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CO₂ Removal from CO₂-CH₄ Gas Mixture Using Different Solvents and Hollow Fiber Membranes

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Commercial hollow fiber membrane modules have been investigated for the selective removal of CO_2 from the CO_2 – CH_4 gas mixture. Experiments were carried out to evaluate the effectiveness of different membrane modules and different solvents for the removal of CO_2 from a gas mixture containing 10% CO_2 and 90% CH_4 . Three different membrane modules with fibers made from polypropylene, super phobic polypropylene, and silicone rubber were used in this study. The effects of gas and liquid flow rates, temperature, solvent type and concentration, and membrane module type were investigated. The experimental results indicated that CO_2 removal increased with increasing the liquid flow rate, decreasing gas flow rate, and increasing solvent concentration. For physical absorption, the CO_2 removal decreased with increasing temperature, whereas temperature had negligible effect using chemical absorption. The polypropylene module showed the best performance in terms of CO_2 flux.

1. Introduction

Carbon dioxide constitutes about 80% of greenhouse gases which absorb infrared radiation causing an increase in earth surface temperature. It is reported that CO₂ emissions, which are produced by industry and power plants using fossil fuels, are increasing due to the significant growth in the production of natural gas as an efficient and environmentally clean fuel supply. Therefore, capture of CO₂ by various techniques has been the focus of research in recent years to avoid further increase of CO₂ levels in the atmosphere. Conventional techniques such as column absorption for CO₂ capture suffer several limitations including high energy consumption. In addition, the interdependence of the two fluid phases to be in contact with each other leads to difficulties such as emulsions, foaming, channeling, and entrainment in absorption columns.³

Membrane-based CO₂ capture is becoming the technology of interest.⁴ With the progress in materials and membrane fabrication techniques, membrane system processes became more attractive compared to conventional separation processes such as amine scrubbing which has been the preferred industrial choice for decades.

Among different membrane modules, hollow fiber membrane modules (HFMM) have been used for the majority of work reported to date. Using a suitable membrane configuration, fluids to be contacted flow on opposite sides of the membrane and the fluid/fluid interface forms at the mouth of each membrane pore. Mass transfer occurs by diffusion across the interface just as in traditional contacting equipment. Only a very small pressure drop across the membrane is required to ensure that the fluid/fluid interface remains immobilized at the mouth of the pore. The overall process can be described in three steps: first, the transfer of the solute gas from the bulk gas phase to the membrane surface; second, transfer through the membrane pores; and last, the transfer from the membrane—liquid interface into the bulk of the liquid.

Advantages membrane contactors offer over columns and other conventional mass transfer equipment include the following: (i) The two fluid flows are independent which makes the available surface area undisturbed at high and low flow rates. In contrast, columns are subject to flooding at high flow rates and unloading at low ones. Emulsion formation does not occur since there is no fluid/fluid dispersion in the membrane contactors. (ii) Scale-up is straightforward with membrane contactors. That is, membrane operations usually scale linearly so that a predictable increase in capacity is achieved simply by adding membrane modules. (iii) Interfacial area is constant and known, which allows performance to be predicted more easily than with conventional dispersed phase contactors. (iv) Substantially higher efficiency (as measured by the height of a transfer unit or HTU) is achieved with membrane contactors than with dispersive contactors. (v) Low solvent holdup is present. (vi) Unlike mechanically agitated dispersed phase columns, membrane contactors have no moving parts.

Most publications reported in the past decade on CO₂ removal using hollow fiber membrane modules (HFMM) were dedicated to CO₂/N₂ mixtures.⁶⁻⁹ Fewer papers addressed CO₂ capture from CO₂/CH₄ mixtures and were mainly devoted to the evaluation of custom-made hollow fibers.¹⁰⁻¹² Therefore, the aim of present study was to carry out comprehensive evaluation and comparison of different commercially available hollow fibers in terms of CO₂ removal efficiency, using different physical and chemical stripping solvents. Water was selected as an example of physical solvents, whereas amines and alkaline solutions were tested as chemical solvents.

