CO_2 desorption process using variations in vacuum pressure. Sampling for the CO_2 desorption process was carried out after steady-state conditions. Analysis of CO_2 gas concentration in DEA was carried out using the chittick titration method.
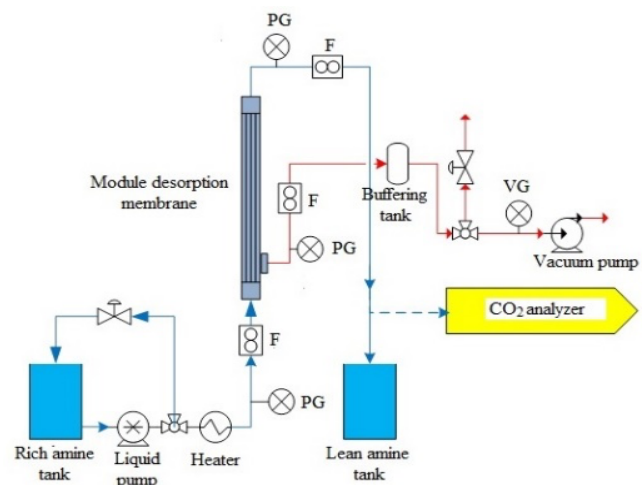

Figure 1. Experimental setup of membrane contactor for CO_2 desorption process

C. Measurement of CO_2 Loading

The concentrations of CO_2 in the DEA solution can be determined by chittick titration method. In this method, the titration was conducted with MO indicator and 1 N HCl solution as a titrant. The amount of CO_2 released from the titration was measured by reading the change of saturated NaCl solution volume inside a U-tube manometer. The concentration of CO_2 in DEA solution were calculated by following equation: [2]

$$\alpha = \frac{mol\ CO_2}{mol\ solvent} = \left[\frac{\frac{(Vgas-VHCl)(P)(273K)}{(101325Pa)(T)\left(22{,}4\frac{L}{mol}\right)}}{C_1 V_1}\right] \quad (1)$$

Where V_{gas} is the change in NaCl volume inside the manometer tube (mL), V_{HCl} is HCl volume added to reach the equivalent point (mL), P is atmospheric pressure (Pa), T is the ambient temperature (K), C_1 is solvent concentration, and V_1 is the volume of the solvent used for titration (mL).
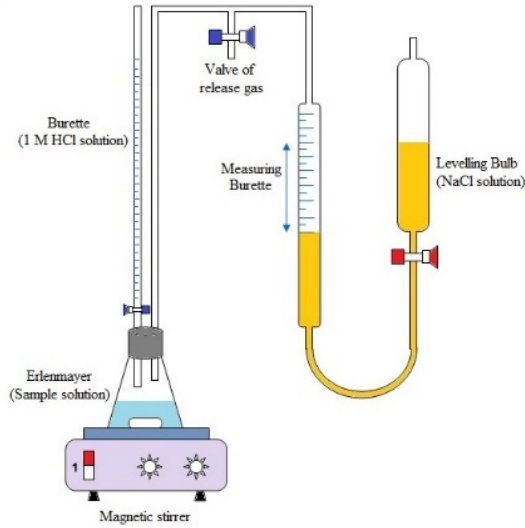

Figure 2. Chittck apparatus of CO_2 analysis

D.   Measurement of flux and CO_2 desorption efficiency

The data of CO_2 concentration in the DEA solution were used to calculate desorption flux and desorption efficiency by using Eq.2 and Eq.3.

$$J_{CO_2} = \frac{(C_{Li}-C_{Lo}) x Q_I}{A_i} \quad (2)$$

$$\eta(\%) = \left(1 - \frac{C_{Lo}}{C_{Li}}\right) x 100 \quad (3)$$

Where J_{co2} is desorption flux (mol/m^{2}s), Q_1 is liquid flowrate (m^3/s), η is desorption efficiency (%), A is the effective surface area of the membrane contactor (m^2), and C_{Li} and C_{Lo} are the liquid phase CO_2 concentration at inlet and outlet of desorber (mol/m^3).

