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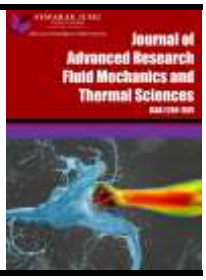

Mixed Amine Absorbent based on Hollow Fiber Membrane Contactor Removes CO_2 from Flue Gas

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ABSTRACT

The application of membrane gas absorption for the synthesis of carbon dioxide from exhaust gases is specifically explored in connection to the combined supply of heat and carbon dioxide to greenhouses. Novel absorption liquids perform better than monoethanolamine when used with easily available, reasonably priced polyolefin membranes from the market in terms of system stability and mass transfer. In this paper, the PTFE (Polytetrafluoroethylene) hollow fiber membrane contactor was decarbonized by three mixed amine absorbents, MDEA-PG, MDEA-PZ, and MDEA-MEA, focusing on optimizing the CO_2 removal process. The order of CO_2 membrane absorption performance of amine absorbent is MDEA-MEA> MDEA-PG> MDEA-PZ. The reasonable mass concentration range of the mixed amine absorption liquid is 30% to 35%. In addition, MEA, MDEA-PG, and MDEA-PZ have the highest CO_2 removal rate when the solute ratio is 1:0.6, which are 43.15%, 32.36%, and 30.55%, respectively. These results indicate significant enhancements in CO_2 capture compared to traditional single-amine solutions.

1. Introduction

N-methyldiethanolamine (MDEA) and monoethanolamine (MEA), sodium glycinate (PG) and piperazine (PZ) four amine absorbents have different CO_2 absorption characteristics. The primary amine MEA has a larger absorption rate , but the amount of CO_2 absorption is low [1].

Membrane contactor is a new type of separation technology developed in recent years. It couples membrane technology and traditional absorption process. It has the advantages of large mass transfer area, small and compact structure. It avoids the problems of liquid flooding, bubbling, and mist entrainment in the traditional absorption tower process and has a good application prospect in the field of biogas decarbonization [2]. The main advancements in membrane processing is the technology developments since in many of helium recovery methods, the current available polymeric and inorganic membranes require permeable selectivity for separation. Helium can be attained directly from natural gas or related gas processing and also from the recycling and recovering from industrial applications [3]. Aziz *et al.,* [4] used activated carbon derived from banana stems as an

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effective adsorbent in removing inorganic pollutants, specifically its performance in removing Pb2+ ions. Studied the fabrication and properties of a low-cost ceramic membrane from clay for pore sizes in the range 0.5-2.7μm was achieved [5]. Although many literatures have reported the research on CO_2 removal by membrane contactor, most of them are limited to normal pressure operation, are taken from the previous studies [6-8]. According to Henry's law, the solubility of CO_2 gas in water is directly related to the pressure. In fact, the current mainstream technology for biogas purification, namely the high-pressure water washing process, operates under a certain pressure (0.8-1.2MPa), are taken from the previous studies [9,10]. Since then, this method has been widely used and studied in the separation of acid gas from mixed gases such as flue gas, natural gas and refinery gas due to its unique advantages. Zhang and Wang [11] summarized the progress of the latest membrane technology for acid gas research, and looked forward to the engineering application prospects of the improved separation process. Ghobadi *et al.,* [12] also analyzed the influence law of the characteristics of using membrane technology to separate CO_2 in flue gas from the experimental system technology, absorbent and membrane structure and materials used. In the development of new fuels, the process of CO_2 removal from biogas purification to biological natural gas has also received close attention from some scholars. Zhao *et al.,* [13] applied membrane modules to carry out biogas purification experiments, investigated the separation performance of CH_4/CO_2 under different pressures, and optimized the design and analysis of the membrane absorption process.

