CERTIFICATION OF APPROVAL

Study on Carbon Dioxide in Several Types of Amines

By

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own work except as specified in the reference and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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ABSTRACT

This project is basically to study the carbon dioxide (CO_2) absorption in several types of amines. In recent years, the importance of the chemical absorption process rises as a competitive process to reduce the discharge of CO_2 into atmosphere. This is due to CO_2 characteristics as it is one of the primary green house gases which represent approximately 83% of anthropogenic effect. Besides, absorption of acid gases such as CO_2 from natural and refinery gases is an important industrial process. Aqueous alkanolamines solutions are the most widely used solvents for the acid gas absorption process. Aqueous alkanolamines that are used in the study are monoethanolamine (MEA), diethanolamine (DEA) and 2-amino-2-methyl-1-propanol (AMP). The objectives of this project are to determine the amount of CO_2 that being absorbed in amines, to determine the changes in inlet and outlet concentration of CO_2 during absorption, to determine the changes in concentration of amines and finally to determine the best mix amines for optimum absorption of CO_2.

The scope of this project includes studying the CO_2 absorption into aqueous alkanolamines and mixed aqueous solutions of alkanolamines with varying temperature and concentration of alkanolamines by measuring the inlet and outlet of CO_2 concentration and concentration of amines after the absorption. This study will also involve the measurement of density of aqueous alkanolamines and its mixture. The findings are then compared to the trend obtained from previous literature. From the result obtained, it can be concluded that as concentration of aqueous amines increases, rate of absorption increases. Theoretically, for temperature effect, as temperature increases, rate of absorption decreases. However, this study does not fully obey the theory due to some conditions which will be discussed thoroughly in this paper. The addition of primary and secondary amines, MEA and DEA into sterically hindered amine, AMP results in significant enhancement of CO_2 absorption rates. Amine blends of MEA and AMP gives better CO_2 absorption rates compared to DEA and AMP blends as MEA being primary amines has stronger effect towards CO_2 absorption.

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ABBREVIATION AND NOTATION

AMP	2-amino-2-methyl-1-propanol
CO_2	Carbon dioxide
D	Diffusion coefficient
DEA	Diethanolamine
GC/MS	Gas chromatograph / mass spectrometer
HCl	Hydrochloric acid
H	Henry's law constant
MDEA	N-methyldiethanolamine
MEA	Monoethanolamine
k_{app}	Apparent rate constant

N_2	Nitrogen
N_2O	Nitrous oxide
NaOH	Sodium hydroxide
r	Reaction rate
RI	Refractive Index

CHAPTER 1
INTRODUCTION

1.1 BACKGROUND

Nowadays, the climate of the earth is warmer than any time in the past thousand years. This change, which is incredibly fast by geological time scales, will be brought about by increase level of carbon dioxide (CO_2) in the earth's atmosphere. Since the Industrial Revolution, man has introduced tremendous amounts of CO_2 into the atmosphere. While some of this gas is assimilated into natural reservoirs, approximately 50% remains airborne.

The most important source of excessive CO_2 is the burning of carbon based fossil fuels for energy production. Fossil-fuel use increases at an exponential rate of 4.3% annually. This consequently causes a doubling of CO_2 concentrations. CO_2 is also a byproduct of all living systems and is normally considered harmless. Besides, CO_2 also is produced in large quantity worldwide by many important industries such as refinery, steel production, and cement production, petrochemical and as well as gas processing plant. Also contributing to the net increase in CO_2 is the deforestation of large land areas each year. CO_2 accumulation in the atmosphere is the most dangerous problem today and these will increase CO_2 concentration which will causes greenhouse effect. The greenhouse effect is a result of the absorption of infrared radiation by the surface of the earth. This absorption causes an increase in atmosphere temperature. This in turn increases the amount of water vapor in the atmosphere. Since water vapor is also a strong absorber of infrared radiation, a positive feedback mechanism is created, leading to further infrared-radiation absorption.

The doubling of atmospheric CO_2 will cause an increase in the mean global temperature of about 30°C to 50°C. As temperature increase, atmospheric circulation patterns are altered which will change local weather patterns. These changes could bring enormous impact on agricultural production. Due to the rise of global temperature, significant portion of the polar ice caps would melt and will result in a rise in sea level. For the past 150 years, CO_2 concentration level in atmosphere has rose to 370 ppm from 280 ppm. This would lead to flooding in coastal areas including major population centers. The repercussions of dramatic climate change are vast, especially to portions of the environment that have already been mismanaged and are currently stressed such as impacts like increased human health effects, loss of habitat, decreases in natural resources and loss of biodiversity. The vulnerability and sensitivity of such climate changes rely on the lack of expectation, the magnitude of change, its rate and frequency. Areas that are temperate become tropical. Desert areas may become more extreme and the sea level will rise above current mean levels. Repercussions of these changes will be in the form of freshwater shortages and shifts in the production of food and wood products. Human will be affected directly by extreme temperatures and weather events. There also will be indirect effects of infective parasites, water borne and food borne diseases and increases levels of detrimental health effects due to air pollution. Thus, immediate action need to be taken to reduce the amount of CO_2 being released to atmosphere. The fact that changes in CO_2 concentrations in the atmosphere could cause changes in the earth's climate has been known for over one hundred years. However, only in the last five to ten years has significant research been done in this field. The problem of the greenhouse effect might be remedied by a reduction in the use of fossil fuel, large scale reforestation to increase the capacity of bionic sink. In order to lessen the severity of the problem, fossil fuel consumption must be curtailed and alternate energy sources developed. However, not much hope is held out for these remedies.

Thus, it is becoming increasingly important to develop economically feasible technology to capture and sequester CO_2 from fossil fuel burning power plants and by removing CO_2 from process stream before it is released to atmosphere. There has been worldwide attention to reduce CO_2 production and with current technologies, CO_2 separation can be performed by several approaches including absorption into liquid solvents, adsorption on solids, permeation through membranes, and chemical conversion. Although there are several different methods that have been proposed for the capture and separation of CO_2, one of the few methods that has been proven to work on industrial scale for capture of CO_2 from flue gas is chemical absorption using certain solvents such as the removal system for the absorption of CO_2 with amines and NH_3 which is an advanced air pollution control device to reduce greenhouse gas emissions. Absorption is a mass-transfer process in which a vapor solute in a gas mixture is absorbed by means of a liquid in which the solute is more less or less soluble. In other words, the absorption of gases is the process of removing one or more components from a mixture of gases and it is achieved by diffusion into a soluble liquid. Reverse absorption process is called stripping or desorption. The important variables affecting the equilibrium of solute are temperature, pressure and concentration. In recent years, absorption of acid gases such as CO_2 from natural and refinery gases is also an important industrial process. For example, the economics of hydrogen and ammonia plants depends heavily on the efficiency of systems used for removal of CO_2 from process plants since separation of CO_2 from ammonia synthesis gas to make it fit for the synthesis reaction and recovering it to meet the later requirements of urea production is a major step in the design of an ammonia plant. Over the past thirty years there have been many innovations in this section of plant, from ordinary water wash to hot potassium solutions to primary and secondary amines. The importance of the chemical absorption process rises as a competitive process to reduce the discharge of CO_2 into atmosphere. There has been a continuous development in technology for CO_2 removal, with the prime focus to achieve higher purity of outlet gas.

Aqueous alkanolamine solutions are the most widely used solvents for the acid gas absorption process and frequently used for removal of acidic gases such as CO_2 and H_2S from gas streams in the natural gas, synthetic and refinery industries. Alkanolamines are weak basic compounds that react with certain acid gases, forming weak chemical bonds. These bonds are easily broken upon mild heating. Alkanolamines are classified depending on number of organic hydrocarbon group attached to the nitrogen atom as primary, secondary or tertiary. It is used primarily to absorb CO_2 and H_2S. Monoethanolamine (MEA) and diethanolamine (DEA) became the dominant commercial absorbents. MEA are **primary amines** with one ethanol group attached to the basic nitrogen atom is the strongest amine. MEA is effective in removing CO_2 but requires a large quantity of heat to regenerate. MEA is a very efficient absorbent and yields low residual CO_2 in the outlet gas.

DEA are **secondary amines** with two ethanol groups or two isopropanol groups respectively attached to the nitrogen atom. The additional alcohol groups draw more of the free electron character away from the nitrogen atom and make the secondary amine somewhat weaker bases. Being a secondary amine, the reactivity is slow compared to primary amines. DEA is used when the specification allows for some CO_2 to be left in treated gas.

N-methyldiethanolamine (MDEA) is **tertiary amines** and has two ethanol groups attached to nitrogen atom along with methyl group. Being a tertiary amine, MDEA does not have a hydrogen atom attached to nitrogen. The CO_2 reaction can therefore only occur after the CO_2 dissolves in water to form a bicarbonate ion. The bicarbonate ion then undergoes an acid base reaction with amine to yield the overall CO_2 reaction. MDEA is a weak base that reacts much faster with hydrogen sulphide than with CO_2. This makes MDEA is particularly selective under the proper design conditions. MDEA can also be used non-selectively for bulk removal of CO_2 from gas streams, but bulk removal requires the aid of absorption enhancing additives. Tertiary amines are less inherently corrosive and can be used in higher concentrations but not a good choice when the raw gas pressure is low or the specification calls for deep CO_2 removal. However, tertiary amines, particularly MDEA are well suited for selective absorption on high pressure gas streams.

2-amino-2-methyl-1-propanol (AMP), which is the hindered form of MEA, is considered as one of the most important hindered amines for the industrial gas treating processes. According to Sharma (1964, 1965) this **sterically hindered** amine has steric effects which influences the stability of the carbamates formed by the amine with CO_2 and it has higher cyclic capacities of CO_2 and appreciable rate of absorption. AMP is considered today is one of the most important hindered amines for natural gas treatment processes.

1.2 PROBLEM STATEMENT

CO_2 is the primary green house gas representing roughly 83% of anthropogenic effect. Combustion of fossil fuels is responsible for about 85% of the world's energy supply and is the most significant source of anthropogenic CO_2 emissions worldwide. As amount of CO_2 is increasing, further consequences such as increase in temperature worldwide, change in local weather patterns and rising of sea level caused by melting of polar ice caps is happening. The severity of consequences of this major climatic change requires the action to be taken to lessen man's input of CO_2 into the atmosphere. Besides, the greenhouse treat is a global problem that recalls for global action. Apart from that, the removal of CO_2 from natural gas, synthesis gas for ammonia production and refinery gases has also become a very important operation in process industry. The importance of chemical absorption process rises as a competitive process to reduce the amount of CO_2 being released to atmosphere.

Although there are several different methods that have been proposed for the capture and separation of CO_2, one of the few methods that has been proven to work and has been mentioned previously is chemical absorption of the acid gas components into solution of alkanolamines. Aqueous alkanolamines solutions are the most widely used solutions for the acid gas absorption process. Industrially important alkanolamines for this operation are monoethanolamine (MEA), diethanolamine (DEA), and N-methyldiethanolamine. Besides, a new class of amines, sterically hindered amines such as 2-amino-2-methyl -1-propanol (AMP) has been introduced as commercially attractive new absorbent over conventional amines.

Aqueous MEA solutions are most extensively used because of its high reactivity with CO_2, low solvent cost and ease of regeneration. However, highly concentrated MEA are not used for the removal of acidic gas such as CO_2 as they can also react with reactor vessels and several process compartments. Besides, maximum CO_2 loading in MEA is also limited. Sterically hindered amine such as AMP has higher cyclic absorption capacity of CO_2 than conventional primary and secondary amines such as MEA and DEA and also appreciable rates of absorption. Thus, nowadays, blends of amines could be applied to enhance the loading capacity and absorption rate of CO_2. Hence, this project is basically to study on CO_2 absorption on several types of amines such as MEA, DEA and AMP. Furthermore, the amine mixtures will then be used for CO_2 removal with the intent of combination the advantages of the amines in the mixture. Amine blends can increase the total amine consideration in solution and thereby enhance the CO_2 loading capability. Consequently, the best amine blends that can be utilized in CO_2 absorption can be obtained from the study.

1.3 OBJECTIVES

Several objectives have to be achieved in doing this project. The objectives are identified and are listed as below:

i) To determine the amount of CO_2 that being absorbed in amines.
ii) To determine the inlet and outlet concentration of CO_2 during absorption.
iii) To determine the changes in concentration of amines.
iv) To determine the best amines and the optimum percentage of mix amines for optimum absorption.

1.4 SCOPE OF STUDY

Basically this study is an experiment based project. This project requires some experiments to be carried out to observe the absorption of CO_2 by using several types of amines which includes MEA, DEA and AMP and also blends of those amines. In the experiment, the densities of amines will be measured at variation of temperature and concentration of amine. Temperature and concentration will be the manipulated variables in comparing the behavior of the CO_2 absorption. These will include the determination of changes of CO_2 concentration after absorption in amines. All of the trend obtained from the results obtained from experiment will be compared to the previous literature. From the experiment that has been carried out, the best amine blends for CO_2 absorption can be determined. Some recommendations are expected to be made throughout the progress of the project.

CHAPTER 2

LITERATURE REVIEW & THEORY

2.1 REACTION MECHANISM

Zwitterion mechanism that was originally proposed by Caplow and reintroduced by Danckwerts was accepted as the reaction mechanism for the carbamate formation between CO_2 with primary and secondary alkanolamines. Besides the primary and secondary alkanolamines, the zwiterrion mechanism was also found to be suitable for modeling the absorption of CO_2 into aqueous AMP solutions according to Saha, Bandyopadhyay and Biswas. The carbamate formation reaction occurs when CO_2 reacts with alkanolamines. Besides, protonated amine is also being produced.

$$CO_2 + 2R_1R_2NH \leftrightarrow R_1R_2NCOO^- + R_1R_2NH_2^+ \qquad (1)$$

Where R_1 is an alkyl and R_2 is H for primary amines and alkyl for secondary amines.

