# ศึกษาสมบัติการละลายของก๊าซคาร์บอนไดออกไซด์ในสารละลาย 2-(ไดเอทิลอะมิโน)เอธานอล

นายภัทร บุญพิพัฒนาพงศ์

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# SOLUBILITY STUDY OF CARBON DIOXIDE IN AQUEOUS 2-(DIETHYLAMINO)ETHANOL (DEAE) SOLUTION

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A Thesis Submitted in Partial Fulfillment of the Requirements
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| Thesis Title | SOLUBILITY STUDY OF CARBON DIOXIDE IN AQUEOUS 2-(DIETHYLAMINO)ETHANOL (DEAE) SOLUTION |
| By | Mr. Pattara Boonpipattanapong |
| Field of Study | Chemical Engineering |
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ภัทร บุญพิพัฒนาพงศ์ : ศึกษาสมบัติการละลายของก๊าซคาร์บอนไดออกไซด์ในสารละลาย 2-(ไดเอทิลอะมิโน)เอธานอล. (SOLUBILITY STUDY OF CARBON DIOXIDE IN AQUEOUS 2-(DIETHYLAMINO)ETHANOL (DEAE) SOLUTION) อ.ที่ปรึกษาวิทยานิพนธ์หลัก: ศ. ดร.สุทธิชัย อัสสะบำรุงรัตน์, อ.ที่ปรึกษาวิทยานิพนธ์ร่วม: ดร.เกรียงไกร มณีอินทร์, 66 หน้า.

ในปัจจุบันนี้ความกังวลในด้านสิ่งแวดล้อมในปัญหาเรื่องการเปลี่ยนแปลงสภาพภูมิอากาศและภาวะโลกร้อนเป็นประเด็นที่สำคัญเนื่องจากมีปริมาณก๊าซคาร์บอนไดออกไซด์ในบรรยากาศจากการปลดปล่อยจากโรงไฟฟ้า การดูดซับทางเคมีที่ใช้สารเอมีนเป็นเทคโนโลยีที่ได้รับการพิสูจน์แล้วว่าใช้ในการกำจัด ก๊าซคาร์บอนดออกไซด์สารละลายเอมีน ที่ใช้ในเชิงพาณิชย์ในปัจจุบัน คือ โมโนเอธาโนลามีนและ เมทิลไดเอธาโนลามีน อย่างไรก็ตามสาร เหล่านี้ยังมีข้อเสียอยู่ เช่น มีความจุวัฏจักรที่ต่ำ และใช้พลังงานในการฟื้นสภาพมาก ดังนั้นจึงได้มีการพัฒนาสารดูดซึมก๊าซคาร์บอนไดออกไซด์ชนิดใหม่ที่ช่วยเพิ่มประสิทธิภาพ ดังนั้นวัตถุประสงค์ของงานวิจัยนี้จึงเป็นการวัดค่าสมบัติการละลายของก๊าซคาร์บอนไดออกไซดในสารละลายน้ำของ 2-(ไดเอทิลอะมิโน)เอธานอล ซึ่งเป็นสารดูดซึมชนิดใหม่ โดยจะทำการวัดค่าที่สภาวะอุณหภูมิ 30-80 องศาเซียลเซียส ความดันย่อยของก๊าซคาร์บอนไดออกไซด์ในช่วง 5-100 กิโลปาสคาล และ ความเข้มข้นของสารละลายที่ 3, 4, และ 5โมลาร์ ค่าสมบัติการละลายของสารละลาย 2-(ไดเอทิลอะมิโน)เอธานอลที่ได้จะนำไปเปรียบเทียบกับ ค่าที่ได้จากสารละลายเอมีนที่ใช้ทั่วไปอย่าง สารละลายโมโนเอธาโนลามีนและ เมทิลไดเอธาโนลามีน ที่สภาวะเดียวกัน จากผลการทดลองจะเห็นว่า 2-(ไดเอทิลอะมิโน)เอธานอล นั้นมีค่าความจุวัฏจักรมากกว่าสารดูดซึมที่ใช้ในเชิงพาณิชย์ทั้ง 2 ชนิด ที่สภาวะของก๊าซเสียที่ออกมาจากโรงไฟฟ้า ซึ่งนั่นหมายความว่า 2-(ไดเอทิลอะมิโน)เอธานอล ใช้พลังงานในการฟื้นสภาพน้อยกว่าและ นำไปสู่การประหยัดค่าใช้จ่ายสำหรับกระบวนการดักจับก๊าซคาร์บอนไดออกไซด์

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# # 5570324721 : MAJOR CHEMICAL ENGINEERING
KEYWORDS: SOLUBILITY / CARBON DIOXIDE / FLUE GAS / ABSORPTION / AMINE SOLUTION

PATTARA BOONPIPATTANAPONG: SOLUBILITY STUDY OF CARBON DIOXIDE IN AQUEOUS 2-(DIETHYLAMINO)ETHANOL (DEAE) SOLUTION. ADVISOR: PROF. SUTTICHAI ASSABUMRUNGRAT, Ph.D., CO-ADVISOR: KREANGKRAI MANEEINTR, Ph.D., 66 pp.

Nowadays, environmental concern on climate change and global warming is the main issue because of the higher content of carbon dioxide discharge from power plant to the atmosphere. Chemical absorption with amine solution is a proven technology to remove carbon dioxide. The commercially used amine solution are monoethanolamine (MEA) and methyl diethanolamine (MDEA). However, it still has disadvantages such as low cyclic capacity and high energy requirement. Therefore, new solvents for carbon dioxide capture have been developed to enhance the amine performance. Consequently, the objective of this study is to measure the solubility of carbon dioxide in an aqueous solution of 2-(Diethylamino)ethanol (DEAE) used as a new solvent. It is measured at 30-80 degrees Celsius and carbon dioxide partial pressures ranging from 5 to 100 kPa. The concentration of aqueous DEAE solution are 3, 4 and 5 M. The solubility data of carbon dioxide in aqueous solution of DEAE are compared with that of aqueous solution of MEA and MDEA at the same conditions. The results show that the DEAE has higher cyclic capacity than that of commercial absorbents at the post combustion power plant flue gas stream conditions. This means DEAE can use less energy for regeneration and lead to high energy saving for carbon capture process.

Department: Chemical Engineering Student's Signature ..............................

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# CHAPTER I

# INTRODUCTION

## 1.1 Effect and Uses of $CO_2$

$CO_2$ is known as one of the greenhouse gases. Heat from radiation of the sun is accumulated in the planet. The result from this case is the cause of higher earth's surface temperature, which leads to the climate change. The $CO_2$ is emitted from many places such as iron and steel industries [1] and the power plants [2].

The disadvantage of the greenhouse class into five groups:

- Sea level rise; the result from melting of the ice caps
- Impacts on agriculture; global warming is the cause of decreasing agricultural productivity
- Reduction of ozone layer; the chemical reaction of greenhouse gas deplete ozone layer
- Increased extreme weather; global warming is the reason of the weather systems of the planet change.
- Ecosystem change; the higher temperature can increase the range of some parasites and diseases

However, the $CO_2$ is used in the carbonated beverages industry. The common way of using $CO_2$ is the fire extinguisher. Nowadays $CO_2$ is used in enhanced oil and gas recovery process [2].

## 1.2 Sources of flue gas

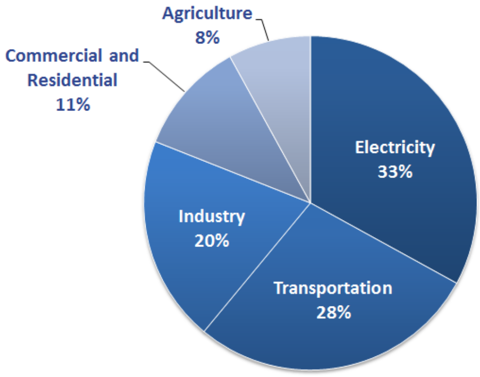


**Figure 1.1** Sources of greenhouse gas [3]

Figure 1.1 gives the sources of flue gas. The two highest quantity of greenhouse gas emission are electricity generation (33%) and the transportation (28%). From Figure 1.1 that shows the important reason to remove the greenhouse gas from the Power station.

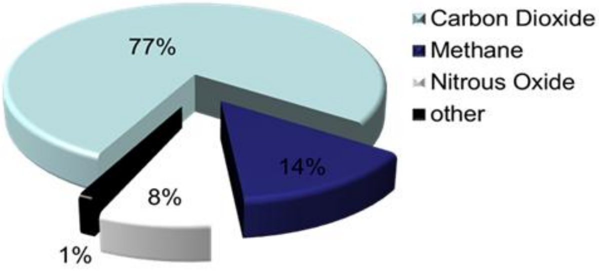


**Figure 1.2** Composition of greenhouse gas [4]

Figure 1.2 gives the composition of greenhouse gas. The highest composition of greenhouse gas is carbon dioxide. From the Figure 1.1 and Figure 1.2, this is the reason why carbon dioxide should be removed from the power station’s flue gas stream.

## 1.3 Flue gas from coal-fired power plant

The flue gas from coal-fired power plants has the concentration of carbon dioxide is about 14% and emitted at atmospheric pressure [5].That the reason why the method to remove the low concentration of carbon dioxide from flue gas stream is tremendous solution for remove carbon dioxide from coal-fired power plant.

## 1.4 Flue gas control technology

Nowadays the various technologies could be used to remove carbon dioxide from flue gas streams. The suitable removal carbon dioxide technologies depend on the properties of the streams and the economics such as temperature of the gas streams, pressure of the gas streams, efficiency of carbon dioxide separation, capital cost, operating cost and corrosion [2]. The various technologies shown in Figure 1.3

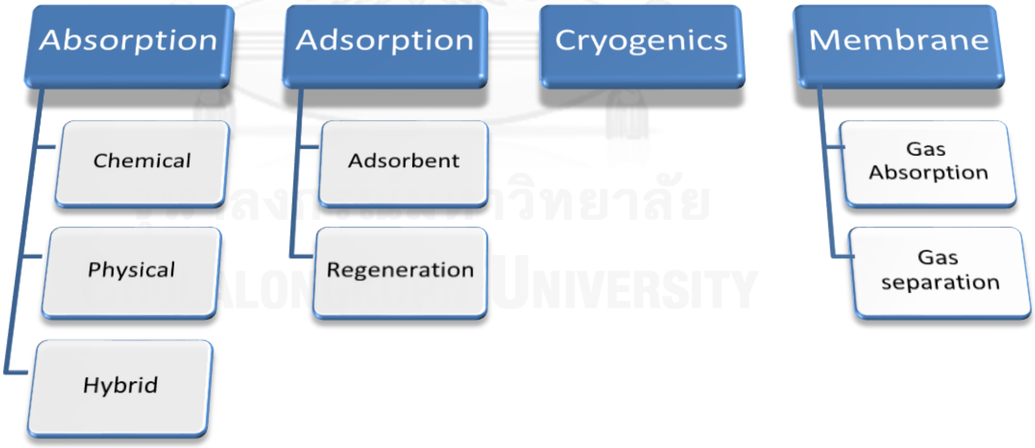


**Figure 1.3** Removal carbon dioxide technologies [6]

### 1.4.1. Absorption technology

Absorption technologies are the widely use to capture carbon dioxide [2], [6]. The absorption technology is shown in Figure 1.4. The procedure of absorption is that flue gas is fed into the liquid solvent in the absorber to capture the carbon dioxide and apply heating in regenerator to recover solution. The kinds of absorption technologies are chemical, physical and hybrid absorption technologies. The chemical absorption usually uses in commercial for carbon dioxide capture in low carbon dioxide concentration, whereas physical absorption usually uses in high partial pressure of carbon dioxide from gas streams.

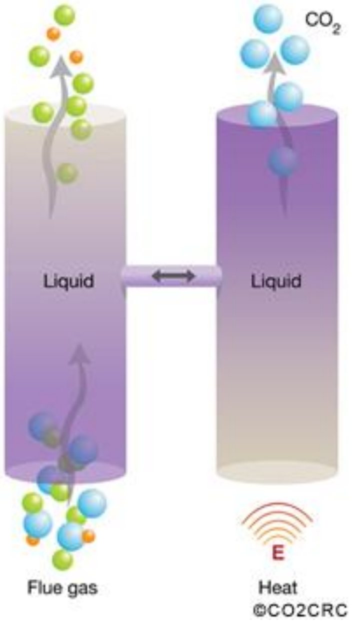


**Figure 1.4** Absorption technology [7]

### 1.4.2. Adsorption technology

The adsorption technology is shown in Figure 1.5. Adsorption technology is an adsorption between the carbon dioxide in gas stream and the active site of a solid adsorbent and regenerated by reducing energy supply. The efficiency of adsorption of carbon dioxide depends on partial pressure, temperature, surface force and absorbent pore size. The types of adsorbent are activated carbon, molecular sieve, alumina and silica gel adsorption technologies. Whereas the types of adsorption regenerations are pressure swing adsorption, temperature swing adsorption and electric swing adsorption technologies [6].

Adsorption is the suitable technique for treating high concentration of $CO_2$ in flue gas streams, whereas poor performance for large-scale separation of $CO_2$ from power plant's flue gas as the result of poor capacity and $CO_2$ selectivity [2].