However, because a single absorbent cannot satisfy the high CO_2 absorption and regeneration capacity, the development of mixed absorbents has also made certain progress. Lu *et al.,* [14] tested adding phosphate and borate to glycine solution separately, which provided higher CO_2 removal rate than GLY solution. At the same time, some researchers added alcohol amine solution additives into the amine-based solution for research, so as to obtain better absorption effect. Chen and Huang [5] used MDEA as the base liquid to compare the CO_2 absorption and absorption rate of mixed amine liquids with three different additives: MEA, DETA and piperazine (PZ). The experimental results show that DETA has the best activation effect, while MEA has the worst activation effect. Chen and Cui [15] compared the CO_2 absorption capacity of PG/MDEA and MEA/MDEA solutions with different component ratios. Among them, the CO_2 removal rate is as high as 99%, and the gas-liquid reaction lasts longer, when the ratio of additive to MDEA is 2/3. Additionally, tertiary amines are considered to be the best activating additives relative to the rest of the amine solution [15]. In short, when selecting a suitable CO_2 absorbent, it should also consider its absorption performance, regeneration energy consumption, economic investment and other factors to make a reasonable choice. The CO_2 absorption performance is dropped when the temperature raised more than 30^oC. However, higher temperatures can have unfavorable effects on the absorption process by decreasing CO_2 solubility, increasing absorbent evaporation, and increasing CO_2 back-pressure [15,16].

In this paper MDEA was selected as the main absorbent, and MEA, PG and PZ were used as additives, respectively, and new mixed amine absorbing liquids MDEA-MEA, MDEA-PG and MDEA-PZ were formed by adding MDEA absorbent solution to the PTFE hollow fiber membrane. The simulated flue gas was decarbonized on the device platform, and the CO_2 membrane absorption characteristics of the three mixed absorbents were investigated.

2. Related Work

2.1 Basic Principle of Membrane Absorption Method

The membrane absorption method is a new separation process that combines the membrane separation method and the traditional absorption method. In this process, the gas-liquid two phases contact and transfer mass at the fixed gas-liquid interface, and flow on both sides of the membrane

respectively. The membrane itself has no selectivity to the gas and only plays the role of isolating the absorbent and the gas. CO_2 diffuses to the liquid phase side through the membrane under the action of the concentration gradient. Theoretically, the membrane pores are the gas that allows one side of the membrane to be separated and can penetrate the other side of the membrane without requiring a lot of pressure. The basic principle is shown in Figure 1 (taking the hydrophobic porous membrane, the gas phase and the liquid phase as an example) [17]. The main driving force for the gas separation of this technology is the concentration difference between the gas phases. The mass transfer process is based on Fick's law and includes the following three steps as shown in Figure 2.

i. First, the solute is transferred from the mixed gas to the surface of the membrane pores;
ii. The solute is then transferred from the membrane by the membrane. The pores diffuse to the gas-liquid two-phase interface;
iii. The solute finally reacts with the absorbent and is carried out of the membrane contactor by the liquid phase [18].

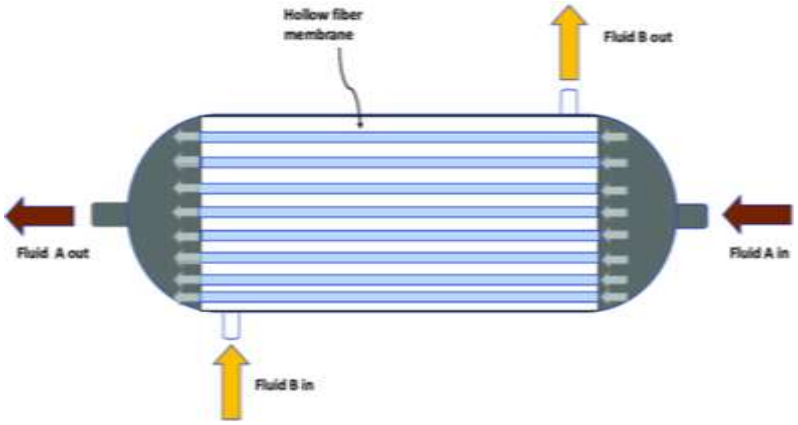


Fig. 1. Schematic of a hollow fiber membrane contactor

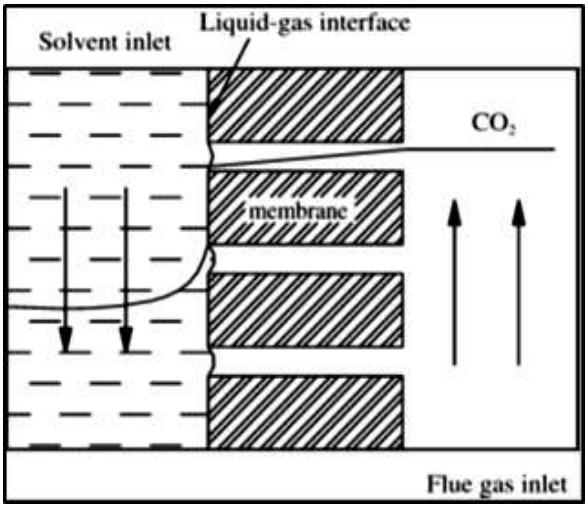


Fig. 2. CO_2 transport in a hollow fiber membrane contactor [18]