1. The first step of the mechanism reaction is the formation of zwitterion.

$$CO_2 + R_1R_2NH \underset{k_{-1}}{\overset{k_2}{\leftrightarrow}} R_1R_2NH^+COO^- \qquad (2)$$

2. Base-catalyzed deprotonation of the zwitterions by any base existing in the solution. The base could be amine, OH^- or H_2O.

$$R_1R_2NH^+COO^- + B \xrightarrow{k_b} R_1R_2NCOO^- + BH^+ \qquad (3)$$

The responding reactions are as follows:

$$R_1R_2NH^+COO^- + R\,R_2NH \xrightarrow{k_{amine}} R_1R_2NCOO^- + R_1R_2NH_2^+ \quad (4)$$

$$R_1R_2NH^+COO^- + H_2O \xrightarrow{k_{H_2O}} R_1R_2NCOO^- + H_3O^+ \qquad (5)$$

$$R_1R_2NH^+COO^- + OH^- \xrightarrow{k_{OH^-}} R_1R_2NCOO^- + H_2O \qquad (6)$$

3. Danckwerts derived the forward reaction rate equation at steady state as:

$$r_{CO_2-amine} = \frac{k_{2,R_1R_2NH}[CO_2][R_1R_2NH]}{1+\dfrac{k_{-1}}{k_{H_2O}[H_2O]+k_{OH^-}[OH]+k_{R_1R_2NH}[R_1R_2NH]}} \qquad (7)$$

4. For normal primary and secondary amines such as MEA and DEA, the carbamates formed (reaction 1) are quite stable. In the case of a hindered amine such as AMP, the carbamate formed is unstable and thus it undergoes carbamate reversion reaction as follows:

$$R_1R_2NCOO^- + H_2O \leftrightarrow R_1R_2NH + HCO_3^- \qquad (8)$$

This condition is due to steric effects that influence the stability of the carbamate formed by the amine with CO_2. According to Sarvori and Savage, due to the instability of the hindered amine, these readily undergo hydrolysis forming bicarbonate and releasing free amine again reacts with CO_2 thus leading to a stoichiometric loading capacity of 1 mol of CO_2/mol of amine with appreciable rate of reaction.

5. Apart from above reactions, CO_2 also reacts in aqueous solutions which consist of hydration of CO_2 and the bicarbonate formation.

   a. Hydration of CO_2

      $CO_2 + H_2O \leftrightarrow HCO_3^- + H^+$ (slow) (9)

   b. Bicarbonate formation

      $CO_2 + OH^- \leftrightarrow HCO_3^-$ (10)

The hydration of CO_2 reaction is very slow and according to Blauwhoff, Versteed & van Swaaij it may usually be neglected. However, the bicarbonate formation reaction is fast and can enhance mass transfer even the concentration of hydroxyl ion is low. The forward reaction rate of bicarbonate formation can be described (Pinsent et al.)

$$r_{CO_2-OH^-} = k^*_{OH^-}[CO_2][OH^-] \tag{11}$$

$$\log_{10} k^*_{OH^-}(m^3kmol^{-1}s^{-1}) = 13.635 - \frac{2895}{T(K)} \tag{12}$$

Due to chemical reaction between CO_2 and amines, neither the free-gas solubility nor the diffusivity of CO_2 in amine solutions can be measured directly. In experiments done by Wang and Li to study the kinetics of absorption of CO_2 into aqueous solution of 2-amino-2-methyl-1-propanol and diethanolamine (DEA), N_2O analogy was used to determine the diffusion coefficient of CO_2 in aqueous AMPD solutions. Actually, the N_2O analogy has been frequently used to estimate the solubility and diffusivity in amine solutions. Henry's law constant and diffusion coefficient in aqueous AMPD was calculated by utilizing solubility and diffusivity of N_2O and CO_2 in water.

$$\left(\frac{H_{CO_2}}{H_{N_2O}}\right)_{AMPD} = \left(\frac{H_{CO_2}}{H_{N_2O}}\right)_{H_2O} \tag{13}$$

$$\left(\frac{D_{CO_2}}{D_{N_2O}}\right)_{AMPD} = \left(\frac{D_{CO_2}}{D_{N_2O}}\right)_{H_2O} \tag{14}$$

Vetsteeg and van Swaaijj proposed, based on available data of solubility and diffusivity of N_2O and CO_2 in water, four convenient equations as follows:

$$H_{N_2O}(kPam^3kmol^{-1}) = 8.5470 \times 10^6 \times \exp(-2284/T(K)) \tag{15}$$

$$H_{CO_2}(kPam^3kmol^{-1}) = 2.8249\times10^6\times\exp(-2044/T(K)) \quad (16)$$

$$D_{N_2O}(m^2s^{-1}) = 5.07\times10^{-6}\times\exp(-2371/T(K)) \quad (17)$$

$$D_{CO_2}(m^2s^{-1}) = 2.35\times10^{-6}\times\exp(-2119/T(K)) \quad (18)$$

Equations (15) – (18) are widely applied in previous study to calculate the corresponding solubility and diffusivity of CO_2 in amine solution. Thus, this analogy could also be applied in the experiment to study the CO_2 absorption in aqueous alkanolamines and the blends of the amines specifically in diffusion of CO_2.

2.2 RELATED STUDY ON CO_2 ABSORPTION

There have been a lot of studies and researches that have been done due to the need to reduce CO_2 being released to atmosphere. All of the studies investigated on mechanism and kinetics of the reaction between CO_2 and aqueous alkanolamine solutions. A wide variety of alkanolamines such as MEA, DEA, MDEA have been used industrially for a number of years according to Kohl and Riesenfeld. A recent advancement in gas treating technology is the application of sterically hindered amines such as AMP which offer absorption capacity, absorption rate, selectivity and degradation resistance advantages over conventional amines for CO_2 removal from gases according to Sartori and Savage. As had been mentioned earlier, all the alkanolamines had its own advantages in absorption and thus the absorption of acid gases in blended amines has specific advantages over the use of single amines. Based on study done by Chakravarthy, Phukan and Weiland in 1985, the additional of small amount of primary amine to conventional tertiary amine such as MDEA can enhance the rate of absorption of CO_2 to large extent without appreciably affecting its own characteristics. Yih and Shen in their paper quoted that compared to MDEA, AMP has the same CO_2 loading capacity but has higher reaction rate constant for the reaction with CO_2. Thus, blends of AMP and MEA might be an attractive new solvent for the acid gases treating process.

From the study done by Xiao, Li and Li in 2000 by using laboratory wetted wall column it was found that the overall reaction rate constant for the reaction with CO_2 increased with concentration of MEA and also increase with temperature. This correlation is shown in Figure 2.1.

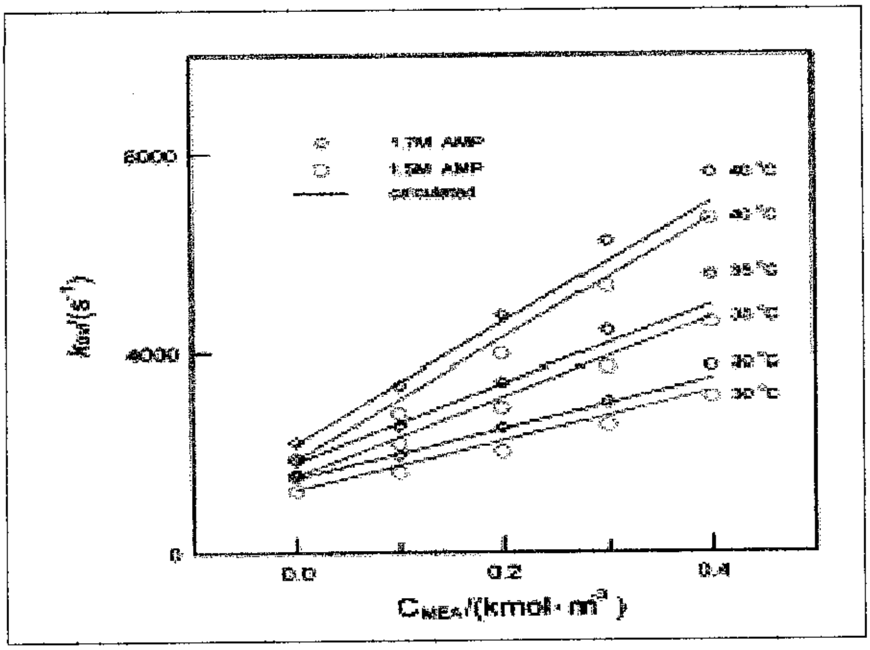

Figure 2.1 Overall Reaction Rate Constant for reaction of CO_2 with AMP + MEA + H_2O as a function of MEA concentration

Wang and Li et al. did a study on kinetics of CO_2 absorption of CO_2 into aqueous solutions of AMP and DEA. This study also used wetted wall column as the experimental apparatus. Based on the result that was obtained, the addition of small amounts of DEA to AMP results in significant increase in the CO_2 absorption rates. Figure 2.2 and Figure 2.3 showed the plot of apparent rate constant vs. the concentration of DEA. The apparent rate constant, k_{app} increases as the temperature and DEA concentration increase. Besides, at the same temperature, the values of k_{app} for AMP with 1.5 kmol/m^3 concentrations are larger than those of AMP with 1.7 kmol/m^3 concentrations at the same DEA concentration. For the purpose of comparison of the effect of MEA and DEA on the absorption of CO_2 in aqueous AMP solutions, the values of k_{app} reported by Xiao et al. were included in Figure

2.3. It can be seen that DEA does not yield that strong effect, as MEA does, on the CO_2 absorption into aqueous AMP solutions.

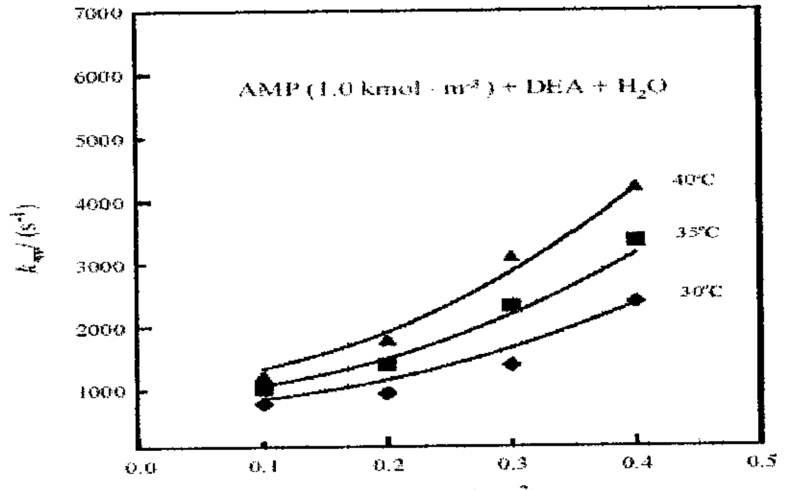

Figure 2.2: The apparent rate constant for the reaction of CO_2 with AMP (1.0kmol/m^3) + DEA as a function of DEA concentration

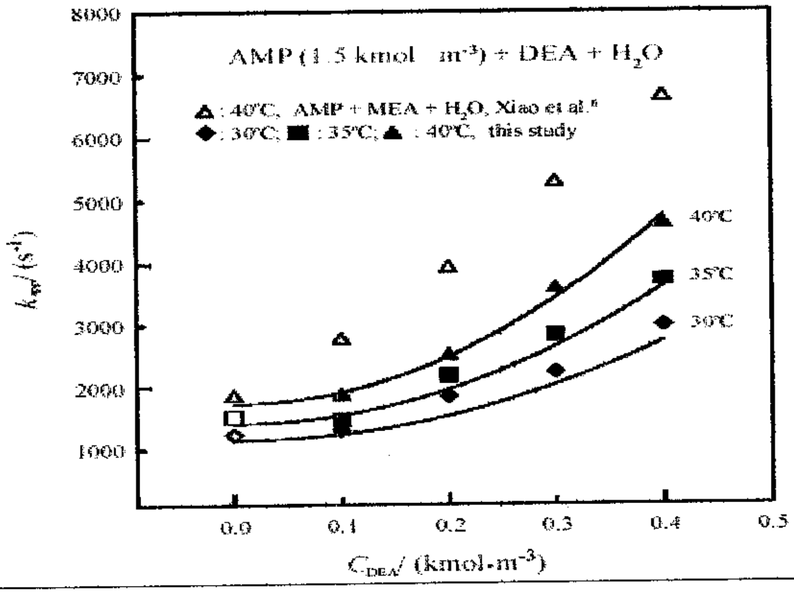

Figure 2.3: The apparent rate constant for the reaction of CO_2 with AMP (1.5kmol/m^3) + DEA as a function of DEA concentration.

CHAPTER 3

METHODOLOGY/ PROJECT WORK

3.1 PROJECT OVERVIEW

In doing this project work, initially intensive researches have been carried out to obtain some overview of the project and basic understanding before pursuing the project. Researches have been done by referring to several related books, research articles and as well as online journals. By comparing the literature reviews obtained, an experimental procedure and analytical procedure was chosen. Although almost all of the previous study done use wetted wall column as the experiment apparatus to study the CO_2 absorption in alkanolamines, a method similar to gas bubbler method was chosen to be used in this study in order to approach the study with different method. Since, there is no similar study on CO_2 absorption that used the bubbler method, only the trend of the result obtained is compared to the previous study as there are no specific researches that used gas bubbler method. Several operating parameters such as solvent concentration, operating temperature and solvent blends were systematically varied in order to investigate their influences towards CO_2 absorption.

The experimental setup is almost similar to gas bubbler method, whereas the gas is bubbled directly into a liquid. This method is best applied to gas-liquid systems that have a large solubility, rapidly approach equilibrium and for which there is a direct and simple method of analysis such as acid-base titration, precipitation titration, spectrophotometry or weight increase. There are several important details in the use of bubbler method that includes temperature. In the experiment, the gas needs to be at the same temperature and this is accomplished by passing the gas through a suitable length of tubing immersed in the water bath before passing into the solvent. Besides, pressure is also considered in this method and the gas is kept at constant by keeping a back pressure on the exit gas equal to the solvent vapor pressure. Time of bubbling is also one of the important factors in gas bubbler method. CO_2 is a gas that requires a longer than average bubbling time to reach saturation in water and aqueous solutions.