2. Experimental Section

2.1. Reagents and Materials. An HFMM based on regular microporous polypropylene (PP) fibers (model G 501, 2.5 in. in diameter and 8 in. in length, 10 200 fibers/module, total membrane area of 1.4 m², Membrana, U.S.A.), a module based on asymmetric PP fibers (2.5 in. in diameter and 8 in. in length, 18 000 fibers/module, model Superphobic G 606, total membrane area of 1.25 m², Membrana, U.S.A.), a module based on regular microporous polypropylene (PP) fibers (model G 478, 1.25 in. in diameter, 9 in. in length, 3600 fibers/module, total membrane area of 0.57 m², Membrana, U.S.A.), and HFMM modules based on silicone rubber (SR) hollow fibers (model

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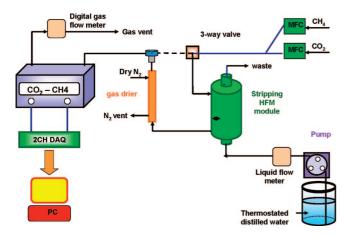


Figure 1. Experimental setup used in the determination of CO₂/CH₄ removal efficiency using stripping liquid.

M40-24000, 24 000 fiber per module, total membrane area of 2.95 m², Nagayanagi Co., Japan) were used. Water, NaOH, and different types of amines were used as stripping solvents.

2.2. Experimental Setup. The experimental setup used in the determination of CO₂ removal is shown in Figure 1. Different flow rates of CO₂ and CH₄ gas mixtures were prepared using mass flow controllers (Alicat Scientific) and fed through the shell side of the module. An online gas drier (Perma Pure, U.S.A.) based on a single Nafion tube with flowing dry nitrogen was used to dry the treated gas stream to avoid water condensation inside the analyzer. Stripping solvents were fed in counter flow arrangement through the tube (lumen) side of the fiber using a peristaltic pump (Masterflex, model 77200) with different flow rates, which were measured using a liquid flow meter (Omega Inc., U.S.A.). The concentrations of carbon dioxide and methane were analyzed simultaneously using a dualchannel CO₂ and CH₄ infrared analyzer (model 602, California Analytical Instruments, U.S.A.). The gas stream was then vented to the atmosphere, keeping the system pressure at 1 atm. Moreover, given the low gas flow rates and the short fiber length, it was reasonably assumed that the pressure drop along the length of the fibers was negligible. The retentate gas flow rate was measured using a digital flow meter (Aallborg, U.S.A.). The inlet gas composition was 10% CO₂ and 90% CH₄ throughout.

3. Results and Discussion

3.1. Physical Absorption. Experimental evaluation of the physical absorption of CO₂ was carried out using a mini PP HFM module (model G 478, 1.25 in. in diameter, 9 in. in length, 3600 fibers/module, Membrana, U.S.A.) with distilled water as the stripping solvent. The gas mixture was passed through the shell compartment, while distilled water was pumped through the tube side of the module.

3.1.1. Effect of Gas and Liquid Flow Rates on Physical Absorption. The obtained data showed significant CO₂ removal rate (as presented in Figure 2) which may be attributed to several factors such as an efficient gas/water contact, the non-circulating arrangement which brings continuous fresh water to the module, and the extent of CO₂ absorption in distilled water. The high CO₂ removal at lower gas flow rates could be explained on the basis of longer gas residence time within the module and consequently higher rate of absorption. Similar behavior is also reported by Yeon et al. ¹³ using the mixture of MEA and TEA solution in PVDF hollow fiber membrane module. On the other hand, the greater CO₂ removal with higher water flow rate is

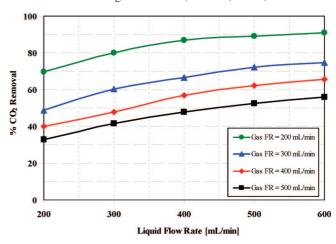


Figure 2. Effect of distilled water flow rate on the efficiency of CO₂ removal at different gas flow rates.

