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The Effect of MDEA/AMP and Span-80 in Water-in-Oil (W/O) Emulsion for Carbon Dioxide Absorption

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ABSTRACT

Emulsion liquid membrane (ELM) has been widely studied as an alternative method for amine absorption technology in the removal of acid gases such as carbon dioxide (CO_2) and hydrogen sulphide (H_2S). However, searching for stable ELM formulation with an enhanced CO_2 absorption remains as challenge. Therefore, in this study, the aqueous solution containing a mixture of methyl diethanolamine (MDEA) and 2-amino-2-methyl-1-propanol (AMP) in sodium hydroxide (NaOH) solution was introduced as a dispersed phase, kerosene as continuous phase and Span-80 acts as a surfactant for the formation of water-in-oil (W/O) emulsion. In this study, the dispersed phase consists of 8% v/v MDEA and 4% v/v AMP and the continuous phase which contains 6% v/v Span-80 produced a stable emulsion and exhibited 65.2% of CO_2 removal. This study indicates that the introduction of blended amine able to produce stable emulsion with an enhanced CO_2 removal.

Keywords: Absorption, amine, carbon dioxide, liquid emulsion, stability

1.0 INTRODUCTION

In the sweetening process of the natural gas, the amines are usually used as a reactant to absorb the carbon dioxide (CO_2) and hydrogen sulphide (H_2S). Alkanaloamines such as monoethanolamine (MEA), diethanolamine (DEA), tri-ethanolamine (TEA), diisopropylamine (DIPA), methyldiethanolamine (MDEA), and diglycolamine (DGA) are commonly amines used for acid gas removal [1, 2, 3]. The effectiveness of alkanolamines is depending on the rate of reaction between CO_2 and the amine, as well as the absorption capacity. A primary amine like aqueous MEA has been used widely because of its high reactivity and low solvent cost. However, this amine has a low loading capacity of CO_2 [2]. It is proved that CO_2 loading in MEA is only 0.5 mol of CO_2 per mol of amine. This value is relatively lower as compared to MDEA which has higher loading capacity of CO_2 (1 mol of CO_2/1 mol of amine). However, this tertiary amine has low performance in CO_2 absorption rate [2]. Thus, blended amine has been introduced to further improve the performance of CO_2 absorption, by combining MDEA with

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MEA, DEA and piperazine, and AMP with MEA and DEA [2]. A study also showed that sterically hindered amines offer better results in term of absorption capacity, selectivity and degradation resistance in the CO_2 separation processes as compared to the conventional amines [4]. It was reported that AMP gave high CO_2 absorption rate and high CO_2 loading [3]. The CO_2 loading in AMP is high (1mol of CO_2 /mol of amine), thus the blend of AMP with MDEA would give high results of CO_2 absorption.

Furthermore, there are some disadvantages if direct amine was used, where the CO_2 loading capacity is low, corrosion may occur, and amine is degraded after several treatments [5]. Thus, to overcome low loading capacity of CO_2, a mixture of primary or secondary alkanolamine with tertiary alkanolamine is suggested in order to enhance the absorption ability of amine. This blended technology combines the higher equilibrium capacity of the tertiary amine for CO_2 with the higher CO_2 reaction rate of the primary or secondary amine [6].

Using water-in-oil (W/O) emulsion, amines in aqueous solution formed a globule and dispersed in a continuous organic phase. Thus, the removal process depends on the transfer of solute (CO_2) through the emulsion. Recently, W/O emulsion has been introduced as an alternative way of CO_2 separation as compared to the conventional separation that gives high simultaneous purification and concentration of the solute. The separation occurs when the solute permeates through the liquid phase from a feed phase to the receiving phase [7]. The effectiveness of emulsion depends on the stability of the emulsion, diffusivity of the adsorbate, which depends on the surface area and the thickness of emulsion. In the W/O emulsion technique, aqueous amine is sealed inside the non-corrosive, organic phase membrane of emulsion. However, the use of emulsion has been limited because of physical instability of emulsion globules caused by fluid shear [8]. The extraction process is also hindered due to the emulsion breakup and the unwanted release of internal receiving phase to the external contributing phase. A surfactant that stabilizes the emulsion also affects the stability and CO_2 absorption. A good surfactant should be soluble in the membrane phase only and not react with the extractant in the membrane phase to prevent the decomposition of the extractant and enhance the efficiency of the emulsion process [9]. Span-80 gives high relatively stable and easily demulsified emulsions and shows less resistance to mass transfer than other surfactant [10]. It also has a low HLB value (4.3) that gives high solubility in oil than water [11, 12].

