EFFECT OF N-METHYL-2-PYRROLIDONE AND METHANOL ON THE CARBON STEEL CORROSION IN HYBRID MONOETHANOLAMINE SOLUTIONS

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DEDICATION

This thesis is dedicated to my supervisor, Ir. Dr Tan Lian See who provided me both technical knowledge and advice to support for my research. It is also dedicated to my family, who encourage me that even the largest task can be accomplished if it is done one step at a time.

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ABSTRACT

Carbon dioxide (CO_2) absorption is a matured technology for CO_2 capture and natural gas purification process. However, the presence of the acid gas in the unit operation poses corrosion issues which need to be addressed to ensure integrity of the operation system is uncompromised. The present study explores and provides comprehensive information on the effects of process parameter variations on the corrosion behaviour of carbon steel in CO_2 absorption systems using methylethanolamine (MEA) blended with methanol (MeOH) or N-methyl-2-pyrrolidone (NMP) as aqueous and hybrid solutions. The process parameters of interest involved amine solutions type, temperature, and CO_2 loading. The gravimetric and electrochemical experiments were carried out to investigate the corrosion behaviour of the carbon steel coupons in these solutions. The results indicated that the corrosion rate of carbon steels immersed in different amine solutions increased as the solution temperature and CO_2 loading rose. The gravimetric results showed that the carbon steel coupons had the lowest corrosion rate were the ones immersed in MEA+MeOH. Both MEA aqueous and hybrid solutions were examined using Raman spectroscopy analysis and the surface of the carbon steel coupons subjected to the corrosion were studied using field emission scanning electron microscopy (FESEM). However, the Electrochemical Impedance Spectroscopy (EIS) results showed an opposite trend from the gravimetric results. This was because the carbon steel immersed in MEA+H_2O had the highest resistance toward corrosion. For the electrochemical measurement method, unlike gravimetric measurement method, the surface of carbon steel coupons after the immersion was not scrubbed and straightaway brought for testing upon lifting from the solution. Hence, the high resistance detected could be due to the initiation of spontaneous passivation on the carbon steel coupons. Multivariable Power Least Squares Method (MPLSM) was adopted to further examine the relationship of the studied parameters to the corrosion behaviour of carbon steels immersed in solutions MEA+H_2O and MEA+MeOH. The results showed a positive correlation for MEA +H_2O with the immersion time as the most prominent effect affecting polarization resistance. As for the MEA+MeOH, it was found that the lower R^2 value of 0.5 indicated that the current measured parameters were not sufficient to represent the response data set. This implies the interaction between parameters could also affect the polarization resistance. The knowledge gained from this project could contribute to industries in reviewing the impact of organic solvents and improving on the corrosion issue in the operation.

ABSTRAK

Penyerapan karbon dioksida (CO_2) adalah teknologi matang untuk menangkap CO_2 serta proses pembersihan gas asli. Walau bagaimanapun, kehadiran gas asid dalam operasi unit menimbulkan isu-isu kakisan yang perlu ditangani oleh industri untuk memastikan integriti sistem operasi tidak dikompromi. Kajian ini meneroka dan menyediakan maklumat yang komprehensif mengenai kesan variasi parameter proses ke atas tingkah laku kakisan keluli karbon dalam sistem penyerapan CO_2 menggunakan methylethanolamine (MEA) dicampur dengan metanol (MeOH) atau N-methyl-2-pyrrolidone (NMP) sebagai penyelesaian akueda dan hibrid. Parameter proses minat melibatkan jenis penyelesaian amine, suhu, dan beban CO_2. Eksperimen gravimetric dan elektrokimia telah dijalankan untuk menyiasat tingkah laku kakisan kupon keluli karbon dalam penyelesaian ini. Hasil eksperimen menunjukkan bahawa kadar kakisan keluli karbon yang tenggelam dalam penyelesaian amine yang berbeza meningkat sebagai suhu penyelesaian dan peningkatan beban CO_2. Keputusan gravimetric menunjukkan bahawa kupon keluli karbon mempunyai kadar kakisan yang paling rendah adalah yang tenggelam dalam MEA+MeOH. Kedua-dua penyelesaian berair dan hibrid MEA telah diperiksa menggunakan analisis spekroskopi Raman dan permukaan kupon keluli karbon yang tertakluk kepada kakisan telah dikaji menggunakan imbasan pelepasan medan mikroskopi elektron (FESEM). Sebaliknya, keputusan Electrochemical Impedance Spectroscopy (EIS) menunjukkan trend yang bertentangan dari hasil gravimetric kerana keluli karbon yang direndam dalam MEA+H_2O mempunyai rintangan tertinggi terhadap kakisan. Untuk kaedah pengukuran elektrokimia, tidak seperti kaedah pengukuran gravimetric, permukaan kupon keluli karbon selepas rendaman tidak disapu dan terus dibawa untuk ujian apabila mengangkat dari penyelesaian. Oleh itu, rintangan tinggi yang dikesan mungkin disebabkan oleh permulaan kepasifan spontan pada kupon keluli karbon. Kaedah Power Least Squares pelbagai variasi (MPLSM) telah diterima pakai untuk mengkaji lebih lanjut hubungan parameter yang dikaji kepada tingkah laku kakisan keluli karbon yang direndam dalam penyelesaian MEA+H_2O dan MEA+MeOH. Keputusan menunjukkan korelasi positif untuk MEA+ H_2O dengan masa rendaman sebagai kesan yang paling menonjol yang mempengaruhi rintangan polarisasi. Bagi MEA+MeOH, ia mendapati bahawa nilai R^2 yang lebih rendah sebanyak 0.5 yang menunjukkan bahawa parameter diukur semasa tidak mencukupi untuk mewakili set data tindak balas. Ini menunjukkan bahawa interaksi antara parameter juga boleh menjejaskan rintangan polarisasi. Adalah dijangkakan bahawa pengetahuan yang diperoleh daripada projek ini boleh menyumbang kepada industri untuk mengkaji kesan pelarut organik dalam meningkatkan isu kakisan dalam operasi.