3.2 METHODOLOGY

The experimental setup is set in water bath in order to maintain the temperature throughout the process. Two filtration flasks are used where one serves as the reference cell and the other one is where the absorption of CO_2 in amines takes place. The experimental setup is shown in figure below:

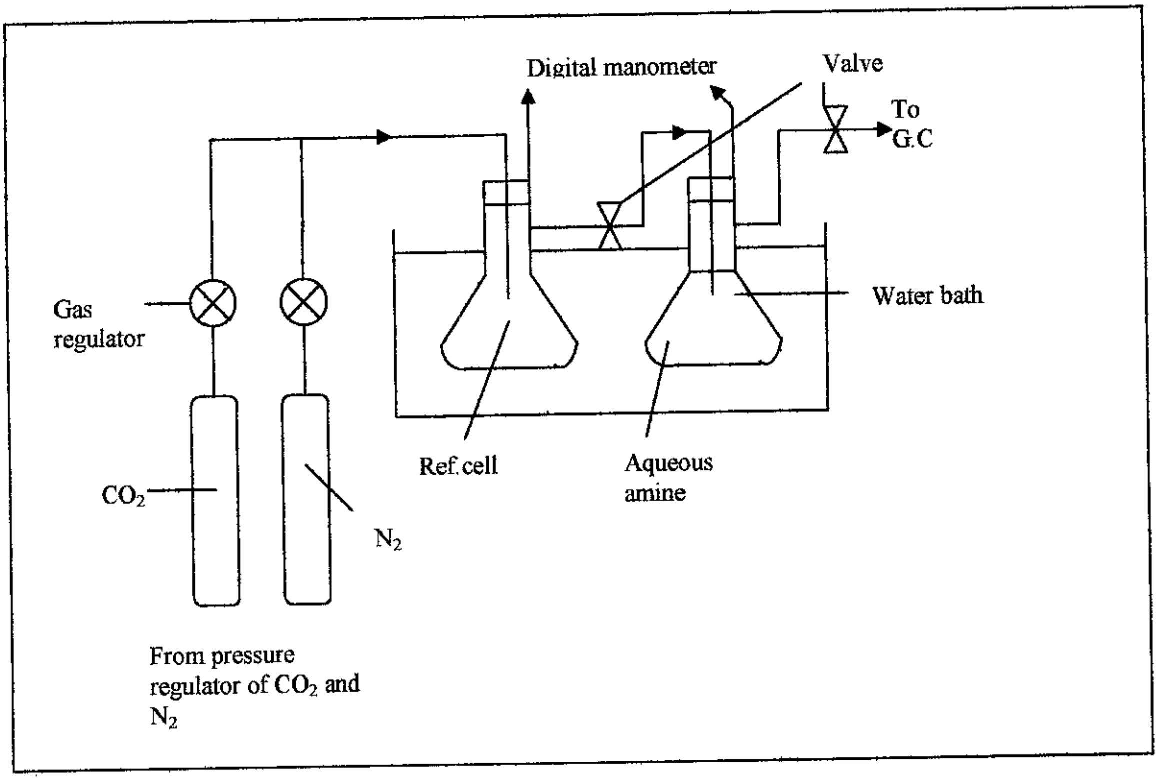

Figure 3.1: Experimental set up for CO_2 absorption in amines

CO_2 cylinder with certified purity of 99.995% from MOX Malaysia was used. N_2 cylinder was also procured from MOX Malaysia. The purity of N_2 used was better than 99%. Monoethanolamine (MEA), diethanolamine (DEA) and 2-amino-2-methyl-1-propanol (AMP) were supplied by R&M Chemical, Essex, UK with purity of 99.7% each. Amine concentrations were determined by titration with standard 5.0

kmol/m^3 of HCl by using phenolphthalein as indicator. The summary of amine concentrations is presented in the preceding table.

Table 3.1: Concentration of pure alkanolamines

Alkanolamines	Concentration, kmol/m^3
Monoethanolamine (MEA)	15.2
Diethanolamine (DEA)	10.15
2-amino-2-methyl-1-propanol	10.46

The first part will deal with preparation of sample and for this case, the preparation of aqueous amine with different concentration. Besides, the mixed amines were also prepared. The amines; MEA and DEA were prepared using distilled water at concentration of 1.5 kmol/m^3, 2.0 kmol/m^3, 2.5 kmol/m^3 3.0 kmol/m^3. The amine blends of MEA and AMP and blends of DEA and AMP were also prepared to check the effect on CO_2 absorption. The AMP concentrations of 1.5 kmol/m^3 were added with MEA and DEA with concentrations of 0.1 kmol/m^3, 0.2 kmol/m^3, 0.3 kmol/m^3 and 0.4 kmol/m^3. For every run, 100 ml of sample was used for CO_2 absorption. The CO_2 absorption were measured over a temperature range of 35°C, 40°C,and 45°C and total pressure of 5-25 kPa.

Firstly, the setup was purged with nitrogen gas to get rid of all the possible gases exist in the cells. After the setup was completely purged, the carbon dioxide gas was introduced into the apparatus. Flow of both gases into the setup was controlled by the gas regulator attached to the cylinders. For each run the equilibrium cell or reference cell was allowed to reach thermal equilibrium with respect to desired temperature and pressure for about 5 minutes. Once it reaches equilibrium, the ball valve connecting to the second cell containing amine was opened to allow gas to enter the cell where the CO_2 absorption will take place. Digital manometers were connected to each of the cell to check on pressure. The setup was ensured to be completely sealed to avoid any possible leaking of gas throughout the experiment. This was checked by using liquid leak detector where the liquid was injected at all the connections and any bubbles formed at the joint will indicate leak at that point.

All the joints and connections were sealed with Teflon tape. The experiment was run until there was no more absorption takes place. This was obtained by observing the pressure of the second cell to drop until it is held constant. Throughout the experiment, water bath was used in order to maintain the temperature in the cells. In this experimental section, a few measurements need to be taken in order to predict the CO_2 absorption behavior that includes:

i) Density measurement

The densities of alkanolamines solution were measured by using digital densitometer. The measurements were performed in constant temperature water bath where temperature is held constant to ±0.2°C. The measurements were done over a temperature range of 35°C, 40°C and 45°C.

ii) Analysis of aqueous alkanolamines after absorption

Due to chemical reaction between CO_2 and amines, solubility and diffusivity of CO_2 in amine solution cannot be measured directly. The N_2O analogy has been frequently used to estimate the solubility and diffusivity of CO_2 in amine solution. The N_2O analogy is the same as has been described in literature review part previously. However, due to unavailability of N_2O, this measurement could not be done. Thus, the amine concentration after absorption of CO_2 was checked by using Refractometer by measuring its Refractive Index (RI). The RI varies depending on wavelength of light and temperature. This measurement gave the total concentration of aqueous alkanolamines and not the individual components present in the solution. The measurements were taken before and after CO_2 absorption. The result was obtained in term of changes in RI. Apart from that, the CO_2 loading of the amine solution was determined by standard titration methods with HCl using phenolphthalein as indicator.

iii) Absorption rate measurement

The concentration of CO_2 at the inlet and outlet of the filtration flask are measured by gas analysis using a gas chromatograph/ mass spectrometer (GC/MS). The gas phase samples are analyzed by gas chromatograph using porapaq-Q column, TCD detector and helium carrier gas. The procedure is the same as had been described previously. After introducing CO_2, the pressure in the experimental set up was reduced when the end hose was connected to GC until pressure is about 25 kPa. This was the maximum allowable pressure that can be applied to the set up. Then, the valve connecting the apparatus to gas cylinder was closed and valve connecting reference cell and sample cell was also closed. The gas was allowed to equilibrate in the reference cell for about five minutes before the valve connecting the cell to the sample cell can be opened. Finally gas sample was taken and analyzed after 10 minutes by using GC/MS.

CHAPTER 4

RESULT AND DISCUSSION

Absorption is a process when two contacting phases are a gas and a liquid. In this study, CO_2 is absorbed by aqueous alkanolamines which includes MEA, DEA and blends of MEA and DEA with AMP. For the first part of the experiment, the applicability of the experimental set up was tested. This was done by absorbing CO_2 into sodium hydroxide, NaOH with concentrations of 0.05M using the experimental setup described in the previous section. CO_2 gas was absorbed into the NaOH solution at operating pressure about 5-10 kPa. As there was no specific regulator for CO_2 cylinder at the moment, this resulted in large fluctuations of CO_2 pressure and it was quite difficult to maintain the pressure at certain point. The pressure could not be higher than this since the stopper for the experimental setup will blow out if excessive pressure was applied. The experiment was run for a few times due to some reasons that occurred such as stopper blew out during the experiment which was most likely affecting the findings of the experiment. Standard acid titrant of 0.1 M hydrochloric acid, HCl was used for titration with NaOH while phenolphthalein acted as indicator. There are reactions that occur during the absorption of CO_2 into aqueous NaOH. It exhibits pseudo first order reaction.

$$CO_2 + 2OH^- \rightarrow CO_3^- + H_2O \qquad (19)$$

There are also reactions of CO_2 in the aqueous solutions.

$$CO_2 + H_2O \leftrightarrow HCO_3^- + H^+ \qquad (20)$$

$$CO_2 + OH^- \leftrightarrow HCO_3^- \qquad (21)$$

For chemical absorption of CO_2 into NaOH solution, the solution turned from pink color to clear end point after titration with HCl. In this part, hydroxide ion are neutralized and followed by conversion of carbonate ions to bicarbonate ions. The difference between a feed sample and a product sample is proportional to CO_2 being absorbed in the NaOH solution. The following are result obtained from the titration of NaOH with HCl.

Initial volume of 0.05 M NaOH = 50 ml
0.1 M HCl needed for titration = 4.3 ml
Product (after titration) volume = 53 ml
Δ of product volume and initial volume = 3 ml

Thus, amount of CO_2 absorbed in NaOH is 3ml. From the finding, it can be concluded that CO_2 absorption took place only in a small amount. This is due to the low concentration of NaOH that was being used as absorption rate is affected by solvent concentration. However, the result obtained cannot be compared as there was no literature review on this. It could be assumed that the experimental procedure can be used in utilizing absorption of CO_2 in aqueous alkanolamines. In doing the experiment, it must be strictly ensure that gas from atmosphere enter the absorption cell is very small and can be neglected.

4.1 Density measurement

The densities of MEA, DEA and blends of AMP with MEA and DEA were measured in various temperatures; 35°C, 40°C and 45°C and the results are presented in Figure 4.1 and Figure 4.2. The densities were measured using digital densitometer with deviation of 0.001 g/cm^3.

The density of aqueous MEA increases as the concentration of MEA increases and decreases as the temperature increases which is shown in Figure 4.1. According to Hsu and Li (1997), for the temperature below 70°C, the density of pure fluids has the order of MEA > H_2O. In Figure 4.2, the density of DEA also increases with the concentration and decreases with increase in temperature. The graph obtained was not too linear due to some factors that affected the measurement such as the actual temperature of the solutions are not the same as the temperature of the water bath and this led to slight deviations in density measurement.

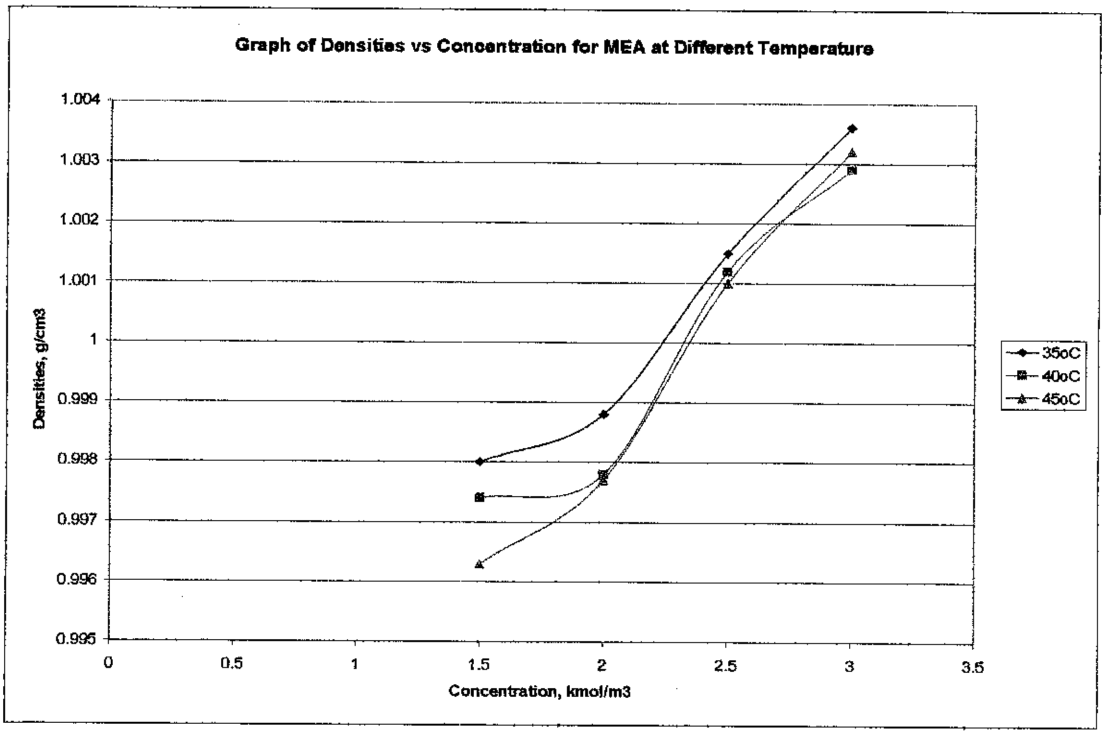

Figure 4.1: Graph of density versus MEA concentration at 35°C, 40°C and 45°C.

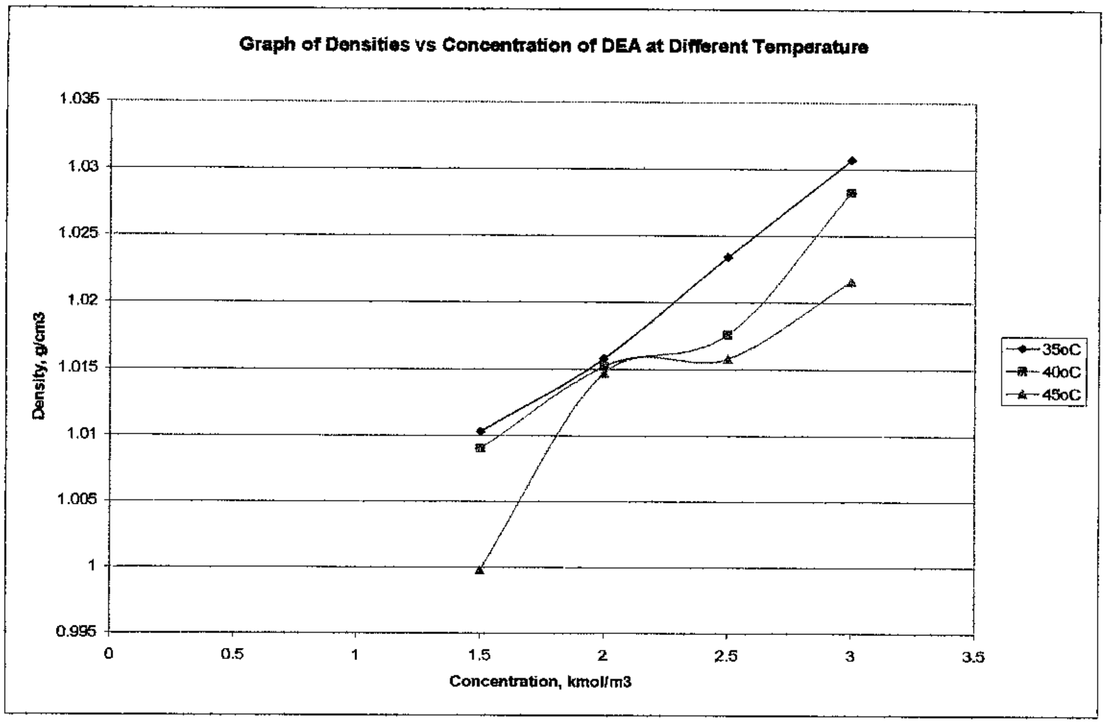

Figure 4.2: Graph of density versus DEA concentration at 35°C, 40°C and 45°C.

