# Parametric Study for Carbon Dioxide Absorption from Natural Gas Using New Solvent

by

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Dissertation submitted in partial fulfillment of the requirement for the Bachelor of Engineering (Hons) (Chemical Engineering)

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## **CERTIFICATION OF APPROVAL**

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A project dissertation submitted to the Chemical Engineering Programme Universiti Teknologi PETRONAS in partial fulfillment of the requirement for the BACHELOR OF ENGINEERING (Hons) (CHEMICAL ENGINEERING)

Approved by,

(Dr Lau Kok Keong)

UNIVERSITI TEKNOLOGI PETRONAS TRONOH, PERAK January 2010

## **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 except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

ONG YUIN YEE

## ABSTRACT

Natural gas (NG) is produced by the anaerobic decomposition of living matter, it is comes out of the ground, is very variable in composition, depending on the location. The composition of natural gas can vary widely, typical composition of natural gas are methane (CH<sub>4</sub>), ethane ( $C_2H_6$ ), propane ( $C_3H_8$ ), butane ( $C_4H_{10}$ ), carbon dioxide (CO<sub>2</sub>), oxygen (O<sub>2</sub>), nitrogen (N<sub>2</sub>), hydrogen sulphide (H<sub>2</sub>S) and rare gases. The CO<sub>2</sub> content in NG is getting higher and higher. In order to achieve natural gas sweetening, absorption is one of the unit operations to reduce concentration of CO<sub>2</sub>. Nowadays, there are several solvent are using to remove CO<sub>2</sub> and H<sub>2</sub>S, such as alkanolamines, potassium carbonate and Sodium hydroxide. Alkanolamines include primary amines, secondary amines and tertiary amines. Primary amines consist of monoethanol amine (MEA) and diglycolamine (DGA), where secondary amines consist of diethanolamine (DEA) and di-isopropylamine (DIPA) and tertiary amines consist of methyl-diethanolamine (MDEA) and triethanolamine (TEA). Experimental work is one of the methods to analyze the solubility of solvent absorb high content of  $CO_2$  from natural gas. There are few type of experimental setup, which are Laboratory-Scale Absorption Unit, Pilot-Scale Absorption Unit, Industrial-Scale Absorption Unit and Atmospheric pressure system. To achieve the accuracy, calibration of each absorption unit must be set before start the experiment. This paper reports the solubility data of CO<sub>2</sub> in the novel type of solvent, which is Stonvent and DEA in the various conditions of temperature, concentration, flowrate and CO<sub>2</sub> partial pressure below atmospheric pressure.

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## ABBREVIATIONS AND NOMENCLATURES

AOAC	Association of Official Analytical Chemist
$a_v$	effective interfacial area per unit volume of packing
DEA	diethanolamine
DGA	diglycolamine
DIPA	di-isopropylamine
<i>G</i> <sub>1</sub>	inert gas molar flow rate
Н	Henry's law coefficient
Ι	ratio of the liquid mass transfer coefficient for absorption with and
	without chemical reaction
k <sub>G</sub>	gas-side mass transfer coefficient
k <sub>L</sub>	liquid-side mass transfer coefficient
$k_L^O$	liquid-side mass transfer coefficient without chemical reactions
K <sub>G</sub>	overall mass transfer coefficient
MDEA	methyldiethanolamine
MEA	monoethanolamine
$N_A$	mass flux of component A
NG	natural gas
Р	total pressure
TEA	triethanolamine
TIPA	triisopropanolamine
$y_A$	mole fraction of component A in the gas bulk
y <sub>A</sub> ,₁	mole fraction of component A on the gas-side of the gas-liquid interface
$y_A^*$	equilibrium mole fraction of component A in gas phase
Z	column height

# CHAPTER 1 INTRODUCTION

#### 1.1 Background Study

Natural gas (NG) is a fossil fuel like oil and coal. Fossil fuels are, essentially, the remains of plants and animals and microorganisms that lived millions and millions of years ago.

There are many different theories as to the origins of fossil fuels. The most widely accepted theory says that fossil fuels are formed when organic matter (such as the remains of a plant or animal) is compressed under the earth, at very high pressure for a very long time. This is referred to as thermogenic methane. Similar to the formation of oil, thermogenic methane is formed from organic particles that are covered in mud and other sediment. Over time, more and more sediment and mud and other debris are piled on top of the organic matter. This sediment and debris puts a great deal of pressure on the organic matter, which compresses it. This compression, combined with high temperatures found deep underneath the earth, break down the carbon bonds in the organic matter. The deeper under the earth's crust, the higher the temperature. At low temperatures (shallower deposits), more oil is produced relative to natural gas. At higher temperatures, however, more natural gas is created, as opposed to oil. That is why natural gas is usually associated with oil in deposits that are 1 to 2 miles below the earth's crust. Deeper deposits, very far underground, usually contain primarily natural gas, and in many cases, pure methane [1].

Natural gas might be considered as a very uninteresting gas and it is colorless, shapeless, and odorless in its pure form [1]. Table 1.1 shows the composition of natural gas.

Component	Percentage
Methane	85
Ethane	8
Butane	1
Propane	0.5
Heavier HCs	0.1
Nitrogen	1
Carbon dioxide	2
Hydrogen	0.1
Oxygen	0.1
Hydrogen sulfide	0.5
Water vapour	1.2
Other gases	0.5

**Table 1.1**: Typical Composition of Natural Gas [2]

Carbon dioxide  $(CO_2)$  absorption is an essential step in many industrial processing operations such as coal gasification, natural gas processing, oil refining, and hydrogen manufacture [4].

Absorption, also known as gas absorption, gas washing and gas scrubbing is a unit operation used in the chemical industry to separate gases by washing or scrubbing a gas mixture with a suitable liquid. Absorption is used to separate gas mixtures; remove impurities, contaminants, pollutants, or catalyst poisons from a gas; or recover valuable chemicals. Thus, the species of interest in the gas mixture may be all components, only the component(s) not transferred, or only the component(s) transferred [3].