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LIST OF ABBREVIATIONS AND CHEMICAL FORMULA

Tscf	-	Trillion of standard cubic feet
NACE	-	National Association Corrosion Engineer
ASTM	-	American Society for Testing & Material
ISO	-	The International Organization for Standardization
MPY	-	Mils penetration per year
SSC	-	Sulfide Stress Cracking
EDX	-	Electron Dispersive X-Ray
EIS	-	Electron Impedance Spectroscopy
CR	-	Corrosion Rate
LPR	-	Linear Polarization Resistance
FESEM	-	Field Emission Scanning Electron Microscopy
USA	-	United State America
MPLSM	-	Multivariable Power Least Squares Method
RMSE	-	Root mean square error

LIST OF SYMBOLS

^{o}C	-	Degree Celsius
g	-	Gram
A	-	Area
D	-	Density
E	-	mass of corroding metal
I_{corr}	-	Measured corrosion current
ΔE/ΔI	-	Polarization Resistance
Ba	-	Empirical Constant
Bc	-	Empirical Constant
R_s	-	electrolytic resistance
R_p/ R_t	-	polarization resistance
C_E	-	electrode capacitance
η	-	activation overvoltage
Z"/ Z_{real}	-	real component
-Z'/Z_{im}	-	imaginary component
j	-	complex number
Z|(ω)|	-	magnitude of impedance
f	-	function of frequency
Φ	-	phase angle
%	-	Percentage
K	-	Rate Constant
R	-	Gas Constant
T	-	Temperature
E_a	-	Activation Energy
A	-	Modified frequency factor
m	-	Mass
v	-	Volume
ρ	-	Density

CHAPTER 1

INTRODUCTION

1.1 Research Background

It is imperative to remove the acidic gases such as carbon dioxide (CO_2) from a gas stream like natural gas to achieve the pipeline standard specification for the business distribution that is applied at industrial scale [4]. The process can also be applied to flue gas from combustion process to alleviate greenhouse gas emission to the environment [4]. High CO_2 content will reduce the market value of natural gas and cause corrosion in the pipeline if the gas is exposed to water [4]. Hence, prudent strategies of CO_2 separation are required to optimize both the capital and the operating expenses of carbon capture technology.

The removal of CO_2 through absorption process using chemical absorbent is more commonly carried out than physical absorbent due to their better performance of CO_2 absorption at low CO_2 partial pressure [5]. Additionally, with the emergence of aqueous alkanolamine solutions, chemical absorption is considered as one of the leading matured carbon scrubbing technologies in recent years [5]. Alkanolamine such as monoethanolamines (MEA), methyldiethanolamine (MDEA), diethanolamine (DEA) and di-2-propanolamine (DIPA) solutions have been frequently used for CO_2 absorption process [5]. However, material corrosion is one of the drawbacks associated these alkanolamine solutions . In the real industry application, when they are in contact with tubing lines and the absorption vessel unit made of carbon steel during the chemical absorption process, the tubing and vessel would be susceptible to the phenomena of corrosion [6, 7]. Carbon steel is usually adopted as the construction material for many industrial units, inclusive of the chemical absorption unit operations. In fact, the production of carbon steel accounts for almost 85 % of the total annual steel production worldwide due to its lower costcompared to other types of alloy [6].