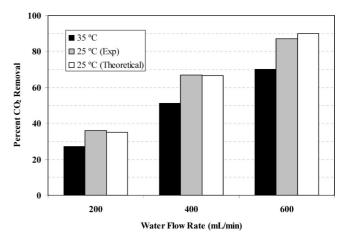


Figure 3. Effect of temperature of distilled water on the efficiency of CO_2 removal. The theoretical values are obtained by multiplying the experimental values obtained at 35 °C by 1.3. This factor corresponds to the increase in the CO_2 solubility between these two temperatures. Gas flow rate = 400 mL/min.

attributed to the faster removal of the absorbed CO_2 away from the surface of the hollow fibers by faster convection. This behavior can also be explained by the fact that, as the liquid flow rate increases, the boundary layer thickness of the liquid phase decreases resulting in an increase in mass transfer as reported by Li et al.⁸

3.1.2. Effect of Temperature on Physical Absorption. The experiments were carried out using a large PP HFM module (Model G501, 10 200 fibers/module, Membrana, U.S.A.) with distilled water as the stripping solvent. For physical absorption, the main contributor to the CO₂ removal rate is its solubility in water. The reported data of CO₂ solubility 14 shows that the solubility increases with decreasing temperature. The solubility of CO₂ (at 10 kPa partial pressure) is increased by 30% and 100% when the temperature decreased from 35 to 25 °C and 10 °C, respectively. These ratios were used to predict the percent removal at 25 and 10 °C from the experimental values obtained at 35 °C. The results presented in Figure 3 show that at a given water flow rate, the % CO2 removal was increased when lowering the water temperature from 35 to 25 °C as predicted from the enhanced CO₂ solubility at lower temperature. To quantitatively validate this assumption, the CO₂ removal data obtained at 35 °C were multiplied by 1.3 (30% increase). This factor corresponds to the increase of CO2 solubility in water when the temperature decreased from 35 to 25 °C. The theoretical values predicted at 25 °C are plotted along with the

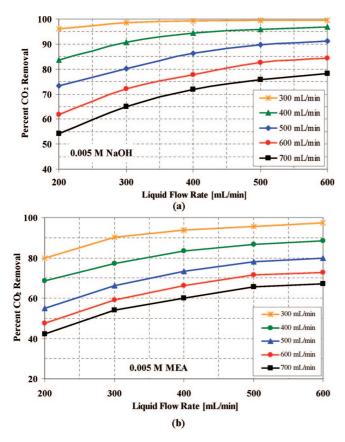


Figure 4. Effect of solvent flow rates on the efficiency of CO₂ removal for NaOH (a) and MEA (b) solutions.

experimentally determined CO_2 removal at 25 °C in Figure 3. The close agreement between the experimental data and the theoretical data calculated on the basis of enhanced solubility confirms that the removal of CO_2 under such conditions is directly proportional to its solubility in water. Similarly, the effect of CO_2 removal at even lower temperature (10 °C) followed the same general trend, namely, the enhanced CO_2 removal upon lowering the water temperature from 35 to 10 °C at a given water flow rate. The theoretical values in this case were obtained by multiplying the experimental values obtained at 35 °C by 2 (100% increase) which corresponds to the ratio between CO_2 solubility at 10 and 35 °C, respectively.

3.2. Chemical Absorption. Sodium hydroxide and monoethanol amine (MEA) solutions were used to assess the combined physical and chemical absorption of CO₂. In both cases the solvent was passed through the tube side of the membrane module (PP G478).