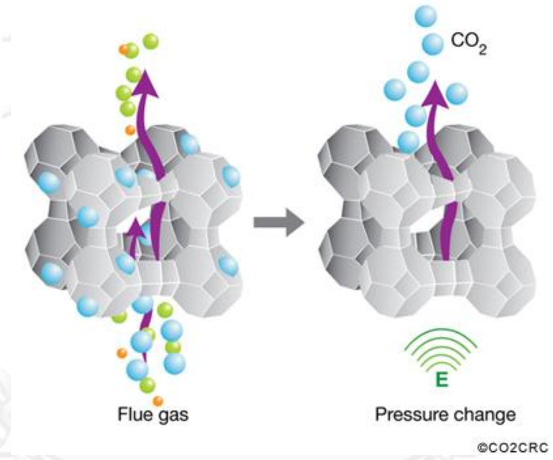


**Figure 1.5** Adsorption technology [7]

### 1.4.3. Cryogenic technology

The Cryogenic technology is shown in Figure 1.6. Cryogenic process is commonly used in commercial for purified carbon dioxide from the high carbon dioxide concentration (> 50%) [6]. The way of cryogenic technology is to freeze the carbon dioxide from the flue gas streams into liquid carbon dioxide [2]. Cryogenic technology is the suitable technique for treating high concentration of $CO_2$ in gas streams and high economic for transportation of $CO_2$. Therefore the removal carbon dioxide from the coal-fired power plant cannot use the cryogenic technology, because of the quantity of energy requirement for refrigeration is unrewarding for the power plant. But in the pre-combustion capture, process is popularly use because of high concentration of carbon dioxide.

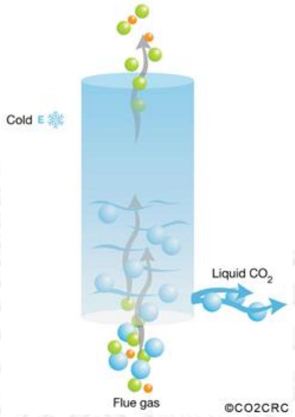


**Figure 1.6** Cryogenic technology [7]

### 1.4.4. Membrane

The membrane is the semi permeable film that is selective permeation under conditions suitable to its function. The applications of membrane for carbon dioxide capture are 2 types:

#### *1.4.4.1 Gas absorption membranes*

Gas absorption membranes are made by micro-porous solid membranes that are used as contacting devices between gas flow and liquid flow. The carbon dioxide diffuses through the membrane and is removed by the absorption liquid, which selectively removes certain components from the gas stream on the other side of the membrane. In contrast to gas separation membranes it is the absorption liquid (not the membrane) that gives the process its selectivity [2].

### *1.4.4.2. Gas separation membranes*

Gas separation membranes rely on differences in physical or chemical interaction between gases and a membrane material, causing one component to pass through the membrane faster than another. The separation of the gases depends on diffusivity – differences in partial pressure from one side of the membrane to other side of the membrane, as the driving force for gas separation [2], as shown in Figure 1.7.

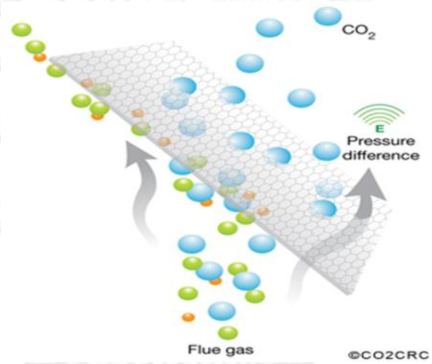


**Figure 1.7** Membrane technology [7]

Membrane technology can be used to capture the carbon dioxide in coal-fired power plant. However membrane technology may not be attractive for large-scale separation of carbon dioxide and has low degrees of separation, therefore multiple stages and/or recycle can help the trouble. But that lead to increase the energy consumption and cost.

From each technology, it can be summarized that chemical absorption technology is the proper economical and suitable technology for carbon dioxide capture from coal-fired power plant flue gas streams.

Moreover, the development of efficient chemical used as the solvent is very vital. Nowadays alkanolamines such as monoethanolamine (MEA) and methyldiethanolamine (MDEA) are well performed for carbon dioxide capture solvent. But these solvents have disadvantages. Therefore, the novel solvents are developed to increase the absorption performance and removal carbon dioxide efficiently. The detail of chemical solvent will be described in next chapter.

### 1.5 Objective of this research

1. Evaluate DEAE's performance for solubility of $CO_2$ and compare the results with the commonly used solvents MEA and MDEA with various operating conditions.

### 1.6 Benefit

1. Improve the existing technology on process that is used in carbon dioxide capture technology from power plant flue gas stream.
2. Use the research's data as the basis information for the design of treating acid gas plants with the novel absorbent.

## 1.7 Research scopes

The research scopes of this experiment are shown in Table 1.1.

**Table 1.1** Research scopes of experiment

| **Operating condition** | **Solubility study** |
|---|---|
| Solution concentration (M) | 3,4 and 5 |
| Partial pressure (kPa) | 5-100 |
| Temperature (˚C) | 30-80 |
| Absorbent | DEAE MEA MDEA |

The other chapter in this thesis will show in more detail. That combines a theory and literature review of absorption process and chemical selection and absorbent development in the chapter 2. Chapter 3 shows the methodology of thesis’s experiment. Solubility of carbon dioxide in DEAE will show and compare the performance for absorption with common absorbent like MEA and MDEA. Latest the performance will conclude in the chapter 4.

# CHAPTER II

# THEORY AND LITERATURE REVIEWS

## 2.1 Absorption technology

### 2.1.1. Basic concept of absorption

The concentration gradient required to produce the mass transfer of component A from the gas phase to the liquid phase, is illustrated in Figure 2.1. The gas diffuses through the thin boundary layer as the gas film and the thin boundary layer as the liquid film. When gas enters to the liquid phase, it can dissolve or it can react with the chemical solvent.

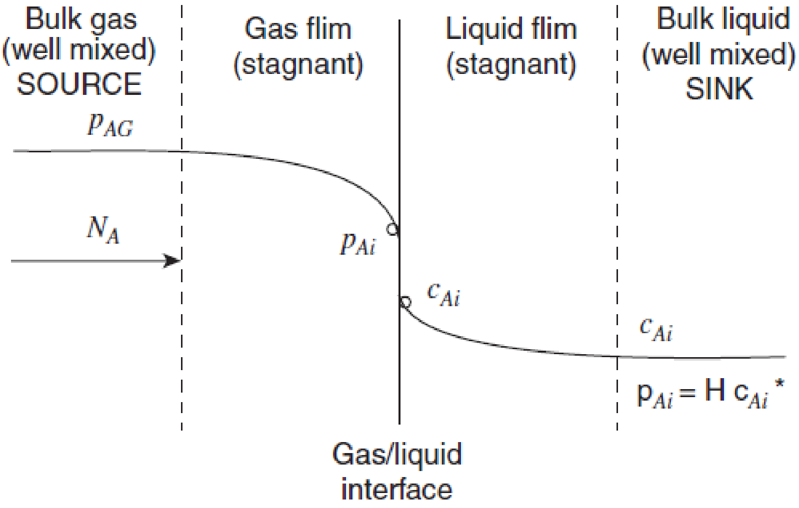


**Figure 2.1** Concentration gradients between two contacting phases where the solute is transferred from gas to liquid. [8]

Figure 2.1 shows a partial pressure gradient from the bulk gas composition, $p_{AG}$, to the interfacial gas composition, $p_{Ai}$, and a concentration gradient in the liquid from, $c_{Ai}$, at the interface to the bulk liquid concentration, $c_A$, L. On the basis of Whitman's second assumption of no resistance to mass transfer at the interfacial surface, $p_{Ai}$ and $c_{Ai}$, are equilibrium concentrations [8].

### 2.1.2 Type of absorption

The type of absorption is classified by the types of liquid absorbents [2, 9].

#### *2.1.2.1. Physical absorption*

The physical absorption occurs when carbon dioxide dissolves in the physical absorbent but does not react with the absorbent. The main advantage of a technology is that physical solvent has more economical in high $CO_2$ partial pressure system [10]. The amount of $CO_2$ absorbed by the solvent is determined by the vapor-liquid equilibrium of the mixture, which is governed by the pressure and temperature [10]. A high carbon dioxide concentration, the carbon dioxide loading capacity is higher than chemical absorption.

#### *2.1.2.2. Chemical absorption*

The chemical absorption uses the solvent, chemically absorbing the carbon dioxide from flue gas stream. This reaction is reversible and this is the reason why the solvent can be regenerated by providing the energy into the solvent [1]. The examples of chemical solvents are monoethanolamine (MEA) and potassium carbonate [11].

The chemical absorption has relatively high selectivity of this form of separation. In addition, a relatively pure $CO_2$ stream could be produced. These factors make chemical absorption well suited for $CO_2$ capture for industrial flue gases that why in this case the suitable for removal low carbon dioxide concentration is chemical absorption technology [9].

### 2.1.3 Process description

In the absorption, the flue gas is fed into the absorber at the bottom and the solvent is fed at the top as shown in Figure 2.2. The reactions start when the solvent and carbon dioxide flow through the column. A clean gas leaves the column at the top, when the carbon dioxide rich solvent leaves the column at the bottom. The rich solvent goes through the heat exchanger to increase the temperature before sending it to the stripper section. The heated rich solvent stream goes to the stripper at the top. In the stripper, steam is used for regeneration process. Finally, separated acid gas gets out of the stripper at the top. The lean MEA leaves the system at the bottom of the stripper and go through the heat exchanger. The MEA and water are added to the lean solvent stream to balance the component before recycling back to the absorber unit [12].

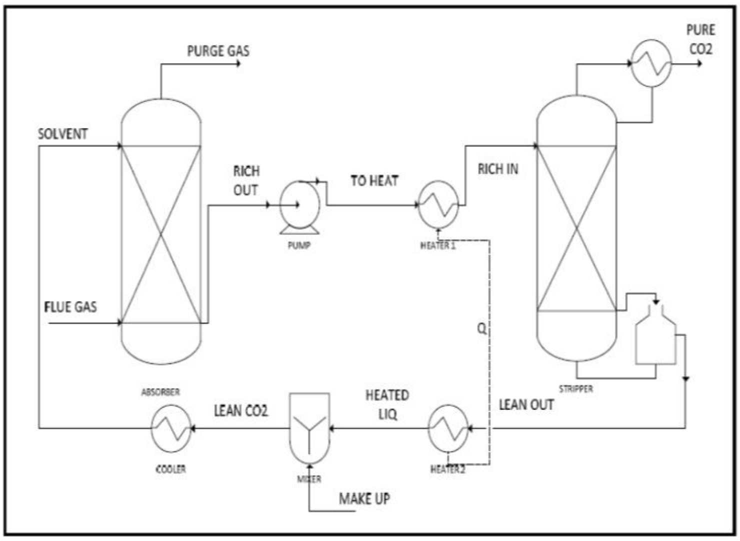


**Figure 2.2** Schematic flow diagram of the absorption process [12]

## 2.2 Solvents for acid gas removal

The main factor to the successful carbon dioxide absorption process is the effective absorbents. The ideal absorbent properties are: huge cyclic capacity or solubility of acid gas, high rate of absorption, high thermal and chemical stability, high water solubility, low corrosion of material of construction, low cost, low environmental effect [13]. The example of chemical and physical are shown in Table 2.1and 2.2, respectively.

**Table 2.1** Commercial chemical absorbents for carbon dioxide capture process [2]

| **Types of chemical solvent** | **Solvent** | **Process conditions** | **Developer/ licensor** |
|---|---|---|---|
| Organic | | | |
| MEA | MEA and chemical inhibitors | 40°C, ambient pressure | Dow Chemical, USA |
| Amine Guard (MEA) | MEA and chemical inhibitors | | Union Carbide, USA |
| Econamine | DGA | 80-120°C, 6.3 MPa | SNEA version by Societe NationalElf Aquitane, France |
| ADIP | DIPA and MDEA | 35-40°C, >0.1 MPa | Shell, Netherlands |
| MDEA | MDEA | | |
| Flexsorb/ KS-1, KS-2, KS-3 | Hindered amine | | Exxon, USA |

| Types of chemical solvent | Solvent | Process conditions | Developer/ licensor |
|---|---|---|---|
| <u>Inorganic</u> | | | |
| Benfield and versions | $K_2CO_3$ | 70-120°C, 2.2-7 MPa | Lurgi, Germany; Eickmeyer and Associates, USA |

Table 2.2 Commercial physical absorbents for carbon dioxide capture process [2]

| Types of physical solvent | Solvent | Process conditions | Developer/ licensor |
|---|---|---|---|
| Rectisol® | Methanol | (-10) until (-70)°C , > 2 MPa | Lurgi and Linde, Germany |
| Purisol® | N-methyl-2-pyrolidone (NMP) | (-10) until (+40)°C , > 2 MPa | Lurgi, Germany |
| Selexol® | dimethyl ethers of polyethylene glycol (DMPEG) | (-40)°C, 2-3 MPa | Union Carbide, USA |
| Fluor solvent | Propylene carbonate | 3.1-6.9 MPa | Fluor, El Paso, USA |

The wide varieties of chemical absorbents are alkanolamines solvents. However, some of them have limitations in low absorption capacity. This is the reason to develop the novel solvents to enhance the absorption capacity. Moreover, inorganic absorbents such as potassium carbonate, potassium hydroxide, sodium carbonate and sodium hydroxide are the alternative way to absorb carbon dioxide.

### 2.2.1 Alkanolamine absorbent

Aqueous amine solutions have been widely used to capture $CO_2$ more than 30 years in the chemical and oil industries [10]. Nowadays amines are used to capture carbon dioxide from the flue gas streams.

The amines widely used to capture carbon dioxide in commercial are monoethanolamine (MEA), diethanolamine (DEA) and methyldiethanolamine (MDEA). Among them, MEA is better than others. These amines have the one amino group and at least one hydroxyl group in their structures. The hydroxyl groups reduce the vapor pressure and increase the water solubility, and the amino group provides for the necessary alkalinity for capture carbon dioxide [14]. The commercial amines as shown in Table 2.3.