2.2 Selection of Membrane Materials

The hollow fiber membrane materials used for gas separation mainly include polymers, inorganic materials and composite membranes. Among them, polymer membranes are the most widely used due to their efficient separation performance, and their physical properties are mainly determined

by their chemical structures. Rahbari-Sisakht *et al.,* [18,19] compared the physical and chemical CO_2 absorption experiments of PS and PVDF membrane contactors before and after modification, and found that the modified membrane material provided higher CO_2 removal when glycerol was used. In addition to efficiency. In the study of the stability characteristics of membrane materials (PP, PVDF and PTFE). Among them, PP membrane material has received the most applications and researches because of its low price and good performance [20,21]. Porcheron *et al.,* [22] successfully assembled and applied a $10m^2$ PTFE pilot-scale membrane module to remove CO_2. In addition, inorganic membrane materials have also been widely used due to their high temperature resistance and thermal stability. Porcheron *et al.,* [22], and Koonaphapdeelert *et al.,* [23] investigated the removal process of CO_2 in a ceramic hollow fiber membrane contactor and found that it still has a strong removal effect under high temperature and chemical environment. For membrane advantages, PTFE still has good release properties under long-term operating conditions [24,25].

3. Material and Method

3.1 Measurement and Instruments

Monoethanolamine (MEA), N-methyldiethanolamine (MDEA), Glycine, piperazine (PZ), carbon dioxide (purity ≥ 99.9%), nitrogen (purity ≥ 99.99%) are of analytical grade. KJ-SH6×40 PTFE type hollow fiber membrane module; QF-1904 type Austrian gas analyzer; HH-S type constant temperature oil bath; LZB-4 type rotor flow meter; YE-60 type bellows pressure gauge.

3.2 Method

The flue gas treatment process is shown in Figure 3. The simulated flue gas (fraction ratio: CO_2:N_2=15%:85%, which is close to the gas composition ratio of the actual power plant exhaust flue gas) is mixed evenly by the buffer bottle, the liquid flow rate, and the gas flow rate was 60 mL/min, 350 mL/ min respectively, the temperature is 30°C, the pressure of a liquid is 0.4 bar and the pressure of gas is 0.2 bar. After the flow is adjusted by the flow meter, it enters the shell side of the membrane contactor, the mixed amine absorption liquid is pumped into the tube side of the membrane wire by the booster pump, and the absorption liquid contacts the simulated flue gas in the reverse flow in the membrane contactor to complete the decarburization process. During the experiment, in order to realize the recycling of the absorbent, a regeneration device was installed at the outlet of the tube side of the membrane module to regenerate the lean liquid of the absorbent. The regeneration method was heating regeneration.

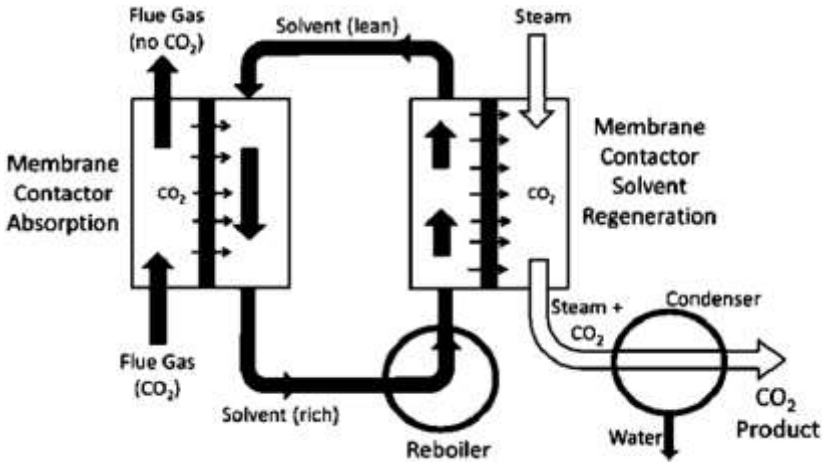


Fig. 3. Membrane contactor processing CO_2 in flue gas

Sampling every 15min, measure the flue gas flow at the inlet of the simulated flue gas and at the outlet of the treated flue gas respectively, and use the Austrian gas analyzer to measure the CO_2 content in the flue gas at the inlet and outlet respectively, and calculate the absorbed CO_2. The amount of gas (ΔV) is converted into the molar amount of matter (Δn) by the ideal gas state equation, then the CO_2 removal rate can be calculated:

$$\eta = (1 - \frac{QoCo}{QiCi}) \times 100\%$$

where η_{CO2} removal rate, %; Q_i and Q_o are the inlet and outlet flue gas flow rates, respectively, mL/min; C_i, C_o are the inlet and outlet CO_2 concentrations, ppm.

Under the condition that the environment is basically stable and the amount of absorption liquid is unchanged, after the membrane absorption runs for a certain period of time, the CO_2 removal rate gradually tends to zero. At this time, the CO_2 concentration in the outlet flue gas basically remains unchanged, and the absorption capacity of the absorption liquid can be considered is saturated, stop the experiment.

4. Results and Discussion

4.1 Effect of Liquid Flow Rate

In this study the effect of liquid volumetric flow rate on CO_2 removal efficiency PTFE hollow fiber membrane contactor was investigated. The liquid volumetric flow rate was varied from 35ml/min to 85 ml/min. Meanwhile, the MEA concentration was 5 Wt.%, CO_2 mole fraction in feed gas was 15%, gas volumetric flow rate was 350 ml/min, utilizing a gas feed with an initial mole percentage of 15% carbon dioxide. The results of the experiment indicate a significant increase in the efficacy of carbon dioxide removal, with a rise from 35% to 91% as the volumetric flow rate of the liquid was increased, as shown in Figure 4.

When liquid flow rate of absorbent increase, liquid velocity inside the shell side increase, %CO_2 removal efficiency increased. An increasing in the liquid flow rate, liquid phase boundary layer at membrane wall decrease which increase the diffused of carbon dioxide through boundary layer. This led to an increase in the liquid mass transfer coefficient and thus improve the CO_2 absorption, as indicated by previous studies [26,27].

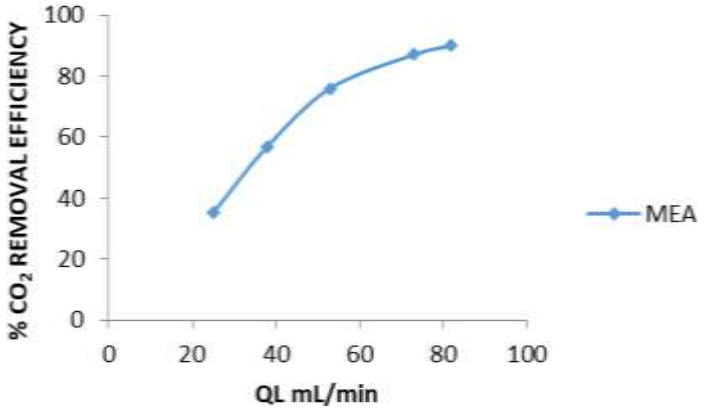


Fig. 4. Effect of liquid flow rate on CO_2 Removal efficiency (Gas flow rate 350 ml/min, CO_2: 15%, liquid temperature: 30°C

4.2 Effect of Gas Flow Rate

This research investigates the effect that the volumetric flow rate of the gas has on the carbon dioxide removal efficiency with which carbon dioxide is removed from a PTFE hollow fiber membrane contactor that makes use of a solvent 5wt.% of MEA. The volumetric flow rate of the gas was controlled to be anything between 250 and 500 NL per minute. The volumetric flow rate was 60 ml/min, CO_2 volume fraction in feed gas was 15%, the temperature was 30˚C .

On the basis of the data , When the volumetric flow rate of gas was raised from 250 NL/min to 500 NL/min it is the observation that the CO_2 removal efficiency reduced from 81% to 58% , as shown in Figure 5.