This study was not rare, but it has been challenging to find good formulation for specific applications, especially for CO_2 removal. Therefore, it is crucial to find the suitable formulation of W/O emulsion which exhibited stable emulsion with enhanced CO_2 absorption. In this study, blended amine (MDEA/AMP) and role of surfactant in stabilizing the emulsion and as resistance in the diffusion were identified. Therefore, this research investigates the effect of W/O formulation on CO_2 absorption performance. The parameters that affect the stability of emulsion and CO_2 absorption were to be determined.

2.0 METHODS

2.1 Emulsion Preparation

An emulsion was prepared according to a method described by Bhatti *et al.*

[13] using different types of amines. The aqueous phase consists of amines in NaOH solution as extractant, while the organic phase consists of kerosene as continuous phase and Span 80 as surfactant. The liquid emulsion membrane was prepared by homogenizing the aqueous and the organic solution. 100 mL of the aqueous phase was prepared by adding amine into 0.1 M NaOH solution. The solution was stirred for 15 minutes. For organic solution, 100 mL of organic phase was prepared by adding Span-80 into the kerosene oil and stirred for 15 minutes. The stirring speed and temperature of the heating plate for aqueous and organic phase solutions were fixed at 700 rpm and 27°C, respectively. For the preparation of emulsion, the high-performance disperser Ultra Turrax® T25 with 18G mixing shaft was used. 100 mL organic phase mixture was placed in the beaker and the aqueous phase mixture was poured dropwise into the beaker containing the organic phase to produce water-in-oil emulsion. Table 1 presents the emulsion formulation and parameter used to determine the percentage removal of CO_2 using different types of single amine, methyldiethanolamine (MDEA), and 2-amino-2-methyl-1-propanol (AMP). Table 2 shows the emulsion formulation of blended amine using different ratios of MDEA and AMP where the amount of MDEA is fixed at 8% v/v and Table 3 consists of emulsion formulation using different amounts of Span-80.

2.2 Emulsion Stability

The stability of emulsion was measured based on visual observation. Sedimentation is an early process that leads to the emulsion breakdown after a certain period of time [14]. It is a process in which droplets move downwards since the droplet density is greater than the density of the continuous phase. Sedimentation was demonstrated by the presence of a layer in the top of the test tube (2 layers) while emulsion breakdown shows another layer in the top and bottom of the test tube (3 layers) [15]. The emulsion condition becomes less homogenous as it starts to settle. The stability test of the emulsion was conducted prior to CO_2 absorption process for different amines, different MDEA/AMP ratio, and different amounts of Span-80. The measurement proceeds by filled in emulsion in the graduated test tubes and left in the room for 24 hours. The determination of emulsion stability was based on the percentage of emulsion sedimentation where the volume of the top layer was measured. The percentage of the separation is determined by Eqn. 1.

$$\% \text{ stability} = (Vt-Vs)/ Vt \times 100 \quad (1)$$

where V_T is the total volume (ml) and V_S is the top layer volume (ml).

The emulsion's viscosity was measured by using Programmable Rheometer Brookfield Model DV-III.

2.3 CO_2 Absorption

In this study, the rotating disc contactor (RDC) column was used for CO_2 absorption in the emulsion. Figure 1 illustrates the schematic diagram of CO_2 absorption system used in this study. The function of RDC is to maintain the stability and homogeneity of the emulsion in the column. The column was filled with 200 mL of emulsion and the flow rate of gas entering the column was fixed at 20 LPM (Litre per minute). Longer contact time was achieved as the CO_2 enters the column from the bottom of the column.