Apart from density measurement of individual aqueous primary and secondary alkanolamines, densities of blends of sterically hindered amine, AMP with MEA and DEA were also being measured using the same method. AMP with concentration of 1.5 kmol/m^3 was mixed with 0.1, 0.2 and 0.3kmol/m^3 of MEA and DEA respectively with ratio of 1:1 in volume. The density of aqueous MEA with AMP showed an increase when the concentration of MEA increases but decrease as temperature increase. This trend is shown in the next graph, Figure 4.3. This agreed well with the result obtained from Xiao, Li and Li (2000). For blends of AMP and DEA, as concentration of DEA increases, the density of the solution also increases and as temperature increases, the density decreases. This result is similar to those obtained by Wang and Li (2002). However, the result does not yield a refined straight line as there were fluctuations in temperature during the measurement. The result for density of AMP and DEA blends are shown in Figure 4.4.

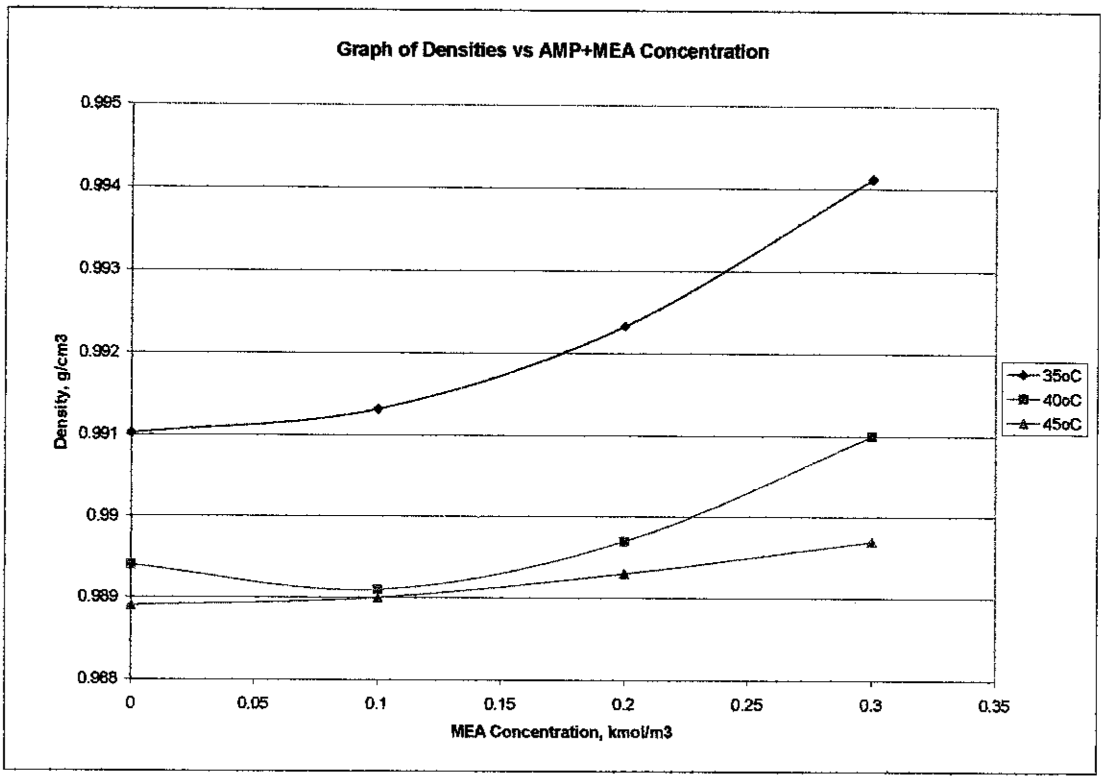

Figure 4.3: Densities of Blends of AMP+MEA as a Function of MEA Concentration

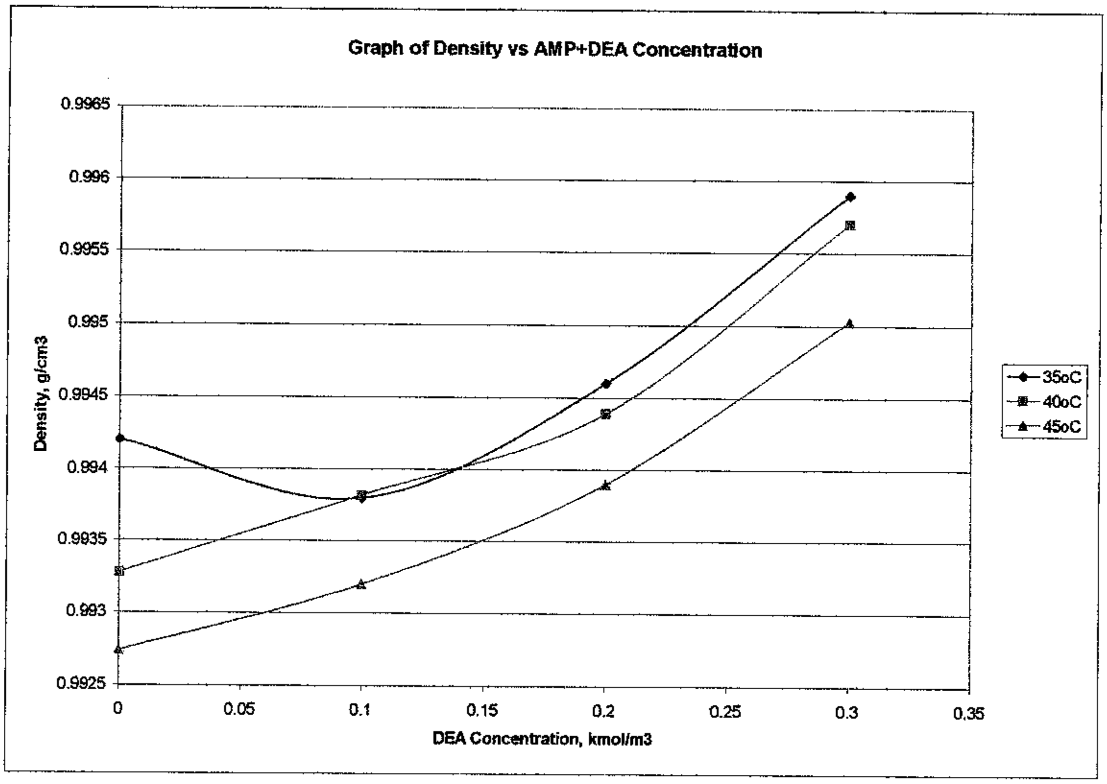

Figure 4.4: Densities of Blends of AMP + DEA as a Function of DEA Concentration

4.2 Effect of Concentration and Temperature

For the subsequent part of the experiment, the absorption of CO_2 into aqueous amine solutions was carried using the apparatus that has been described in the previous section. After being purged with N_2 to get rid of the possible other gases in the set up, CO_2 was introduced into the cell. CO_2 was regulated in the reference cell for about five minutes before releasing it into the sample cell containing amine. The operating pressure was 25 kPa while temperature was varied at 35°C, 40°C and 45°C. The justifications of using 25 kPa as the operating pressure was due to incapability of experimental set up to withstand pressure more than stated pressure. As the pressure variation was limited, the absorption of CO_2 was assumed to be completed when the pressure reaches 0 kPa.

The aqueous amines of MEA and DEA used for the absorption with concentrations of 1.5, 2.0, 2.5 and 3.5 kmol/m^3 were then being analyzed by using Refractometer to check on its Refractive Index (RI). When light travels from one medium such as glass to another phase such as liquid, it undergoes a change of velocity and if the angle of incidence is not 90°, a change of direction occur. For a given interface, angle, temperature and wavelength of light the amount of deviation or refraction will depend on the composition of the liquid. The critical angle depends on the species concentration whereas as the critical angle changes, the proportions of reflected and refracted light change. Applying this principle, it concludes that with increase of solution concentration, the refractive index becomes high in proportion to it. Refractive index of the medium is the ratio between the sine of refraction angle β and the sine of incident angle α to the normal line when the refractive index of air at the atmospheric pressure is 1 and a beam of light penetrates a certain medium χ.

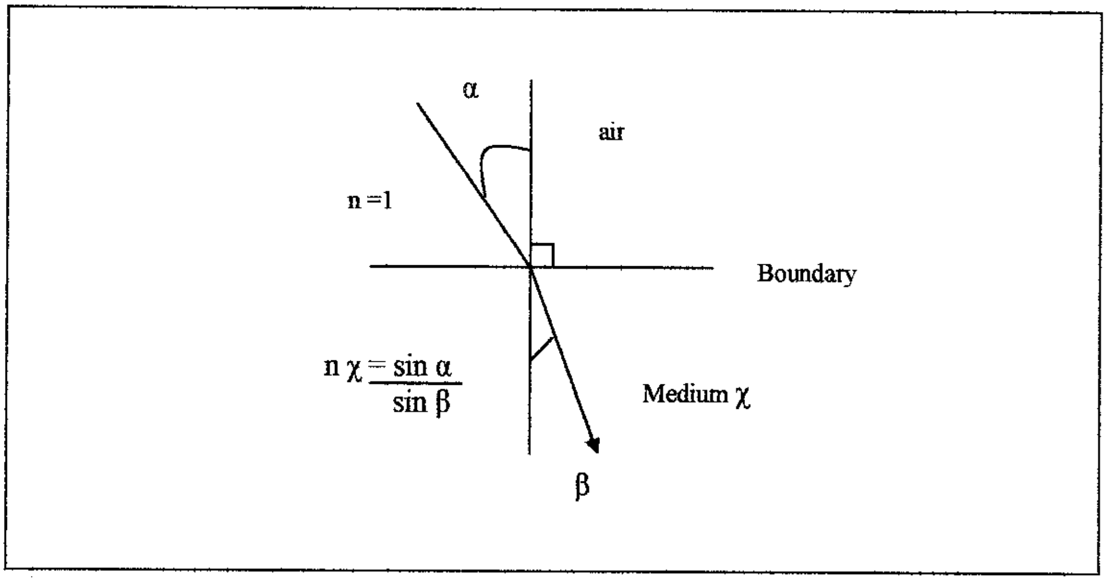

Figure 4.5: Refraction phenomenon

Standard solutions of amine ranging from 0.5 to 3.0 kmol/m^3 in concentration were prepared and the RI corresponds to the concentration were measured using refractometer in order to construct the standard curve. From the standard curve, it was clearly see that the RI has almost linear relationship with aqueous amines concentration whereas as the concentration increases, RI also increases. The results obtained were expressed as the changes in Refractive Index for aqueous amines as the measurements were made before absorption and after absorption occurred. Below are the results obtained from this part of the experiment:

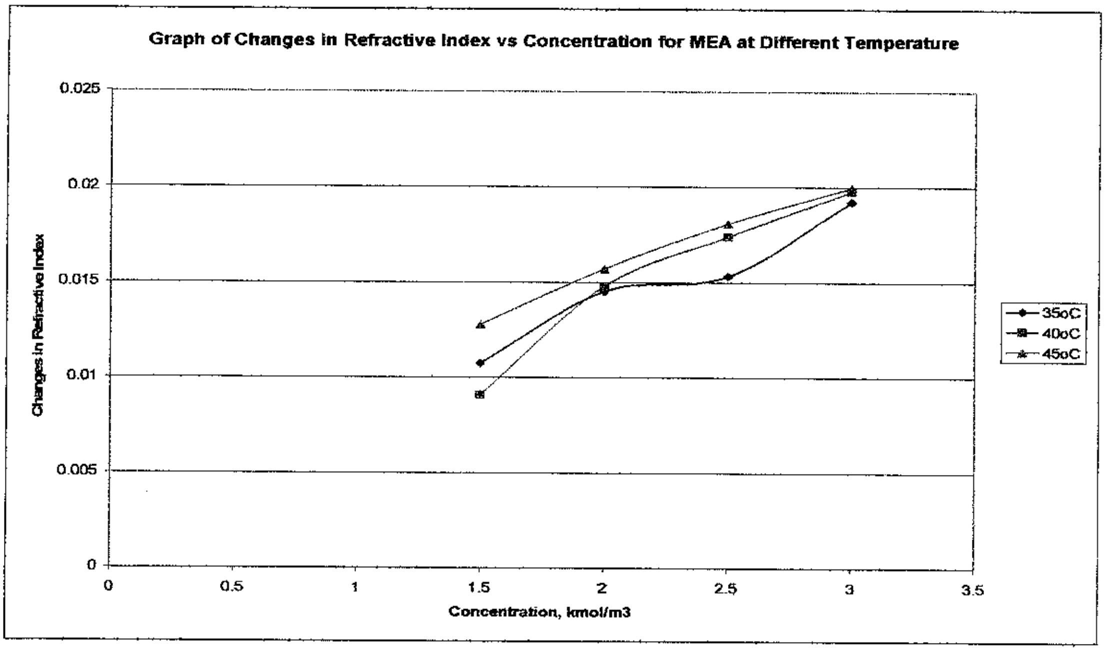

Figure 4.6: Graph of Changes in Refractive Index vs Concentration for MEA

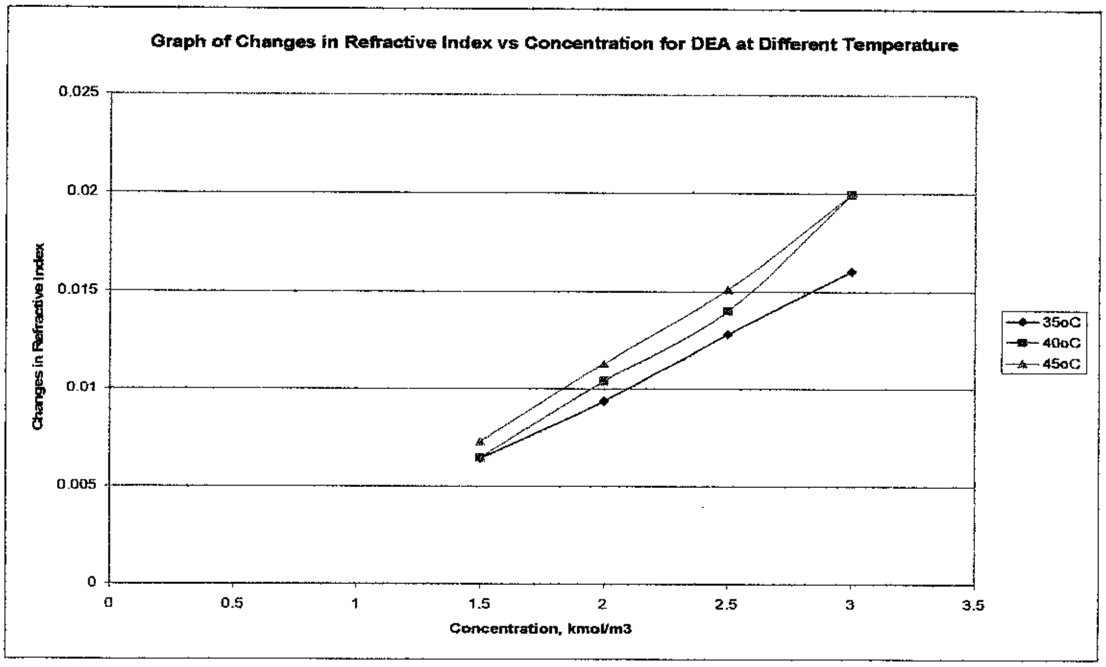

Figure 4.7: Graph of Changes in Refractive Index vs Concentration for DEA

As observed from Figure 4.6 and Figure 4.7, the change in RI increases as the concentration of solvent increases. The change in RI represents the change of amines concentrations and since the changes in RI are positive, it indicates that the concentration of total amine increases after CO_2 absorption process. Since RI gives the measurement of total amine system rather than individual components in the aqueous solutions, it cannot be concluded that amine concentration itself increases as the absorption occurred. This is because; there were chemical reactions that took place during the absorption. The reaction of CO_2 with aqueous alkanolamines yields other products such as bicarbonate ion, carbamate ion, protonated amine and free amine. The reactions involved include:

Formation of carbamate ion and protonated amine:

$$CO_2 + 2R_1R_2NH \leftrightarrow R_1R_2NCOO^- + R_1R_2NH_2^+$$

Formation of bicarbonate ions from hydration of CO_2 with water:

$$CO_2 + OH^- \leftrightarrow HCO_3^-$$

The formation of those products would be the contributing conditions towards the positive increase in the RI or in other words, increase of total solvent concentration. Based on this, it can be further explained that more bicarbonate and carbamate formed signify that more CO_2 is being absorbed by aqueous amine solutions and thus increasing the total concentration of the solution. For RI measurement, only MEA and DEA were being utilized since RI measurement gave the reading of total concentration instead of individual concentration. Thus, it is quite inappropriate to use RI in determining the concentration of amine blends as the solution contained two type of amines initially which is AMP with either MEA or DEA. For amine blends, different method was used to analyze the samples which will be described later.