The findings of this research are consistent with those acquired from previous studies carried out by Peyravi *et al.,* [27], Izaddoust and Keshavarz [28], and Mohammaddoost *et al.,* [29]. Because of the rise in carbon dioxide concentration at the gas-liquid interface. The residence time of the carbon dioxide decreases, resulting in the absorption of a small amount of carbon dioxide across the membrane and its conversion into a solvent [30].

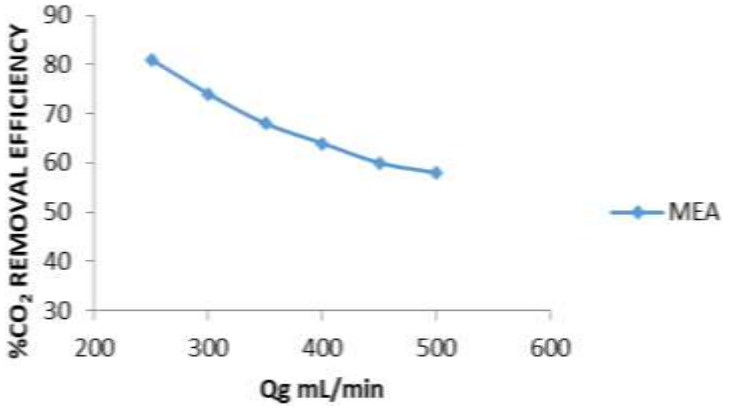


Fig. 5. Effect of gas flow rate on CO_2 Removal efficiency (Liquid flow rate: 60 ml/min, CO_2: 15%, liquid temperature: 30°C

4.3 The Effect of Mixed Amine Absorbent Concentration on CO_2 Membrane Absorption

In Figure 6(a), the three mixed amine absorbents with mass concentrations of 15%, 25%, and 35% were prepared in 4Liter each, and the solute ratio of MDEA: MEA/PG/PZ was 1:1, and the CO_2 removal rate on the hollow fiber membrane contactor platform was measured. The results are shown in Figure 4. Sodium glycinate (PG) is prepared from Glycine and NaON in a mass ratio of 1:1. At the mass concentration of was 15%, the comprehensive CO_2 removal rates of MDEA-MEA, MDEA-PG, and MDEA-PZ were 22.05%, 20.52%, and 18.25%, respectively, which were higher than that of a single MDEA absorbent at this concentration; the mass concentration was At 25%, the comprehensive CO_2 removal rates of the three mixed amine absorbents were 35.35%, 28.84%, and 24.87%, respectively, which were higher than that of the single MDEA absorbent at this concentration; when the mass concentration is 35%, the comprehensive CO_2 removal rates of the three mixed amine absorbents are 48.65%, 42.35% and 34.9%, respectively, which are higher than that of the single MDEA absorbent at this concentration. It can be seen that when the concentration is high (35%), the increase in the comprehensive removal rate of CO_2 of the mixed absorbent actually decreases, and the increase in decarburization performance begins to slow down. Increases in absorbent concentration nearly correlate with an increase in viscosity in terms of the overall mass transfer resistance. As the mixed

solution concentration rose, the solution became more viscous and less able to diffuse and dissolve CO_2. At high absorbent concentrations, the liquid film resistance dominates the total mass transfer resistance, which in turn controls the overall reaction rate [31]. It is foreseeable that if the concentration of the mixed solution is further increased, the growth rate of their CO_2 membrane absorption will further decrease, so the reasonable concentration of the mixed amine absorption solution is between 25% and 35%.

In Figure 6(b), Experiments conducted over a long period of operation at temperature 30°C demonstrated that PG,PZ absorbents at different concentrations have a lower ability to absorption performance compared to MEA. Because MEA has high reaction rate with CO_2. Moreover, the descending in CO_2 absorption performance is related to the decreasing in the solute concentration at the solid-liquid interface that is caused by more time. Hence, the CO_2 adsorption rate would decrease consequently.

