SEPARATION OF CARBON DIOXIDE USING EMULSION LIQUID MEMBRANE CONTAINING METHYLDIETHANOLAMINE AND 2-AMINO-2-METHYL-1-PROPANOL

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A thesis submitted in fulfilment of the
requirement for the award of the degree of
Master of Engineering (Gas)

Faculty of Chemical and Energy Engineering
Universiti Teknologi Malaysia

JULY 2017

Dedicated to my beloved father and mother, Mohd Najib bin Sujak and Nahariah bt Bohari,

My husband, Nur Izham bin Makhtar, my son and all my siblings for their love and encouragement.

ACKNOWLEDGEMENT

All praise due to Allah, I have successfully completed my thesis. I am so blessed to Allah who gave me tremendous courage and spirit while facing the entire obstacle all this while.

In preparing this thesis, I was in contact with many people, researchers and academicians. They have contributed towards my understanding and thoughts. In particular, I wish to express my sincere appreciation to my supervisor, Assoc. Prof. Dr. Khairul Sozana Nor binti Kamarudin for encouragement, guidance and critics. Without her continued support and interest, this thesis would not have been the same as presented here. I am also very thankful to the technical staff, Mr. Ahmad Fadzli bin Akbar and Mr. Redhuan Ramlee for assistance in the laboratory works. I am grateful to the Ministry of Higher Education for supporting my Master project.

My sincere appreciation also extends to all my friends for their view, support and tips. Unfortunately, it is not possible to list all of them in this limited space. I am grateful to all my family members too.

All peace is upon to all of you.

ABSTRACT

Carbon dioxide (CO_2) removal is important in industry to optimize the capital and the operating costs of a process, to meet gas specifications and also for environmental purpose. The most developed CO_2 capture technology that the separation process can be easily adapted to the existing plant is pre-combustion capture. In natural gas industry, amine absorption method is widely used in pre-combustion capture technology (sweetening) to remove the CO_2 from natural gas. However, due to amine corrosive nature, alternative is needed for CO_2 separation. Therefore, emulsion liquid membrane (ELM) was introduced to overcome the corrosion problem. However, separation ability of emulsion needs to be investigated. In this study, water in oil (w/o) emulsion was prepared. 2-amino-2-methyl-1-propanol (AMP) and methyldiethanolamine (MDEA) in sodium hydroxide solution form an aqueous phase. The organic phase consists of a mixture of kerosene and Span-80 as surfactant. In this study, the effects of amines mixture concentration, Span-80 concentrations and retention time on CO_2 absorption capacity were investigated. ELM was prepared by homogenizing the aqueous phase into the organic phase at speed of 10 000 rpm for 5 minutes. For CO_2 absorption study, 200 ml of ELM was tested in a rotating disc contactor column with the supply of 20 psig CO_2/CH_4 gas mixture. The stability of the MDEA-AMP emulsion was determined and the CO_2 absorption by the emulsion was measured. This study showed that using 8% v/v MDEA and 4% v/v AMP with 6% v/v Span-80, 64.98% of CO_2 was removed at the emulsion stability of 78.00%. This study also proved that the presence on amines did not lead to any corrosion of the stainless steel.

ABSTRAK

Penyingkiran karbon dioksida (CO_2) adalah penting dalam industri bagi mengoptimumkan kos modal dan kos operasi, serta memenuhi spesifikasi gas dan juga untuk tujuan alam sekitar. Teknologi tawanan CO_2 yang paling membangun yang mana proses pemisahan boleh dijalankan dengan mudah dan disesuaikan dengan loji sedia ada adalah teknologi tawanan pra-pembakaran. Dalam industri gas asli, kaedah penyerapan amina digunakan secara meluas dalam teknologi tawanan pra-pembakaran untuk menyingkirkan CO_2. Walau bagaimanapun, disebabkan sifat hakisan amina, kaedah alternatif diperlukan untuk penyerapan CO_2. Oleh itu, cecair membran emulsi (ELM) diperkenalkan untuk mengatasi masalah hakisan ini. Namun, keupayaan pengasingannya perlu dikaji. Dalam kajian ini, emulsi air dalam minyak (w/o) telah disediakan. Fasa akueus mengandungi 2-amino-2-metil-1-propanol (AMP) dan metildietanolamina (MDEA) dalam larutan natrium hidroksida. Fasa organik pula mengandungi campuran kerosin dan Span-80 sebagai surfaktan. Dalam kajian ini, kesan kepekatan campuran amina, kepekatan Span-80 serta masa tahanan terhadap kapasiti penyerapan CO_2 telah disiasat. ELM telah disediakan dengan cara penghomogenan fasa akueus ke fasa organik pada kelajuan 10 000 rpm selama 5 minit. Untuk kajian penyerapan CO_2, 200 ml ELM telah diuji di dalam turus penyentuh cakera berputar dengan bekalan aliran 20 psig campuran gas CO_2/CH_4. Kestabilan emulsi MDEA-AMP telah ditentukan dan penyerapan CO_2 oleh emulsi telah diukur. Kajian ini menunjukkan bahawa dengan menggunakan 8% v/v MDEA dan 4% v/v AMP dengan 6% v/v Span-80, 64.98% daripada CO_2 telah disingkirkan pada kestabilan emulsi 78.00%. Kajian ini juga membuktikan kehadiran amina, tidak mengakibatkan sebarang kakisan pada keluli tahan karat.