Table 1 Emulsion formulation using different types of amines

Formulation / Condition	**Specification**
Aqueous phase (100 mL)	Amine used: MDEA, AMP,
Ratio Amine to NaOH	8 % v/v: 92 % v/v
Organic phase (100 mL)	
Ratio Kerosene to Span-80	92 % v/v: 8 % v/v
Emulsification Time	5 minutes
Emulsification Speed	10 000 rpm
Absorption Time	1 minute

Table 2 Emulsion formulation using various amine composition

MDEA: AMP	**Aqueous Phase MDEA: AMP: NaOH (100 mL)**	**Organic Phase Kerosene: Span-80 (100 mL)**
8:0	8% v/v: 0% v/v: 92% v/v	92% v/v: 8% v/v
8:2	8% v/v: 2% v/v: 90% v/v	92% v/v: 8% v/v
8:4	8% v/v: 4% v/v: 88% v/v	92% v/v: 8% v/v
8:6	8% v/v: 6% v/v: 86% v/v	92% v/v: 8% v/v
8:8	8% v/v: 8% v/v: 84% v/v	92% v/v: 8% v/v
Emulsification time	5 min	
Emulsification speed	10 000 rpm	
Absorption time	1 min	

Table 3 Emulsion formulation using different amount of Span-80

Span-80	**Aqueous Phase MDEA: AMP: NaOH 100 mL**	**Organic Phase Kerosene: Span-80 100 mL**
8	8% v/v: 4% v/v: 88% v/v	92% v/v: 8% v/v
6	8% v/v: 4% v/v: 88% v/v	98% v/v: 6% v/v
4	8% v/v: 4% v/v: 88% v/v	96% v/v: 4% v/v
2	8% v/v: 4% v/v: 88% v/	94% v/v: 2% v/v
Emulsification time	5 min	
Emulsification speed	10 000 rpm	
Absorption time	1 min	

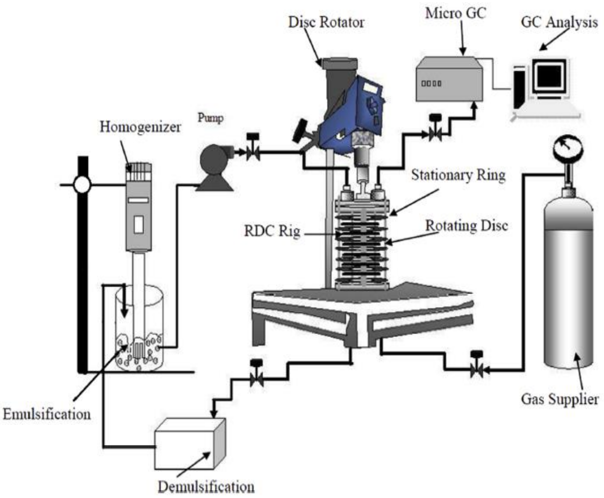

Figure 1 The schematic diagram for CO_2 absorption system [13]

The speed of the rotating disc was kept at 450-500 rpm range. Gas chromatography (GC) was used to determine the amount of CO_2 entering and leaving the RDC. The percentage of CO_2 absorption was calculated based on the amount of CO_2 leaving the column by using Eqn. 2.

Percentage of CO2 absorption:

$$\frac{A_r - A_e \times 100\%}{A_r} \qquad (2)$$

where,

A_r: Area of Reference (µV/s)
A_e: Area of Emulsion (µV/s)

3.0 RESULTS AND DISCUSSION

3.1 Effect of Emulsion Formulation on Stability and Viscosity

In the preparation of W/O emulsion for CO_2 removal, the two immiscible liquids were emulsified, which gives the energy to form a stable emulsion through the fragmentation of one phase into another. Emulsion breakdown was indicated by the presence of three layers as shown in Figure 2 (a), while the sedimentation was indicated by the presence of another layer on the top of the test tube (2 layers) as shown in Figure 2 (b). The stability of emulsion was measured after 24 hours.

Figure 2 Emulsion stability (a) emulsion breakdown and (b) sedimentation

Interfacial shear between the continuous phase and dispersed phase caused the interfacial layer to thin and in some cases, breakdown. Moreover, it was reported that stable emulsion resulted in high CO_2 removal [16] thus, it is crucial to get stable emulsion in this study. In this study, Span-80 has been chosen as a surfactant in emulsion because it has low hydrophile-lipophile balance (HLB) value of 4.3, which gives higher solubility in oil, as compared to water [11, 12]. The emulsion can be stabilized by the absorption of surfactant molecule at the interface between oil and water, thus giving low free energy to the phase boundary. However, the solute diffusivity from external phase into internal phase is reduced when the interface layer becomes thicker, thus decreasing the efficiency of extraction process.