The amines can absorb carbon dioxide by mechanisms as shown below: To form carbamate anion and bicarbonate anion [15] that have the equations as followed:

Carbamate anion

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

Where $R_1$, $R_2$ are alkyl group

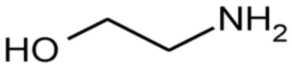
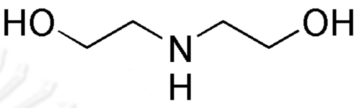
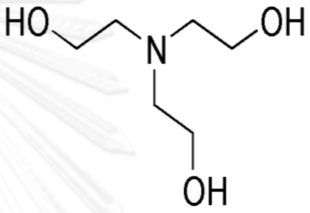
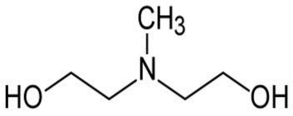
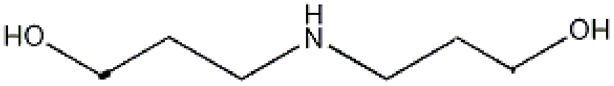
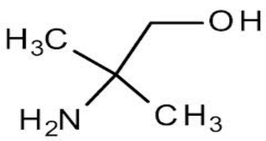
Bicarbonate anion

$$R_1R_2NH + CO_2 + H_2O \leftrightarrow HCO_3^- + R_1R_2NH_2^+$$

Two reactions have the different rate of reaction and heat of reaction [15]. The tertiary amine can be only bicarbonate form because it does not have hydrogen atom at N location. But the primary and secondary can be in both forms. The carbamate can be unstable and change to be bicarbonate form and this depends on structure of amines [15]. The mole ratio of captured carbon dioxide by amine absorbent in carbamate form is 0.5 (mole carbon dioxide per mole amines) and from bicarbonate form is 1 (mole carbon dioxide per mole amines) theoretically. The kinetic of carbamate formation is faster than bicarbonate formation and unstable carbamate form can change to bicarbonate form by hydrolysis [16].

The factors of amine's disadvantages are corrosion, high absorbent degradation and use too much energy to regenerate [2]. The more than 60% of the total energy consumption in capture carbon dioxide by alkanolamine solvent process is the energy to regenerate the solvent [17] That is the reason why the developer need to increase carbon dioxide absorption capacities, absorption rate and degradation endurance, with the decrease in corrosion and energy to regenerate.

**Table 2.3** Conventional alkanolamines [14]

| Name | Acronym | Chemical structure |
|---|---|---|
| Monoethanolamine | MEA |  |
| Diethanolamine | DEA |  |
| Triethanolamine | TEA |  |
| Methyldiethanolamine | MDEA |  |
| Diglycolamine | DGA |  |
| Diisopropanolamine | DIPA |  |
| 2-Amino-2-Methyl-1-Propanol | AMP |  |

### 2.2.2 Alkaline process

The widely used chemicals of alkaline absorbents are potassium carbonate, sodium carbonate and aqueous ammonia. In this group potassium carbonate is more often to use than other chemicals [17]. The mechanism of carbon dioxide capture by potassium carbonate has equation as followed:

$$K_2CO_3 + CO_2 + H_2O \leftrightarrow 2KHCO_3$$

Potassium carbonate process is a suitable technique for treating low carbon dioxide concentration flue gas stream and fast reaction at high temperature and low carbon dioxide absorption rate at ambient temperature [2]. That is the reason why alkaline process is not used to capture carbon dioxide from power plant's flue gas stream.

It can be concluded that the type of chemical that develop to be novel absorbents for absorb carbon dioxide from flue gas stream is the alkanolamine.

### 2.3 Literature review of novel absorbents

The suitable absorbent for carbon dioxide capture from power plant's flue gas stream is amine. Many researchers developed various alkanolamines as novel absorbent because hydroxyl group increases the distribution in alkanolamine-$H_2O$-$CO_2$ system [15]. Ratio of carbamate anion on alkanolamine depend on type of amine and the primary amine (such as MEA) has the carbamate anion ratio > secondary amine (such as DEA) > tertiary amine (such as MDEA), respectively [1]. The tertiary alkanolamine is the favorite type because it can be only bicarbonate anion, which leads to increase the solubility of carbon dioxide capture and greater in cyclic capacity [13].

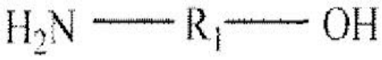


**Figure 2.3** General structure of alkanolamine

An outstanding structure of alkanolamine for capture carbon dioxide is including steric hindrance and R equal two or three carbons [15]. Because increasing the chain length is enhancing carbamate stability on R group more than four carbons [13]. The increasing steric hindrance on alkanolamine can increase the rate of absorption because it's increase unstable carbamate anion [15]. The changing the amount of $NH_2$ group does not affect the solubility of alkanolamine [13]. The 2-(Diethylamino)ethanol is the novel absorbent for study in this research.

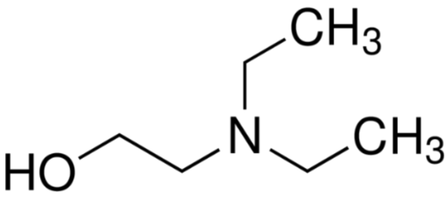
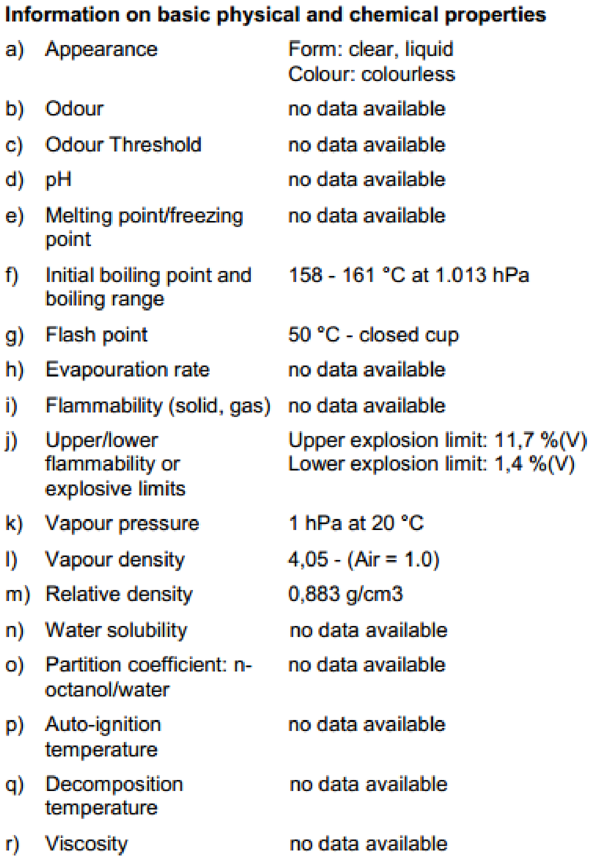


**Figure 2.4** The 2-(Diethylamino)ethanol structure

The physical and chemical properties of 2-(Diethylamino)ethanol is shown below:

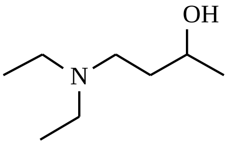


**Information on basic physical and chemical properties**

| | | |
|---|---|---|
| a) | Appearance | Form: clear, liquid<br>Colour: colourless |
| b) | Odour | no data available |
| c) | Odour Threshold | no data available |
| d) | pH | no data available |
| e) | Melting point/freezing point | no data available |
| f) | Initial boiling point and boiling range | 158 - 161 °C at 1.013 hPa |
| g) | Flash point | 50 °C - closed cup |
| h) | Evapouration rate | no data available |
| i) | Flammability (solid, gas) | no data available |
| j) | Upper/lower flammability or explosive limits | Upper explosion limit: 11,7 %(V)<br>Lower explosion limit: 1,4 %(V) |
| k) | Vapour pressure | 1 hPa at 20 °C |
| l) | Vapour density | 4,05 - (Air = 1.0) |
| m) | Relative density | 0,883 g/cm3 |
| n) | Water solubility | no data available |
| o) | Partition coefficient: n-octanol/water | no data available |
| p) | Auto-ignition temperature | no data available |
| q) | Decomposition temperature | no data available |
| r) | Viscosity | no data available |

**Figure 2.5** The physical and chemical properties of 2-(Diethylamino)ethanol

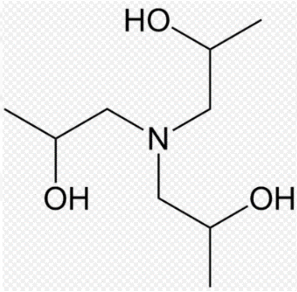
The novel absorbents from other researchers are shown below:



4-diethylamino-2-butanol
(DEAB)

**Figure 2.6** Structure of DEAB as novel absorbent from [18].

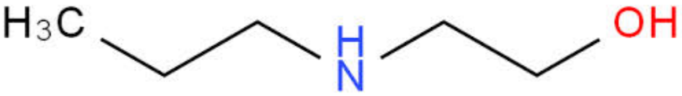
The new solvents are expected to provide much higher $CO_2$ absorption capacity and higher cyclic capacity than that of the conventional amine, MEA. It can be deduced that the new solvents have superior performance in terms of absorption and cyclic capacities, which should lead to decrease the absorbent circulation rate and energy efficiency for absorbent regeneration at the partial pressure of carbon dioxide ranging from 15 to 100 kPa and the all of solutions are measured at 40, 60 and 80°C [18].



Triisopropanolamine (TIPA)

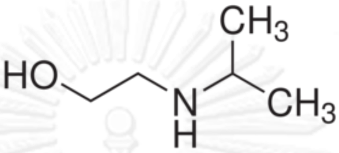
**Figure 2.7** Structure of TIPA as a novel absorbent from [19]

The solubility of TIPA at 313.15, 323.15 and333.15 K with $CO_2$ partial pressure ranging from 1 to 3000 kPa were compared with common amines such as MEA and MDEA, the $CO_2$ loading capacity of aqueous TIPA solutions was much better than that in aqueous MEA and aqueous MDEA solutions at higher $CO_2$ partial pressures [19].



2-(propylamino)ethanol (PAE)

**Figure 2.8** Structure of PAE from [15]



2-(isopropylamino)ethanol (IPAE)

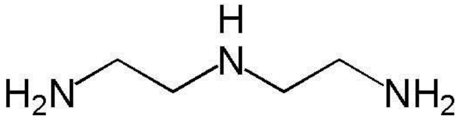
**Figure 2.9** Structure of IPAE from [15]

IPAE and PAE are compared with common amines as MEA, the $CO_2$ loading capacity of aqueous IPAE solutions was much better than that in aqueous MEA. IPAE solutions showed high $CO_2$ solubility because of the predominant production of bicarbonate ($HCO_3^-$) at 313 K, 373 K and 393 K with $CO_2$ partial pressure ranging from 5 kPa to 0.2 MPa [15].

1,6-Hexanediamine

**Figure 2.10** Structure of 1,6-Hexanediamine as a novel absorbent from [13]

1,6-hexanediamine, N,N-dimethyl showed a high cyclic loading of 0.81 and 0.85 moles $CO_2$/moles amine respectively. That is shown 1,6-hexanediamine, N,N-dimethyl has a better performance than MEA, DEA and Piperazine. The absorption carbon dioxide experiment performs at 30°C and regenerates at 90°C with $CO_2$ partial pressure ranging from 1 to 40 kPa [13].



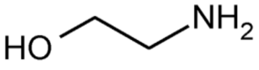
Diethylenetriamine (DETA)

**Figure 2.11** Structure of DETA as a novel absorbent from [20]

The solubility of carbon dioxide in aqueous DETA is shown that the $CO_2$ loading increased with increasing $CO_2$ partial pressure, and decreased with increasing temperatures. It was also found that solubility of the $CO_2$ in the solvents studied were appreciably greater than in typical amine solvents such as MEA and MDEA. The carbon dioxide absorption experiment performs at 313.2 K, 333.2 K and 353.2 K with $CO_2$ partial pressure ranging from 1 to 900 kPa [20].

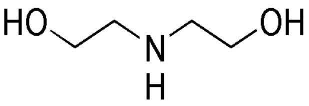
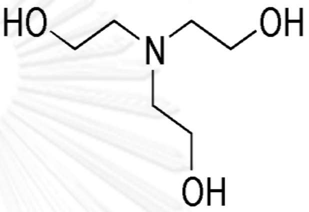
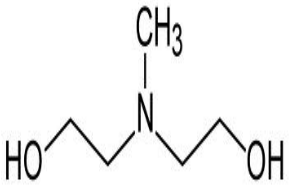
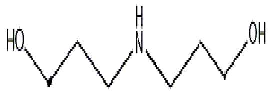
The operating conditions in each literature's work are shown in Table 2.4 that will be the guide to design operating condition on this work.

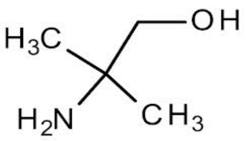
**Table 2.4** Condition of literature review in conventional alkanolamines absorption

| name | acronym | chemical structure | condition |
|---|---|---|---|
| Monoethanolamine | MEA |  | $T^1$ = 40-120°C<br>${P_{CO_2}}^2$ =<br>0 -1000 kPa<br>$Conc.^3$= 2.5-5 M<br>[18, 21, 22, 23, 24] |

---

[1] T = operating temperature
[2] $P_{CO_2}$ = partial pressure of carbon dioxide
[3] Conc. = concentration of absorbent solution

| name | acronym | chemical structure | condition |
|---|---|---|---|
| Diethanolamine | DEA |  | T = 25-120°C<br>$P_{CO_2}$= 1.33-5681 kPa<br>Conc.= 0.5-5 M [25] |
| Triethanolamine | TEA |  | T =50-100 °C<br>$P_{CO_2}$ =5-5490 kPa<br>Conc.= 2-5 M [26] |
| Methyldiethanolamine | MDEA |  | T = 40-120 °C<br>$P_{CO_2}$= 0.001-6600 kPa<br>Conc.= 1-4.28 M [27, 28] |
| Diglycolamine | DGA |  | T = 50-100 °C<br>$P_{CO_2}$= 1.6-5890 kPa<br>Conc.= 60 wt% [29] |
| Diisopropanolamine | DIPA |  | T = 298 and 353 K<br>$P_{CO_2}$= 2.5-955 kPa<br>Conc.= 10-33 wt% [30] |

| name | acronym | chemical structure | condition |
|---|---|---|---|
| 2-Amino-2-Methyl-1-Propanol | AMP |  | T = 20-80°C<br>$P_{CO_2}$ = 1-100 kPa<br>Conc.= 2-3 M<br>[31] |

The solubility of carbon dioxide in aqueous DEAE solution is compared with that of the commercial absorbent amine, MEA and MDEA, which is usually provided to base case in most alkanolamine based $CO_2$ capture studies [13, 15, 20]. The experimental section including apparatus, chemical and methodology to investigate the performance of carbon dioxide absorption in aqueous DEAE solution will be discussed in Chapter 3.