The inverse operation, called stripping or desorption, is employed when it is desired to transfer volatile components from a liquid mixture to gas. Absorbers are frequently coupled with the strippers to permit regeneration (or recovery) and recycling of the absorbent. Because stripping is not perfect, absorbent recycled to the absorber contains species present in the vapor entering the absorber [3].

#### **1.2 Problem Statement**

- In many industries, carbon dioxide content from Natural Gas must be removed because it reduces the energy content and form H<sub>2</sub>CO<sub>3</sub> when react with water, which is corrosive to the pipeline and equipment as well.
- Alkanolamine solution is inadequate because it has absorption limitations. Carbon dioxide in alkanolamine need to be removed to the environment in regeneration step and this will cause Green House effect and Global Warming.

## 1.3 Objectives

Carry out experiment to evaluate the performance of a novel solvent in terms of carbon dioxide solubility with difference parameters.

## 1.3 Scope of Study

The project will be utilizing the fundamental knowledge in Solubility and Absorption System. The scopes of study include the following:

- Study mass transfer from gas-side to liquid side.
- Study on the different types of experimental setup.
- Investigate the CO<sub>2</sub> solubility of novel solvent with difference parameters.

## **CHAPTER 2**

## LITERATURE REVIEW & THEORY

#### 2.1 Absorption Theory



Figure 2.1: Process Flow of a typical flue gas decarbonization system [4]

Figure 2.1 show a process flow of a typical flue gas decarbonization system using amine as a solvent. The upstream absorption stage cools the  $CO_2$  stream and removes particulates from it. Next, the cooled and cleaned stream enters the absorption tower, where it make contact with the alkanolamine solvent in countercurrent flow. The gas to be absorbed enters the absorber at its bottom, flow up, and leaves at the top. The solvent enters the top of the absorber, flow down, and emerges at the bottom.  $CO_2$  is chemically bound to the solvent by the exothermic reaction of the gas with the amine in the solvent. The liquid amine  $CO_2$ -rich solvent then leaves the bottom of the absorber and passes into the stripping tower via a cross heat exchanger. In the  $CO_2$  stripper, the mixture is heated with stream to liberate the  $CO_2$  from the solvent as the as the acid gas. This step is carried out at lower pressure than the previous absorption step, to enhance desorption of  $CO_2$  from the liquid. The  $CO_2$  is now ready for the further steps of compression, transport from the power plant to a storage site, and sequestration. The hot lean amine solution then flows through the cross heat exchanger, where it is contacted with the rich

amine solution from absorber. The lean amine solution from the cross heat exchanger is then returned to the top of the absorption tower [4].

## 2.2 Equipment

Absorption and stripping are conducted in trayed towers, packed columns, spray towers, bubble columns, and centrifugal contactors [3].

A trayed tower also know as plate column is a vertical, cylindrical pressure vessel in which vapor and liquid, which flow countercurrently, are contacted on a series of metal trays or plates. Liquid flows across each tray, over an outlet weir, and into downcomer, which takes the liquid by gravity to the tray below. Gas flows upward through openings in each tray, bubbling through the liquid on the tray. Figure 2.2 shows the five possible vapor-liquid flow regimes for a contacting tray. The most common and favored regime is the *froth regime*, in which the liquid phase is continuous and the gas passes through in the form of jets or a series of bubbles. The *spray regime*, in which the gas phase is continuous, occur for low weir heights (low liquid depth) at high gas rates. For low gas rates, the bubble regime can occur, in which the liquid is fairly quiescent and bubbles rise in swarms. At high liquid rates, small gas bubbles may be undesirably emulsified. If bubble coalescence is hindered, an undesirable foam forms. Ideally, the liquid carries no vapor bubbles (occlusion) to the tray below, the vapor carries no liquid droplets (entrainment) to the tray above, and there is no sweeping of liquid through the openings of the tray. With good contacting, equilibrium between the exiting vapor and liquid phases is approached on each tray [3].



Figure 2.2: Possible vapor-liquid flow regimes for a contacting tray: (a) spray; (b) froth; (c) emulsion; (d) bubble; (e) cellular foam [3]

A packed column is a vertical cylindrical pressure vessel containing one or more sections of a packing material over whose surface the liquid flows downward by gravity, as a film or as droplets between packing elements. Vapor flows upward through the wetted packing, contacting the liquid. The sections of packing are contained between a lower gas-injection support plate, which holds the packing, and an upper grid or mesh hold-down plate, which prevents packing moving. A liquid distributor, placed above the hold-down plate, ensures uniform distribution of liquid as it enters the packed section. If the depth of packing is more than about 20ft, liquid channeling may occur, causing the liquid to flow down the column mainly near the wall, and gas to flow mainly up the center of the column, thus greatly reducing the extent of vapor-liquid contact. In that case, a liquid redistributor should be installed [3].



Figure 2.3: Random packing materials [3]

Figure 2.3 show various dumped-type packing elements and construction materials are commonly utilized, which are intended to create large internal surface but a small pressure drop in order to facilitate mass transfer between the two streams. Figure 2.4 show the structured or arranged packing may be made of corrugated metal or plastic sheets providing a large number of regularly arranged channels but range of other geometry exists. In plate towers, liquid flow from plate to plate in cascade trend and gases bubble through the flowing liquid at each plate through a mass of dispersers (e.g. hole in sieve tray, slits in bubble-cap tray) or through a cascade of liquid as in a shower deck tray [3].



Figure 2.4: Structured packing materials [3].