Two types of amines were used in the preparation of emulsion. Each formulation consists of 8 % of MDEA and varied ratio of AMP in NaOH solution, and the organic phase that consists of 92 % kerosene and 8 % Span-80. Span-80 acts as a surfactant to stabilize the emulsion. Emulsion stability refers to the ability of emulsion droplets to homogenously disperse in a continuous phase. As mentioned earlier, coalescence, creaming or sedimentation is one-step occurrence before emulsion breakdown. As reported by Aroua *et al*. [3], AMP gave a higher CO_2 absorption rate and high CO_2 loading when combined with MDEA. The CO_2 loading in AMP is high (1 mol of CO_2 /mol of amine), thus the blend of AMP with MDEA would give higher results of CO_2 absorption. However, the stability of the emulsion containing blended amine should also be observed. A study by Dolmat [17] on single amine also found that 8 % v/v of MDEA in dispersed phase was the best formulation for CO_2 removal. Therefore, in this study, several samples were prepared with the amount of MDEA which was fixed at 8 % v/v, with varied amount of AMP. Table 4 shows the stability of emulsion containing different ratios of MDEA and AMP.

Table 4: Emulsion stability and viscosity with different MDEA-AMP ratio

MDEA: AMP	**Stability (%)**	**Viscosity (cP)**
8:0	66	225
8:2	70	645
8:4	78	864
8:6	80	908
8:8	92	1112

Table 4 shows the percentage stability of emulsion increases as the amount of AMP increases. The presence of amine (AMP/MDEA) in aqueous solution also affects the viscosity of the emulsion. The viscosity of emulsions containing blended amines (MDEA/AMP) increases as the quantity of AMP increases. If the emulsion is viscous, it forms a more stable emulsion. As reported by Mohamed *et al*. [18], viscosity affected emulsion stability. In addition, as reported by Shi *et al*. [19], water content has a strong influence on viscosity of crude oil, and the viscosity of water-in-oil emulsion increases gradually as water content increases. However, too viscous emulsion may reduce the diffusion of solute. It is also important to note that viscosity of emulsion is far different from the individual liquids. The viscosity of kerosene (1.64 cP) is relatively low as compared to NaOH solution (87 cP), AMP (147 cP) and MDEA (101 cP).

The stability of emulsion was determined after 24 hours by visual observation. In each sample, two layers were formed in which the bottom layer is thicker than the top layer, which indicates sedimentation has occurred. As shown in Table 4, a sample with 8 % MDEA and 8% AMP shows the highest viscosity (1112 cP) and stability (92 %). The results also indicated that combination of MDEA/AMP in dispersed phase produced higher emulsion stability than single amine as stated by Chakravarti [6]. The effect of Span-80 amount on viscosity and stability of emulsion was also observed using 8 % MDEA and 4 % AMP in 100 mL NaOH as aqueous solution on next section.

3.2 CO_2 Removal

CO_2 was removed by means of absorption. The absorption in the emulsion can be described as a transfer of CO_2 from the continuous phase into the dispersed phase through oil-water interphase. Then, CO_2 reacted with MDEA and AMP in the dispersed phase to produce bicarbonates (HCO_3^-). MDEA has a higher loading capacity (1 mol of amine/ 1 mol of CO_2) than conventionally used amine like MEA. Theoretically, MDEA does not react directly with CO_2 since MDEA is a tertiary amine, but it acts as a base that catalysed the hydration of CO_2.

According to Sema *et al*. [20], the reaction is essentially base-catalysed CO_2 hydrolysis, and MDEA does not combine with CO_2, thus leading to low absorption. As reported by Ali Khan *et al*. [21], MDEA has the lowest reaction rate compared to MEA and AMP. Due to the low reaction between MDEA and CO_2, MDEA is commonly combined with activator such as piperazine (PZ) or sterically hindered amine of AMP to improve the reaction with CO_2. The hindered amine carbamates undergo hydrolysis forming bicarbonate and releasing free amine since AMP has low stability constants. The fast reaction of AMP will quickly absorb the CO_2. Then, the free amine molecule will react faster with CO_2. The reaction of CO_2 with AMP could result in three reactions: the formation of carbamate, the formation of bicarbonate, and the reversion of carbamate to bicarbonate or formation of the carbonate ion [22].