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LIST OF ABBREVIATION

AMP	-	2-amino-2-methyl-1-propanol
CH_4	-	Methane
CO_2	-	Carbon dioxide
DEA	-	Diethanolamine
ELM	-	Emulsion Liquid Membrane
GC	-	Gas chromatography
H_2S	-	Hydrogen Sulfide
HLB	-	Hydrophile-lipophile Balance
LNG	-	Liquefied Natural Gas
MDEA	-	Methyldiethanolamine
NaOH	-	Sodium hydroxide
o/w	-	oil-in-water
PZ	-	Piperazine
RDC	-	Rotating disc contactor
TEA	-	Triethanolamine
w/o	-	water-in-oil
w/o/w	-	water-in-oil-in-water

CHAPTER 1

INTRODUCTION

1.1 Research Background

Natural gas is a mixture of hydrocarbon gases that mainly consists of methane as a major component and carbon dioxide (CO_2), nitrogen (N_2) and hydrogen sulfide (H_2S) as minor components (impurities). As reported by Rufford et al. (2012), the natural gas needs to undergo treatment to remove the impurities before the natural gas saleable. The presence of carbon dioxide in transmission pipeline cannot exceed 3%. In LNG production cryogenic plant, the CO_2 concentration in natural gas should be less than 50 ppmv before entering the plant in order to prevent the formation of dry ice which can cause severe operational problems in gas processing plant (Rufford *et al*., 2012). The United Nation's Intergovernment Panel on Climate Change (IPCC) stated that the concentration of CO_2 in natural gas should be lower than 2% by volume in order to prevent any pipeline corrosion (IPCC, 2015).

Removal of acid gas in natural gas can be done in several different processes namely physical absorption, chemical absorption, adsorption, and membrane separation (Mortaheb *et al*., 2012). Physical absorption occurs by contacting the gas mixture with the liquid solvent. The gas will diffuse into the liquid where the mass

transfer takes place at the interface between the liquid and the gas. The limitation of this process depends on the solubility of the gases, temperature and pressure. In chemical absorption, the absorbate that reacts with the substances need to be removes. It has limited absorption due to the stoichiometry of the reaction and the concentration of its reactant. In other condition, the physical absorption will combine with chemical absorption. Adsorption is another removal process that involved the adhesion of the molecules from a gas, liquid, or dissolved solid to a surface. Through this process, a thin film of the adsorbate formed on the surface of adsorbent. The latest technology that has been used nowadays is membrane separation due to its simplicity process, ease to control, and easy to scale up as compared to conventional technology. This membrane separation involves the separation of the gas or liquid via solid and relatively thin membranes in the molecular arrangements where it depends on the pore size of the membrane.

In the sweetening process of the natural gas, the amines are usually used as reactant to absorb the carbon dioxide (CO_2) and hydrogen sulphide (H_2S). Monoethanolamine (MEA), diethanolamine (DEA), tri-ethanolamine (TEA), diisopropylamine (DIPA), methyldiethanolamine (MDEA), and diglycolamine (DGA) are common type of amines used for acid gas removal. However, amine based absorption technique has also some disadvantages where the CO_2 loading capacity is low, corrosion may occur and amine is degraded after several treatment. Thus, to overcome low loading capacity, 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 (Chakravaty et al., 1985). As reported by Aroua et al. (2002), lower reaction rate between CO_2 and MDEA was overcome by the addition of a sterically hindered amine like 2-amino-2-methyl-1-propanol (AMP) due to the fast reaction that gives high CO_2 absorption.

In addition, the presence of acid gases (CO_2 and H_2S) would further increase the corrosion rate. The carbonic acids produced for example, can induce the corrosion

of iron. Consequently, corrosion had become one of the major problems in industries where failure of the equipment such as absorber, tray or pipeline occurred, thus reduce the efficiency of the process.