## 2.3 Solvent

In term of solvent, there are few type of solvent are selected to remove  $CO_2$  through absorption. There are three type of alkanolamine, which are primary amine, secondary amine and tertiary amine. It is important in gas absorption industries to reduce various costs such as supplying new amines, replacing the lost amines due to evaporation or chemical degradation, repairing the corrosion damages, operation problems and also, regeneration of used alkanolamine solutions. The primary amines (RNH<sub>2</sub>) include monoethanolamine (MEA) and diglycolamine (DGA). MEA, one of the most frequently used solvents for CO<sub>2</sub> capture, has been the traditional solvent of choice for CO<sub>2</sub> absorption and H<sub>2</sub>S removal in general and is effective at low pressure. It is the cheapest technical and the most capture reaction heat. MEA has the lowest boiling point, so there may be solvent carryover in the CO<sub>2</sub> removal and regeneration steps. Depending on the conditions, MEA can remove H<sub>2</sub>S to less than 4 ppmv while removing CO<sub>2</sub> to less than 100ppmv. MEA systems generally require a reclaimer to remove degraded product from circulation. Typical solution strength ranges from 10 to 20 weight % with a maximum rich loading of 0.35 mole acid gas/mole MEA. DGA also removes CO<sub>2</sub>, H<sub>2</sub>S, COS and mercaptans. Typical solution strengths are 50-60 weight %, which result in lower circulation rates and less energy required for stripping as compared with MEA. DGA also requires reclaiming to remove the degradation product [5]. Chemical Reaction of primary amines is show as below.

$$CO_2 + RNH_2 \leftrightarrow RNCOOH$$
 (2.1)

$$RNCOOH + OH^{-} \leftrightarrow HCO_{3}^{-} + RNH_{2}$$

$$(2.2)$$

The secondary amines ( $R_2NH$ ) include diethanolamine (DEA) and di-isopropylamine (DIPA). DEA is a solvent which removes both CO<sub>2</sub> and H<sub>2</sub>S but requires higher pressure than MEA to meet overhead specification. Because DEA is a weaker amine than MEA, so it have lower capture reaction heat and this makes the use of DEA more economical in the regeneration step than using MEA. Typical solution strength range from 25 to 35 weight % with a maximum rich loading of 0.35 mole/mole. DIPA is another secondary amine that removes COS. Solution are low in corrosion and require relatively low energy for regeneration. The most common applications for DIPA are in the ADIP and SULFINOL [5]. Chemical Reaction of secondary amines is show as below.

$$CO_2 + R_2 NH_2 \leftrightarrow R_2 NCOOH$$
 (2.3)

$$R_2NCOOH + OH^- \leftrightarrow HCO_3^- + R_2NH_2$$
(2.4)

The tertiary amines ( $R_3N$ ) include methyl-diethanolamine (MDEA) and triethanolamine (TEA). MDEA is often used to selectively remove  $H_2S$ , especially for cases with high  $CO_2$  to  $H_2S$  ratio in the sour gas. MDEA can remove  $H_2S$  to 4ppm while maintaining or less  $CO_2$  in the treated gas using relatively less energy for regeneration than that for DEA. However tertiary amines react more slowly with  $CO_2$ , but they circulated more quickly than primary and secondary amines. Typical solution strengths are 40-50 weight % with a maximum rich loading of 0.55 mole/mole. Because MDEA is not prone to degradation, corrosion is low and reclaimer is unnecessary. Operating pressure can range from atmospheric, typical of tail gas treating units, to over 1000 psia [5]. Chemical Reaction of tertiary amines is show as below.

$$CO_2 + R_3NH_2 \leftrightarrow R_3NCOOH$$
 (2.5)

$$R_3NCOOH + OH^- \leftrightarrow HCO_3^- + R_3NH_2$$
(2.6)

Potassium Carbonate ( $K_2CO_3$ ) solution was once widely used for  $CO_2$  absorption from natural gas. The primary reason for removing  $CO_2$  was to provide a raw material for subsequent processing. In the  $CO_2$ -recovery process, the alkali carbonate was partially converted to bicarbonate in the absorber and back to the carbonate again in the regenerator, which was heated by steam. Two packed absorbers in series were low  $CO_2$ recovery efficiency and high regeneration steam requirement. The basic reaction chemistry for aqueous hot potassium carbonate solution and  $CO_2$  is represented by the following reversible reaction [8].

$$CO_2 + K_2CO_3 + H_2O \leftrightarrow 2KHCO_3$$
 (2.7)

$$CO_2 + CO_3^{2-} + H_2O \leftrightarrow 2HCO_3^{-}$$
 (2.8)

$$CO_3^{2-} + H2O \leftrightarrow HCO_3^- + OH^-$$
 (2.9)

$$CO_2 + OH^- \leftrightarrow HCO_3^-$$
 (2.10)

The other solvent can be used to absorb  $CO_2$  is Sodium hydroxide (NaOH). The absorption of carbon dioxide is a common system to remove  $CO_2$ . Chemical Reaction of NaOH and  $CO_2$  are show as below [10].

$$CO_2 + NaOH \rightarrow NaHCO_3$$
 (2.11)

$$NaOH + NaHCO_3 \rightarrow Na_2CO_3 + H_2O$$
 (2.12)

#### 2.4 Experimental Setup

The CO<sub>2</sub> absorption experiments were conducted in four different scale absorption units:

#### 2.4.1 Laboratory-Scale Absorption Unit

The main component of the unit is an absorption column with an internal diameter of 0.019 m. The column make of acrylic plastic was packed with Sulzer-EX (Sulzer Brother Ltd.) wire gauze laboratory structured packing of which the geometric area and element height are 1700  $m^2/m^3$  and 0.55 m, respectively. The total height of the packing section was 1.10 m (20 packing elements). To achieve maximum mass-transfer performance, the packing elements were staggered at 90° with respect to the previous one. Since the gas phase CO<sub>2</sub> concentration profile is required for K<sub>G</sub>a<sub>v</sub> determination, five sampling points were installed along the column height [6].

#### 2.4.2 Pilot-Scale Absorption Unit

The absorption experiments also took place in a 0.10-m internal diameter acrylic column packed with Gempak 4A stainless steel (Glitsh, Inc.) structured packings. The height of the packing section was varied between 0.98 (four packing elements) and 2.21m (nine packing elements) depending upon the desired  $CO_2$  removal target. An orifice-type liquid distributor with a maximum drip-point density of 1528 points/m<sup>2</sup> was installed at the top of the column. The absorption column was designed in such a way that the  $CO_2$  concentration in the gas phase and temperature of the liquid solvent could be measured every 0.20 m along the column [6].