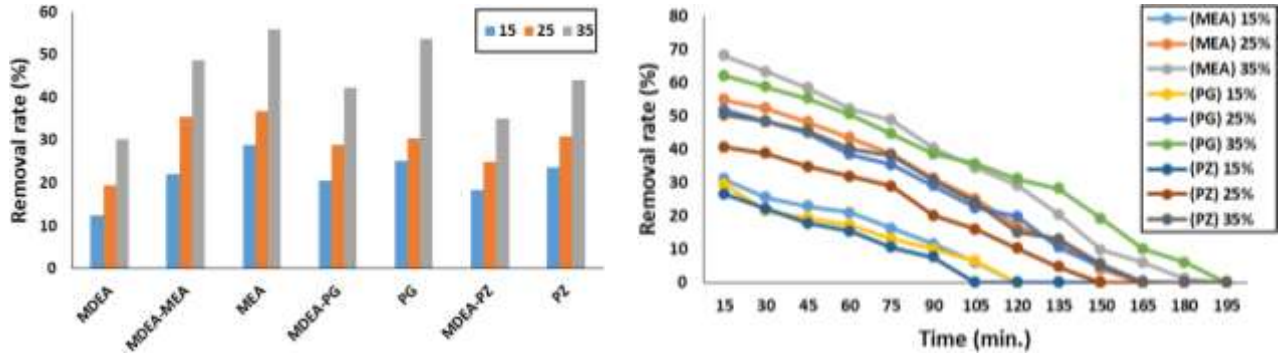
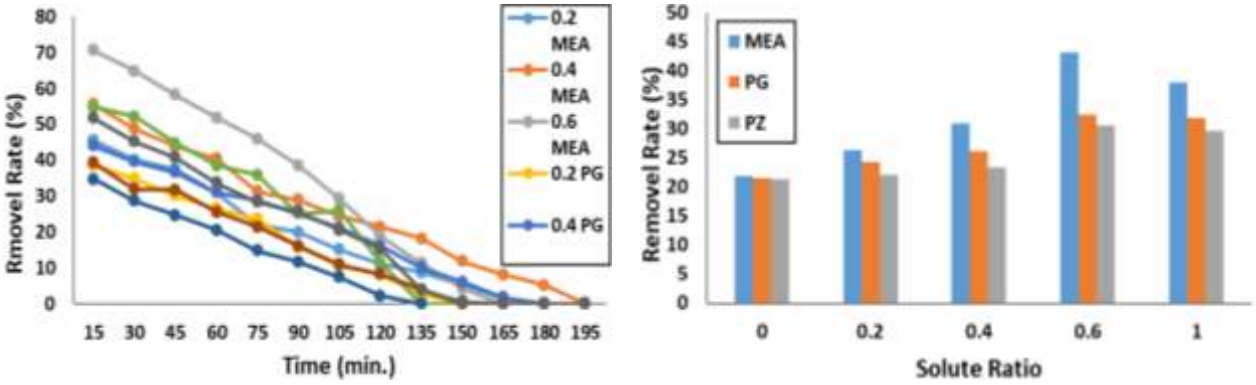


Fig. 6. CO_2 absorption performance of mixed amine absorbent with different concentration and comparison with single absorbent

4.4 Influence of Solute Ratio

Prepare 4 Liter of three mixed amine absorption solutions (mass concentration of 20%) with the solute ratios of 1:0.2, 1:0.4, and 1:0.6 (molar ratio), respectively, and measure the CO_2 removal rates of the three absorption solutions. The results are shown in Figure 7. From Figure 7, it can be seen that under different sampling times, the measurement points of the CO_2 removal rate of the mixed amine absorbing solution with a ratio of 1:0.6 are mostly scattered outside the area sandwiched by the CO_2 removal rate of the absorbing solution with a ratio of 1:0 and 1:1. , and the reaction period is shortened. In addition, the measurement points of the CO_2 removal rate of the mixed amine absorbent under the two ratios (1:0.4 and 1:0.2) are basically distributed in this area, which shows that the mixed amine absorbent of the same solute has different ratios. Different CO_2 membrane absorption properties the average removal rates of MDEA-MEA, MDEA-PG and MDEA-PZ in each ratio are 1:0.6>1:1>1:0.4>1:0.2>1:0, when the ratio is 1:0.6 The comprehensive removal rate of CO_2 was the highest, which were 43.15%, 32.36% and 30.55%, respectively, and its CO_2 absorption performance was higher than that of the ratio of 1:1. It shows that within an appropriate range, increasing the solute ratio of the additive can improve the decarburization performance of the mixed amine absorbent. The material exchange rate is slowed down and the reaction period is elongated, and this change is more obvious when the solute ratio is high. At this time, the decarburization advantage of the high ratio mixed amine absorption solution is weakened by this change. Therefore, the optimal solute ratio is MDEA:MEA/PG/PZ is equal 1:0.6.



