

ELUCIDATION OF THE EFFECT OF OPERATING CONDITIONS
ON THE FOAMING BEHAVIOR OF METHYL DIETHANOLAMINE
(MDEA) PIPERAZINE SOLVENT BLEND USED FOR CO₂
ABSORPTION IN THE PRESENCE OF BICINE

NOOR SARA ANISAH BINTI AZMI

CHEMICAL ENGINEERING
UNIVERSITI TEKNOLOGI PETRONAS

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Of Methyl Diethanolamine (MDEA) Piperazine Solvent Blend Used For CO₂
Absorption In The Presence Of Bicine**

by

Noor Sara Anisah Binti Azmi

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Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

JANUARY 2021

Universiti Teknologi PETRONAS,
32610, Bandar Seri Iskandar,
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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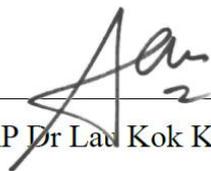
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Chemical Engineering Programme
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in partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(CHEMICAL ENGINEERING)

Approved by,



(AP Dr Lau Kok Keong)

UNIVERSITI TEKNOLOGI PETRONAS
BANDAR SERI ISKANDAR, PERAK

January 2021

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.



NOOR SARA ANISAH BINTI AZMI

ABSTRACT

Foaming of amine solutions is a major problem for amine gas sweetening plants. However, studies on the effect of operating conditions on foaming behavior are still limited in literature. Therefore, this project aims to study the effect of temperature and gas flowrate on foaming behavior in Methyl Diethanolamine (MDEA) Piperazine solvent in the presence of Bicine. A bench scale experimental set-up was used to conduct the experiment with temperature ranging from 40 °C - 90°C and gas flowrate ranging from 1-5 SCFH. The aqueous amine solvent blend includes 25 wt% of MDEA and 5 wt% of PZ. A degradation product which is Bicine was added into the blend to initiate foaming. The foaming behavior was analyzed in terms of foam height which was characterized as foaminess coefficient (Σ) and foam stability. From the experiment, it was found that the increase in temperature did not affect the foaminess coefficient of the MDEA+PZ solution as not much foam was formed. However, the increase in temperature showed a decrease in foam stability. Aside from that, the increase in gas flowrate showed a decrease in foaminess coefficient and an increase in foam stability. From this study, optimizing the operating conditions showed high potential of reducing foaming in amine solvents.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The emission of greenhouse gases such as carbon dioxide (CO₂), fluorinated gases (F-gases), nitrous oxide (N₂O) and methane (CH₄) has always been a global concern. CO₂ makes up the largest percentage of the greenhouse gases which is produced mainly from the burning of fossil fuels for energy. It is estimated that worldwide emissions of CO₂ from fossil fuel will increase up to 40.2 GT (Gross Tonnage) by the year 2030 (Khan, Hailegiorgis, Man, & Shariff, 2018). Due to the increasing demand of energy through the burning of fossil fuels, more CO₂ is being released to the atmosphere, which is contributing to climate changes, melting of glaciers, rising of sea levels and other concerning phenomenon.

Globally, the power production sector is ranked first among stationary CO₂ producers, followed by the cement industry and the refining industry (Straelen, Geuzebroek, Goodchild, Protopapas, & Mahony, 2009). The iron and steel industry and petrochemicals industry are also large producers of CO₂. CO₂ emitted at refineries comes from a variety of sources which includes furnaces and boilers, utilities, fluid catalytic cracker and hydrogen manufacturing. Several technologies have been developed to remove CO₂ in refineries through the process of absorption, adsorption, distillation, membrane separation and hydrates. One of the most widely used strategy is by means of chemical absorption. Presently, amine scrubbing is a well-established technique commercially utilized in which aqueous alkanolamines and their mixtures are widely used in absorption process (Khan, Hailegiorgis, Man, Shariff & Garg, 2016). Amine scrubbing or also known as amine gas sweetening process are usually

utilized in natural gas processing plant or refineries to remove sour gases, H₂S and CO₂. Aqueous alkanolamines such as monoethanolamine (MEA), 2-amino-2-methyl-1-propanol (AMP), diethanolamine (DEA), di-isopropanolamine (DIPA), N-methyl-diethanolamine (MDEA), and triethanolamine (TEA), are some of the widely-used chemical absorbents to remove H₂S and CO₂ (Chowdhury, Yamada, Higashii, Goto & Onoda (2013). However, MDEA is commonly used in gas sweetening process as it is proven in literature to have a few benefits during operation, compared to other solvents which will be further discussed in the literature review.