3.2.1. Effect of Gas and Liquid Flow Rates on Chemical Absorption. Figure 4a,b shows the effect of solvent flow rate on the efficiency of CO₂ removal for different gas flow rates. The results seem to be similar to those presented in Figure 2 for distilled water and show the following two general trends: (i) enhanced CO₂ removal at lower gas flow rates which is attributed to longer gas residence time and (ii) the effect of gas flow rate which is more pronounced at lower liquid flow rates (e.g., 200 mL/min), which are less efficient to sweep away the permeated CO₂.

3.2.2. Effect of Solvent Concentration. The removal rate using chemical solvents is generally controlled by CO_2 solubility in the solution and CO_2 reaction with the alkaline solution (combined effect of physical dissolution and chemical reaction). The experimental results of the effect of solvent concentration on the efficiency of CO_2 removal are presented in Figure 5a,b.

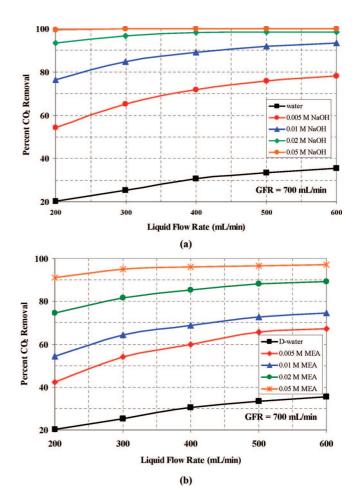


Figure 5. Effect of solvent concentration on the efficiency of CO₂ removal for NaOH (a) and MEA (b) solutions.

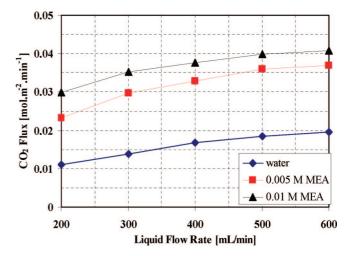


Figure 6. Effect of liquid flow rate on CO_2 flux for water and MEA solution with different concentrations. Gas flow rate = 600 mL/min.

As expected, the higher the alkali concentration, the higher the efficiency of CO₂ removal. Similar behavior was reported by several researchers.^{6,15,16} Moreover, even the lowest tested concentration (0.005 M) exhibited significantly higher removal efficiency compared to that of distilled water.

Figure 6 presents CO₂ flux as a function of liquid flow rate for water and different concentrations of MEA. The results show that the absorption flux in the presence of MEA (with chemical reaction) is higher than that when water is used as a solvent (physical). As expected, the difference between physical and chemical absorption increases with increasing the concentration

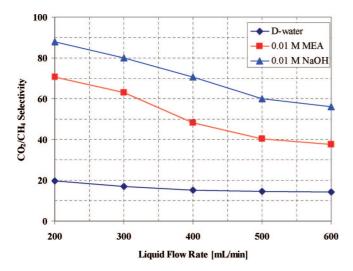


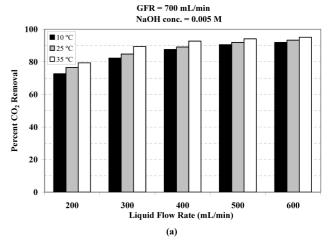
Figure 7. Effect of liquid flow rate on CO₂/CH₄ selectivity for water and MEA solution with different concentrations. Gas flow rate = 600 mL/min.

of MEA indicating higher absorption as shown Figure 6. The $\rm CO_2$ flux ratio between chemical and physical absorption is referred to as the enhancement factor (E). The calculated enhancement factor based on the experimental data presented in Figure 6 ranged from 1.8 to 2.1 for 0.005 M MEA solution and from 2.1 to 2.7 for 0.01 M MEA.

The fiber dimensions, transport and equilibrium properties, and reaction kinetics for the system presented in Figure 6 have been used to calculate the theoretical approximate enhancement factor based on the analysis presented by Versteeg el at. 17 The theoretical values for E were found to be 1.8 and 2.7 for 0.005 and 0.01 M MEA solution, respectively. It is clear that the theoretical values of the enhancement factor are in good agreement with those obtained on the basis of the experimental data.