(a) (b)

Fig. 7. Comparison of CO_2 absorption performance of mixed amine absorbents with different proportions

4.5 Optimization of Mixed Amine Absorption Liquid System

In order to further analyze the CO_2 membrane absorption characteristics of the mixed amine absorbing liquid, three mixed amine absorbents with a solute ratio of 1:0.6 were selected, and their mass concentration gradients were further refined to 10%. At 50% mass concentration, the comprehensive removal rate during the reaction period is shown in Figure 8. It can be seen from Figure 4 that with the increase of the concentration of the mixed amine absorbing liquid, the comprehensive CO_2 removal rates of the three absorbents MDEA-MEA, MDEA-PG, and MDEA-PZ gradually increased, but when the mass concentration further increased, the growth rate of removal rate began to show a clear downward trend, and the comprehensive removal rate of CO_2 began to stabilize. The above phenomenon can be explained by the Gp value (the ratio of the time that CO_2 penetrates from the membrane gas/liquid interface into the fiber membrane filament axis to the average residence time of the absorbing liquid in the fiber membrane). The smaller the penetration depth of CO_2 during the contact time with the liquid, the less frequently it reacts with free amine molecules at the gas/liquid interface. As the concentration increases, although the CO_2 absorption capacity of the mixed absorbing solution increases, when the concentration increases to a certain extent, the Gp value also begins to increase significantly. Therefore, considering the cost of the absorbent and the service life of the membrane (the high concentration of absorbent is also more corrosive to the mousse), the optimal mass concentration value of the mixed absorbent in the future practical application process is 40%.

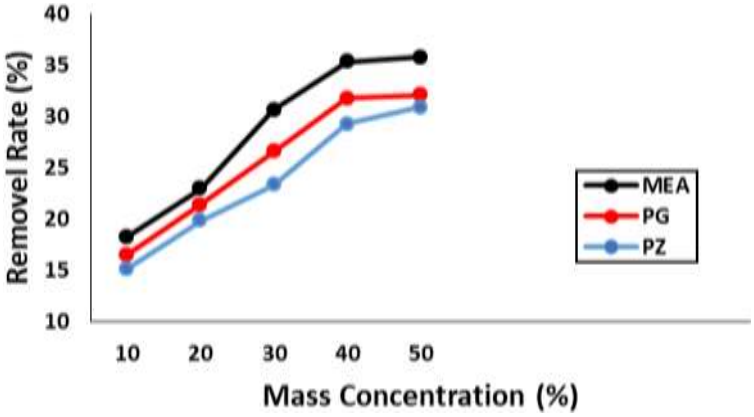


Fig. 8. CO_2 comprehensive removal rate with concentration of three mixed amine absorbents

5. Conclusions

This paper applies the membrane technology method with strong application prospects, analyzes the current research status of CO_2 absorption by membrane technology at home and abroad, and designs a set of hollow fiber membrane contactor absorption technology . The experimental operating system has studied the CO_2 absorption performance of different absorbents experimental perspective. For mixed amine absorbents, appropriate addition of amine activators with free H ions can effectively improve the CO_2 membrane absorption performance. hrough the above research work, the following conclusions are drawn.

i. The CO_2 membrane absorption performance of the three mixed amine absorbents is in the order of MDEA-MEA > MDEA-PG > MDEA-PZ. When the concentration is higher, although the comprehensive removal rate of CO_2 in the mixed amine absorbing liquid is higher, the growth rate of the removal rate begins to slow down.
ii. The effect is obtained on the characteristic distribution of some membrane technologies applied to CO_2 separation.
iii. The experiments demonstrate that increasing the liquid flow rate while maintaining a constant gas flow rate enhances the CO_2 removal effectiveness, reducing CO_2 levels at the gas phase outlet and increasing the total mass transfer coefficient. Conversely, increasing the gas flow rate without altering the liquid flow rate results in higher CO_2 levels at the gas phase outlet, with minimal impact on the mass transfer coefficient.

Future investigations should encompass a broader range of experimental conditions, including temperature and pressure variations, to elucidate their impact on the efficacy of mixed amine absorbents in hollow fiber membrane systems. Additionally, developing and testing new amine activators could potentially enhance the efficiency of CO_2 capture further while minimizing operational costs and energy requirements.

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