#### 2.4.3 Industrial-Scale Absorption Unit

The absorption unit consisted of both absorption and regeneration sections. The experimental result in this study was generated from the absorption section of which the main component is an industrial-scale absorption column made of a 0.25-m (10-in.) internal diameter stainless steel pipe. The column was packed with six elements of Sulzer BX gauze structured packings (Sulzer Brothers Ltd.). The total height of the packing section was 1.02 m. Five gas sampling points were installed along the column to allow for  $CO_2$  concentration measurement [6].

#### 2.4.4 Atmospheric pressure system

The solubility data were measured using an atmospheric pressure apparatus. The cell was a thermostated glass reactor with internal diameter of 30mm in the bottom, 66mm in the top, and the height of 210mm. The temperature in the reactor was fixed within  $\pm 0.1^{\circ}$ C by a temperature controller, adjusting the temperature in the thermostated water bath. The volume of the whole cell was about 270cm<sup>3</sup> and the volume of the solution inside the cell, was 75cm<sup>3</sup>. The proper flows of CO<sub>2</sub> and Argon were adjusted (Flow Controller, Supelco, VCD 1000) and mixed in a mixing tube to make the proper CO<sub>2</sub> partial pressures.

The total gas flow in all experiments was about  $150 \text{cm}^3/\text{min}$ . The resulting gas stream pass through a saturator at room temperature then, the saturated gas stream, was bubbled into the alkanolamine solution. The outlet gas, passed through a room temperature condenser to return evaporated water into the cell. To ensure that equilibrium had been reached, the experimental time was kept at least 4h prior to sampling the liquid phase. The CO<sub>2</sub> content in the liquid phase was determined by the addition of excess amounts of phosphoric acid into solutions and then by measuring the evolved volume of CO<sub>2</sub> by a graduated buret. The correction of the temperature and pressure was incorporated. In this work, the water partial pressure above the alkanoamine solution was determined using Rault's law, and the vapor pressures of TIPA and MEA and piperazine were neglected. The gas absorption experiments were carried out at the temperatures ranging from 30 to 70°C, various amine concentrations and blends and the partial pressures of CO<sub>2</sub> between 10 and 80kPa [7].

#### 2.5 Solubility

As the solubility data have a very important role in the design of industrial gas absorption units thus, many researchers have been attracted to this field. For example, Austgen et al. measured the solubility of  $CO_2$  in aqueous mixtures of MDEA with MEA or diethanolamine (DEA) at 40 and 80°C. Jou et al. measured the equilibrium solubility of H<sub>2</sub>S and CO<sub>2</sub> in various triethanolamine (TEA) solutions at 25, 50, 75, 100 and 125 °C. Similar studies have been done by Xu et al., Liu et al., Roberts and Mather, and Teng and Mather.[7]

#### 2.6 Sample analysis

The concentration of  $CO_2$  in the gas phase along the absorption column was measured by an infrared (IR) gas analyzer which was installed as close as possible to the sampling point. To assure that there was no liquid entering the sample lines, a test tube allowing visual observation was installed between the sampling point and analyzer. During the experiments, the gas compositions at different level in the absorption column were sampled by switching the sampling point from one part to another, and readings were taken after a steady state for each level was reached [11].

The standard method was used to analyzer the compositions of the NaOH solutions. The alkali content (hydroxide and carbonate) in liquid sample was determined by titration (methyl orange end point) with standard 1.0N HCl solutions. To determine the hydroxide content, the carbonate ions were first precipitated by adding excess barium chloride solution. The sample was then titrated with standard1.0N HCl solutions using phenolphthalein as the indicator. The latter titration directly gave the hydroxide content. The different between the first and second titrations gave the carbonate content [11].

In the case of the amine solutions, the total amine concentration was determined by titration with standard 1.0N HCl solutions using methyl orange as the indicator. The  $CO_2$  content in the liquid sample was then determined by the standard method given by the Association of Official Analytical Chemist (AOAC). This involved acidifying a precisely measured quantity of the sample by adding excess HCl solution. The  $CO_2$  gas released was collected in a precision gas burette. The amount of released  $CO_2$  was later used to calculate the  $CO_2$  loading of the amine solution [11].

#### 2.7 Overall Mass Transfer Coefficient (K<sub>G</sub>a<sub>V</sub>)

The mass flux of component A  $(N_A)$  at steady state can be represented in terms of gasside mass transfer coefficient  $(k_G)$ , total pressure (P), and phase driving forces [11]:

$$N_{A} = k_{G} P(y_{A} - y_{A}, i)$$
(2.13)

Where  $y_A$  and  $y_A$ , t represent the mole fraction of component A in the gas bulk and mole fraction of component A on the gas-side of the gas-liquid interface, respectively. The mass flux can also be expressed in terms of the overall mass transfer coefficient (K<sub>G</sub>) and equilibrium mole fraction of component A in gas phase  $(y_A^*)$  as follows [11]:

$$N_A = K_G P(y_A - y_A^*)$$
(2.14)

The relationship between the overall mass transfer coefficient and the individual-phase coefficients can be given as [11]:

$$\frac{1}{K_G} = \frac{1}{k_G} + \frac{H}{k_L} \tag{2.15}$$

Where H and  $k_L$  are Henry's law coefficient and liquid-side mass transfer coefficient, respectively. In the case of chemical absorption, such as CO<sub>2</sub> absorption into hydroxide or amine solutions, the overall mass transfer coefficient can be expressed as a function of the term called enhancement factor *I* [11]:

$$\frac{1}{K_G} = \frac{1}{k_G} + \frac{H}{Ik_L^0}$$
(2.16)

Where  $k_L^o$  denotes the liquid-side mass transfer coefficient without chemical reactions. The enhancement factor *I* is defined as the ratio of the liquid mass transfer coefficient for absorption with and without chemical reaction. In general, *I* is a strong function of concentration and packing mass transfer as well as of the reaction phenomena in the liquid film [11].