3.2.3. CO₂/CH₄ Selectivity. Figure 7 presents CO₂/CH₄ selectivity, which was calculated based on the ratio of permeance ¹⁸ of CO₂ and that of CH₄, for different solvents (water, 0.01 M MEA, and 0.01 M NaOH). When water is used as the stripping solvent, the selectivity is almost constant (about 17) which is consistent with the ratio of the solubility of CO₂ and CH₄ in water. However, CO₂/CH₄ selectivity is much higher when MEA or NaOH solutions are used due to the fact that only CO₂ reacts (not CH₄) with MEA and NaOH. The selectivity is higher for NaOH due to higher reaction rate of CO₂ with NaOH. The selectivity decreases with increasing liquid flow rate since the residence time for the liquid in the fiber is less, resulting in less reaction time.

3.2.4. Effect of Temperature on Chemical Absorption. The effect of temperature on CO₂ removal using both solvents was also studied, and the results are presented in Figures 8a,b. Contrary to the results obtained with distilled water, the CO₂ removal showed slight increase with temperature. Unlike physical absorption where solubility was the main factor in removal rate (Figure 3), this behavior is attributed to the combined effect of solubility and chemical reaction. With increasing temperature, the solubility decreases whereas the reaction rate increases. For NaOH and MEA solutions, the increased effect due to chemical reaction seemed to compensate for the decreased effect of CO₂ solubility at higher temperatures. Similar trends were reported in the literature for different chemical solvents. ^{15,19,20} The effect of temperature on diffusion coefficients (gas and liquid), reaction rate, and solubility were considered through mathematical modeling elsewhere. ²¹



GFR = 700 mL/min MEA con. = 0.01 M

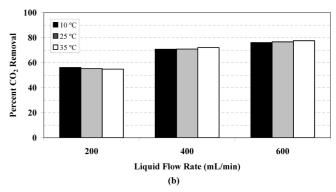


Figure 8. Effect of temperature on the efficiency of CO₂ removal for NaOH (a) and MEA (b) solutions.

3.3. Comparison of Different Commercial Modules. Comparison between different fibers and modules (PP G501, Superphobic G606 and SR M40-24,00) for the efficiency of carbon dioxide removal using distilled water, 0.05 M potassium carbonate solution, and 0.05 M sodium hydroxide solution was carried out using a 10% CO₂–90% CH₄ gas mixture at a flow rate equal to one shell volume (mL) per minute for each module, respectively. This is to provide a basis for meaningful comparison between different commercial modules having different volumes of the tube and shell compartments.

The results obtained for the three commercial modules are presented in Figure 9. As expected, the CO_2 flux increases when chemical absorbents (potassium carbonate and sodium hydroxide solutions) are used as compared to physical absorbent (water). Moreover, the CO_2 flux was higher using porous fibers (in PP and Superphoic modules) than that when nonporous Silicon Rubber fibers are used. The Superphobic module is expected to have a dense layer at the outer surface which is expected to impose an additional diffusion barrier to the gas transport resulting in lower CO_2 flux than that when PP module is used.

3.4. Comparison of Different Amine Solutions. In addition to the common MEA, DEA, and ethylenediamine (EDA), three additional amines were tested using a PP mini-module G478. These amines contain a progressively greater number of nitrogen atoms as well as both primary and secondary amine functionality. The data presented in Figure 10 shows that the removal rate of the newly tested amines increases with increasing the number of nitrogen atoms: tetraethylenepentamine (TEPA) > triethylenetetramine (TETA) > diethylenetriamine (DETA). Moreover, all these new amines showed significantly higher removal efficiency than those obtained with the known MEA,

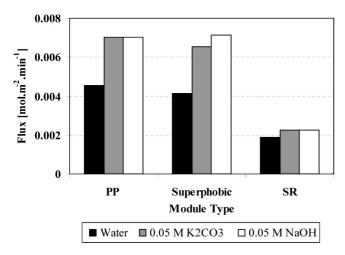


Figure 9. CO_2 flux obtained with different commercial HFMM using water, 0.05 M potassium carbonate, and sodium hydroxide as stripping liquids, respectively. Gas flow rate = 1 shell volume (mL)/min and liquid flow rate = 2 tube volume (mL)/min.