From the result obtained, it was observed that as the changes in RI increases with concentration, the solubility of CO_2 also increases with concentration of aqueous amines and led to increase in changes of RI. This condition will be explained by using Le Châtelier's principle where when a chemical system in a state of equilibrium is disturbed; it reattains equilibrium by undergoing a net reaction that reduces the effect of the disturbance. In this case for concentration effect, the system which was assumed at equilibrium was disturbed by a change in concentration of aqueous amines; the system reacts in the direction that reduces the change where as the concentration of aqueous amine, the system reacts to consume some of it. From Le Châtelier's principle, it was proven that by increasing the concentration of aqueous alkanolamines, more products are formed and that is, shift equilibrium position to the right. This is the reason behind the increase of changes in RI with amines concentration as more CO_2 is absorbed into the solution to produce carbamate, bicarbonate and protonated amine.

Usually gas is more soluble at colder temperature. The temperature dependence of solubility is usually explained using Le Chatelier's principle. The principle states that when a system at equilibrium is placed under stress, the equilibrium will shift in a way that relieves that stress. In this case, the "stress" is the addition of heat. Le Chatelier's principle predicts that heating the solution mixture will shift the equilibrium in favor of dissolution, to remove the added heat. The same sort of analysis can be applied to same sort of gases such as CO_2. Considering heat as a component of the equilibrium system, a rise in temperature adds heat to the system and a drop of temperature removes heat from the system. Therefore, a temperature increase favors the endothermic or heat absorbing direction and a temperature decrease favors the exothermic or heat releasing direction. The heat absorbed or released when a gas dissolves in liquid has essentially two contributions that include:

i) **Energy is absorbed to open a pocket in the solvent**. Solvent molecules attract each other. Pulling them apart to make a cavity will require energy, and heat is absorbed in this step for most solvents. Water is a special case- *it already contains open holes* in its network of loose hydrogen bonds around room temperature. For water, very little heat is required to create pockets that can hold gas molecules.

ii) **Energy is released when a gas molecule is popped into the pocket.** Intermolecular attractions between the gas molecule and the surrounding solvent molecules lower its energy, and heat is released. The stronger the attractions are, the more heat is released. Water is capable of forming hydrogen bonds with some gases, while organic solvents often can't. A larger amount of heat is released when a gas molecule is placed in the pocket in water than in organic solvents.

There is usually net absorption of heat when gases are dissolved in organic solvents, because the pocket-making contribution is bigger. Le Chatelier's principle predicts that when heat is absorbed by the dissolution process, it will be favored at higher temperature. Solubility is expected to increase when temperature rises. Gas absorption is usually an exothermic process.

$$\text{Exothermic: Reactants} \leftrightarrow \text{Products} + \text{heat}$$

For temperature effect towards absorption, absorption is favored at low temperature compared to high temperature. However, the results obtained contradicted the theory where as the temperature increases, the CO_2 solubility in aqueous amines is found to be increased. This is caused by Refractive Index itself which varies and depends on temperature. When solution was measured by the refractometer, it read different results from measurement to measurement owing to change in the temperature of the sample. Besides, aqueous amines is quite volatile that sometimes badly affects the measurement such as the measurement temperature becomes unstable and the measurement value gradually increases because the prism and the pheripheral part of the refractometer were cooled. Apart from that, there was a slight difference in temperature between the prism and sample which led to inaccuracy of the results obtained. To overcome this, thermostatic circulating tank can be used or soaking the sample container in the thermostatic bath to even the sample and the prism in temperature as near as possible. It also can be assumed that the absorption process does not reach its equilibrium state yet.

For the next part of the experiment, the concentration of CO_2 gas at initial and concentration of gas after certain period was compared. In this section, the same experiment as had been described previously was used except for connecting the end hose to Gas Chromatograph/ Mass Spectrometer (GC/MS). The result was obtained based on reduction of CO_2 fraction after certain period of time.

$$Fraction(CO_2) = \frac{Amount\_of\_Gas\_Component}{Total\_Amount\_of\_GasMixture}$$

$$\mathrm{Re}duction\_of\_Fraction(CO_2) = \frac{\Delta Amount\_of\_the\_Gas\_COmponent}{Total\_Amount\_of\_Gas\_Mixture}$$

Larger reduction of fraction of gas components signify higher absorption rate. The calculation done was based on the peak area yielded from GC/MS since peak area most likely represents the amount of gas existing in the sample. Below are the formula used in order to calculate peak area ratio of CO_2.

$$Peak\_Area\_Ratio(CO_2) = \frac{Peak\_Area\_of\_CO_2}{Total\_Peak\_Area\_of\_the\_Gas\_Mixture}$$

$$\mathrm{Re}duction\_of\_Peak\_Area\_Ratio(CO_2) = \frac{\Delta Peak\_Area\_of\_CO_2}{Total\_Peak\_Area\_of\_the\_GasMixture}$$

Due to time constraint, this experiment only used amine blends to examine its absorption behavior as time did not permitted to study on individual amines. Blends of AMP with MEA and DEA were used for this experiment to observe their absorption rate with variation of concentration and temperature. After the setup was being purged with N_2 to flush out all possible gases that present in the apparatus, CO_2 was introduced into the cell at pressure of 25 kPa. After 10 minutes of contact time, the sample gas was analyzed by using GC/MS and the results obtained were based on the reduction peak area of CO_2. It was assumed that there was no leakage in the experimental set up during the absorption process which meant that the amount of gas entering the system remained in it until 10 minutes of contact times. The results obtained were given in Figure 4.8 and Figure 4.9 for blends of AMP with MEA and DEA respectively.

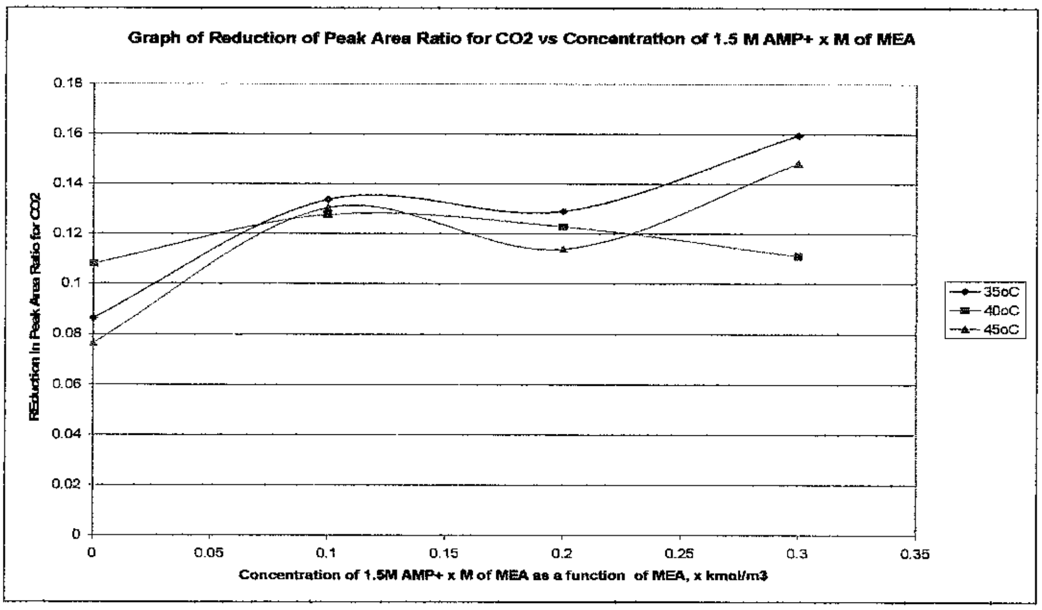

Figure 4.8: Reduction of Peak Area Ratio for AMP+MEA Blends as a Function of MEA Concentrations

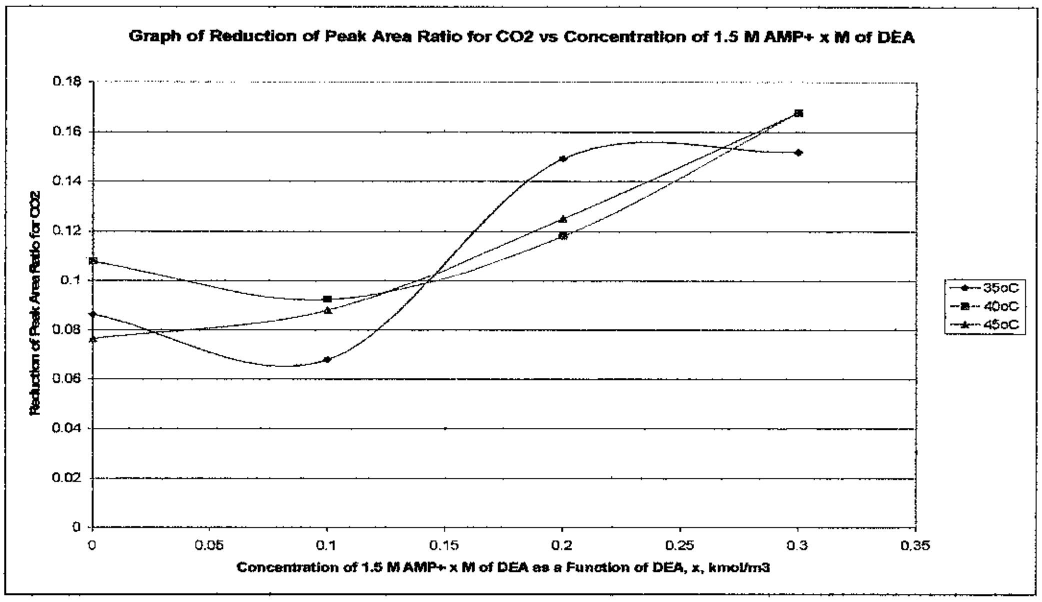

Figure 4.9: Reduction of Peak Area Ratio for AMP+DEA Blends as a Function of DEA Concentrations

From the result obtained, it can be seen roughly that for AMP+MEA and AMP+DEA blends exhibit relationship of as concentration of MEA and DEA increases, the reduction of peak area ratio also increases. These signify the increase in rate of CO_2 absorption in aqueous blends of alkanolamines. As the temperature increases, the reduction of peak area ratio decreases. These agree with the theory which is as the temperature increases, the rate of absorption decreases. As the absorption of CO_2 is an exothermic reaction, low temperature favors the solubility of CO_2 into aqueous alkanolamines solution. However, the graph line obtained for both blends are not linear. This might be due to some errors that occurred during the handling of experiment. Among errors that might be affecting the experimental result are the fluctuations of pressure which will influence the stability of temperature. As had been discussed, temperature played an important role in determining rate of absorption. Apart from that, there might be some air present in the apparatus which would decrease rate of absorption consequently. When comparing the effect of addition of small amount of MEA and DEA into AMP aqueous solutions, mostly MEA yield stronger effect compared to DEA as being a primary amine. The relationship of this statement is shown in Figure 4.10 – 4.12

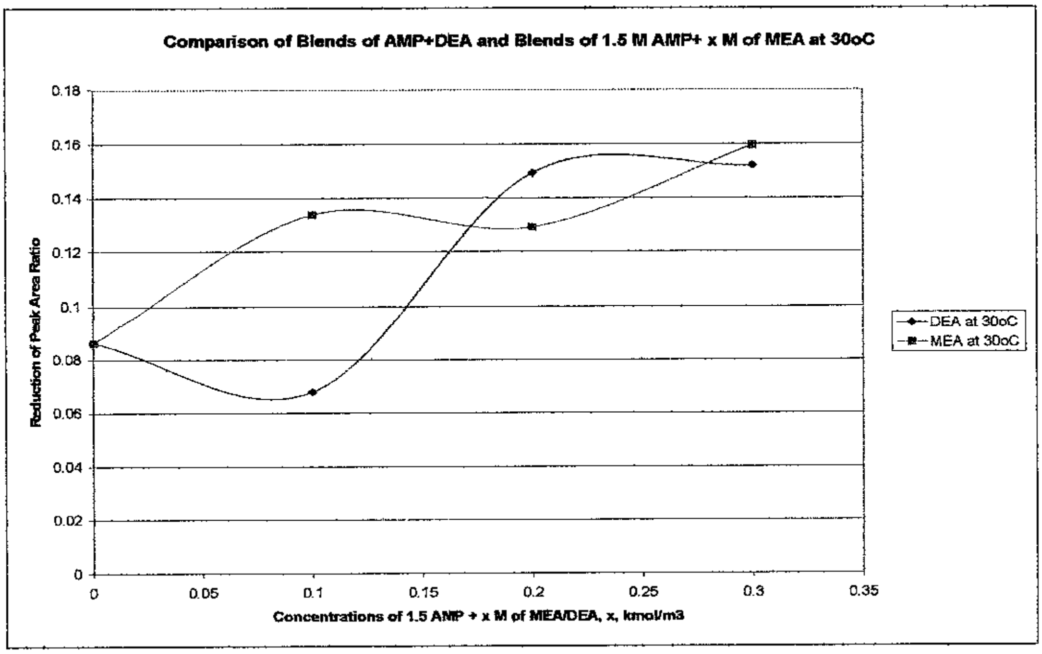

Figure 4.10: Comparison of MEA and DEA Addition into 1.5 M Aqueous AMP towards CO_2 Absorption at 30°C.