Just like any other plant operations, operating an amine gas sweetening plant will come with its' sets of problems. One of the greatest operational problems in amine absorption process is foaming. Foaming can be triggered by various contaminants such as corrosion inhibitors, amine degradation products and excessive antifoam agent (Keewan, Banat, Alhseinat, Zain, & Pal, 2018). Some of the corrosion inhibitors studied on includes fatty acid-based (Bis(2-Hydroxyethyl) cocoalkylamine; BHCL) and hydrocarbon-based (HCB) corrosion inhibitors. While for amine degradation products, they include, bicine, ammonium thiosulfate, hydrochloric acid, acetic acid and others as stated in Thitakamol & Veawab's (2008) study. Process parameters such as solution temperature, solution volume, gas flow rate, alkanolamine concentration, CO₂ loading of solution, and alkanolamine type could also affect foaming behavior as studied by Thitakamol & Veawab, (2008).

Based on plant experiences, foaming was reported to cause unfavourable impacts on the integrity of plant operation which will lead to an increase in capital investment and operation cost (Thitakamol and Veawab, 2008). Unfortunately, research on foaming behavior in amine solvents are still limited in literature and are mostly reported through plant experience. Therefore, there is a need in more research regarding foam behavior in amine solvent.

1.2 Problem Statement

It is widely known that foaming in amine solvent is a major operational problem in amine gas sweetening plant. Foaming will lead to excessive loss of absorption solvents, premature flooding, decrease in plant throughput, off-specification of products and high absorption solvent carryover to downstream plants (Thitakamol & Veawab, 2008).

In the natural gas processing plants and refineries, foaming in amine gas sweetening process is usually mitigated with the use of anti-foaming agents, however it is found that anti-foaming agent appears to become less effective over time which is thoroughly explained by von Phul & Cummings (2007).

Studies on foaming behavior, especially on the effect of operating parameters, temperature and gas flowrate in amine solvents are limited in literature. Foaming behavior are mostly reported from operator experience and presented as technical review while experimental-based research remains limited (Thitakamol & Veawab, 2008).

Although there are several studies that were done to investigate the effect of temperature on the foam height, they showed contradicting results when compared to each other. The varying results could stem from a variety of different factors that could affect the foaming behavior, alongside temperature. Moreover, only two studies studying the effect of gas flowrate on foam height were found in literature where both studies also showed contradicting results. Aside from that, the effect of bicine as the degradation product on foam height is also still limited in literature.

The lack of experimental based research and concrete information on the foaming behavior in amine solvent signifies how important it is to study the foaming behavior in amine solvent.

1.3 Objectives

The objectives of this project include the following:

- i. To evaluate the effect of different temperature on the foam height and foam stability in N-methyl diethanolamine (MDEA) Piperazine solvent blend in the presence of Bicine.
- ii. To evaluate the effect of different gas flowrate on the foam height and foam stability in N-methyl diethanolamine (MDEA) Piperazine solvent blend in the presence of Bicine.

1.4 Scope of Study

The purpose of this study is to investigate the effect of temperature and gas flowrate on the foaming behavior in MDEA solvent with the use of a bench-scale experimental set up. The ranges of temperature and gas flowrate that will be varied throughout the experiment will be between 40-90 °C, which represents the operating conditions in an absorber column (Thitakamol & Veawab, 2008) and 1-5 SCFH respectively.

The pressure, solution volume and solution concentration were held constant throughout the experiment to eliminate their effect on the foaminess coefficient. Thitakamol & Veawab (2008) found that adding degradation products or corrosion inhibitors in amine solutions, increased foaming tendency up to 23% as clean amine solutions did not foam. Therefore, bicine which is a degradation product of amine, with concentration of 20,000 ppm, was used as the foaming initiator throughout the experiment.

The foam height in amine solvent will be calculated using a foaming model that was derived from the Bikerman model (Thitakamol & Veawab,2008) The foam height will be represented as foaminess coefficient in the result and discussion section.

CHAPTER 2

LITERATURE REVIEW

2.1 CO₂ Capture Technologies

In efforts of satisfying the rising global demand for energy while reducing environmental impacts such as greenhouse gas (GHG) emissions, mitigation technologies have been developed. With CO₂ being the major contributor to GHG emission, this section will emphasize the current technologies available for CO₂ capture.