Considering an element of column with height dZ, the mass balance can be given as [11]:

$$N_A a_{\nu} dZ + G_1 d[y_A / (1 - y_A)$$
(2.17)

$$K_{G}a_{\nu}P(y_{A} - y_{A}^{*})dZ = G_{1} dY_{A}$$
(2.18)

Where  $a_{\psi}$  and  $G_1$  represent the effective interfacial area per unit volume of packing and inert gas molar flow rate, respectively. From equation (2.18), the overall transfer coefficient per unit volume of packing ( $K_G a_{\psi}$ ) can be defined as [11]:

$$K_{G}a_{w} = \left\{ \frac{G_{4}}{\left[ P(y_{A} - y_{A}^{*}) \right]} \right\} \left\{ \frac{dY_{A}}{dZ} \right\}$$
(2.19)

In this study, CO<sub>2</sub> absorption was conducted in a tested column. The CO<sub>2</sub> concentration in the gas phase along the column was measured, interpreted in term of mole ratio (Y<sub>A</sub>) and subsequently plotted as functions of column height (Z), called the CO<sub>2</sub> concentration profile. The slope of the profile expressing concentration gradient (dY<sub>A</sub>/dZ) at a particular y<sub>A</sub> is then used for evaluating the  $K_{\mathcal{C}}\alpha_{\psi}$  value according to equation (2.19). It should be noted that  $y_{\mathcal{A}}^*$  in the above equation was assumed to be zero since removal of CO<sub>2</sub> in this case is considered a moderately rapid reaction where CO<sub>2</sub> content in liquid bulk is zero [11].

## **CHAPTER 3**

## METHODOLOGY

## 3.1 RESEARCH METHODOLOGY



Figure 3.1: Project Process Flow

## 3.2 EXPERIMENTAL PROCEDURE

# 3.2.1 Experimental of Atmospheric Pressure System

## 3.2.1.1 Start-up Procedure

1. Before experiment start up, ensure all valves are closed.				
2. Power supply of thermostated water bath is switched on and the temperature is fixed by adjusting the temperature, which is ranging from 30 to 60°C.				
3. The condenser is opened.				
4. The volume of 250ml empty round bottom flask is weighed and recorded.				
5. The volume of 50ml solvent is prepared and poured into a round bottom flask.				
6. The round bottom flask with solvent is weighed again and recorded.				
7. The condenser, thermometer and round bottom flask are connected together immediately.				
8. The round bottom flask is immersed inside the thermostated water bath.				

# **3.2.1.2 Running the CO<sub>2</sub> Solubility Experiment**

1. The CO <sub>2</sub> regulator is opened slowly and set below atmospheric pressure.				
2. The $CO_2$ gas from the tank is passed through flow meter, which is set at approximately 50 cc/min to 150 cc/min and bubbled into the				
solvent.				
3. Temperature change is recorded at an interval of 10min.				
4. The outlet gas is passed through the condenser and the evaporate solvent is recovered back into the round bottom flask.				
5. To ensure the solvent is saturated, the experiment time is set at least 4 hours.				
6. The CO <sub>2</sub> regulator is turned off and the thermometer is removed from the round bottom flask after 4 hours.				
7. The round bottom flask is also removed from the thermostated water bath where it is weighed and recorded.				
8. The power supply for the thermostated water bath is switched off.				



Figure 3.2: Experimental setup of atmospheric pressure system

Experiment	Conc CO <sub>2</sub> Feed (%)	Flow rate (cc/min)	Partial Pressure of CO <sub>2</sub> (kPa)	Temperature of Water Bath (°C)	Experiment	Conc CO <sub>2</sub> Feed (%)	Flow rate (cc/min)	Partial Pressure of CO <sub>2</sub> (kPa)	Temperature of Water Bath (°C)
1	100	100	20	30	26	100	100	50	40
2	100	100	30	30	27	100	100	50	50
3	100	100	50	30	28	100	100	50	60
4	100	100	70	30	29	100	100	70	30
5	100	100	20	40	30	100	100	70	40
6	100	100	30	40	31	100	100	70	50
7	100	100	50	40	32	100	100	70	60
8	100	100	70	40	33	30	50	30	30
9	100	100	20	50	34	50	50	30	30
10	100	100	30	50	35	100	50	30	30
11	100	100	50	50	36	30	100	30	30
12	100	100	70	50	37	50	100	30	30
13	100	100	20	60	38	100	100	30	30
14	100	100	30	60	39	30	150	30	30
15	100	100	50	60	40	50	150	30	30
16	100	100	70	60	41	100	150	30	30
17	100	100	20	30	42	30	50	30	30
18	100	100	20	40	43	30	100	50	30
19	100	100	20	50	44	30	150	70	30
20	100	100	20	60	45	50	50	20	40
21	100	100	30	30	46	50	100	30	40
22	100	100	30	40	47	50	150	50	40
23	100	100	30	50	48	100	50	70	40
24	100	100	30	60	49	100	100	20	50
25	100	100	50	30	50	100	150	30	50

 Table 3.1: Experimental Setting

### 3.3 MILESTONE

For the first semester, I will more focus on the literature research, gathering information on the theory of absorption, type of absorption experimental, solvent analysis and develop equation base on gas diffusion and mass transfer. Literature review is done by gather as much as possible through journal, online research and reference book on  $CO_2$  absorption. There are several kind of absorption unit need to studies and select a most effectiveness absorption unit. Besides, the selectivity of solvent also important to remove the maximum  $CO_2$  content in NG.  $CO_2$  content is calculate through equation of gas diffusion and mass transfer.

While the second semester is more covers the implementation of the finding. The data of the experiment are collected and need to analyze whether the value is feasible or not. Construct a graph to determine the trend of different type of solvent and ability to absorb high content of  $CO_2$ .