III.   RESULTS AND DISCUSSION

The CO_2 desorption process from DEA solution using polypropylene hollow fiber membrane contactor with a vacuum regeneration method at various vacuum pressures and solvent flow rates was investigated. CO_2 stripping process from a solvent (rich amine stream) to regenerate the solvent, so that the solvent can be reused for the absorption process thus reducing the solvent cost.

In general, the important factors affecting the desorption process were operating temperatures, pressures, and flow rates. High temperatures were preferred since they reduced the solubility of CO_2 in the DEA solution and low pressures increased the partial pressure of CO_2 in the DEA

solution so that CO_2 was easily released. In this research, rich amine (CO_2 loading: 0.20, 0.24, and 0.27 mol CO_2/mol DEA) was fed to the lumen side of the membrane contactor with the varying liquid flow rates of 100 - 500 mL/min. The operating temperature was 70°C to allow solubility reduction of CO_2 in the solvent while the vacuum pressure varied from 10 to 50 cmHg.

The result shows that liquid flowrates had strong positive effects on desorption fluxes (Fig. 3 to Fig. 5). Desorption flux increased with the increase in liquid flowrates for varying CO_2 loading and had insignificant effects on changes in vacuum pressures 10 to 50 cmHg at the temperature of 70°C.
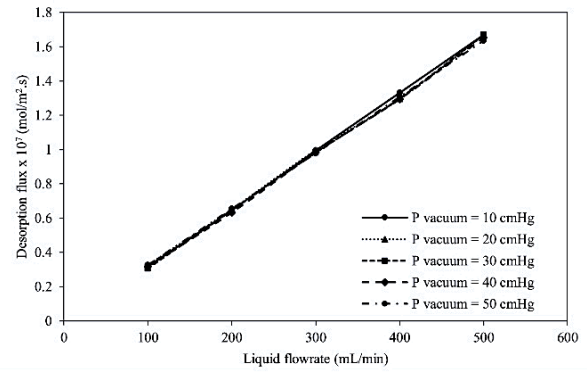

Figure 3. Effect of liquid flowrate at varied vacuum pressure to desorption flux (α=0.20 mol CO_2/mol DEA; T=70^{0}C; DEA=30% wt)

This behavior was expected since more CO_2 was introduced into the separation system at higher liquid flowrate that increase the driving force for mass transport. In addition, the increase in turbulence at higher liquid flowrate reduced film resistance thickness that increased desorption flux. The similar result was reported by Naim et al. [12] and Rahbari-Sisakht et al. [17] that the increase of liquid flowrates from 50 to 200 mL/min improved desorption fluxes from 1.5 x 10^{-5} to 1.57 x 10^{-5} mol/m^{2}s respectively, while the effect of sweep gas flowrate was insignificant.

The effect of liquid flowrate on desorption flux at varied CO_2 loading (0.24 and 0.27 mol CO_2/mol DEA) is depicted in Fig.4 and Fig.5.
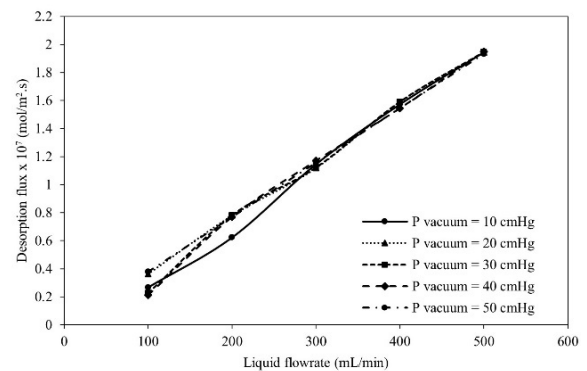

Figure 4. Effect of liquid flowrate at varied vacuum pressure to desorption flux (α=0.24 mol CO_2/mol DEA; T=70^{0}C; DEA=30% wt)