Recently, liquid membrane has been introduced as alternative way of CO_2 separation as compared to conventional separation. Liquid membrane gives high simultaneous purification and concentration of the solute. The separation occurs when the solute permeate through the liquid phase from a feed phase to the receiving phase (Kislik, 2010). The effectiveness of liquid membrane depends on the stability of the emulsion, diffusivity of the adsorbate that depends on the surface area and the thickness of membrane.

1.2 Problem Statement

Alkanaloamines such as monoethanolamine (MEA), diethanolamine (DEA), and methyldiethanolamine (MDEA) have been widely used in pre and post combustion CO_2 capture especially in natural gas industry. However, the effectiveness depends on the rate of reaction between CO_2 and the amine, and the absorption capacity. A primary amine like aqueous monoethanolamine (MEA) has been used widely because of its high reactivity and low solvent cost but this amine has low loading capcity of CO_2. The CO_2 loading in MEA is low which only 0.5 mol of CO_2 per mol of amine. Compared to MEA, MDEA is a tertiary amine that has higher loading capacity of CO_2 (1mol of CO_2/1mol of amine) but low in CO_2 absorption rate (Mandal et al., 2001). Thus, blended amine has been introduces to further improve the performance of CO_2 absorption, by combining MDEA with MEA, DEA and piperazine, and AMP with MEA and DEA (Mandal et al., 2001). It was reported that AMP gave highly CO_2 absorption rate and high CO_2 loading (Aroua *et al*., 2002). The CO_2 loading in AMP is high (1 mol of CO_2 /mole of amine), thus the blend of AMP with MDEA gives high results of CO_2 absorption. However, the use of blended amine also causes corrosion

resulting in destruction of material or equipment because of the presence of acid gases such as CO_2 (Gunasekaran et al., 2013).

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Based on the above stated problem, alternative method is required for effective CO_2 separation and at the same time it can prevent corrosion. Emulsion containing amine is a promising method where metal surfaces were protected from corrosion and at the same time able to remove CO_2 using amine absorption. In the ELM technique, aqueous amine is sealed inside the non-corrosive, organic phase membrane of emulsion. Surfactant that stabilizes the emulsion also affects the stability and CO_2 absorption. Therefore, this research investigates the potential use of MDEA and AMP for CO_2 absorption in emulsion liquid membrane. And parameters that affect the stability of emulsion and CO_2 absorption were to be determined.

1.3 Research Objective

The objectives of this study are:

1. To formulate a stable emulsion liquid membrane (ELM) containing a mixture of amine for CO_2 removal and can overcome corrosion problem.
2. To evaluate the performance of emulsion in carbon dioxide-methane separation in rotating disc contactor.

1.4 Scope of Study

In order to achieve the first objectives, the emulsion was prepared. MDEA and AMP selected as extractant whereas Span-80 was selected as a surfactant to stabilize

the emulsion. The stability of the emulsion was determined by conducting physical observation for the formation of sedimentation or phase separation within 24 hours to prove the effectiveness of emulsion. Corrosion study was also conducted to determine the effectiveness of emulsion in preventing corrosion using Corrosion Study Kit from SOLTEQ.

To evaluate the efficiency of CO_2 separation, rotating disc contactor (RDC) was used. A gas mixture of CO_2/CH_4 of ratio 70/30 was used and the amount of CO_2 absorbed was calculated from gas chromatography (GC) results.

1.5 Significant of Study

This technique would be an option for CO_2 separation. A suitable formulation that produces stable emulsion is able to prevent corrosion as well as performed well in CO_2 separation. In general, appropriate emulsification produces small particles that provides large surface area for diffusion and separation, thus increase the performance for CO_2 separation. The use of blended of amine such as tertiary alkanolamine (MDEA) and sterically hindered amine (AMP) would also improve the absorption of CO_2. Therefore, the findings from this study on CO2 separation using emulsion liquid membrane provides an alternative for better improvement in the CO_2 separation process.

1.6 Thesis Outline

This thesis consists of five chapters. The first chapter consists of research background, problem statement, the objectives and scope of the study. Chapter 2 discussed a literature review about CO_2, alkanolamines, CO_2 capture technology and details about ELM. The materials and methods for ELM preparation, stability and performance of ELM were reported under Chapter 3. In Chapter 4, the results and discussion were presented. This chapter discussed the stability of emulsion based on mixture of amine (MDEA/AMP) and surfactant (Span-80) concentration; viscosity of emulsion liquid membrane; the performance of ELM using various MDEA/AMP concentrations and surfactant (Span-80) concentration; absorption time; mass transfer coefficient; and the corrosion study. The final chapter provides conclusions for a current study and recommendation for future work.

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