# CHAPTER III
# EXPERIMENTAL

This chapter is shown the experimental procedure in this research. This chapter has been separated into five sections; chemicals, apparatus, experiment conditions, method of experiment and analysis of solubility of $CO_2$ in aqueous alkanolamine solvent.

## 3.1 Chemical

The chemicals in this work are separated into 2 sections; gases, and inorganic chemical as absorbents used in each experiment. The details will be present below.

### 3.1.1 Gases

Carbon Dioxide is supplied from Praxair Inc. with 99.5% purity. For control of $CO_2$ partial pressure, Nitrogen is supplied from Praxair Inc. with 99.5% purity, which is subject to manage $CO_2$ partial pressure for use in each experiment.

### 3.1.2 Absorbents and inorganic chemical

The Monoethanolamine are obtained from Sigma Aldrich with 99% purity. The Methyldiethanolamine are obtained from Sigma Aldrich with 99% purity. The 2-(Diethylamino)Ethanol come from comes from Merch with 99+% purity. Inorganic, 1N hydrochloric (HCl) is obtained from RCI Labscan limited.

## 3.2 Apparatus

### 3.2.1 Circulator and water bath

The Lauda water circulators heat, hold the water temperature and control at desired temperature. The saturation cell and reactor cell are immersed in the Memmert water bath with temperature range of 0-100°C.

### 3.2.2 Saturator and reactor set

$CO_2$ and $N_2$ were fed into both test tubes as the saturation cell and reactor cell, which including flasks, test tubes and rubber corks for absorption the gas mixture ($CO_2$ and $N_2$).

### 3.2.3 Condenser set

During absorption process the water need to be recovered to maintain the concentration of solution in the reactor cell.

### 3.2.4 Mass flow meter

The mass flow meters in this work are electric AALBORG GFM17 gas flow meters that is calibrated by Agilent Technology ADM100 Universal Mass Flow meter with flow range of 0.5 to 1,000 mL/min and accuracy of ±3% of reading.

## 3.3 Experimental conditions

### 3.3.1 The effect of reaction temperature in a range of 30 – 80 °C on solubility of $CO_2$

In this study, the effects of reaction temperature on a carbon dioxide treatment of flue gas stream will be investigated. By varies in a range of 30 – 80°C to find out the optimal absorption and regeneration temperature on $CO_2$ absorption process. Table 3.1 shows the condition for study on the effect of reaction temperature.

**Table 3.1** Absorption conditions

| Absorption | |
|---|---|
| Temperature (°C) | 30, 40, 60 and 80 |
| Reaction time (hrs.) | 8-18 |
| $CO_2$ partial pressure (kPa) | 5, 15, 30, 50, 75 and 100 |
| solvent concentration (M) | 3-5 |
| Type of solvent | DEAE, MEA and MDEA |

### 3.3.2 The effect of $CO_2$ partial pressure in a range of 5 – 100 kPa on solubility of $CO_2$

By varying the $CO_2$ partial pressure at 5, 15, 30, 50, 75 and 100 kPa, the results could identify the performance and the effect on absorption. Table 3.1 shows the $CO_2$ partial pressure.

### 3.3.3 The effect of solvent concentration on solubility of $CO_2$

The effect of solvent concentration will be studied for the optimal condition of $CO_2$ absorption. Table 3.1 shows the solvent concentration.

### 3.3.4 The absorption performance of DEAE as the new solvent

In this work, DEAE is presented as new solvent that needs to evaluate the performance for $CO_2$ absorption by comparing with the commercial alkanolamines, MEA and MDEA used as the base case solvents. Table 3.1 shows the types of solvent.

## 3.4 Method of experiment

For procedure of this study, feed the absorbent into a reactor cell and place the reactor cell into the constant temperature water bath. Feed $N_2$ and $CO_2$ as the mixture. The partial pressure of $CO_2$ and $N_2$ can be controlled by using the mass flow meters, and feed this mixture to the reactor cell after flowing through the water saturator cell until it reaches to equilibrium about 12-18 hours. To analyze the equilibrium solubility data, titration with 1M HCl is used with sample from the solution.

## 3.5 Solubility of $CO_2$ in aqueous alkanolamine solvent analysis

The solubility of $CO_2$ for each sample is evaluated as follows: the liquid sample is brought from reactor cell using a 2 or 3 mL of sample. Then, methyl orange is used for indicator about 3-4 drops. After that, excess 1N HCl is added into the liquid sample and all of the $CO_2$ that is released from the sample is collect in the burette for measurement as shown in Figure 3.1. The amount of emitted $CO_2$ is calculated in g-mol. The solubility of $CO_2$ and concentration of sample solution are evaluated by using the method outline by Association of Official Analytical Chemists [32]. From these results, the g-mole ratio of $CO_2$ to alkanolamine in the liquid phase is described in the next chapter. The solubility of $CO_2$ is reported in the average form of three equilibrium data points.

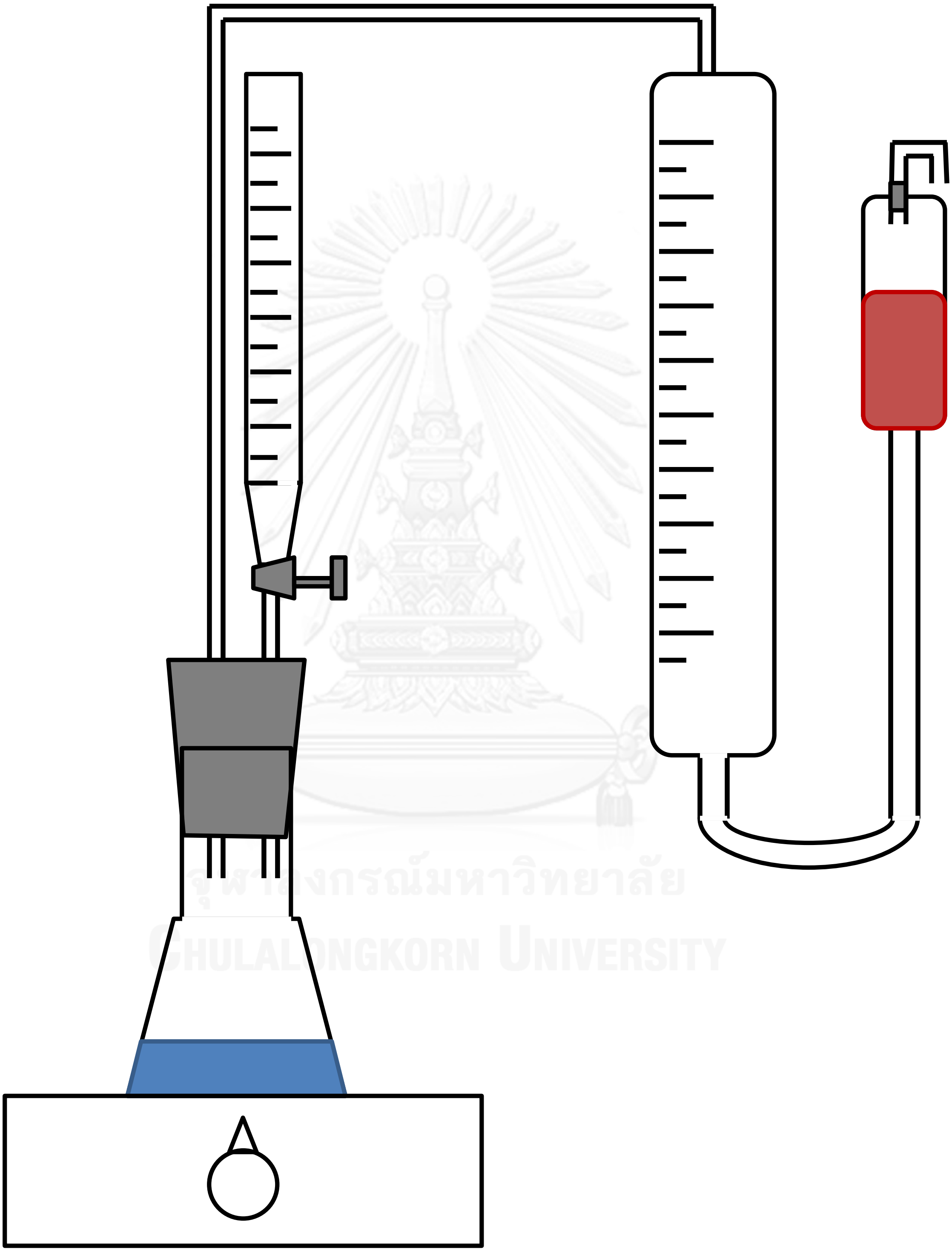


Figure 3.1 Solubility of $CO_2$ measurement apparatus [32]

# CHAPTER IV

# RESULTS AND DISCUSSIONS

Solubility study of carbon dioxide in aqueous 2-(Diethylamino)ethanol (DEAE) solution from absorption process is analyzed. The effects of absorption conditions on solubility of carbon dioxide are investigated. In addition, the performance of the DEAE solution is compared to the commercially used solvents (MEA and MDEA) and discussed in this chapter.

## 4.1 Verification of solubility study

This section is aimed to verify the method of the experiment for making sure the result from this apparatus can be reliable. The solubility of $CO_2$ or $CO_2$ loading ($\alpha$) in aqueous 5.0M MEA is measured at the same operating conditions with previous literature to compare the reliability of this experiment. The errors in this work are from the determination of the $CO_2$ partial pressure and the measurement of the volume as well as the composition of the $CO_2$ released from liquid sample. The solubility data of $CO_2$ in aqueous 5.0M MEA is shown in Table 4.1. The data is compared with the data from literature [15, 21, 22, 23, 24] at the same conditions in Figure 4.1-4.3.

The index to verify the method of this experiment is the percent average absolute deviation (%AAD) [33]. The %AAD between this work and literature obtained by [21] is investigated from the following equation:

$$\%AAD = \frac{100}{n} + \sum_{n=1}^{n} \left| \frac{P_{ref} - P_{exp}}{P_{ref}} \right| \tag{4.1}$$

Where n is the amount of data points

$P_{ref}$ is the data points from reference.

$P_{exp}$ is the data points from this experiment.

The %AAD from 40°C to 80°C and $CO_2$ partial pressure from 5 kPa to 100 kPa for this experimental apparatus is 2.67% and the maximum percentage error is 11.05%. It can be concluded that this method and experiment is reliable enough for other conditions in this study.

**Table 4.1** The solubility of $CO_2$ in aqueous 5.0M MEA solution

| Temperature | 30°C | | 40°C | | 60°C | | 80°C | |
|---|---|---|---|---|---|---|---|---|
| Solvent | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) |
| 5.0 M MEA | 5.00 | 0.498 | 4.90 | 0.491 | 5.20 | 0.44 | 5.10 | 0.371 |
| | 13.90 | 0.557 | 13.80 | 0.523 | 15.80 | 0.475 | 16.00 | 0.443 |
| | 31.70 | 0.560 | 30.40 | 0.532 | 31.00 | 0.513 | 30.40 | 0.456 |
| | 51.40 | 0.583 | 49.50 | 0.557 | 49.90 | 0.537 | 50.90 | 0.484 |
| | 74.30 | 0.597 | 76.10 | 0.570 | 76.10 | 0.542 | 75.80 | 0.491 |
| | 99.60 | 0.611 | 100.30 | 0.590 | 101.10 | 0.549 | 99.50 | 0.537 |