Rotating Disc Contactor (RDC) column was used to conduct the CO_2 absorption study. The emulsion was placed in the column and a mixture of CO_2 gases was allowed to get into contact for absorption to occur. The amount of CO_2 absorbed was determined from the Gas Chromatography (GC) results. The initial result showed that CO_2 removal using single amine is almost the same (52.3 % and 51.8 % for MDEA and AMP respectively) as shown in Figure 3. Based on 1-minute absorption time, the result shows that the absorption rate of MDEA-CO_2 is slightly higher than that of AMP-CO_2. The result is consistent with a study conducted by Rodriguez *et al*. [23] where they also reported that AMP in individual systems gave low absorption rates.

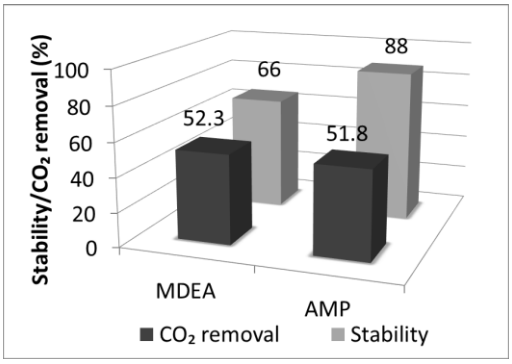

Figure 3 Emulsion stability and percentage of CO_2 removal

However, AMP offers an additional advantage over MDEA, particularly for CO_2 removal, due to the fact that the CO_2-AMP reaction rate is much faster than the CO_2-MDEA reaction rate [24]. Figure 4 shows that the blended MDEA/AMP mixture in emulsion improved the percentage of CO_2 removal. As stated by Mandal *et al*. [2], MDEA has an advantage of removing more CO_2 where it has a high equilibrium loading of 1.0 mol of CO_2 per mol of amine. However, the reaction rate of MDEA is low, hence the MDEA needs to blend with AMP which possess high CO_2 absorption rate and high CO_2 loading capacity [16].

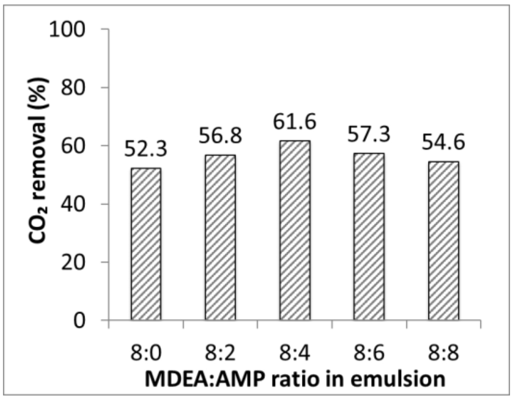

Figure 4 Emulsion characteristics at different MDEA-AMP ratio

In the emulsion formulation, it is expected that CO_2 removal increases as more amines (MDEA and AMP) in the dispersed phase react with CO_2. However, the results shows that CO_2 removal initially increased but began to decrease when the amount of AMP reached 6 % v/v. The percentage of CO_2 removal is the lowest when the emulsion consisted of single amine (AMP) in the dispersed phase (Figure 3). This condition is due to the viscosity of the emulsion; as more MDEA/AMP are present in the dispersed phase, the viscosity of the solution also increases and the emulsification procedure, highly viscous solution requires more energy to form a good dispersion in a continuous phase. Thus, as the fixed emulsification parameters are fixed, the size of droplet size in the emulsion would be larger for more viscous solution. Consequently, the total surface area would be less, hence reducing the percentage of CO_2 removal. In addition, as the droplet size increases, emulsion homogeneity also decreased, which also leads to low CO_2 removal. In case of blended amines, emulsion containing 8 %v/v MDEA with 4 %v/v AMP showed the highest CO_2 removal (61.6 %) while emulsion containing 8% v/v MDEA with 8% v/v exhibited the lowest CO_2 removal (54.6 %). On the other hand, for single amine emulsion, the percentage of CO_2 removal for emulsion containing MDEA-only is 52.3 %. These results shows that blended amine in W/O emulsion improved the percentage of CO_2 removal where blended AMP with tertiary amine will give high absorption capacity as stated by Aroua et. al. [3] and Xiao et. al. [4]. However, viscosity plays significant influence on the formation of emulsion droplets, emulsion stability and directly affects the overall performance of CO_2 removal.