## **CHAPTER 4**

## **RESULT & DISCUSSION**

In this experimental work, there are two types of solvents namely Stonvent and DEA. Both the solvents are tested in atmospheric pressure. Stonvent is a novel type of solvent, which is generated by locally. One of the advantages of Stonvent, there is no regeneration required throughout the process after absorbing  $CO_2$ . Figure 4.1 shows the solid particle form when  $CO_2$  has been absorbed by Stonvent. Meanwhile, Figure 4.2 shows the solid particle when it is dissolved in water.



Figure 4.1: Solid particle form after absorbed CO<sub>2</sub>



Figure 4.2: Solid particle dissolved in water

In contrast, DEA is secondary amines in Alkanolamine solution. It is an organic and colorless compound. DEA act as a weak base as compared to MEA which is a strong base when compared to MEDA. DEA is more economical in the regeneration process when compared to MEA. This is due to the fact that DEA requires less energy (i.e. heat) than MEA during regeneration. Generally, CO<sub>2</sub> will be released to the environment in the regeneration process which would bring harm to the environment causing Green House effect and Global Warming.

The following figures show the initial and completion of the reaction of Stonvent and DEA is the  $CO_2$  absorption.



Figure 4.3: Initial stage of CO<sub>2</sub> absorption by Stonvent solution



Figure 4.4: Completion of CO<sub>2</sub> absorption by Stonvent solution after 4 hours



Figure 4.5: Initial stage of CO<sub>2</sub> absorption by DEA solution



Figure 4.6: Completion of CO<sub>2</sub> absorption by DEA solution after 4 hours

In order to compare the higher solubility of  $CO_2$  in both the solvents, parameters such as temperature, pressure, concentration and flowrate are used as a basis of study. The data below is a summary of the experimental data obtained as part of the experiment to compare the absorption of  $CO_2$  in both the solvents.

On the experimental ran on Stonvent, the first part is to vary the pressure (i.e. 20 kPa to 70 kPa) at constant concentration and flowrate. The experiment is followed by varying the temperature (i.e.  $30^{\circ}$ C to  $60^{\circ}$ C) while other parameters are set constant (i.e. concentration and flowrate). Next, parameters (i.e. temperature and pressure) are at constant while concentration and flowrate is varied. Table 4.1 till Table 4.4 summarizes all the experimental processes mentioned.

As for DEA, parameters such as pressure and temperature are set constant while flowrate and concentration are varied. Table 4.5 and 4.6 summarizes the data from the experimental work.

Table 4.7 and Table 4.8 compares the experimental work on the solubility of both the solvent as mentioned earlier.

# 4.1 CO<sub>2</sub> solubility data in a solution of Stonvent

Concentration (%)	Flow rate (cc/min)	Pressure (kPa)	Temperature (°C)	Loading (g of CO <sub>2</sub> / g of Stonvent)
100	100	20	30	0.1467
100	100	30	30	0.1501
100	100	50	30	0.1463
100	100	70	30	0.1344
100	100	20	40	0.1395
100	100	30	40	0.1450
100	100	50	40	0.1409
100	100	70	40	0.1381
100	100	20	50	0.1346
100	100	30	50	0.1354
100	100	50	50	0.1312
100	100	70	50	0.1279
100	100	20	60	0.1248
100	100	30	60	0.1263
100	100	50	60	0.1325
100	100	70	60	0.1249

Table 4.1: Solubility of  $CO_2$  in aqueous solution of Stonvent from 20kPa to 70kPa

<b>Concentration</b> Flow rate		Pressure	Temperature	Loading (g of CO <sub>2</sub> /
(%)	(cc/min)	(kPa)	(°C)	g of Stonvent)
100	100	20	30	0.1467
100	100	20	40	0.1395
100	100	20	50	0.1346
100	100	20	60	0.1248
100	100	30	30	0.1501
100	100	30	40	0.1450
100	100	30	50	0.1354
100	100	30	60	0.1263
100	100	50	30	0.1463
100	100	50	40	0.1409
100	100	50	50	0.1312
100	100	50	60	0.1325
100	100	70	30	0.1344
100	100	70	40	0.1381
100	100	70	50	0.1279
100	100	70	60	0.1249

Table 4.2: Solubility of CO<sub>2</sub> in aqueous solution of Stonvent from  $30^{\circ}$ C to  $60^{\circ}$ C

Concentration (%)	Flow rate (cc/min)	Pressure (kPa)	Temperature (°C)	Loading (g of CO <sub>2</sub> / g of Stonvent)
30	150	30	30	0.0337
50	150	30	30	0.0642
100	150	30	30	0.1424
30	100	30	30	0.0432
50	100	30	30	0.0684
100	100	30	30	0.1501
30	50	30	30	0.0334
50	50	30	30	0.0674
100	50	30	30	0.1474

Table 4.3: Solubility of  $CO_2$  in aqueous solution of Stonvent from 30% to 100%

**Table 4.4:** Solubility of  $CO_2$  in aqueous solution of Stonvent from 50cc/min to150cc/min

Concentration (%)	Flow rate (cc/min)	Pressure (kPa)	Temperature (°C)	Loading (g of CO <sub>2</sub> / g of Stonvent)
30	50	30	30	0.0334
30	100	30	30	0.0432
30	150	30	30	0.0337
50	50	30	30	0.0674
50	100	30	30	0.0684
50	150	30	30	0.0642
100	50	30	30	0.1474
100	100	30	30	0.1501
100	150	30	30	0.1424

# 4.2 CO<sub>2</sub> solubility data in a solution of DEA

Concentration (%)	Flow rate (cc/min)	Pressure (kPa)	Temperature (°C)	Loading (g of CO <sub>2</sub> / g of DEA)
30	150	30	30	0.1108
50	150	30	30	0.1214
100	150	30	30	0.1418
30	100	30	30	0.1091
50	100	30	30	0.1197
100	100	30	30	0.1398
30	50	30	30	0.1081
50	50	30	30	0.1191
100	50	30	30	0.1388