By comparing Fig. 4 and Fig. 5 it can be concluded that the difference in CO_2 loadings had a significant effect on its desorption flux. The increase in desorption flux at

higher CO_2 loading was mainly due to the increase in the driving force. For example, at a vacuum pressure of 50 cmHg and a flowrate of 500 mL/min, the desorption flux at CO_2 loading of 0.2 and 0.27 mol CO_2/mol DEA reaches 1,6362 x 10^{-7} mol/m^{2}s dan 2.2013 x 10^{-7} mol/m^{2}s, respectively. The increase in the value of the desorption flux indicated that the increase in CO_2 loading from 0.20 to 0.27 mol CO_2/mol DEA can increase desorption flux up to 25.669% as presented in Fig. 6.
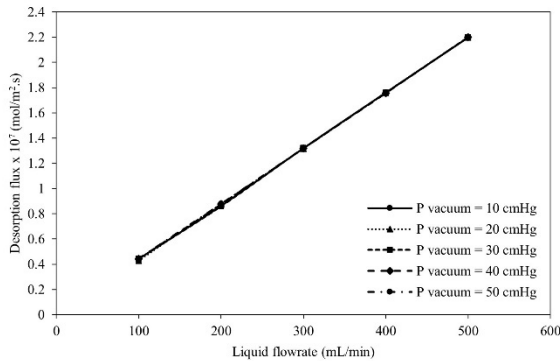

Figure 5. Effect of liquid flowrate at varied vacuum pressure to desorption flux (α=0.27 mol CO_2/mol DEA; T=70°C; DEA=30% wt)
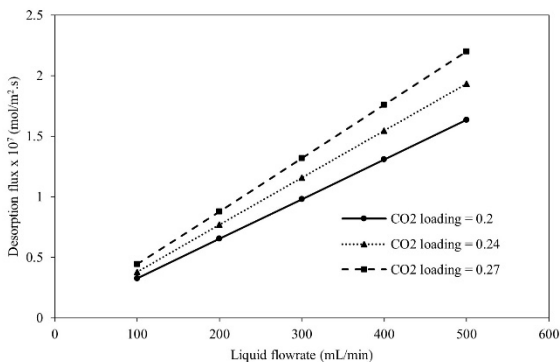

Figure 6. Effect of liquid flowrate at varied CO_2 loading to desorption flux (P$_{vacuum}$ = 50 cmHg; T=70^{0}C; DEA=30% wt)

On the other hand, the effect of vacuum pressure (from 10 to 50 cmHg) on desorption fluxes was insignificant. The desorption process was carried out at a high temperature, in this case, 70°C, to release CO_2 from the DEA solution. The large amount of liberated CO_2 forms a film layer on the liquid-membrane side which increases mass transfer resistance therefore a higher level of vacuum pressures was required to strip CO_2 [23].

The effect of liquid flowrate on desorption efficiency is presented in Fig. 7 to Fig. 9.
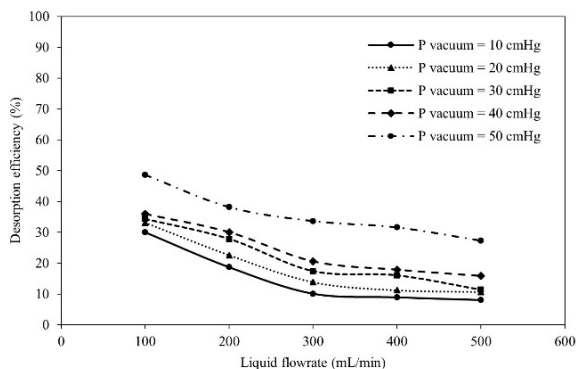