Consequently, the chemical absorption units made up of carbon steel are susceptible to corrosion in the real industry applications [7].

Carbon steel corrosion is a non-desired phenomenon that causes degradation of pipes, and unit operations in the plants. From economic perspective, it is very important to understand the corrosion behavior of carbon steel since every significant investment on plant's equipment or system counts [8].

Various types of corrosion can happen in an amine-based CO_2 absorption unit. These include uniform corrosion, erosion-corrosion, pitting corrosion, intergranular corrosion, crevice corrosion, , and stress corrosion cracking (SCC) [9]. Generally, catastrophic failure of major equipment or a plant due to corrosion problem is intolerable. To further elaborate, the corrosion in CO_2 absorption plants poses significant negative impacts on the industrial economics as well as the safety of plant personnel. The plants could suffer from the losses of revenue due to equipment downtime, cost of repair for the corroded process component, production losses and shortening of equipment life span [10]. According to Greck [11], revenue loss up to RM 1.36 million per day was reported in an amine plant with the production capacity of 100 MMscf (million standard cubic feet) due to corrosion-related repairs. The World Factbook 2017 also provided the information as in Figure 1.1 which shows the data of global cost of corrosion by different categories. The global cost of corrosion is estimated to be RM 10.19 trillion per year [11].

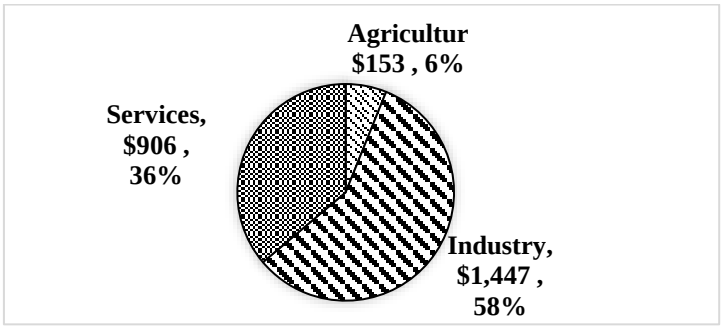

Figure 1.1 Data of cost corrosion study from National Association Corrosion Engineer (NACE) corrosion report. Adopted from Impact, N.E, 2017 [8].

In recent years, a new dedicated hybrid formulation is adopted by Total to improve CO_2 absorption performance of chemical absorption process. It is a replacement for the conventional alkanolamine aqueous solvent that allows to remove various acid gases simultaneously. The hybrid solvent is a mixture of physical and chemical absorbents whereby its capability to remove CO_2 is optimized [12]. There are numerous studies highlighting different hybrid solvents which are composed of amines such as MEA, Aminomethyl propanol (AMP), and, Dimethylethanolamine (DMEA) with acetal compounds are adopted for CO_2 absorption. One of the acetal compounds, 2,5,7,10-tetraoxaundecane (TOU) plays as a physical absorbent is observed to improve the performance of CO_2 absorption significantly compared to other compounds [13]. Another study also found that hybrid solvent consisted of MEA, N-Methyl-2-pyrrolidone (NMP) and water was able to improve the CO_2 removal from natural gas with high CO_2 concentration, compared to the common MEA aqueous solvent [14]. Additionally, hybrid solvent of MEA mixed with methanol (MeOH) was reported to be able to increase the CO_2 absorption performance [15]. With these findings, MEA is regarded as the first-generation benchmark and most well-known amine that makes up hybrid solvents for CO_2 capture. The characteristics such as high reactivity and fast absorption rate are among the major reasons for alkanolamine (or MEA specifically) to be widely used in CO_2 absorption processes [16, 17]. However, MEA is very corrosive and this is affirmed by the literatures [18]. Although hybrid solvents had been proven to improve CO_2 absorption performance, the high corrosion potential of MEA could potentially limit its application for CO_2 capture. Therefore, prior to the expansion of the applications of MEA hybrid solvents, it is necessary to understand the corrosion behaviour of these solvents, especially when they are in contact with carbon steel.