As stated by Johansson (2013), there are in principle, four options for CO₂ capture which are, pre-combustion processes, post combustion processes, oxyfuel combustion and chemical-looping combustions. Presently, most focus is on post combustion as it is deemed to be the only feasible option in a short-term perspective. (Johansson, 2013). Post combustion process includes separating CO₂ from flue gas or fuel gas stream by processes such as absorption, adsorption, membrane separation, chemical looping, hydrate-based separation, biochemical methods and cryogenic distillation (Jinadasa, Jens & Halstensen, 2018).

Currently, the most mature technology for CO₂ capture is absorption process as the basic technology is well known (Jindasa et. al, 2018). This is also supported by Quintella, Hatimondi, Musse, Miyazaki, Cerqueira & de Araujo Moreira (2011), stating that absorption and adsorption are the most used capture technologies, having more patent applications and articles published than other capture technologies. In addition to that, Rochelle (2009) stated, as cited by Yu, Huang & Tan (2012) that chemical absorption using aqueous alkanolamine solutions is proposed to be the most

applicable technology for CO₂ capture before 2030. Hence, the following sections of this literature review will be highlighting CO₂ capture using alkanolamine solutions.

2.2 CO₂ Capture Using Alkanolamines

2.2.1 Types of Alkanolamines

There are several types of alkanolamines that are commercially utilized. They are classified into 3 categories which are primary, secondary and tertiary. The types of alkanolamines are listed in Table 2.1.

Table 2.1: Types of Alkanolamines

Category	Types of Alkanolamines
Primary	Monoethanolamine (MEA)
	2-amino-2-methyl-1-propanol (AMP)
Secondary	Diethanolamine (DEA)
	Di-isopropanolamine (DIPA)
	Piperazine (PZ)
Tertiary	N-methyl-diethanolamine (MDEA)
	Triethanolamine (TEA)

Recently, MDEA+PZ blend has become the more preferred amine solvent compared to other amines as it has proven to be beneficial for regeneration process. It is deemed favorable due to its' high thermal stability, high CO₂ equilibrium loading capacity of up to 1.0 mole of CO₂ per mole of amine, low vapor losses and low enthalpy of reaction with CO₂ (Khan et. al, 2018).

It is well known that one of the major problems with the use of alkanolamines in an amine gas sweetening plant is foaming in the absorber column. Foaming will pose adverse impacts on the integrity of the plant. Thus, foaming in alkanolamines will be further discussed in the following sections of this literature review.

2.3 Foam Theory

2.3.1 Foam Characteristics

Foam is a colloidal system with agglomeration of closed gas bubbles being dispersed in a liquid where each bubble is separated by a thin liquid film, called lamella (Thitakamol & Veawab, 2008). Foams can be categorized into two types which are Kugelschaum and Polyederschaum.

Initially, the Kugelschaum or also known as sphere foam is produced in freshly prepared systems and consists of small roughly, spherical bubbles (Pugh, 2005) separated by the lamella (liquid film between the bubbles) which has a thickness approximately equal to the diameter of the gas bubble (Thitakamol & Veawab, 2008). Pugh (2005) states that initially, sphere foam is formed and dispersed as dilute bubbles in the liquid before the structure gradually changes into polyhedral gas cells with thin flat walls known as the polyederschaum.

According to Thitakamol & Veawab (2008), polyederschaum is produced when there is drainage of liquid in the lamella making the lamella thinner. A Plateau border (PB) will be formed when the film walls drain until they meet at 120° forming a junction point of the interconnecting channels (Pugh, 2005). As a result, a honeycomb network of bubbles is formed and allows liquid to flow around the interconnected PB structure. Polyederschaum is located between Kugelschaum and the gas phase and is prone to foam coalescence and rupture. Figure 2.1 shows the (a) structure of foam and (b) the Plateau border, adapted from Pugh (2005).