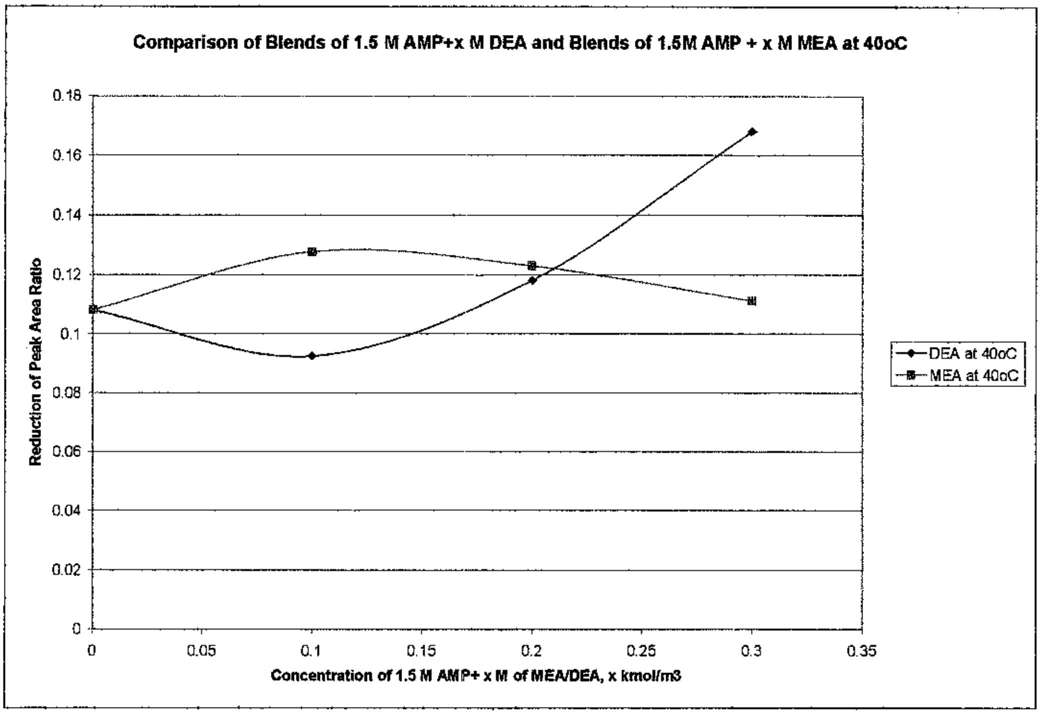

Figure 4.11: Comparison of MEA and DEA Addition into 1.5 M Aqueous AMP towards CO_2 Absorption at 40°C.

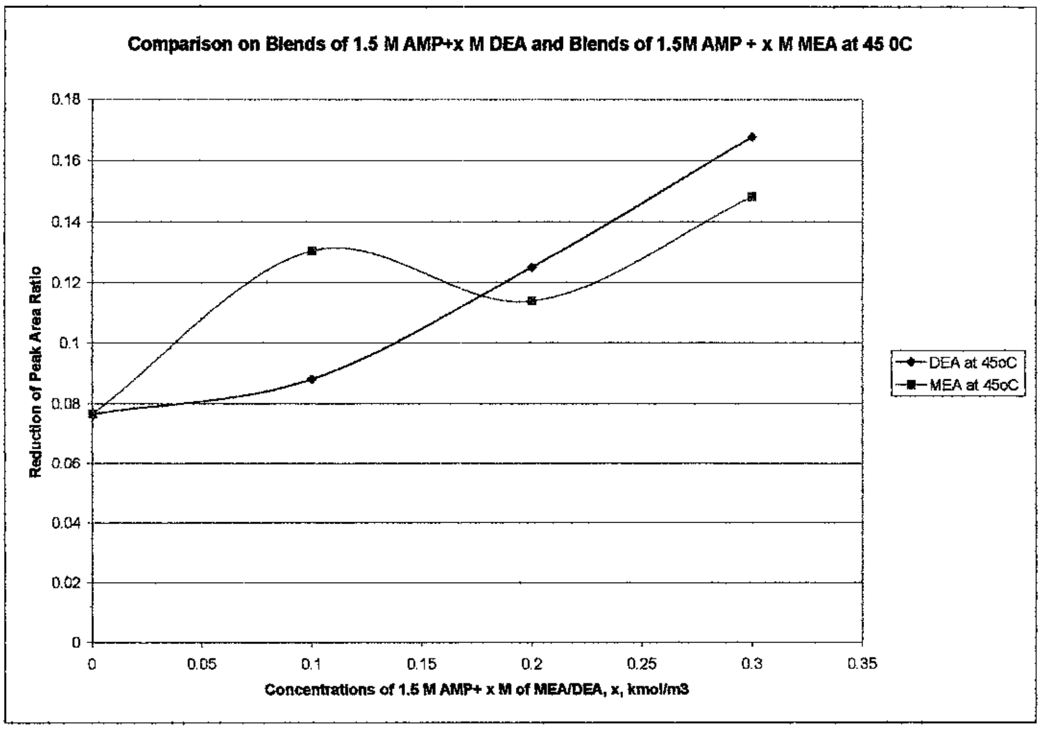

Figure 4.12: Comparison of MEA and DEA Addition into 1.5 M Aqueous AMP towards CO_2 Absorption at 45°C.

For the last part of the experiment, CO_2 loading in amine were determined by using standard titration method with 2.0 kmol/m^3 of HCl. 10 ml of sample after absorption was taken and phenolphthalein was used as the indicator. Addition of phenolphthalein into the sample yield light pink solution which indicates the base behavior of the solution. The samples were then titrated with HCl until clear solution was formed. The amount of HCl needed for the titration of each sample was recorded and the total volume of the solution after titration completed was also recorded. During the titration, gas was released from the sample when HCl was introduced and this clearly indicates that CO_2 was absorbed into the aqueous alkanolamines during the experiment. In acid base reactions that form a gaseous product, the driving force is the formation of a gas and water because both products remove ions from solution. In the CO2-amine-H2O solutions, amine reacted with CO2 to form mainly the protonated amine (AMH+), bicarbonate ion (HCO3-), and carbamate anion (AMCO2-), where the quantitative ratio of bicarbonate ion to carbamate anion strongly influenced the CO2 loading in the amine solutions.

Since bicarbonate formed in the reaction between aqueous alkanolamine and CO_2 is not too stable, when the ionic carbonate is treated with HCl, the product that formed from the reaction is CO_2. This is why gaseous product which is CO_2 was released during the titration. Figure 4.13, 4.14 and 4.14 exhibits the result obtained from this part of experiment. From the graph, the results obtained are as the concentration of solution increases, the total volume after titration decreases. While as the temperature increases, the total volume after titration also decreases. The findings actually violate the theory. This might be due to as the titration was done in open air, the atmospheric air might enter the solutions and causing inaccuracy of the result. Besides, some of the samples used are stored for quite a time before being titrated. The possibility of air entering the solution was very high and consequently affected the result yielded.

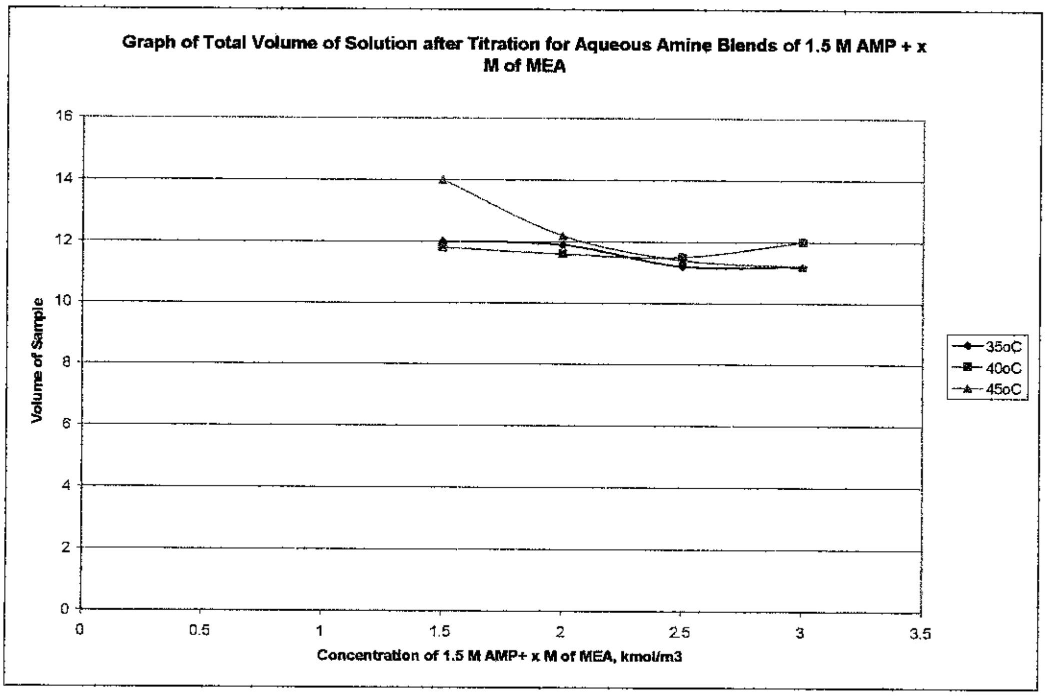

Figure 4.13: Total Volume of Sample Solution after Titration for Aqueous Blends of 1.5 AMP + x M of MEA at Different Temperature

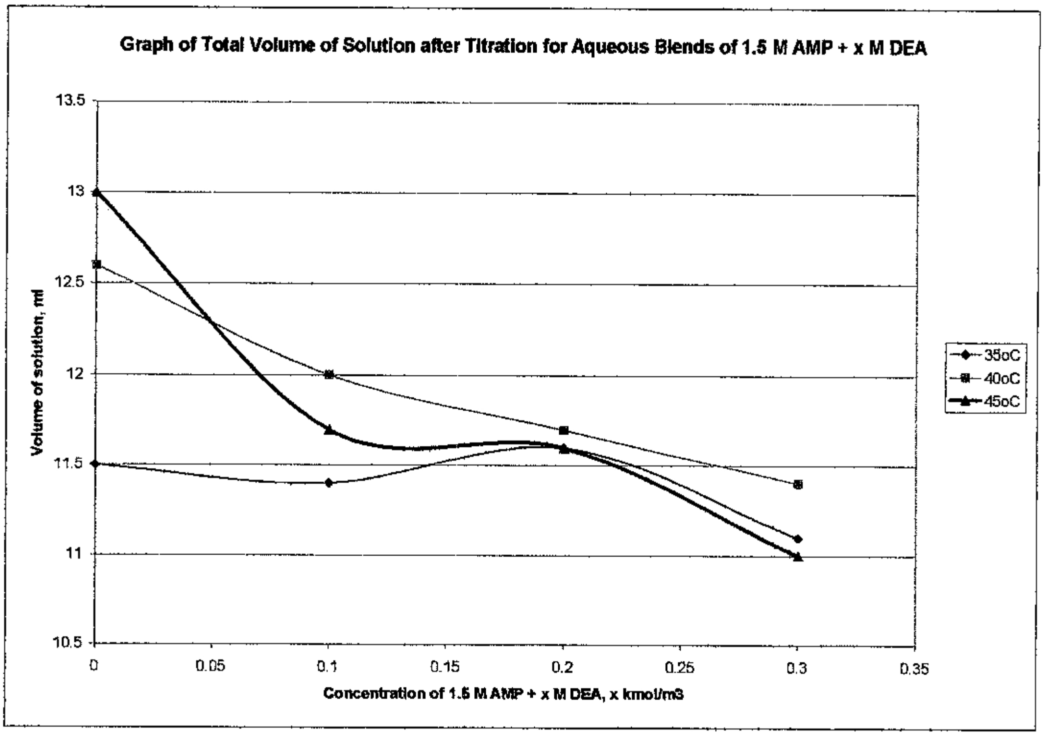

Figure 4.14: Total Volume of Sample Solution after Titration for Aqueous Blends of 1.5 AMP + x M of MEA at Different Temperature

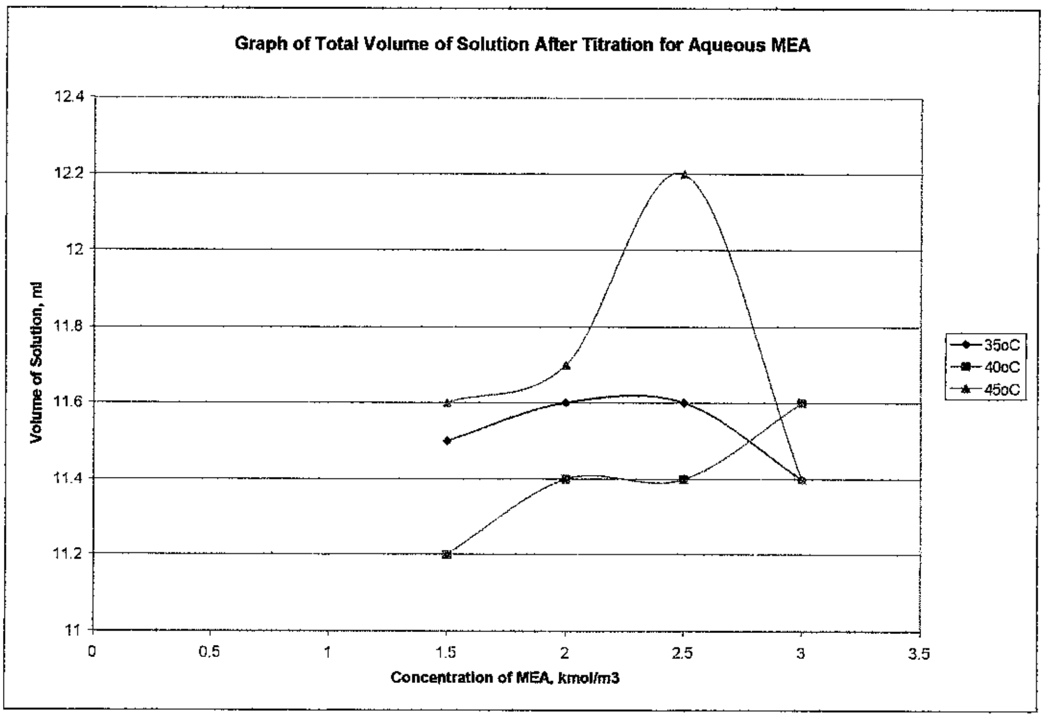

Figure 4.15: Total Volume of Sample Solution after Titration for Aqueous MEA at Different Temperature