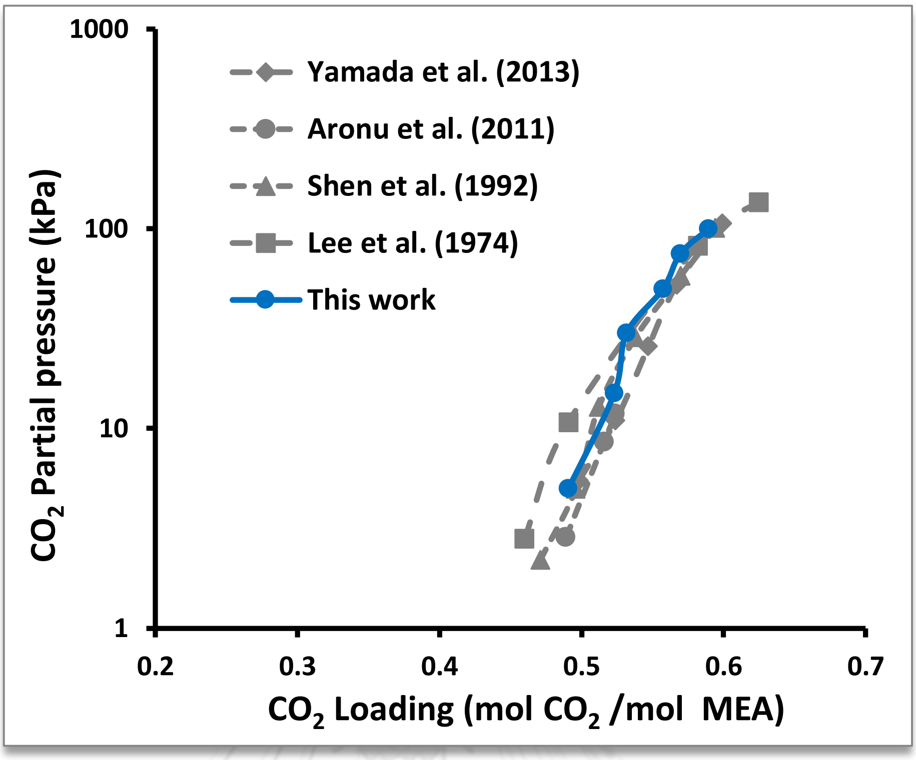


Figure 4.1 The solubility of $CO_2$ in aqueous 5.0M MEA solution at 40°C

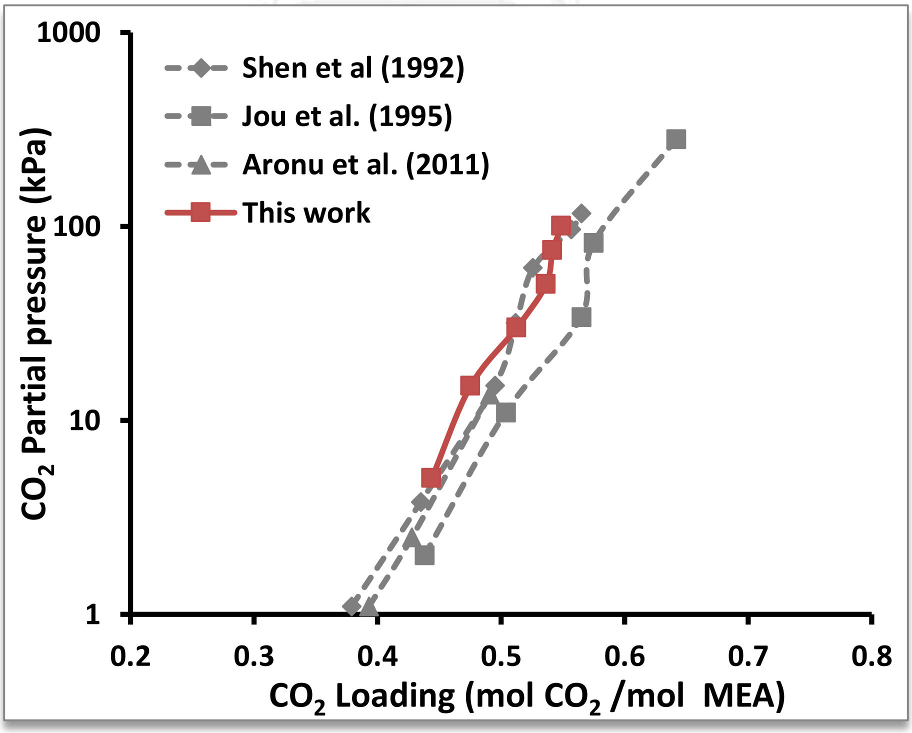


Figure 4.2 The solubility of $CO_2$ in aqueous 5.0M MEA solution at 60°C

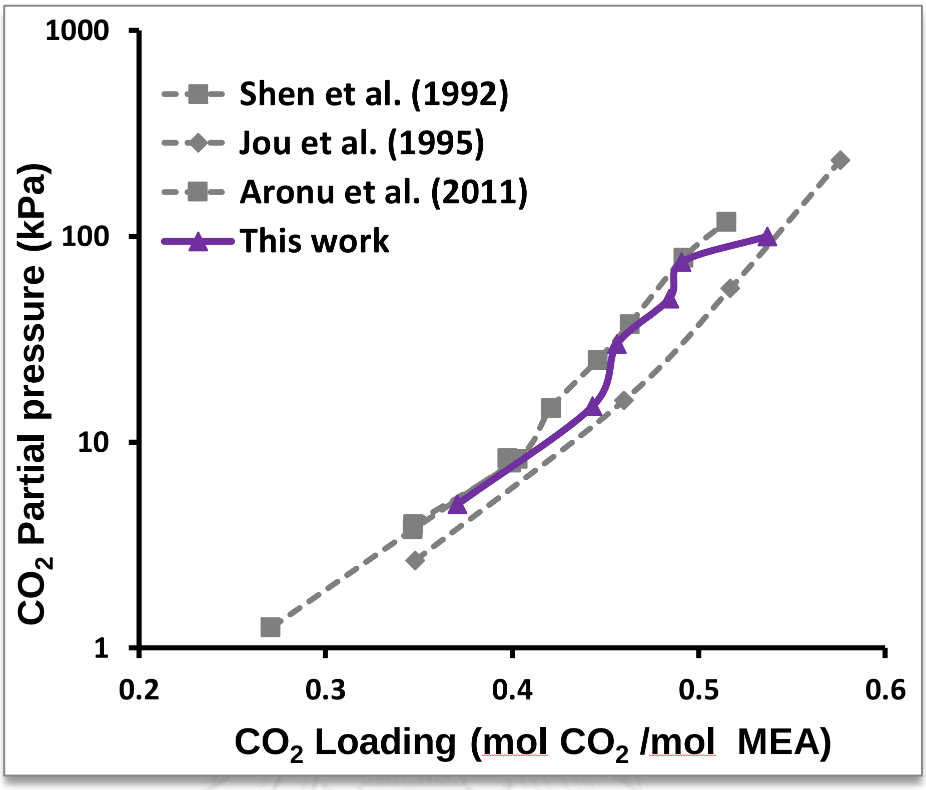


**Figure 4.3** The solubility of $CO_2$ in aqueous 5.0M MEA solution at 80°C

### 4.2 The effect of absorption conditions on solubility of carbon dioxide

This section studies the effect of absorption conditions on solubility of carbon dioxide. The solubility of $CO_2$ or $CO_2$ loading in aqueous solution of DEAE is measured at various temperatures, $CO_2$ partial pressures and concentrations for investigating the behavior of DEAE solution.

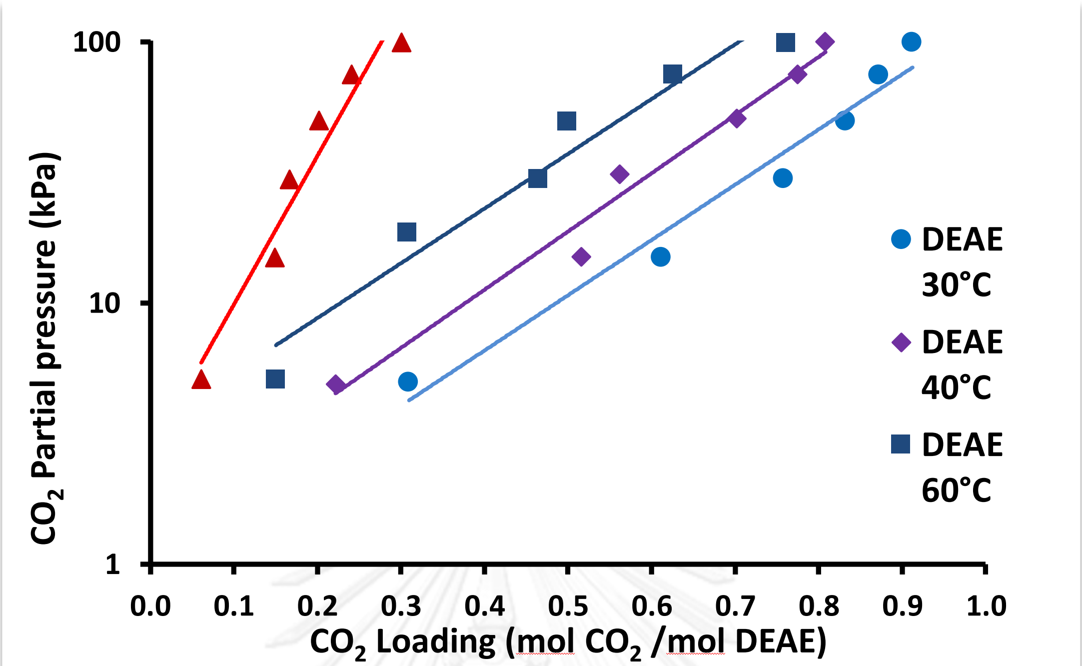


**Figure 4.4** The solubility of $CO_2$ in aqueous 5.0M DEAE solution

**Table 4.2** The solubility of $CO_2$ in aqueous 5.0M DEAE solution

| Temperature | 30°C | | 40°C | | 60°C | | 80°C | |
|---|---|---|---|---|---|---|---|---|
| Solvent | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) |
| 5.0 M DEAE | 5.07 | 0.309 | 4.89 | 0.222 | 5.10 | 0.151 | 5.12 | 0.061 |
| | 15.60 | 0.612 | 15.11 | 0.516 | 18.79 | 0.307 | 14.97 | 0.149 |
| | 30.28 | 0.758 | 31.32 | 0.562 | 29.97 | 0.465 | 29.89 | 0.167 |
| | 50.55 | 0.832 | 51.01 | 0.702 | 49.87 | 0.499 | 50.06 | 0.202 |
| | 74.82 | 0.872 | 75.41 | 0.775 | 75.43 | 0.626 | 75.13 | 0.241 |
| | 98.24 | 0.912 | 100.70 | 0.808 | 99.72 | 0.761 | 99.96 | 0.301 |

### 4.2.1 The effect of temperature on solubility of carbon dioxide

The solubility of $CO_2$ in aqueous 5.0M DEAE solution is shown in Table 4.3. The effect of temperature depending on the solubility of $CO_2$ in aqueous 5.0M DEAE solution is shown in Figure 4.4. It can be seen that solubility increases with the decrease in temperature. According to the above result, the reason is described by Henry’s constant (H) from the following equation [34]:

$$\ln H = C_1^H + \frac{C_2^H}{T} + C_3^H \ln T + C_4^H T \qquad (4.2)$$

The constants in equation 4.2 are shown in Table 4.3 [34]

**Table 4.3** Temperature Dependence of Henry’s Constants

| | $C_1^H$ | $C_1^H$ | $C_1^H$ | $C_1^H$ |
|---|---|---|---|---|
| $CO_2$-$H_2O$ | 170.7126 | -8477.711 | -21.95743 | 0.005 |
| $CO_2$-MEA | 89.452 | -2934.6 | -11.592 | 0.016 |

The Figure 4.5 shows the temperature dependency of Henry’s constants of $CO_2$ in water and in MEA from the equation 4.2.

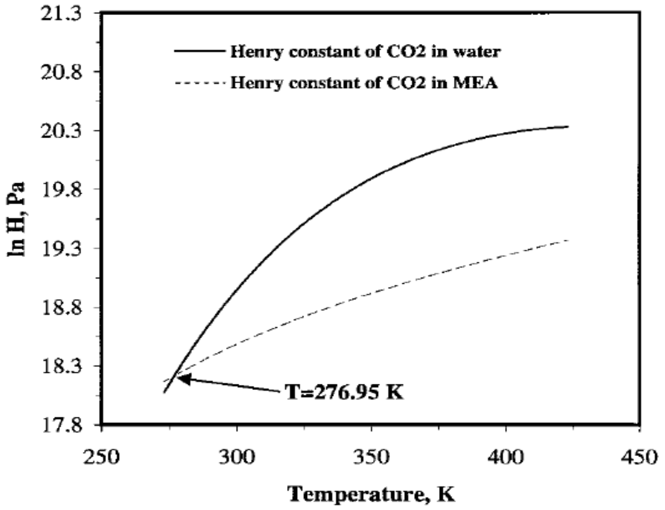


**Figure 4.5** Temperature dependence of Henry's constants of $CO_2$

It's become clearly that when temperature increases leading to the increase of H and from the Henry's law when the H increases with the decrease of solubility. From these results, its can explain why the $CO_2$ absorption process uses the high temperature to take $CO_2$ out from the solution. From Figure 4.3, the suitable temperature for regeneration is 80°C. Because the solvent at 80°C has the lowest amount of $CO_2$ in the solution. And the absorption temperature is about 40°C. Because the commercial $CO_2$ removal process use the MEA at 40°C as absorption condition and it is not different between $CO_2$ loading at 30°C and 40°C in significance.

### 4.2.2 The effect of $CO_2$ partial pressure on solubility of carbon dioxide

According to Figure 4.4, it shows that solubility increase with an increase in $CO_2$ partial pressure. The amines can absorb carbon dioxide by mechanisms as shown below: form carbamate anion and bicarbonate anion [15] that have the equations as followed:

Carbamate anion

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

Bicarbonate anion

$$R_1R_2NH + CO_2 + H_2O \leftrightarrow HCO_3^- + R_1R_2NH_2^+$$

Where $R_1$, $R_2$ are alkyl group

According to Le Chatelier's principle, the equilibrium of the above reaction will shift to the forward if $CO_2$ of the reaction is increased.

According to Figure 4.4, at the 30°C, the greatest absorption condition, the solubility of $CO_2$ at 100 kPa is better than $CO_2$ loading at 5 kPa about 195%. And at the $CO_2$ partial pressure condition of the post combustion stream (at 5 to 15 kPa of $CO_2$ partial pressure which as the range of flue gas released from post combustion power plants) the solubility of $CO_2$ at 15 kPa is better than $CO_2$ loading at 5 kPa about 98%.