In order to produce consistent and stable emulsion, a suitable amount of emulsifier should be included in the emulsion formulation. As mixture of 8 %v/v MDEA with 4 %v/v AMP shows highest percentage of CO2 removal, it has been selected for further investigation for their stability in a varying amount of emulsifier, as shown in Table 3. The selection of an appropriate emulsifier is one of the important decisions when formulating the emulsion [25]. Span-80 was chosen as a surfactant in the formulation because it has low hydrophilic-lipophilic balance (HLB) value (4.3) that gives higher solubility in oil than water. The emulsion was stabilized by the absorption of surfactant molecule at the interface between oil and water, thus giving low free energy to the phase boundary. Figure 5 shows the effect of Span-80 on the viscosity, emulsion stability and percentage of CO_2 removal. The stability of emulsion increases steadily as the amount of Span-80 increases. According to Li *et al*. [26], the stability of the emulsion and the viscosity increases by the proportion of surfactant in the organic phase, which explains why the emulsion stability increases as the amount of Span-80 increased.

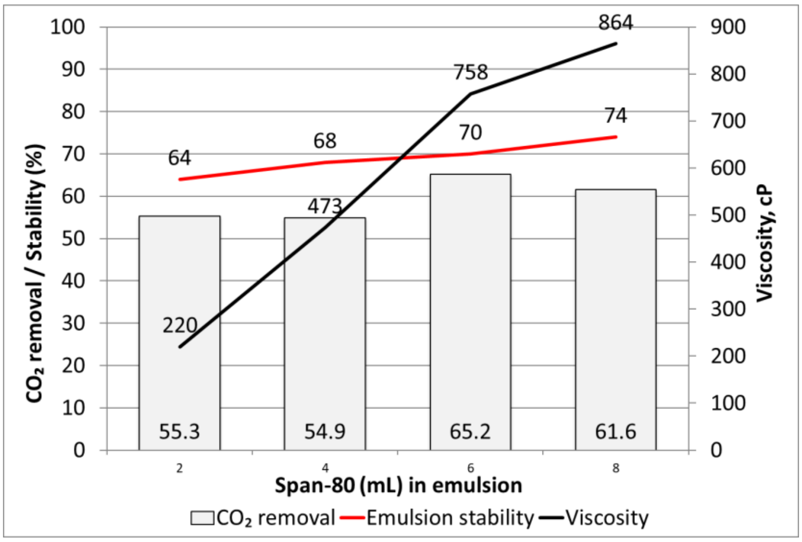

Figure 5 The effect of Span-80 on viscosity, stability, and CO_2 removal

Emulsion containing 6% v/v Span-80 shows the highest CO_2 absorption (65.2%). As stated by Skelland and Meng [27], the increased viscosity significantly decreases the diffusivity for Newtonian fluids. Thus, the addition of more Span-80 reduces the solute diffusivity and decreases the extraction rate, thus reducing the efficiency of CO_2 separation process as shown at 8% v/v Span-80. Ansel *et al.* [28] also proposed that the size of emulsion droplets is directly proportional to the velocity of sedimentation process where large emulsion droplets decreased the total surface area, therefore reducing the absorption of carbon dioxide.

4.0 CONCLUSION

The use of blended amines such as MDEA and AMP in W/O emulsion may enhance the removal of CO_2. In this study, the stability of the emulsion increases as the amount of amine increased. The CO_2 removal of 61% can be achieved by using 12% of amines (8% MDEA/ 4 % AMP). Furthermore, the CO_2 absorption of resulting W/O emulsion has been further improved by varying the amount of Span-80, reached 65.2% of CO_2 removal using 6% v/v Span-80.

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