Figure 7. Effect of liquid flowrate at varied vacuum pressure to

desorption efficiency (α=0.20 mol CO_2/mol DEA; T=70^{0}C; DEA=30% wt)
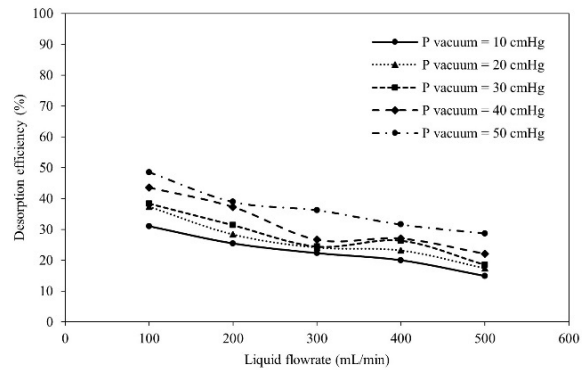

Figure 8. Effect of liquid flowrate at varied vacuum pressure to desorption efficiency (α=0.24 mol CO_2/mol DEA; T=70^{0}C; DEA=30% wt)
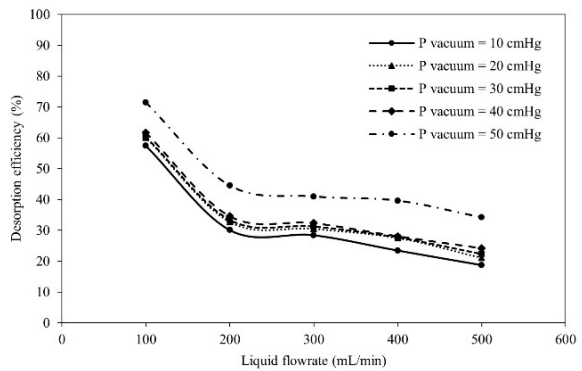

Figure 9. Effect of liquid flowrate at varied vacuum pressure to desorption efficiency (α=0.27 mol CO_2/mol DEA; T=70^{0}C; DEA=30% wt)

According to Fig. 7 – Fig. 9, the negative effect of the increase of liquid flowrate on desorption efficiency was observed. The maximum desorption efficiency of 71.45% was achieved at the lowest liquid flow rate of 100 mL/min, CO_2 loading of 0.27 mol CO_2/mol DEA, and vacuum pressure of 50 cmHg respectively. The reduction in desorption efficiency at higher liquid flowrate was estimated due to the decrease in solvent residence time in the membrane contactor module thus affecting mass transfer coefficient. This explanation is also supported by the report presented by Fang, et.al.[5], Khaisri, et.al.[19], Mansourizadeh and Ismail [4] in the literature.

Chung et.al. [25] and Koonaphapdeelert et.al. [26] stated that the CO_2 desorption efficiency can be caused by a combination of the effect between a decrease in the mass transfer coefficient due to a decrease in Reynold's number and an increase in gas-liquid contact time. The higher gas-liquid contact time occurs if the liquid flow rate is lower, this causes in the desorption process more carbamate and protonated absorbent molecules in the bulk liquid of rich amine solution thereby increasing the driving force of CO_2 mass transfer [16].

Fig. 7 to Fig. 9, shows a decrease in CO_2 desorption efficiency against an increase in liquid flow rate. For example, at a vacuum pressure of 50 cmHg, desorption efficiency decreases with varied CO_2 loading 0.2; 0.24; 0.27 moles CO_2/mol DEA which is 43.925%; 40.873%; 52.165% respectively, for more details presented in Fig. 10 the effect of liquid flow rate on desorption efficiency on

CO_2 loading variations. This significant decrease can be caused by the influence of changes in the reaction equilibrium constant, desorption reaction rate, and solvent viscosity. An increase in solvent viscosity can decrease the molecules diffusivity of CO_2 in DEA solution at a certain temperature based on the Stokes-Einstein correlation [27]:

$$\frac{D_{CO2} x \mu_L}{T} = constant \qquad (4)$$

Where D_{CO2} is the diffusivity coefficient of CO_2 in the liquid, μ_L is the viscosity of the liquid, and T is the temperature.