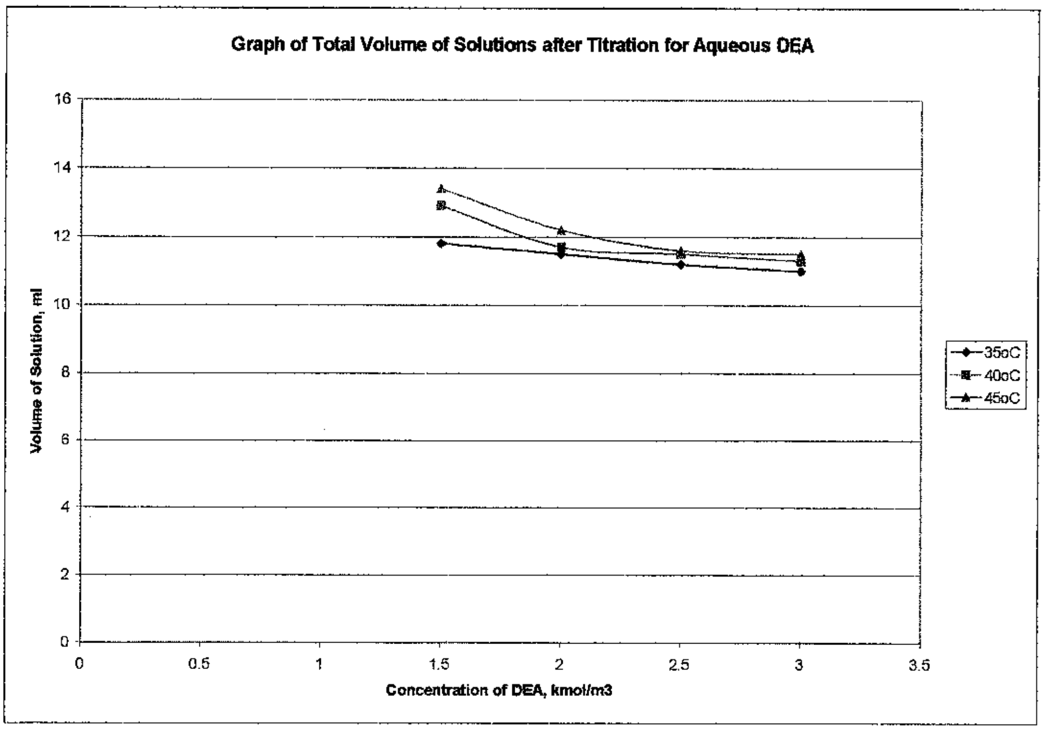

Figure 4.16: Total Volume of Sample Solution after Titration for Aqueous MEA at Different Temperature

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

Absorption of CO_2 into aqueous alkanolamines; MEA, DEA and blends of MEA and AMP and blends of AMP and DEA were investigated by using gas bubbler method at variation of temperature which is 35°C, 40°C and 45°C. Different concentration of amines is used and also mixtures of sterically hindered amine, AMP with primary and secondary amines were also being utilized. The concentrations for primary and secondary amines were varied from 1.5 kmol/m^3 to 3.0 kmol/m^3. While sterically hindered amine, AMP with concentration of 1.5 kmol/m^3 was mixed with secondary and primary amine with concentration ranging from 0.1 kmol/m^3 to 0.3 kmol/m^3 respectively. Densities of individual amines and blended amine system were also being measured. From the result that has been obtained, it can be concluded that, for density of individual amines, as concentration of amines increase, density of solution increase as well. For blended amine system, or MEA + AMP system, the density of amine blends increase as the concentration of MEA increases and decreases as the temperature increase. Same condition applies for blends of DEA + AMP. The absorption by using gas bubbler method is chosen as it can provide larger interfacial area between liquid and gas, and it is quite simple. However, continuous modification and improvement has to be made to improvise the findings of the experiment and the experiment itself. All the result obtained is compared to predicted values from literature to ensure that it obeys the theory of the absorption. From the result obtained, it can be concluded that as concentration increases, rate of absorption increases. Theoretically for temperature effect, as temperature increases, rate of absorption decreases. However, this study does not fully obey the theory due to some conditions that have been discussed previously. The addition of primary and secondary amines, MEA and DEA into sterically hindered amine, AMP results in significant enhancement of CO_2 absorption rates. Amine blends of MEA and AMP gives better CO_2 absorption rates compared to DEA and AMP blends as MEA being primary amines has stronger effect towards CO_2 absorption.

5.2 RECOMMENDATIONS

A few recommendations have been made in order to improve the experimental section done in the future. The apparatus that is used in the experiment should be the one that can sustain higher pressure. This is due to limitation of glassware as the one that is used in the experiment could not withstand high pressure. The maximum allowable pressure is only 30 kPa. It will certainly limit the pressure variation during the experiment. Besides, if more pressure introduced rather than 30 kPa, the apparatus will be popped out to release the excessive pressure which will soon affected the result.

For preparation of aqueous alkanolamines, instead of using distilled water, double distilled water that degassed by boiling should be used. It will leads to more accurate result as the amount of air in water is discharged and minimized. Besides, the preparation of aqueous alkanolamines could be done in Nitrogen Flushed Box to create an O_2 free environment as O_2 content will affect the result and thus more reliable findings will be obtained. It also better if the experiment can be carried out in this O_2 free environment, however, there is some limitations since the entry point of Nitrogen flushed box is quite small, thus it limit the size of equipment that can be used.

For further improvement of the methodology, flow meter needs to be used to measure the flow rate of gas entering the set up. Flow rate being one of the important variable that will affect the absorption rate need to be considered in this project work. Thus, more findings can be obtained by introducing gas flow rate as the operating variable. Apart from that, gas diffusivity and solubility of CO_2 should be determined to study the reaction kinetics of CO_2 in alkanolamines. However, measurement of CO_2 diffusivity and solubility could not be determined directly as there is reaction that take place between CO_2 and amines. Hence, based on previous researches and studies that have been done, N_2O analogy can be used to estimate the solubility and diffusivity of CO_2 in amine solutions. These physical properties would be needed and required to model rate of CO_2 absorption together with its reaction kinetics.

This research can be extended further if more time is allocated to finish the experiment and there are no limitations or constraints on the experimental apparatus and equipment. For this study, due to limited experimental analytical apparatus such as Gas Chromatograph/ Mass Spectrometer (GC/MS), the experiment was delayed and time given is not sufficient to completely finish the experiment. Extended research could be done on variation and combination of amines to exactly determine the best amine blend that can optimize CO_2 absorption and also determination of reaction kinetics of the absorption.

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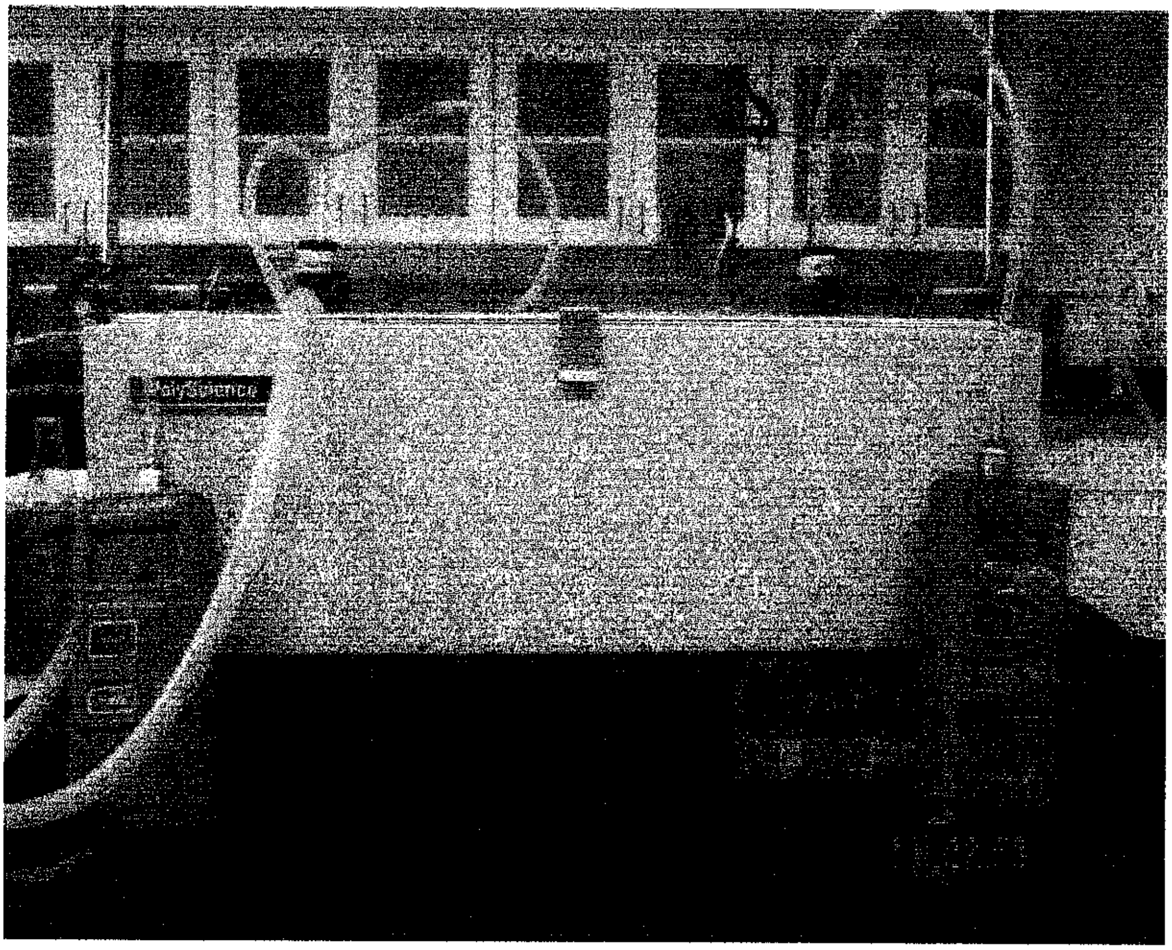
APPENDICES

Appendix A:

Equipment Set up



Appendix B:

1. Density Measurement

i) MEA

Temperature	Concentration, kmol/m^3	Density, g/cm^3
35 °C	1.5	0.9980
	2.0	0.9988
	2.5	1.0015
	3.0	1.0036
40 °C	1.5	0.9974
	2.0	0.9978
	2.5	1.0012
	3.0	1.0029
45 °C	1.5	0.9963
	2.0	0.9977
	2.5	1.0010
	3.0	1.0032

i) DEA

Temperature	Concentration, kmol/m^3	Density, g/cm^3
35 °C	1.5	1.0103
	2.0	1.0158
	2.5	1.0234
	3.0	1.0307
40 °C	1.5	1.0090
	2.0	1.0152
	2.5	1.0176
	3.0	1.0283
45 °C	1.5	0.9988
	2.0	1.0147
	2.5	1.0158
	3.0	1.0216

iii) AMP + MEA

Temperature	Concentration, kmol/m^3	Density, g/cm^3
35 °C	1.5 AMP + 0.0 MEA	0.99102
	1.5 AMP + 0.1 MEA	0.99132
	1.5 AMP + 0.2 MEA	0.99233
	1.5 AMP + 0.3 MEA	0.99412
40 °C	1.5 AMP + 0.0 MEA	0.9894
	1.5 AMP + 0.1 MEA	0.9891
	1.5 AMP + 0.2 MEA	0.9897
	1.5 AMP + 0.3 MEA	0.9910
45 °C	1.5 AMP + 0.0 MEA	0.9889
	1.5 AMP + 0.1 MEA	0.9890
	1.5 AMP + 0.2 MEA	0.9893
	1.5 AMP + 0.3 MEA	0.9897

iv) AMP + DEA

Temperature	Concentration, kmol/m^3	Density, g/cm^3
35 °C	1.5 AMP + 0.0 DEA	0.9942
	1.5 AMP + 0.1 DEA	0.9938
	1.5 AMP + 0.2 DEA	0.9948
	1.5 AMP + 0.3 DEA	0.9959
40 °C	1.5 AMP + 0.0 DEA	0.9933
	1.5 AMP + 0.1 DEA	0.9938
	1.5 AMP + 0.2 DEA	0.9944
	1.5 AMP + 0.3 DEA	0.9957
45 °C	1.5 AMP + 0.0 DEA	0.9927
	1.5 AMP + 0.1 DEA	0.9932
	1.5 AMP + 0.2 DEA	0.9939
	1.5 AMP + 0.3 DEA	0.9950

Appendix C:

Refractive Index Measurement

i) MEA

Temperature	Concentration, kmol/m^3	Changes in RI
35 °C	1.5	0.01074
	2.0	0.01452
	2.5	0.01533
	3.0	0.01924
40 °C	1.5	0.00907
	2.0	0.0148
	2.5	0.01741
	3.0	0.01972
45 °C	1.5	0.01279
	2.0	0.01572
	2.5	0.01809
	3.0	0.01998

i) DEA

Temperature	Concentration, kmol/m^3	Changes in RI
35 °C	1.5	0.00642
	2.0	0.00937
	2.5	0.01284
	3.0	0.00605
40 °C	1.5	0.00647
	2.0	0.01040
	2.5	0.01403
	3.0	0.01998
45 °C	1.5	0.00731
	2.0	0.01132
	2.5	0.1513
	3.0	0.01998

Appendix D:

GC/MS Data

i) AMP + MEA

$$\text{Peak Area Ratio of } CO_2 \text{ (initial concentration)} = \frac{149786471}{151266457} = 0.98823$$