1.2 Problem Statement

In the previous studies, the corrosion phenomena of carbon steels in the aqueous alkanolamine solvent had been investigated by probing the effects of absence or presence of water, CO_2 loading as well as process temperature [5, 19, 20]. Furthermore, the emergence of alkanoamine hybrid solvents has also started to gain

attention for application of CO_2 capture in recent years [12]. Even so, the corrosion behaviour of these alkanolamine hybrid solvent is scarely available. Such missing data has challenged the design of engineering materials which can sustain corrosion in post-combustion capture (PCC) technology and natural gas purification process. Therefore, it is the interest of this study to further elucidate the corrosion behaviour of carbon steel when in contact with alkanolamine hybrid solvent. In this study, the alkanolamine hybrid solvents composed of physical absorbents (NMP and MeOH) and chemical absorbent (MEA) were chosen. NMP was able to improve the CO_2 removal from acid gases through absorption process [14], while MeOH was able to decrease the rate of deprotonation hereby enhancing the diffusivity and solubility of CO_2 in it [15]. As of MEA, it was reckoned to have high corrosiveness and regarded as the first-generation benchmark [16, 17, 18].

1.3 Research Objectives

The objectives of this study are:

i. To characterize the surface morphology changes of carbon steel in MEA aqueous solvent (MEA-H_2O) and MEA hybrid solvents (MEA-NMP-H_2O, MEA-NMP, MEA-MeOH-H_2O & MEA-MeOH).

ii. To investigate the effect of the process condition (i.e. temperature and CO_2 loading) on the corrosion of the carbon steel in MEA aqueous solvent (MEA-H_2O) and MEA hybrid solvents (MEA-NMP-H_2O, MEA-NMP, MEA-MeOH-H_2O & MEA-MeOH) based on gravimetric techniques.

iii. To evaluate the electrochemical characteristics of carbon steel in the MEA solvent with the least corrosion impact and the significance of process condition to the corrosion behaviour.

1.4 Scope of Study

To establish the design of this study, the specific concentration of chemical absorbent of MEA for both the aqueous and hybrid solvents was fixed at 20 wt% which it was the median point of the concentrations commonly adopted (10-30 wt%) in previous studies [21]. Furthermore, it was also reported that the optimal MEA concentration in water was in the range of 20-30 wt% [22]. Therefore, concentrations of the MEA aqueous and hybrid solvents studied in this work are as listed as follows:

1. 20 wt% MEA + 80 wt% H_2O (named as MEA-H_2O solvent)

2. 20 wt% MEA + 40 wt% NMP + 40 wt% H_2O (named as MEA-NMP-H_2O solvent)

3. 20 wt% MEA + 80 wt% NMP (named as MEA-NMP solvent)

4. 20 wt% MEA + 40 wt% MeOH+ 40 wt% H_2O (named as MEA-MeOH-H_2O solvent)

5. 20 wt% MEA + 80 wt% MeOH (named as MEA-MeOH solvent)

The gravimetric technique was employed to compare the effect of process conditions of the carbon steels. Then, these carbon steels' surface were characterized via Field Emission Scanning Electron Microscope (FESEM) and the solutions were characterized via Raman Spectroscopy. Subsequently, the MEA solution which had the least corrosion of the gravimetric outcome was further analyzed via the electrochemical technique. Finally, the significance of each process parameters was determined using the multivariable power least squares method (MPLSM). Ultimately the criteria of the best solvent was justified throughout the quantum of the investigations.

1.5 Significance of Study

The addition of the organic solvent into the amine-based solvent was found to improve the CO_2 absorption process effectively. The findings had been encouraging

enough to merit further investigation on the corrosion behaviour of carbon steels in these organic solvents. This study will provide a greater level of understanding for the industries to review the potential technique in developing new corrosion-resistant materials or technologies, thereafter.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Ooi Z.L**, Tan P.Y, Tan L.S & Yeap S.P. (2020). Amine-based solvent for CO_2 absorption and its impact on carbon steel corrosion: A perspective review. *Chinese Journal of Chemical Engineering*, 1;28(5), 1357-67. (Indexed by SCOPUS).

Indexed Conference Proceedings

1. **Tan P.Y,** Tan L.S, Yeap S.P, Kow K.W, Shariff A.M, & Wong M.K. (2020). Corrosion of Carbon Steel in Hybrid N-Methyl-2-Pyrrolidone-Monoethanolamine Solutions for Carbon Dioxide Absorption Unit Application. *IOP Conference Series: Materials Science and Engineering* 2020, 736(2), 22-86).