Table 4.5: Solubility of  $CO_2$  in aqueous solution of DEA from 30% to 100%

Table 4.6: Solubility of  $CO_2$  in aqueous solution of DEA from 50cc/min to 150cc/min

Concentration (%)	Flow rate (cc/min)	Pressure (kPa)	Temperature (°C)	Loading (g of CO <sub>2</sub> / g of DEA)
30	50	30	30	0.1081
30	100	30	30	0.1091
30	150	30	30	0.1108
50	50	30	30	0.1191
50	100	30	30	0.1197
50	150	30	30	0.1214
100	50	30	30	0.1388
100	100	30	30	0.1398
100	150	30	30	0.1418

# 4.3 Comparison of CO<sub>2</sub> solubility data in solutions of Stonvent & DEA

Solvent	Concentration (%)	Flow rate (cc/min)	Pressure (kPa)	Temperature (°C)	Loading (g of CO <sub>2</sub> / g of Solvent)
	30	100	30	30	0.0432
	50	100	30	30	0.0684
Stonvent	100	100	30	30	0.1501
	30	100	30	30	0.1091
	50	100	30	30	0.1197
DEA	100	100	30	30	0.1398

Table 4.7:	Solubility of CO <sub>2</sub> in aqueous solutions of Stonvent & DEA from 30% to	0
	100%	

**Table 4.8:** Solubility of  $CO_2$  in aqueous solutions of Stonvent & DEA from 50cc/minto 150cc/min

Solvent	Concentration (%)	Flow rate (cc/min)	Pressure (kPa)	Temperature (°C)	Loading (g of CO <sub>2</sub> / g of Solvent)
	100	50	30	30	0.1474
	100	100	30	30	0.1501
Stonvent	100	150	30	30	0.1424
	100	50	30	30	0.1388
	100	100	30	30	0.1398
DEA	100	150	30	30	0.1418

#### 4.4 CO<sub>2</sub> solubility in various parameters using Stonvent



#### 4.4.1 Varying with pressure

Figure 4.7: CO<sub>2</sub> Loading versus Pressure of pure Stonvent at 100cc/min

Figure 4.1 shows the solubility of  $CO_2$  using pure Stonvent with different pressure and temperature, which are set as 30°C, 40°C, 50°C and 60°C. The figure show the pressure increase from 20kPa to 30kPa, the  $CO_2$  loading will slightly increase and decrease after 30°C.

#### 4.4.2 Varying with temperature



Figure 4.8: CO<sub>2</sub> Loading versus Temperature of pure Stonvent at 100cc/min

Figure 4.2 shows the amount of  $CO_2$  absorbs by pure Stonvent with different temperature and pressure, which are 20kPa, 30kPa, 50kPa and 70kPa. It show that when the temperature increasing, the solubility will decrease. From the experimental view, when the temperature increase, the solid will form faster and more hardness. At the same time, the outlet of piping is easily block compare to low temperature.

#### 4.4.3 Varying with concentration



Figure 4.9: CO<sub>2</sub> Loading versus Concentration of Stonvent at 30kPa and 30°C

Figure 4.3 shows the amount of  $CO_2$  absorbs by Stonvent with different concentration and flowrate, which are 50cc/min, 100cc/min and 150cc/min at 30kPa and 30°C. It shows when concentration increasing gradually, the solubility also increase. From the experimental view, when the concentration decreases, the solid form more trend to crystalline.

#### 4.4.4 Varying with flowrate



Figure 4.10: CO<sub>2</sub> Loading versus Flowrate of Stonvent at 30kPa and 30°C

Figure 4.4 shows the amount of  $CO_2$  absorbs by Stonvent with different flowrate and concentration, which are 30%, 50% and 100% at 30kPa and 30°C. It show that  $CO_2$  loading increasing when concentration from 50ccmin to 100cc/min and decease from 100cc/min to 150cc/min. From experimental view, when the flowrate increase, the solid will form more softness and outlet of piping blocking also reduces if compare to high flowrate.

### 4.5 CO<sub>2</sub> solubility in various parameters using DEA



## 4.5.1 Varying with concentration

Figure 4.11: CO<sub>2</sub> Loading versus Concentration of DEA at 30kPa and 30°C

Figure 4.5 shows the amount of  $CO_2$  absorbs by DEA with different concentration and flowrate, which are 50cc/min, 100cc/min and 150cc/min. Its shows the solubility increase when the concentration increases gradually.

## 4.5.2 Varying with flowrate



Figure 4.12: CO<sub>2</sub> Loading versus Flowrate of DEA at 30kPa and 30°C

Figure 4.6 shows the amount of  $CO_2$  absorbs by DEA with different flowrate and concentration, which are 30%, 50% and 100%. Its shows  $CO_2$  loading increase when the flowrate increases gradually.



#### 4.6.1 Varying with concentration

**Figure 4.13:** CO<sub>2</sub> Loading versus Concentration of Stonvent & DEA at 30kPa, 30°C and 100cc/min

Figure 4.7 shows the amount of  $CO_2$  absorbs by Stonvent and DEA with different concentration, which are 30%, 50% and 100% at 30°C, 30kPa and 100cc/min. Its shows  $CO_2$  loading increase when the concentration increases gradually. In 30% and 50% of solutions, it shows the  $CO_2$  solubility of DEA is higher than Stonvent but in 100% of solutions, the  $CO_2$  solubility if Stonvent is higher than DEA.

#### 4.6.2 Varying with flowrate



**Figure 4.14:** CO<sub>2</sub> Loading versus Flowrate of Stonvent & DEA at 30kPa, 30°C and 100% of solution

Figure 4.8 shows the solubility of  $CO_2$  using Stonvent and DEA with different flowrate which are set as 50cc/min, 100cc/min and 150cc/min. For Stonvent, the  $CO_2$  loading increase from 50cc/min to 100cc/min and decrease from 100cc/min to 150cc/min. For DEA, its shows  $CO_2$  loading increase when the flowrate also increases. The result shows that Stonvent achieve higher solubility than DEA when the parametric is 30kPa, 30°C, 100cc/min and 100% of solution.