Temperature	Concentration	Peak area for CO_2	Total Peak Area	Reduction in Peak Area Ratio
35°C	1.5 M AMP + 0.0 M MEA	155059057	162631720	0.086300
	1.5 M AMP + 0.1 M MEA	162626220	190078181	0.133711
	1.5 M AMP + 0.2 M MEA	159298976	185421329	0.129110
	1.5 M AMP + 0.3 M MEA	159299819	192211699	0.159457
40°C	1.5 M AMP + 0.0 M MEA	158636728	190428938	0.108000
	1.5 M AMP + 0.1 M MEA	161133945	187239681	0.127654
	1.5 M AMP + 0.2 M MEA	166049568	191897347	0.122926
	1.5 M AMP + 0.3 M MEA	165253767	188408979	0.111132
45°C	1.5 M AMP + 0.0 M MEA	154379180	181232911	0.076500
	1.5 M AMP + 0.1 M MEA	153729810	179221204	0.130460
	1.5 M AMP + 0.2 M MEA	216397430	247500870	0.113900
	1.5 M AMP + 0.3 M MEA	158272194	188398765	0.148140

ii) AMP + DEA

Temperature	Concentration	Peak area for CO_2	Total Peak Area	Reduction in Peak Area Ratio
35°C	1.5 M AMP + 0.0 M DEA	155059057	162631720	0.08630
	1.5 M AMP + 0.1 M DEA	157339355	170952325	0.06790
	1.5 M AMP + 0.2 M DEA	156319399	186323967	0.14926
	1.5 M AMP + 0.3 M DEA	155081670	182862419	0.15200
40°C	1.5 M AMP + 0.0 M DEA	158636728	190428938	0.10800
	1.5 M AMP + 0.1 M DEA	165581684	197306323	0.09240
	1.5 M AMP + 0.2 M DEA	162215434	191013644	0.11805
	1.5 M AMP + 0.3 M DEA	160230734	195326367	0.167907
45°C	1.5 M AMP + 0.0 M DEA	154379180	181232911	0.07650
	1.5 M AMP + 0.1 M DEA	156579767	173932596	0.08880
	1.5 M AMP + 0.2 M DEA	157762200	182770281	0.12506
	1.5 M AMP + 0.3 M DEA	15190368	184752100	0.167723

```
Data : AINA.D01  04/10/25 13:46:44
Sample           : MEA 2.5M
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time          Area     Height A/H(sec)    MK %Total Name
   1   0.752   0.725 -  1.083      58579284   13701360    4.275   S     26.59
   2   0.948   0.925 -  0.992        480497     341456    1.407   T      0.22
   3  23.743  23.725 - 23.858      22947698   19653769    1.168         10.42
   4  23.936  23.858 - 24.383     136872126   15516784    8.821   SV    62.13
   5  24.158  24.125 - 24.242       1432317     586403    2.443   T      0.65
-----------------------------------------------------------------------------
  Total                           220311922                            100.00
```

```
Data : AINA.D02  04/10/25 15:28:45
Sample           : CO2 initial
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time         Area      Height A/H(sec)    MK %Total Name
    1   1.504   1.383 -  1.675    171067718   16295434   10.498        94.30
    2   1.707   1.675 -  1.967     10346955    3423340    3.022    V    5.70
-------------------------------------------------------------------------------
  Total                           181414673                            100.00
```

```
Data : AINA.D03  04/10/25 16:15:43
Sample           : MEA-3.0m(45c)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time        Area      Height A/H(sec)    MK %Total Name
   1   1.041   1.017 -  1.158     24371726    19701886    1.237          14.16
   2   1.238   1.158 -  1.692    146045610    16301928    8.959   SV     84.84
   3   1.456   1.425 -  1.533      1716009      737868    2.326   T       1.00
--------------------------------------------------------------------------------
  Total                          172133346                              100.00
```

```
Data : AINA.D06  04/10/26 15:32:08
Sample           : 2.5DEA(40C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time        Area      Height A/H(sec)    MK %Total Name
   1   1.003   0.992 -  1.117    29079076    26062947    1.116         15.21
   2   1.207   1.117 -  1.383   154771818    17389636    8.900     V   80.97
   3   1.415   1.383 -  1.667     7305538     1764201    4.141     V    3.82
--------------------------------------------------------------------------------
  Total                         191156431                              100.00
```

```
Data : AINA.D08  04/10/26 16:02:58
Sample           : 1.5 AMPO(40C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time         Area     Height A/H(sec)    MK %Total Name
   1   0.940   0.925 -  1.050     23363194   23148058    1.009          12.27
   2   1.147   1.050 -  1.317    158636728   17105544    9.274     V    83.30
   3   1.353   1.317 -  1.608      8429016    2133578    3.951     V     4.43
--------------------------------------------------------------------------------
  Total                          190428938                             100.00
```

```
Data : AINA.D09  04/10/26 16:37:53
Sample           : 1.5 AMP+0.2MEA(40C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time         Area      Height A/H(sec)     MK %Total Name
   1   1.138   1.117 -  1.242     17171519    24321744    0.706            8.95
   2   1.345   1.242 -  1.517    166049568    17237432    9.633     V     86.53
   3   1.550   1.517 -  1.808      8676260     2132410    4.069     V      4.52
---------------------------------------------------------------------------------
  Total                          191897347                               100.00
```

```
Data : AINA.D10  04/10/26 17:03:12
Sample           : 1.5 AMP+0.3MEA(40C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time         Area     Height A/H(sec)    MK %Total Name
   1   1.071   1.058 -  1.175     14672993   23436729    0.626           7.79
   2   1.278   1.175 -  1.450    165253767   17223640    9.595     V    87.71
   3   1.482   1.450 -  1.733      8483018    2170588    3.908     V     4.50
-------------------------------------------------------------------------------
  Total                          188409779                             100.00
```

```
Data : AINA.D11  04/10/27 09:57:16
Sample           : INITIAL CO2
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time          Area       Height A/H(sec)     MK %Total Name
   1   1.163   1.150 -  1.250       9696871     15225867    0.637           5.71
   2   1.365   1.250 -  1.542     152757444     16696762    9.149      V   89.95
   3   1.578   1.542 -  1.808       7377073      1698532    4.343      V    4.34
----------------------------------------------------------------------------------
   Total                          169831388                               100.00
```

```
Data : AINA.D12  04/10/27 10:19:48
Sample           : INITIALCO2(45C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time         Area     Height A/H(sec)   MK %Total Name
   1   1.950   1.858 -  2.383    149486471 ✓ 16678761    8.963   S    98.82
   2   2.161   2.125 -  2.225      1779986     797142    2.233   T     1.18
-------------------------------------------------------------------------------
  Total                          151266457                           100.00
```

```
Data : AINA.D13  04/10/27 11:25:44
Sample           : AMP1.5M(45C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time          Area      Height A/H(sec)    MK %Total Name
   1   1.078   1.058 -  1.192      25244169    23986852    1.052          13.93
   2   1.278   1.192 -  1.742     154379180    17004665    9.079   SV     85.18
   3   1.489   1.458 -  1.550       1609562      724927    2.220   T       0.89
----------------------------------------------------------------------------------
  Total                           181232911                              100.00
```

```
Data : AINA.D14  04/10/27 12:01:07
Sample           : AMP1.5M+0.2MEA(45C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time        Area      Height A/H(sec)    MK %Total Name
   1   1.204   1.183 -  1.350     24750087    14779655    1.675       100.00
-------------------------------------------------------------------------------
  Total                            24750087                          100.00
```

```
Data : AINA.D15  04/10/27 12:33:04
Sample          : AMP1.5M+0.1MEA(45C)
ID              : amine
Operator        : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time          Area     Height A/H(sec)    MK %Total Name
   1   1.029   1.017 -  1.142      17732137   24204358    0.733           9.89
   2   1.235   1.142 -  1.408     153729841   17047058    9.018     V    85.78
   3   1.443   1.408 -  1.700       7759226    1785977    4.345     V     4.33
--------------------------------------------------------------------------------
  Total                           179221204                             100.00
```

```
Data : AINA.D16  04/10/27 13:21:15
Sample           : AMP1.5M+0.1DEA(45C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time        Area      Height A/H(sec)   MK %Total Name
   1   0.930   0.917 -  1.017      9513357   15065502    0.631         5.47
   2   1.137   1.017 -  1.308    156579767   17203782    9.101    V   90.02
   3   1.345   1.308 -  1.600      7839462    1762773    4.447    V    4.51
-------------------------------------------------------------------------------
  Total                          173932586                            100.00
```

```
Data : AINA.D17  04/10/27 14:40:13
Sample           : AMP1.5M+0.2DEA(45C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time        Area     Height A/H(sec)    MK %Total Name
   1   1.113   1.100 -  1.225    16726153   23777680    0.703            9.15
   2   1.320   1.225 -  1.492   157762200   17321415    9.108    V      86.32
   3   1.527   1.492 -  1.842     8281929    1789907    4.627    V       4.53
----------------------------------------------------------------------------------
  Total                         182770281                               100.00
```

```
Data : AINA.D18  04/10/27 15:15:21
Sample          : AMP1.5M+0.3DEA(45C)
ID              : amine
Operator        : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time          Area     Height A/H(sec)    MK %Total Name
   1   1.082   1.058 -  1.200      26270477   24303837    1.081          14.22
   2   1.289   1.200 -  1.467     151590368   17440075    8.692     V    82.05
   3   1.494   1.467 -  1.733       6891256    1713974    4.021     V     3.73
------------------------------------------------------------------------------
  Total                           184752100                           100.00
```

```
Data : AINA.D19  04/10/27 15:45:29
Sample           : AMP1.5M+0.3MEA(45C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time         Area     Height A/H(sec)    MK %Total Name
   1   0.954   0.942 -  1.067     22245942   25731765    0.865         11.81
   2   1.162   1.067 -  1.333    158272194   17635217    8.975    V    84.01
   3   1.367   1.333 -  1.625      7880630    1798393    4.382    V     4.18
--------------------------------------------------------------------------------
  Total                          188398765                             100.00
```

```
Data : AINA.D20  04/10/27 16:41:45
Sample           : 3.0DEA(45C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time          Area     Height A/H(sec)    MK %Total Name
    1  1.003   0.983 -  1.117      23639453   23642105    1.000         12.77
    2  1.209   1.117 -  1.642     159784327   17651191    9.052    SV   86.32
    3  1.414   1.383 -  1.475       1685804     760633    2.216    T     0.91
-----------------------------------------------------------------------------
  Total                           185109583                            100.00
```

```
Data : AINA.D21  04/10/27 17:09:57
Sample           : 1.5AMP+0.1MEA(40C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time          Area      Height A/H(sec)    MK %Total Name
   1   0.955   0.942 -  1.067      18333575    24228172    0.757            9.79
   2   1.164   1.067 -  1.333     161133945    17763303    9.071    V     86.06
   3   1.368   1.333 -  1.608       7772162     1825637    4.257    V      4.15
--------------------------------------------------------------------------------
  Total                           187239681                             100.00
```

```
Data : AINA.D22  04/10/28 12:08:38
Sample           : 1.5AMP+0.1DEA(35C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time          Area      Height A/H(sec)    MK %Total Name
   1   1.046   1.033 -  1.142      11858660    19287451    0.615           6.94
   2   1.249   1.142 -  1.708     157339355    17117363    9.192   SV     92.04
   3   1.459   1.425 -  1.533       1754309      771906    2.273   T       1.03
------------------------------------------------------------------------------
  Total                           170952325                              100.00
```

```
Data : AINA.D23  04/10/28 12:36:56
Sample           : 1.5AMP+0.2DEA(35C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time          Area     Height A/H(sec)    MK %Total Name
   1    1.039   1.025 -  1.150      22613516   24705048    0.915         12.14
   2    1.246   1.150 -  1.417     156319399   17325374    9.023     V   83.90
   3    1.451   1.417 -  1.692       7391053    1707082    4.330     V    3.97
-------------------------------------------------------------------------------
  Total                            186323967                            100.00
```

```
Data : AINA.D24  04/10/28 13:01:07
Sample           : 1.5AMP+0.3DEA(35C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time          Area     Height A/H(sec)    MK %Total Name
   1  0.928   0.917 -  1.042      20370099   23143349    0.880          11.14
   2  1.135   1.042 -  1.308     155081670   17513301    8.855      V   84.81
   3  1.339   1.308 -  1.583       7410651    1789919    4.140      V    4.05
--------------------------------------------------------------------------------
  Total                          182862419                            100.00
```

```
Data : AINA.D25  04/10/28 13:26:40
Sample           : 1.5AMP(35C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time          Area     Height A/H(sec)    MK %Total Name
   1   1.226   1.125 -  1.400     155059057   17623541    8.798         95.34
   2   1.431   1.400 -  1.692       7572663    1770854    4.276    V     4.66
-----------------------------------------------------------------------------------
  Total                           162631720                             100.00
```

```
Data : AINA.D26  04/10/28 13:53:05
Sample           : 1.5AMP+0.3MEA(35C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time          Area      Height A/H(sec)    MK %Total Name
   1   0.945   0.933 -  1.058      24456189    26013223    0.940          12.72
   2   1.154   1.058 -  1.325     159299819    17873567    8.913     V    82.88
   3   1.358   1.325 -  1.708       8455692     1820633    4.644     V     4.40
----------------------------------------------------------------------------------
  Total                           192211699                              100.00
```

```
Data : AINA.D27  04/10/28 14:20:06
Sample           : 1.5AMP+0.2MEA(35C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time         Area       Height A/H(sec)   MK %Total Name
   1   0.920   0.908 -  1.033     18179481     23033068    0.789          9.80
   2   1.129   1.033 -  1.300    159298976     17991150    8.854   V     85.91
   3   1.332   1.300 -  1.592      7942872      1878418    4.228   V      4.28
--------------------------------------------------------------------------------
  Total                          185421329                              100.00
```

```
Data : AINA.D28  04/10/28 14:52:27
Sample           : 1.5AMP+0.1MEA(35C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time      Area    Height A/H(sec)    MK %Total Name
   1   0.951   0.925 -  1.067   25792052  21860216   1.180          13.57
   2   1.159   1.067 -  1.625  162426220  18158818   8.945    SV    85.45
   3   1.362   1.325 -  1.433    1860910    830558   2.241    T      0.98
--------------------------------------------------------------------------------
   Total                       190079181                          100.00
```

```
Data : AINA.D29  04/10/28 15:21:28
Sample           : 1.5AMP+0.1DEA(40C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time         Area      Height A/H(sec)   MK %Total Name
   1   0.961   0.942 -  1.075     29701769    26930449    1.103          15.05
   2   1.170   1.075 -  1.617    165581684    18354554    9.021   SV    83.92
   3   1.373   1.342 -  1.433      2022870      919484    2.200   T      1.03
---------------------------------------------------------------------------
  Total                          197306323                              100.00
```

```
a : AINA.D30  04/10/28 15:57:49
ple           : 1.5AMP+0.2DEA(40C)
              : amine
rator         : Mohd FAB
hod File Name : AINA.MET

* Peak Report ****
NO R.Time  I.Time - F.Time        Area     Height A/H(sec)    MK %Total Name
1   0.961   0.950 -  1.075    22970561   25203991    0.911        12.03
2   1.169   1.075 -  1.625   166215434   18382349    9.042   SV   87.02
3   1.373   1.342 -  1.442     1827649     837181    2.183   T     0.96
-----------------------------------------------------------------------
otal                         191013644                           100.00
```

```
Data : AINA.D31  04/10/28 16:25:27
Sample           : 1.5AMP+0.3DEA(40C)
ID               : amine
Operator         : Mohd FAB
Method File Name : AINA.MET

**** Peak Report ****
 PKNO R.Time  I.Time - F.Time        Area     Height A/H(sec)   MK %Total Name
   1  0.449   0.433 -  0.558     27764741   24641642    1.127          7.05
   2  0.659   0.558 -  0.825    160230734   18571039    8.628    V   40.70
   3  0.862   0.825 -  0.933      7330892    1945519    3.768    V    1.86
   4  0.947   0.933 -  1.075     34820565   24700155    1.410    V    8.84
   5  1.158   1.075 -  1.325    154751492   19060178    8.119    V   39.30
   6  1.359   1.325 -  1.642      8821741    1988518    4.436    V    2.24
-------------------------------------------------------------------------------
  Total                         393720163                          100.00
```

195326367

0.822323