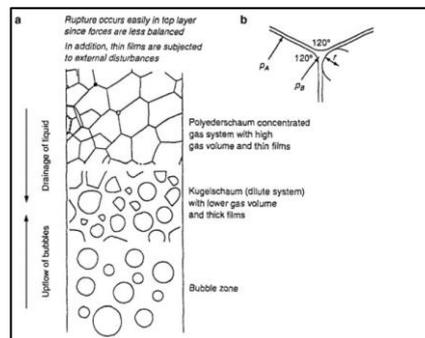


Figure 2.1: (a) Structure of foam in a column and (b) Plateau borders

2.3.2 Foam Mechanism

As described in the literature work by Thitakamol & Veawab (2008), buoyancy, surface and hydrostatic force are vital principle forces in foam formation. Foam is formed by purging gas into liquid through a diffuser or an orifice. The buoyancy force (F_{buoy}) will lift a bubble from the diffuser through the bulk liquid which is a function of density difference between liquid and gas (ρ), bubble volume (V_{bub}) and gravitational acceleration (g) which can be expressed as shown in equation (1):

$$F_{buoy} = \rho V_{bub} g \quad (1)$$

In order to free the bubble from the diffuser, buoyancy force must surpass the hydrostatic force and the surface force (F_{surf}) which is produced by surface tension of liquid solution (γ) and capillary perimeter (l), as expressed in equation (2):

$$F_{surf} = \gamma l \quad (2)$$

When the capillary force increases, liquid will flow from the lamella to the PBs which will lead to thin lamella thickness and foam rupture due to acceleration of foam drainage. Aside from capillary force, gravitational and hydroequilibrium force also causes drainage.

2.3.3 Foam Stability

According to Thiele, Brettschneider, Repke, Thielert & Wozny (2003), foam stability is subject to two different processes which are film thinning and coalescence. Coalescence reduces the total surface area and thus decreasing surface energy which means the foams are thermodynamically unstable. However, it is the opposite for foam stability affected by surface elasticity, Marangoni effect, surface and bulk viscosity, repulsive Coulombic force and gravitational force (Thitakamol & Veawab, 2008).

The ability of a surface to resist thinning process due to a surface tension gradient is called surface elasticity (E). In other words, it is a change in surface tension with respect to a change in surface area (A) which can be expressed by equation (3):

$$E = 2A(d\gamma/dA) \quad (3)$$

The Marangoni effect can be defined as the phenomenon that the surface tension gradient causes a liquid flow in the lamella. It provokes a surface flow from the non-thinned (low surface tension) area to the thinned (high surface tension) area, which works against drainage and restores the film (Thiele et. al, 2003).

In addition to that, bulk viscosity and surface viscosity also play an important role in foam stability. In general, high bulk viscosity is favorable since it will slow down the drainage due to gravitational force which in turn, slows down the formation of foam (Thitakamol & Veawab, 2008). However, an increase in bulk viscosity can lead to a very high increase in surface viscosity and eventually destroying surface elasticity. Consequently, it will decrease foam stability. Other external forces that affect foam stability include, Coulombic forces which slows down the gravity drainage and the gravitational force which does the opposite.

2.4 Foaming in Alkanolamines

2.4.1 Physical Properties Affecting Foaming Behavior

Foaming behavior in alkanolamines are usually characterized by studying the effect of different variables to the foam volume (which also represents foam height) and foam stability. To obtain a better understanding on the foam behavior, there are several physical properties that is studied in conjunction with the foam volume and foam stability in alkanolamine. The physical properties include, solution viscosity, solution density and surface tension.

Generally, it is found that the higher the solution viscosity, the higher the tendency for the solution to foam. A study done by Thitakamol & Veawab (2008) shows that increasing the concentration of MEA solution, increased bulk viscosity which retards the foam collapse caused by gravity drainage. In turn, the foam height increases due to faster rate of drainage. The increase in bulk viscosity also enhanced the foam stability which also increases the foam height. Alhsenait, Pal, Ganesan & Banat (2015) also came to the same conclusion in their study about the effect of degradation products on foaming in MDEA solution. It was found that, as the number of carbon atoms in an organic acid added to the solution increased, the foam height increased due to the increase in surface viscosity.

Besides solution viscosity, solution density affects foam height as well. From Sedransk Campbell, Lapidot, & Williams (2015) literature work on foaming in CO₂-loaded amine solvents degraded thermally under stripper conditions, an increase in solution density resulted an increase in foam thickness. Similar result was found by Alhsenait et. al (2015). Thitakamol & Veawab (2008) also obtained the same findings where they studied the effect of CO₂ loading on the foam height. As CO₂ loading increases, the density of the solution increases which results in an increase in foam height. This is due to the increase in buoyancy force of the bubbles, thus promoting foam formation.