## **CHAPTER 5**

## **ERROR & RECOMMENDATION**

#### 5.1 Error

- 1. Solvent is oxidation when the opening of container is open.
- 2. Liebig condenser is the most basic-cooled design in laboratory, so it cannot cool down all the solvent when perform in high temperature.
- 3. Impurity gases are occur inside piping.
- 4. The flowrate show fluctuating when the pressure is too low.
- 5. The flowrate is decrease when there are solid form block inside of the outlet pipe.
- 6. The flowrate is decrease when the pressure of  $CO_2$  regulator is drop.
- 7. Back flow is occur when the opening of the  $CO_2$  gas is switch off.

### 5.2 Recommendation

- 1. Opening of container must tighten immediately to avoid the oxidation of solvent.
- 2. Graham condenser must used to replace the Liebig condenser to achieve more accuracy because it has a spiral coil running the length of the condenser.
- 3. All the piping must purge using nitrogen gas before start experiment.
- 4. The flowrate must always adjust back to the actual reading when perform low pressure.
- 5. The solidification inside the piping must clean immediately to avoid inconsistent flowrate.
- 6. CO<sub>2</sub> regulator is checked at regular interval to avoid pressure drop.
- 7. Outlet of piping must take out before switch off the opening of the gas.

### CHAPTER 6

## CONCLUSION

In most applications, the choice of contacting device is between trayed tower and a packed column. Packed columns should be considered for corrosive services where ceramic or plastic materials are preferred over metals, in services where foaming may be severe if trays used, and when pressure drop must be low, as in vacuum or nearambient-pressure operations. Otherwise, trayed towers, which can be designed and scaled up more reliably, are preferred. Although structured packings are quite expensive, they may be the best choice for a new installation when pressure drop must be very low or for replacing existing tray (retrofitting) when a higher capacity or degree or degree of separation is required in an existing column. Trayed towers are preferred when liquid velocities are low, while columns with random packings are best for high liquid velocities. Solubility is the term describing the maximum amount of solute dissolved in saturated solution at a specific parameter. When reporting solubility values, it is important to state the temperature, pressure, concentration and flowrate at which the solution is being made because it can affect solubility. From the experimental result, it shows the trend for absorption capacity of Stonvent is directly proportional with increasing pressure of CO<sub>2</sub> in the range between 20kPa to 30kPa. However, it shows a decreasing trend after 30kPa. As for another parameter being tested, it shows that absorption capacity of Stonvent decreases with increasing temperature. On the concentration of Stonvent, the test result shows that when the concentration decreases, the absorption capacity of Stonvent also decreases. On the last parameter being tested, it shows increasing absorption capacity when the flowrate of CO<sub>2</sub> gas increases from 50cc/min to 100cc/min and the absorption capacity starts to decrease after 100cc/min. For DEA, its show a direct relationship where increasing flowrate will result in increasing absorption capacity. This applies to the concentration, when concentration increases, the absorption capacity also increases. In conclusion, Stonvent has highest CO<sub>2</sub> absorption capacity at 30kPa, 30°C, 100cc/min and 100% of solution, which is 0.1501 g of CO<sub>2</sub>/g of Stonvent as compared to DEA, that is 0.1398 g of CO<sub>2</sub>/g of DEA.

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# APPENDICES

# Appendix 1: Gantt chart and Milestone for First Semester

		20-	27-	3-	10-	17-	24-	31-	7-	14-	21-	28-	5-	12-	19-	26-	2-
No.	ACTIVITIES / WEEKENDING	Jul	Jul	Aug	Aug	Aug	Aug	Aug	Sep	Sep	Sep	Sep	Oct	Oct	Oct	Oct	Nov
		1	2		3	4	5	6	7	8		9	10	11	12	13	14
1	Select of Project Topics																
2	Submission of FYP Topics Proposal																
3	Accept the Project Topics Assigned																
4	Preliminary Research Work																
5	Submission of Preliminary Report																
6	Information Searching & Literature Review on			H1N1													
	Theory of CO <sub>2</sub> Absorption			SU							MII						
	Type of solvent for CO <sub>2</sub> Absorption			SPE							IS C						
	Theory of Gas Diffusion & Mass Transfer			ENSIC							EM BI						
7	Submission of Progress Report			Ň							REA						
8	Seminar			PER							ĸ						
9	Studies on the novel type of Absorption apparatus			IOD													
10	Learn to operate Absorption Unit																
11	Preparation of Interim Report & Oral Presentation																
12	Submission of Interim Report																
13	Final Oral Presentation																

Process

Milestone

No.	ACTIVITIES / WEEKENDING	25- Jan	1- Feb	8- Feb	15- Feb	22- Feb	1- Mar	8- Mar	15- Mar	22- Mar	29- Mar	5- Apr	12- Apr	19- Apr	26- Apr	24- May	31- May	7- Jun
		1	2	3	4	5	6	7		8	9	10	11	12	13	17	18	19
1	Gather all the apparatus needed																	
2	Conduct experimental work and collect all the data																	
2	Submission of Progress Report 1																	
3	Analyst the data and construct trend																	
4	Verification result																	
	If Yes: Discussion & Conclusion								MID S									
	If No: Repeat the experiment								EM B									
5	Submission of Progress Report 2								REAK									
6	Poster Exhibition																	
7	Improve project																	
8	Submission of Dissertation (CD Softcopy & Soft Bound)																	
9	Oral Presentation																	
10	Submission of Project Dissertation (Hard Bound)																	

# Appendix 2: Gantt chart and Milestone for Second Semester

Process

Milestone



Appendix 3: Structure of Gas Absorption Pilot Plant

- 3 -

Appendix 4: Flow Diagram of Atmospheric Pressure System