**Table 4.4** The solubility of $CO_2$ in aqueous 3.0M, 4.0M and 5.0M DEAE solutions

| Temperature | 30°C | | 40°C | | 60°C | | 80°C | |
|---|---|---|---|---|---|---|---|---|
| Solvent | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) |
| 5.0 M DEAE | 5.07 | 0.309 | 4.89 | 0.222 | 5.10 | 0.151 | 5.12 | 0.061 |
| | 15.60 | 0.612 | 15.11 | 0.516 | 18.79 | 0.307 | 14.97 | 0.149 |
| | 30.28 | 0.758 | 31.32 | 0.562 | 29.97 | 0.465 | 29.89 | 0.167 |
| | 50.55 | 0.832 | 51.01 | 0.702 | 49.87 | 0.499 | 50.06 | 0.202 |
| | 74.82 | 0.872 | 75.41 | 0.775 | 75.43 | 0.626 | 75.13 | 0.241 |
| | 98.24 | 0.912 | 100.70 | 0.808 | 99.72 | 0.761 | 99.96 | 0.301 |

| Temperature | 30°C | | 40°C | | 60°C | | 80°C | |
|---|---|---|---|---|---|---|---|---|
| Solvent | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) | α (mol/mol) | $P_{CO_2}$ (kPa) |
| 4.0 M DEAE | 4.91 | 0.576 | 3.96 | 0.411 | 5.36 | 0.157 | 4.44 | 0.063 |
| | 15.40 | 0.749 | 14.71 | 0.712 | 15.52 | 0.350 | 15.10 | 0.165 |
| | 34.63 | 0.858 | 30.01 | 0.753 | 29.70 | 0.389 | 30.14 | 0.173 |
| | 50.14 | 0.911 | 49.76 | 0.837 | 49.60 | 0.494 | 47.60 | 0.218 |
| | 74.05 | 0.916 | 74.05 | 0.870 | 73.86 | 0.630 | 74.90 | 0.294 |
| | 99.06 | 1.022 | 99.06 | 0.920 | 99.09 | 0.776 | 99.78 | 0.347 |
| 3.0 M DEAE | 5.60 | 0.710 | 5.32 | 0.626 | 5.16 | 0.408 | 4.72 | 0.105 |
| | 15.07 | 0.841 | 15.57 | 0.795 | 16.93 | 0.509 | 14.17 | 0.200 |
| | 30.07 | 0.870 | 30.07 | 0.814 | 32.97 | 0.651 | 28.61 | 0.262 |
| | 47.55 | 0.922 | 47.55 | 0.844 | 48.34 | 0.735 | 49.43 | 0.387 |
| | 73.60 | 0.969 | 74.08 | 0.880 | 73.43 | 0.758 | 74.35 | 0.467 |
| | 98.09 | 1.027 | 98.55 | 0.944 | 96.65 | 0.785 | 99.40 | 0.510 |

### 4.2.3 The effect of solution concentration on solubility of carbon dioxide

The solubility of $CO_2$ at various concentrations of DEAE solution is shown in Table 4.4 and the data in this table are plotted in Figure 4.6-4.11. The concentration effect on the solubility of $CO_2$ in aqueous DEAE solutions are shown in Figure 4.6-4.11.

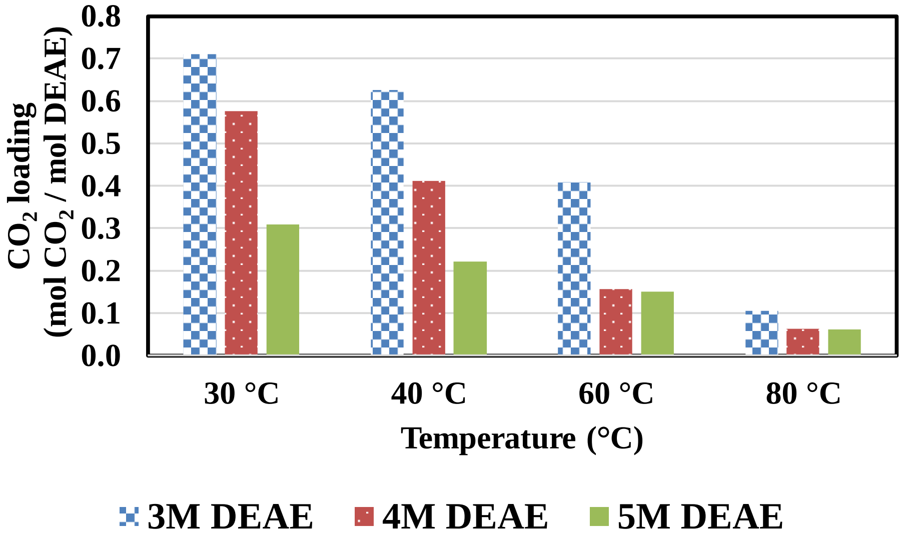


**Figure 4.6** The concentration effect on the solubility of $CO_2$ in aqueous DEAE solutions at 5 kPa $CO_2$ partial pressure

In the Figure 4.6, at 5 kPa and 30°C, the greatest absorption condition, the solubility of $CO_2$ in aqueous 3.0M is better than $CO_2$ loading in aqueous 4.0M and 5.0M about 23.3% and 130%, respectively. In the same way, at 5 kPa and 80°C, the smallest absorption condition or desorption condition, the $CO_2$ loading in aqueous 3.0M is better than $CO_2$ loading in aqueous 4.0M and 5.0M about 66.7% and 72.1%, respectively.

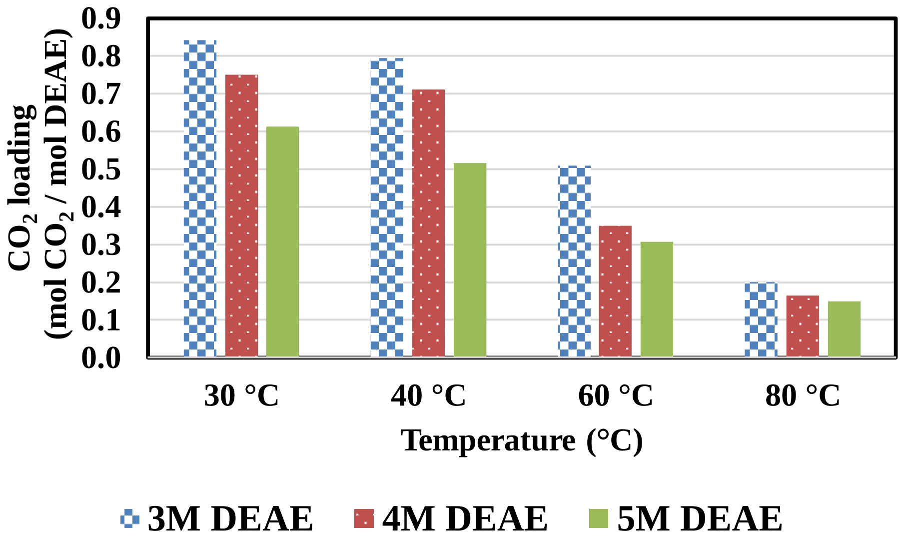


**Figure 4.7** The concentration effect on the solubility of $CO_2$ in aqueous DEAE solutions at 15 kPa $CO_2$ partial pressure

According to Figure 4.7, at 15 kPa and 30°C, the solubility of $CO_2$ in aqueous 3.0M is greater than $CO_2$ loading in aqueous 4.0M and 5.0M about 12.3% and 37.4%, respectively. In the same way, at 15 kPa and 80°C, the lowest absorption condition or regeneration condition, the $CO_2$ loading in aqueous 3.0M is better than $CO_2$ loading in aqueous 4.0M and 5.0M about 21.2% and 34.2%, respectively.

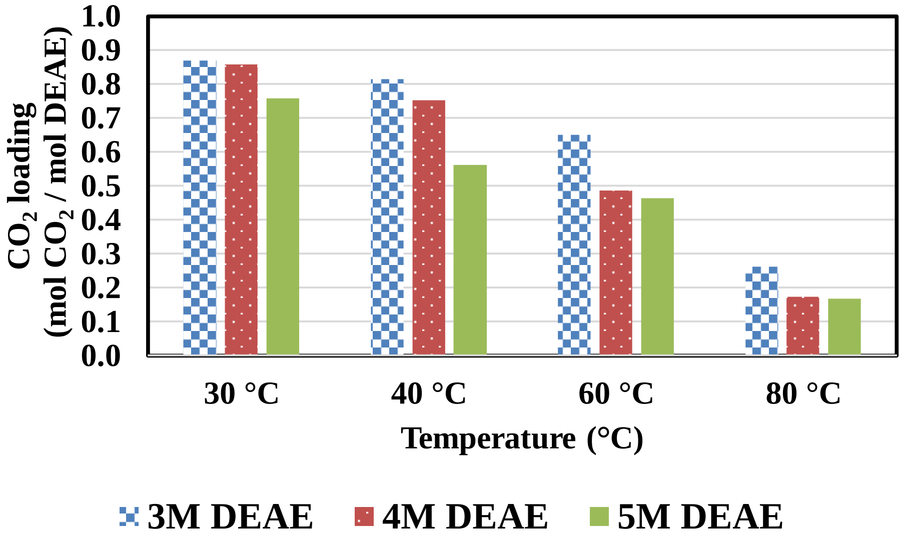


**Figure 4.8** The concentration effect on the solubility of $CO_2$ in aqueous DEAE solutions at 30 kPa $CO_2$ partial pressure

Figure 4.8 is shown that at 30 kPa and 80°C, the regeneration condition, $CO_2$ loading in aqueous 3.0M is better than $CO_2$ loading in aqueous 4.0M and 5.0M about 51.4% and 56.9%, respectively. In the same behavior, at 30 kPa and 30°C, the solubility of $CO_2$ in aqueous 3.0M is better than $CO_2$ loading in aqueous 4.0M and 5.0M about 1.4% and 14.8%, respectively.

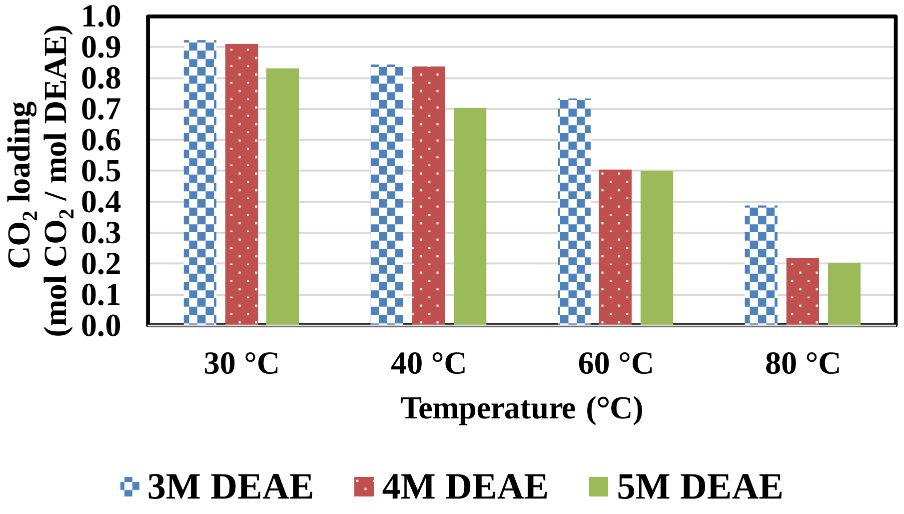


**Figure 4.9** The concentration effect on the solubility of $CO_2$ in aqueous DEAE solutions at 50 kPa $CO_2$ partial pressure

In the Figure 4.9, at 50 kPa and 80°C, the regeneration condition, the $CO_2$ loading in aqueous 3.0M is greater than $CO_2$ loading in aqueous 4.0M and 5.0M about 77.5% and 91.6%, respectively. Also, at 50 kPa and 30°C, the solubility of $CO_2$ in aqueous 3.0M is better than $CO_2$ loading in aqueous 4.0M and 5.0M about 1.2% and 10.8%, respectively.

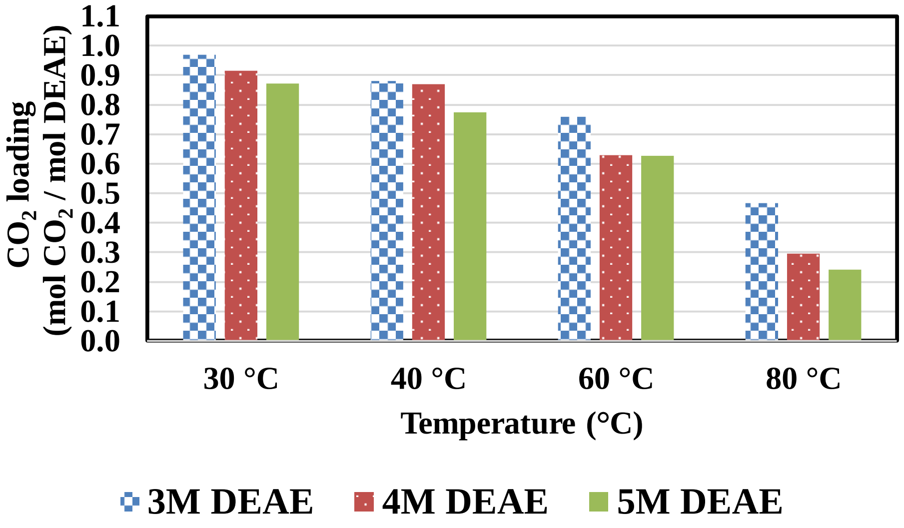


**Figure 4.10** The concentration effect on the solubility of $CO_2$ in aqueous DEAE solutions at 75 kPa $CO_2$ partial pressure

According to the Figure 4.10, at 75 kPa and 30°C, the solubility of $CO_2$ in aqueous 3.0M is greater than $CO_2$ loading in aqueous 4.0M and 5.0M about 5.8% and 11.1%, respectively. Also, at 75 kPa and 80°C, the regeneration condition, the $CO_2$ loading in aqueous 3.0M is better than $CO_2$ loading in aqueous 4.0M and 5.0M about 58.8% and 93.8%, respectively.