Furthermore, surface tension plays a big role in promoting foam formation. Several literatures such as literature work by Alhsenait et. al (2015) and Keewan et. al (2018), have associated the increase in foaming with the decrease in surface tension. According to Thiele et. al (2003) surface tension causes a pressure difference across a curved surface (surface of the bubble), with a higher pressure on the inside of the bubble. Due to the pressure difference, a capillary flow to the Plateau borders will lead to thinning and rupture of the lamellae and causing foam collapse. Thus, the higher the surface tension, the lesser the tendency for the solution to foam.

2.4.2 Effect Of Operating Parameters On Foaming Behavior

Operating parameters temperature and gas flowrate are usually varied in studying foaming behavior as they show significant effect on foaming. The effect of temperature and gas flowrate has been studied by Thitakamol & Veawab (2008) and Keewan et. al (2018) as well as Sedransk Campbell, Lapidot & Williams (2015).

Thitakamol & Veawab (2008) studied the effect of temperature on foaming height in 5 kmol/m³ MEA solution loaded with 0.20 mol/mol and 0.4 mol/mol CO₂. From their study, they found that as the solution temperature increases from 40°C to 90°C, the foam height decreases considerably. This is due to the poor foam stability which is caused by reduced bulk viscosity and the vigorous movement of the molecules at elevated temperature. It was also found that as temperature increases, surface tension and density decreased, which would enhance foam formation. However, the effect of surface tension and density was regarded as insignificant compared to the effect of viscosity.

Interestingly, the study done by Keewan et. al (2018) shows contradicting results to Thitakamol & Veawab's work. Keewan et. al (2018) did a comparative study on the foaming tendency in 50 wt % MDEA in the presence of fatty-acid based and hydrocarbon based corrosion inhibitors. The effect of temperature was also investigated on both corrosion inhibitors and it was found that as temperature increased from 15°C to 75°C, the foam volume increases. This was mainly influenced by the decrease in surface tension as opposed to what Thitakamol & Veawab have stated. However, McCarthy & Trebble (1996) and Yanicki & Trebble (2006) obtained similar results to the study done by Keewan et al.

Studies done on the effect of gas flowrate on the foam height in amine solvent is found to be very limited in literature. Only 2 studies were found on the effect of gas flowrate which were conducted by Thitakamol & Veawab (2008) on MEA 2.0 and 5.0 kmol/m³ and Sedransk Campbell et al. (2015). Both studies also showed contradicting results where the former's work showed a decrease in foam height with an increase in gas flowrate while the latter showed the opposite. Thus, more investigation on the

effect of gas flowrate on foam height should be conducted to obtain a comprehensive understanding on its' effect with respect to different external variables.

2.4.3 Effect Of Additives On Foaming Behavior

Aside from operating parameters, the effect of different types of additives have been investigated on foaming behavior in alkanolamines. The additives include corrosion inhibitors and amine degradation products.

Since gas sweetening plants are very susceptible to corrosion, corrosion inhibitors are used to protect metallic surfaces from corrosion in amine systems (Keewan et. al, 2018). It is believed that corrosion inhibitor is one of the major contributor to foaming in amine gas plant. Keewan et. al (2018) investigated the effect of corrosion inhibitors on foaming behavior in aqueous MDEA solution. The corrosion inhibitors that were studied on include fatty acid based (Bis(2-Hydroxyethyl) cocoalkylamine; BHCL) and hydrocarbon based (HCB) corrosion inhibitors. They found that as the concentration of corrosion inhibitor increases, both foamability and stability increases. However, when nitrogen flowrate increases, the foam height in MDEA solution which contains HCB increases but it is the opposite for the solution that contains BHCL. Therefore, it can be said that different types of corrosion inhibitors will affect foam height differently, depending on the type of variable that was varied.

In another study by Alhsenait et. al (2015), the effect of MDEA degradation products on foaming behavior in MDEA solution were investigated. The MDEA degradation products include, N,N,N-tris-(hydroxyethyl), ethylenediamine (THEED), hydroxyethyl ethylenediamine (HEED), N,N'-bis-(hydroxyethyl), piperazine (bHEP), N,N-bis-(2-hydroxyethyl), glycine (bicine), acetic acid and octanoic acid. From the study it was found that there was an increment in foam volume and foam stability when there is an addition of THEED and HEED. However, bHEP's and bicine's effect on foam volume was insignificant but greatly affected the increase in foam stability. Octanoic acid and acetic acid increased foam volume and breaking time where

octanoic acid had a greater influence compared to acetic acid due to the greater decrease of surface tension by octanoic acid. In addition to that, mixing THEED and HEED decreased the increase in foam volume and breaking time as compared to separately adding both degradation products. Generally, all the degradation products studied, increased the surface tension and viscosity of the solution. Hence, a more detailed evaluation should be done for different types of degradation products in order to obtain a better understanding regarding foaming behavior.