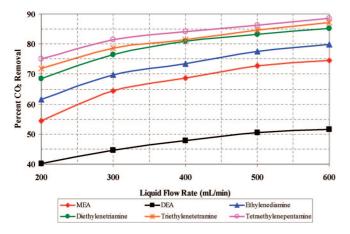


Figure 10. Comparison of the efficiency of CO_2 removal using different amine solutions. Amine concentration = 0.01 M and gas flow rate = 700 mL/min.

DEA, and ethylenediamine at the same molar concentration (0.01 M). These results are consistent with the recently published²² total capacity of 1.08, 1.83, 2.51, and 3.03 (moles of CO_2 /mole amine) of EDA, DETA, TETA, and TEPA, respectively.

The minimum liquid flow rate needed for complete CO_2 removal, $\nu_{L,min}$ [dm³/s], for a given solvent, can be calculated assuming very large mass transfer rate between gas and liquid so that the liquid leaving the module is in equilibrium with the feed gas.²³ $\nu_{L,min}$ is given by the following equation.

$$\nu_{\rm L,min} = \frac{G_{\rm M} y_{\rm CO_2,inlet}}{[CO_2]_{\rm T}} \tag{1}$$

Here, $G_{\rm M,inlet}$ [mol/s] is the molar flow rate of gas, $y_{\rm CO_2,inlet}$ is the CO₂ mole fraction in the feed gas, and [CO₂]_T is the summation of physical solubility ($H_{\rm CO_2}p_{\rm CO_2}$) and chemical solubility. $H_{\rm CO_2}$ is the Henry's law constant (= 3.36×10^{-4} mol/(kPa dm³)) and $p_{\rm CO_2}$ [kPa] is the partial pressure of CO₂ in the feed gas. The calculated ratio of the minimum flow rate of the solvent to that of water using the capacity data reported²² and eq 1 are shown in Figure 11 for 0.01, 0.1, and 1 M amine solution. The minimum solvent flow rate, $\nu_{\rm L,min}$, decreases with increasing the solvent capacity as a result of increasing the amine functionality in the molecule. As the amine concentration

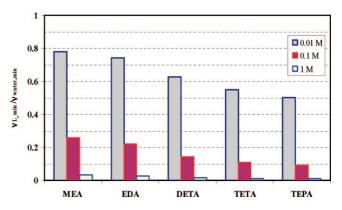


Figure 11. Calculated ratio of the minimum flow rate of the solvent to that of water for different amines.

increases, less solvent is needed for CO₂ removal, as presented in Figure 11. In addition, as the molar concentration of amine increases (0.01–1 M), the contribution of chemical solubility becomes more dominant.

4. Conclusions

A comprehensive study on the removal of CO_2 from mixture of gas streams using different commercial membrane contactors and different solvents has been conducted. The removal efficiency has been evaluated on the basis of solvent and membrane type, operating temperature, gas and liquid flow rates, and solvent concentrations.

The results showed that the CO_2 removal decreased with increasing temperature for physical absorption, whereas temperature had little effect for chemical absorption. The CO_2 removal increased with increasing the liquid flow rate, decreasing gas flow rate, and increasing solvent concentration. Among the membrane modules used, the polypropylene module showed the best performance in terms of CO_2 flux, followed by Superphobic and Silicon Rubber modules, respectively. The solvent with higher number of amino groups showed better performance in term of CO_2 removal.

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