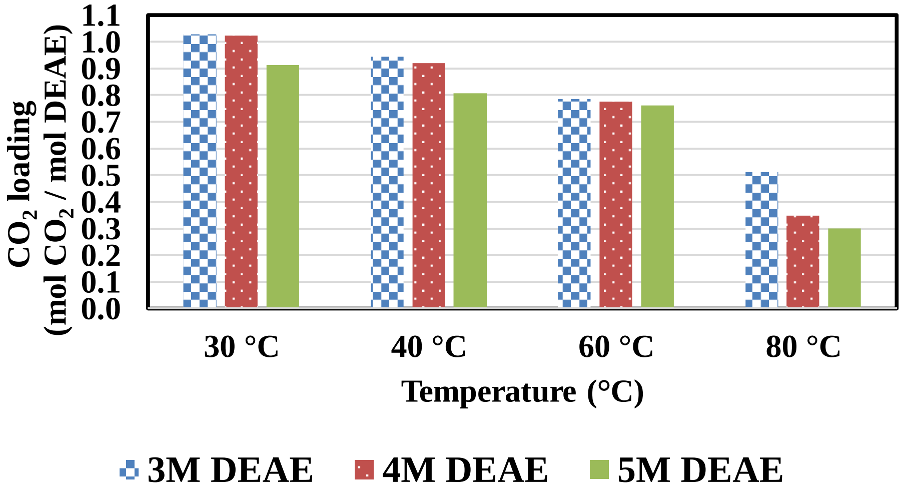


**Figure 4.11** The concentration effect on the solubility of $CO_2$ in aqueous DEAE solutions at 100 kPa $CO_2$ partial pressure

In the Figure 4.11, at 100 kPa and 30°C, the solubility of $CO_2$ in aqueous 3.0M is better than $CO_2$ loading in aqueous 4.0M and 5.0M about 0.5% and 12.6%, respectively. In the same way, at 100 kPa and 80°C, the regeneration condition, the $CO_2$ loading in aqueous 3.0M is better than $CO_2$ loading in aqueous 4.0M and 5.0M about 47.0% and 69.4%, respectively.

In the above results are shown the fact that lower concentration has a higher $CO_2$ loading in all conditions. As the DEAE concentration increased, the corresponding solubility decreased because of the $CO_2$ loading definition as shown below:

$$CO_2 loading\ (\alpha) = \frac{mole\ CO_2}{mole\ amine} \tag{4.3}$$

An increase in solution concentration is equivalent to an increase in mole of amine and free amines are added to react with $CO_2$ and mole $CO_2$ absorbed will be increased in solution. However, the denominator increases faster than numerator in the equation 4.3 because amine is excess.

### 4.3 The performance comparison of DEAE with commercial absorbents

The solubility of $CO_2$ in aqueous DEAE solution is compared to commercially used amine absorbents, MEA and MDEA. The performance comparison of DEAE as the potential absorbent will be discussed in this section.

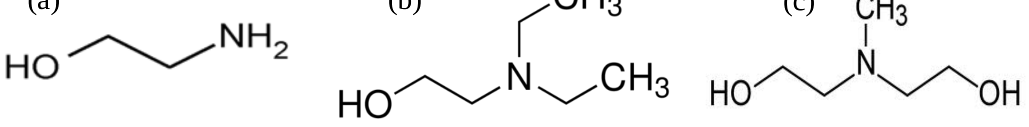


**Figure 4.12** The structure of amine absorbents: (a) MEA, (b) DEAE and (c) MDEA

The Figure 4.12 is shown the molecular structures of the different amines for this study. From the structures, they have the same backbone for $NC_2OH$. The difference among DEAE, MEA and MDEA is the substituent or functional group to the amino backbone of the amine. It leads to the changing of $CO_2$ loading capacity in aqueous amine solution [35].

### 4.3.1 The solubility of $CO_2$ in aqueous DEAE versus MEA and MDEA solutions

According to the Table 4.4, and 4.5, the solubility of $CO_2$ in aqueous 5.0M DEAE solution is compared to that of MEA solution. The $CO_2$ loading in aqueous 5.0M DEAE solution at 30°C and 40°C are less than that of aqueous MEA between 5 and 15 kPa as $CO_2$ partial pressure, which as the range of flue gas released from post combustion power plants [2]. However, at 30°C and 40°C and higher $CO_2$ partial pressure than 15 kPa, DEAE has greater $CO_2$ loading up to 49.3% and 36.9% (at 30°C and 40°C, respectively) compared to that of MEA at the same conditions. Also, at 60°C and 80°C DEAE has lower absorption capacity up to 66% and 83.6%, respectively.

The solubility of $CO_2$ in aqueous 4.0M DEAE solution is compared to that of MEA solution. The absorption capacity in aqueous 4.0M DEAE solution at 40°C are less than that of aqueous MEA at 5 kPa as $CO_2$ partial pressure. Although, at 40°C and higher than 5 kPa, DEAE has better absorption capacity up to 47.2% compared to that of MEA at the same conditions. Also, at 60°C and 80°C DEAE has lower $CO_2$ loading up to 63.5% and 82.6%, respectively.

The absorption capacity of aqueous 3.0M DEAE solution is compared to that of MEA solution and MDEA solution. The results are shown in Figure 4.13 for absorption condition (40°C) and Figure 4.14 for regeneration condition (80°C). The Figure 4.13 shows that the absorption capacity in aqueous 3.0M DEAE solution is better than that of MEA solution at 40°C. Also, the solubility of $CO_2$ in aqueous 3.0M DEAE solution is better than that of MDEA solution. At 40°C and $CO_2$ partial pressure lower than 50 kPa the $CO_2$ loading in aqueous 3.0M MEA solution is better than that of MDEA solution. However, The Figure 4.14 shows that at 80°C with $CO_2$ partial pressure between 5 and 100 kPa in term of $CO_2$ loading the winner is MEA, the runner up is DEAE and the last is MDEA respectively.

It can be seen that from the overview of the result, at the low temperature DEAE has a greater in absorption with much $CO_2$ loading and outstanding in regeneration with low $CO_2$ loading at high temperature. Because the absorption capacity for ternary amine (DEAE) is 1.0 mole of $CO_2$ per mole of amine in theory, higher than those of primary (MEA) and secondary amines where the $CO_2$ loading capacity lies between 0.5–1.0 moles of $CO_2$ per mole of amine in theory [36]. The DEAE and MDEA as ternary amine is brought to compare. The solubility of $CO_2$ in aqueous 3.0M MDEA solution is shown in Table 4.6. The absorption capacity of aqueous 3.0M DEAE solution is compared to that of MDEA solution. It can be seen that at the all conditions, DEAE has a greater in absorption with much higher $CO_2$ loading than MDEA. Because DEAE has a functional group of the substituent to the amino group of the sterically hindered amine for $CO_2$ absorption than MDEA.

**Table 4.5** The solubility of $CO_2$ in aqueous 3.0M, 4.0M and 5.0M MEA solutions

| Temperature | 30°C | | 40°C | | 60°C | | 80°C | |
|---|---|---|---|---|---|---|---|---|
| Solvent | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) |
| 5.0 M MEA | 5.00 | 0.498 | 4.90 | 0.491 | 5.20 | 0.44 | 5.10 | 0.371 |
| | 13.90 | 0.557 | 13.80 | 0.523 | 15.80 | 0.475 | 16.00 | 0.443 |
| | 31.70 | 0.560 | 30.40 | 0.532 | 31.00 | 0.513 | 30.40 | 0.456 |
| | 51.40 | 0.583 | 49.50 | 0.557 | 49.90 | 0.537 | 50.90 | 0.484 |
| | 74.30 | 0.597 | 76.10 | 0.570 | 76.10 | 0.542 | 75.80 | 0.491 |
| | 99.60 | 0.611 | 100.30 | 0.590 | 101.10 | 0.549 | 99.50 | 0.537 |
| 4.0 M MEA | 6.78 | 0.535 | 6.98 | 0.492 | 6.22 | 0.43 | 6.85 | 0.362 |
| | 15.61 | 0.561 | 15.55 | 0.543 | 15.49 | 0.460 | 15.91 | 0.422 |
| | 30.52 | 0.565 | 30.62 | 0.563 | 30.38 | 0.511 | 30.09 | 0.441 |
| | 48.99 | 0.573 | 49.88 | 0.593 | 50.38 | 0.522 | 50.64 | 0.477 |
| | 73.63 | 0.598 | 74.64 | 0.602 | 72.73 | 0.552 | 73.28 | 0.491 |
| | 97.67 | 0.635 | 99.65 | 0.625 | 98.57 | 0.562 | 99.30 | 0.520 |
| 3.0 M MEA | 5.12 | 0.530 | 5.32 | 0.498 | 4.87 | 0.45 | 6.06 | 0.372 |
| | 15.78 | 0.568 | 14.43 | 0.557 | 15.53 | 0.475 | 15.18 | 0.428 |
| | 30.59 | 0.606 | 31.21 | 0.575 | 31.36 | 0.510 | 29.93 | 0.445 |
| | 49.56 | 0.643 | 49.40 | 0.595 | 51.68 | 0.552 | 47.84 | 0.477 |
| | 76.00 | 0.673 | 74.77 | 0.611 | 73.00 | 0.563 | 74.38 | 0.494 |
| | 98.80 | 0.700 | 98.56 | 0.646 | 97.83 | 0.581 | 98.94 | 0.512 |

**Table 4.6** The solubility of $CO_2$ in aqueous 3.0M MDEA solution

| Temperature | 30 °C | | 40 °C | | 60 °C | | 80 °C | |
|---|---|---|---|---|---|---|---|---|
| Solvent | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) | $P_{CO_2}$ (kPa) | α (mol/mol) |
| 3.0 M MDEA | 4.64 | 0.264 | 5.04 | 0.218 | 5.56 | 0.100 | 5.11 | 0.072 |
| | 13.87 | 0.464 | 16.08 | 0.398 | 13.14 | 0.166 | 14.47 | 0.090 |
| | 29.55 | 0.666 | 30.01 | 0.506 | 30.14 | 0.259 | 30.25 | 0.110 |
| | 49.68 | 0.725 | 49.87 | 0.588 | 48.02 | 0.312 | 50.62 | 0.146 |
| | 74.05 | 0.748 | 74.90 | 0.684 | 73.63 | 0.408 | 75.28 | 0.189 |
| | 99.06 | 0.815 | 99.78 | 0.735 | 97.67 | 0.478 | 99.93 | 0.230 |

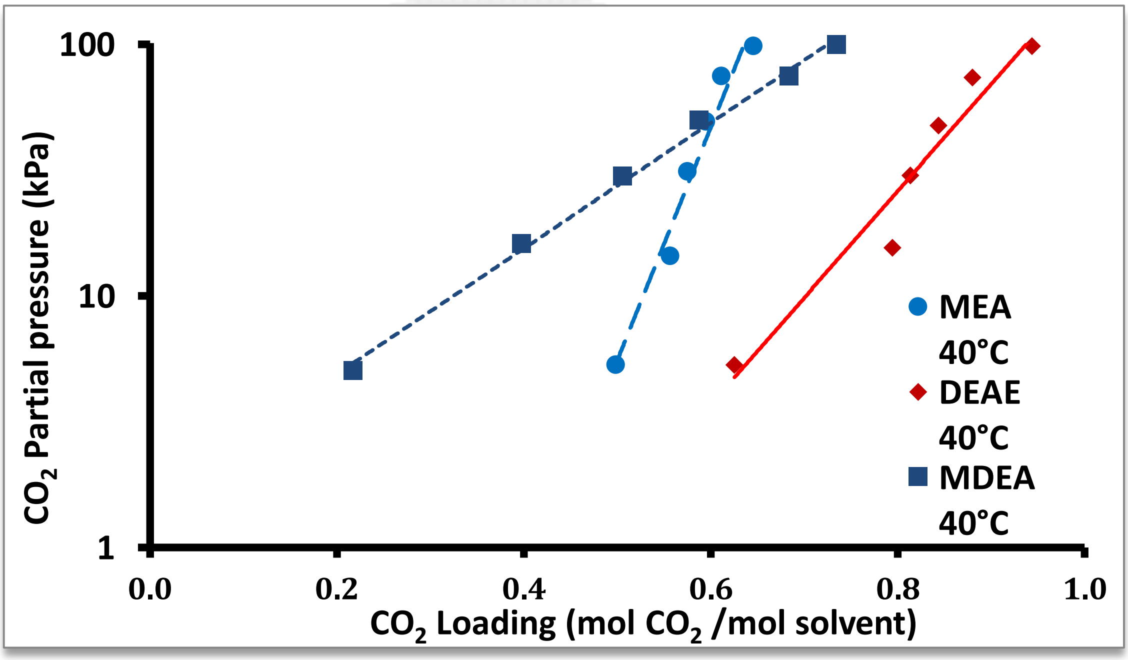


**Figure 4.13** The solubility of $CO_2$ in 3.0M DEAE solution comparing with that in 3.0M MEA solution and 3.0M MDEA solution at 40°C