The diffusivity coefficient of CO_2 is related to the rate of desorption of CO_2 passing through the membrane pore, therefore a decrease in diffusivity will decrease the mass transfer coefficient of desorption.
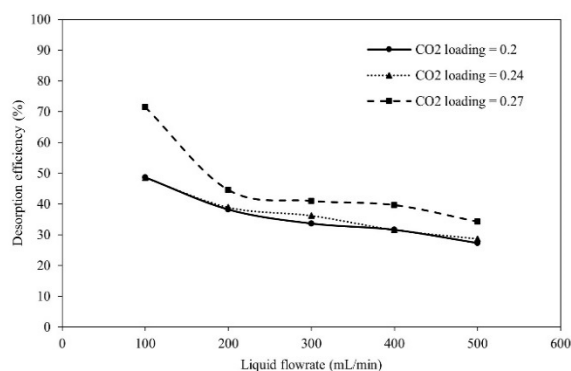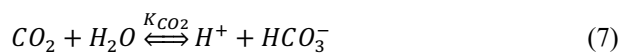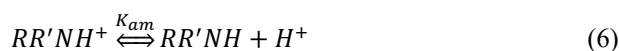

Figure 10. Effect of liquid flowrate at varied CO_2 loading to desorption efficiency (P_{vacuum} = 50 cmHg; T=70^{0}C; DEA=30% wt)

In the CO_2 absorption process, a reaction occurs between DEA solution which is a secondary alkanolamine and CO_2 gas as in the following reaction:

$$2RR'NH + CO_2 \overset{K_{ov}}{\Longleftrightarrow} RR'NCOO^- + RR'NH^+ \qquad (4)$$

$$RR'NCOO^- + H_2O \overset{K_{carb}}{\Longleftrightarrow} RR'NH + HCO_3^- \qquad (5)$$

$$RR'NH^+ \overset{K_{am}}{\Longleftrightarrow} RR'NH + H^+ \qquad (6)$$

$$CO_2 + H_2O \overset{K_{CO2}}{\Longleftrightarrow} H^+ + HCO_3^- \qquad (7)$$

Where K_{ov}, K_{carb}, K_{am}, K_{CO2} are the equilibrium constants of reversible overall reactions, carbamate hydrolysis, protonated amine dissociation and CO_2 hydrolysis, respectively.

In the absorption process, the stability of carbamates is influenced by the carbamate equilibrium constant (Eq. 5). As the equilibrium constant increases, the carbamate stability decreases and the concentration of bicarbonate in the rich amine solution increases. A large concentration of bicarbonate will increase the rate of CO_2 desorption reaction because bicarbonate decomposition is easier when the temperature is above 60°C [28]. These phenomena will increase the partial pressure equilibrium of CO_2 in the solution which will increase the driving force of CO_2 mass transfer, and consequently improve the desorption efficiency.

IV. CONCLUSION

The special design of a hollow fiber membrane contactor was constructed for the CO_2 desorption process from rich DEA solution. In this work, the effect of operating conditions such as liquid flowrate, CO_2 loading, temperature, and vacuum pressure on mass transfer rate and desorption efficiency was investigated. The result shows that the desorption flux of CO_2 increases with increasing liquid flowrate, CO_2 loading, and vacuum pressure. The highest desorption flux was achieved at a CO_2 loading of 0.27 mol CO_2/mol DEA with a solvent flow rate of 500 mL/min, and a vacuum pressure of 50 cmHg of 2.2013 x 10^{-7} mol/m^{2}s. Desorption efficiency increases with decreasing liquid flowrate, increasing vacuum pressure and CO_2 loading. The highest desorption efficiency was achieved at a CO_2 loading of 0.27 mol CO_2/mol DEA with a solvent flow rate of 100 mL/min and a vacuum pressure of 50 cmHg of 71.45%. The results show that the CO_2 desorption process using a membrane contactor can be an innovative solution to improve the efficiency of CO_2 desorption.

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