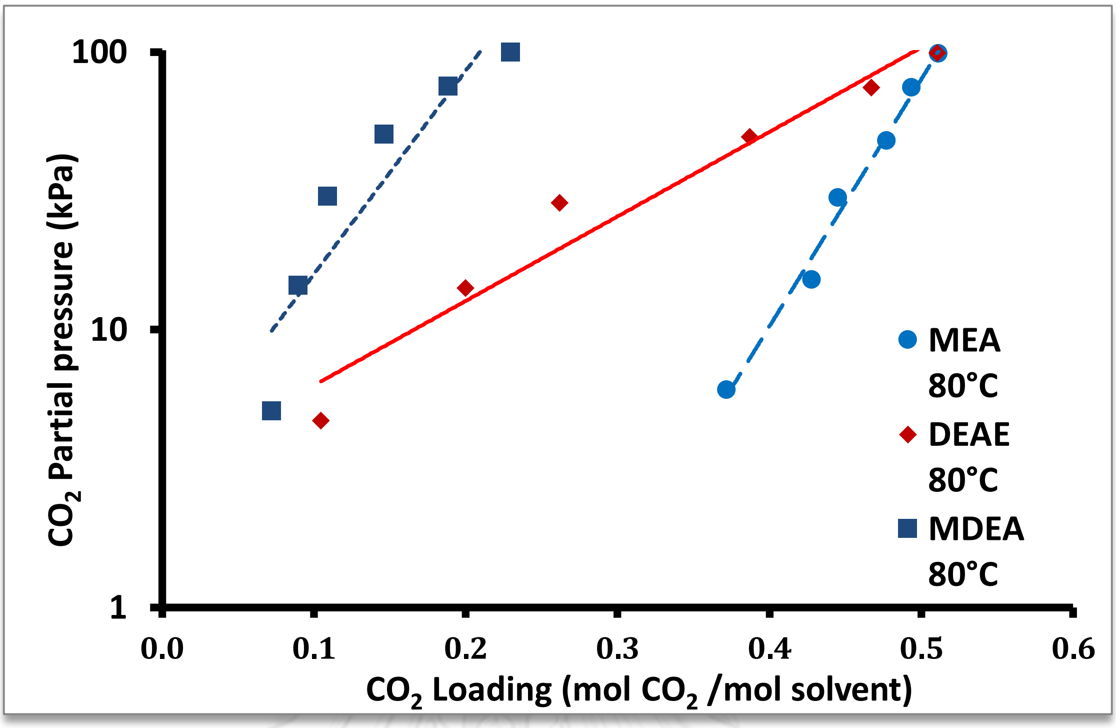


**Figure 4.14** The solubility of $CO_2$ in 3.0M DEAE solution comparing with that in 3.0M MEA solution and 3.0M MDEA solution at 80°C

### 4.3.2 The cyclic capacity of $CO_2$ in aqueous DEAE versus MEA and MDEA solutions

The cyclic capacity is the value of the difference between carbon dioxide solubility in absorption condition and carbon dioxide solubility in regeneration condition. It's the major factor to evaluate the new alkanolamine absorbent as a potential absorbent. An increase in cyclic capacity will lead to the decrease in absorbent circulation rate in the plant. The increasing cyclic capacity will reduce the overall capital cost, operating cost and lead to the decrease in carbon dioxide capture cost [13] from the following equation [15]:

$$cyclic\ capacity = \alpha_{\mathrm{abs}} - \alpha_{\mathrm{regen}} \tag{4.4}$$

When $\alpha_{\mathrm{abs}}$ is the $CO_2$ loading (mole $CO_2$/mole amine) at absorption condition.

$\alpha_{\mathrm{regen}}$ is the $CO_2$ loading (mole $CO_2$/mole amine) at regeneration condition.

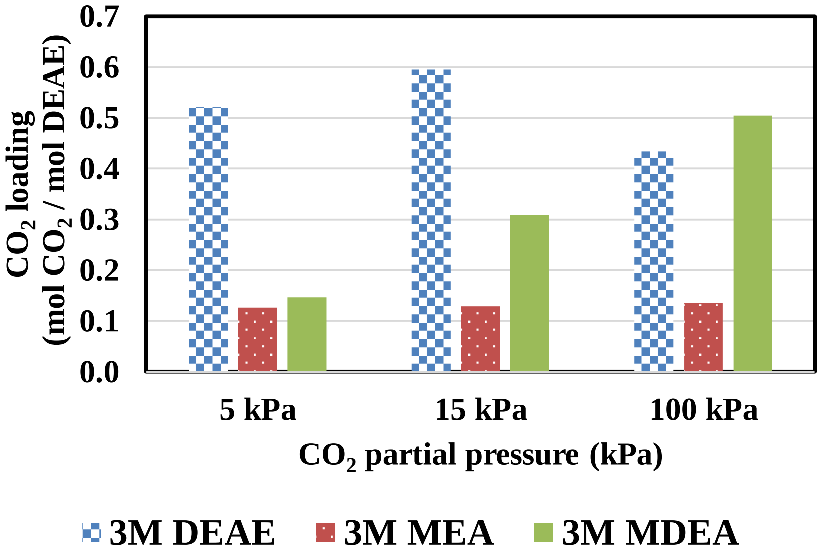


**Figure 4.15** The cyclic capacity of $CO_2$ in aqueous DEAE versus MEA and MDEA solutions

The solubility data of $CO_2$ compared to commercial amine, MEA and MDEA, at various conditions in terms of cyclic capacity are presented in Table 4.7 and Figure 4.15. From the Figure 4.15, DEAE has a much higher cyclic capacity than that of MEA and MDEA. Except at 100 kPa as $CO_2$ partial pressure, MDEA has a cyclic capacity that is 14.2% higher than DEAE at the same conditions. However, at 5 to 15 kPa, which as the range of flue gas released from post-combustion power plants DEAE has a cyclic capacity that is 257% and 92.8%, respectively than that of MDEA.

**Table 4.7** Solubility results of $CO_2$ in MEA and DEAE at 30 - 80 °C

| Temperature | 30°C | | 40°C | | 60°C | | 80°C | | Cyclic* Capacity (mol/ mol) |
|---|---|---|---|---|---|---|---|---|---|
| Solvent | $P_{CO_2}$ (kPa) | α (mol/ mol) | $P_{CO_2}$ (kPa) | α (mol/ mol) | $P_{CO_2}$ (kPa) | α (mol/ mol) | $P_{CO_2}$ (kPa) | α (mol/ mol) | |
| 3.0 M MEA | 5.12 | 0.530 | 5.32 | 0.498 | 4.87 | 0.45 | 6.06 | 0.372 | 0.126 |
| | 15.78 | 0.568 | 14.43 | 0.557 | 15.53 | 0.475 | 15.18 | 0.428 | 0.129 |
| | 30.59 | 0.606 | 31.21 | 0.575 | 31.36 | 0.510 | 29.93 | 0.445 | 0.130 |
| | 49.56 | 0.643 | 49.40 | 0.595 | 51.68 | 0.552 | 47.84 | 0.477 | 0.118 |
| | 76.00 | 0.673 | 74.77 | 0.611 | 73.00 | 0.563 | 74.38 | 0.494 | 0.118 |
| | 98.80 | 0.700 | 98.56 | 0.646 | 97.83 | 0.581 | 98.94 | 0.512 | 0.134 |
| 3.0 M DEAE | 5.07 | 0.309 | 4.89 | 0.222 | 5.10 | 0.151 | 5.12 | 0.061 | 0.161 |
| | 5.60 | 0.710 | 5.32 | 0.626 | 5.16 | 0.408 | 4.72 | 0.105 | 0.521 |
| | 15.07 | 0.841 | 15.57 | 0.795 | 16.93 | 0.509 | 14.17 | 0.200 | 0.594 |
| | 30.07 | 0.870 | 30.07 | 0.814 | 32.97 | 0.651 | 28.61 | 0.262 | 0.552 |
| | 47.55 | 0.922 | 47.55 | 0.844 | 48.34 | 0.735 | 49.43 | 0.387 | 0.457 |
| | 73.60 | 0.969 | 74.08 | 0.880 | 73.43 | 0.758 | 74.35 | 0.467 | 0.413 |
| | 98.09 | 1.027 | 98.55 | 0.944 | 96.65 | 0.785 | 99.40 | 0.510 | 0.433 |

| Temperature | 30°C | | 40°C | | 60°C | | 80°C | | Cyclic* |
|---|---|---|---|---|---|---|---|---|---|
| Solvent | $P_{CO_2}$ (kPa) | α (mol/ mol) | $P_{CO_2}$ (kPa) | α (mol/ mol) | $P_{CO_2}$ (kPa) | α (mol/ mol) | $P_{CO_2}$ (kPa) | α (mol/ mol) | Capacity (mol/ mol) |
| 3.0 M MDEA | 4.64 | 0.264 | 5.04 | 0.218 | 5.56 | 0.100 | 5.11 | 0.072 | 0.146 |
| | 13.87 | 0.464 | 16.08 | 0.398 | 13.14 | 0.166 | 14.47 | 0.090 | 0.308 |
| | 29.55 | 0.666 | 30.01 | 0.506 | 30.14 | 0.259 | 30.25 | 0.110 | 0.397 |
| | 49.68 | 0.725 | 49.87 | 0.588 | 48.02 | 0.312 | 50.62 | 0.146 | 0.442 |
| | 74.05 | 0.748 | 74.90 | 0.684 | 73.63 | 0.408 | 75.28 | 0.189 | 0.496 |
| | 99.06 | 0.815 | 99.78 | 0.735 | 97.67 | 0.478 | 99.93 | 0.230 | 0.505 |

*Cyclic capacity is the difference of $CO_2$ loading between absorption and desorption conditions. In this work, they are represented by 40°C and 80°C, respectively.

According to the results which present the absorption capacity of new solvent compared to the commercial solvents, it can be seen that at some partial pressures especially at less than 15 kPa the new solvent has lower absorption capacity than that of MEA and MDEA at 30°C and 40°C. However, in term of cyclic capacity which is more important for performance comparison, the new solvent has higher cyclic capacity than that of MEA for 312.1% and 361.3% at 5kPa and 15kPa, respectively. Also, in term of cyclic capacity the new solvent has higher cyclic capacity than that of MDEA for 257% and 92.8% at 5kPa and 15kPa, respectively. The high cyclic capacity will lead to reduce the absorbent circulation in the $CO_2$ removal process. Therefore, a smaller absorber can be applied. The increasing cyclic capacity will reduce the overall capital cost, operating cost and lead to the decrease in carbon dioxide capture cost [13].

Therefore, this can be concluded that at all conditions for removal $CO_2$ from coal-fired power plant, DEAE as a new solvent has higher performance compared to the commercial solvent, MEA and MDEA.

# CHAPTER V

# CONCLUSIONS AND RECOMMENDATIONS

## 5.1 Conclusions

In this work, the 2-(Diethylamino)ethanol (DEAE) is selected to be a new $CO_2$ absorbent and the solubility of carbon dioxide in aqueous 3.0M, 4.0M and 5.0M DEAE solutions are measured at 30°C, 40°C, 60°C and 80°C and the partial pressure of carbon dioxide ranging from 5-100 kPa.

According to the results and discussions, the reliability of this work will be proved. The $CO_2$ loading in aqueous 5.0M MEA is measured at the same operating condition with previous literature to compare the reliability of this experiment. The %AAD from 40 °C to 80 °C and $CO_2$ partial pressure from 5 kPa to 100 kPa for this experimental apparatus is 2.67% and the maximum percentage error is 11.05%. It can be concluded that the data from this work can be applicable.

According to the results, the solubility of $CO_2$ increases as partial pressure of $CO_2$ increases and the highest absorption partial pressure of $CO_2$ is 100 kPa with the $CO_2$ loading about 0.912 mole $CO_2$ / mole DEAE in aqueous 5.0M DEAE solution. In contrast, $CO_2$ loading decreases with the increases of temperature and the smallest absorption capacity temperature is 80°C with the $CO_2$ loading about 0.061 mole $CO_2$ / mole DEAE. In the same way, $CO_2$ loading decreases with the increases of concentration with the minimum $CO_2$ loading about 0.061 mole $CO_2$ / mole DEAE in aqueous 5.0M DEAE solution too.

The novel absorbent, DEAE has a higher cyclic capacity than that of MEA and MDEA at conditions for removing $CO_2$ from post combustion power plant. It can be concluded that DEAE has an outstanding performance in term of cyclic capacity leading to the reduction of liquid circulation rate and decrease in energy consumption for absorbent regeneration. Finally, this can reduce the cost in $CO_2$ removal process from post- combustion power plant.

### 5.2 Recommendations

The novel absorbent, DEAE is the great absorbent which in term of solubility. However, there are many factors that need to investigate such as kinetic of absorption, corrosion, solvent degradation, viscosity and mass transfer. Therefore, the method to study the factors should be considered in future work.

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# APPENDIX

# APPENDIX A

# CALCULATION OF THE $CO_2$ LOADING

## Calculation of the $CO_2$ loading by titration

The $CO_2$ loading will be calculated by measure the $CO_2$ that is released from sample solution with 1.0N HCl titration. And the equation for calculate the $CO_2$ loading is shown in A.1.

$$CO_2 loading = \frac{(V_{CO_2}*273)}{(V_{HCl}*1*22.4*298)} \tag{A.1}$$

Where $CO_2$ loading = mole $CO_2$ / mole amine

22.4 = mole of gas / volume at Standard Temperature and Pressure (mole $CO_2$ / L)

273 = temperature at STP condition (K)

298 = temperature at ambient condition (K)

1 = mole of amine / volume (mole amine / L)

$V_{CO_2}$ = volumetric of $CO_2$ in the sample solution (mL)

$V_{HCl}$ = volumetric of HCl in the sample solution (mL)

Example: Calculation of $CO_2$ loading

Data from titration: $V_{CO_2}$ (mL) = 126

$V_{HCl}$ (mL) = 5.6

The $CO_2$ loading = $\frac{(126*273)}{(5.6*1*22.4*298)}$ = 0.920 mole $CO_2$ / mole amine

# APPENDIX B

# LIST OF PUBLICATIONS

Pattara Boonpipattanapong, Kreangkrai Maneeintr and Suttichai Assabumrungrat "Solubility of Carbon Dioxide in a 5M Aqueous 2-(Diethylamino)Ethanol Solution" Proceedings of the International Conference on Business and Industrial Research 2014, Thai-Nichi Institute of Technology, Bangkok, Thailand, May 15-16, 2014.

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