2.5 Research Gap

Based on detailed literature review, literature on foaming behavior in amine solvents remains limited to this day even though foaming is a major problem in amine gas sweetening plant. As discussed previously, there are already several studies on the effect of temperature on foaming behavior in amine solvent. However, some of the studies show contradicting results to each other where it could be due to the different variables involved during the experimental runs. Thus, there is still no concrete understanding about the effect of temperature on the foaming behavior. In addition to that, only 2 studies were found for the effect of gas flowrate on the foam height where each study experimented on different types of alkanolamine solution also showed contradicting results. Moreover, the effect of bicine as a foaming initiator on foaming behavior is also still limited and need more studying upon as it is one of the degradation products in amine. The summary for literature works done on foaming behavior in alkanolamine solution and details on variables that were experimented on is listed in Table 2.2.

The limited literature work, especially experimental-based studies on the effect of temperature and gas flowrate in the presence of bicine, will further drive the purpose to continue this project. This project will be focused on studying the effect of temperature and pressure on the foaming behavior in MDEA+PZ blend where the experimental procedure will be covered in the methodology section.

Table 2.2: Summary of literature works on foaming behavior in alkanolamine solution.

Reference	Details
<p>McCarthy, J., & Trebble, M. A. (1996). An experimental investigation into the foaming tendency of diethanolamine gas sweetening solutions. <i>Chemical Engineering Communications</i>, 144(1),159-171.</p>	<p>Experimental based research</p> <ul style="list-style-type: none"> ▪ Temperature: 20 – 85 °C ▪ Pressure: 0.1 – 3 MPa ▪ Alkanolamine type: DEA ▪ Degradation product: organic acids and HEP. ▪ Additive: antifoam agent & corrosion inhibitor
<p>von Phul, S. A., & Cummings, A. L. (2007). Control of foaming in amine systems.</p>	<p>Technical review</p> <ul style="list-style-type: none"> ▪ Use of chemical anti-foam ▪ Emphasis on removing foam-causing agents.
<p>Thitakamol, B., & Veawab, A. (2008). Foaming behavior in CO₂ absorption process using aqueous solutions of single and blended alkanolamines. <i>Industrial & engineering chemistry research</i>, 47(1), 216-225.</p>	<p>Experimental based research</p> <ul style="list-style-type: none"> ▪ Solution volume ▪ Gas flowrate ▪ MEA concentration ▪ CO₂ loading ▪ Temperature: 40 - 90°C. ▪ Different types of blended alkanolamines ▪ Addition of degradation products (includes bicine)
<p>Chen, X., Freeman, S. A., & Rochelle, G. T. (2011). Foaming of aqueous piperazine and monoethanolamine for CO₂ capture. <i>International Journal of Greenhouse Gas Control</i>, 5(2), 381-386.</p>	<p>Experimental based research</p> <ul style="list-style-type: none"> ▪ Different concentration of Piperazine (PZ) ▪ Additives: corrosion inhibitors & hydrocarbons
<p>Alhseinat, E., Pal, P., Ganesan, A., & Banat, F. (2015). Effect of MDEA degradation products on foaming behavior and physical properties of aqueous MDEA solutions. <i>International Journal of Greenhouse Gas Control</i>, 37, 280-286.</p>	<p>Experimental based research</p> <ul style="list-style-type: none"> ▪ Addition of MDEA degradation products (includes bicine) ▪ Study on physical properties such as density, surface tension & viscosity.

<p>Keewan, M., Banat, F., Alhseinat, E., Zain, J., & Pal, P. (2018). Effect of operating parameters and corrosion inhibitors on foaming behavior of aqueous methyldiethanolamine solutions. <i>Journal of Petroleum Science and Engineering</i>, 165, 358-364.</p>	<p>Experimental based research</p> <ul style="list-style-type: none"> ▪ Addition of fatty-acid based (BHCL) corrosion inhibitors. ▪ Addition of hydrocarbon based (HCB) corrosion inhibitors
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CHAPTER 3

METHODOLOGY

3.1. Project Flowchart

In this project, a bench-scale experimental set up were used to investigate the effect of temperature and gas flowrate on the foaming behavior in MDEA+PZ solvent blend. After reviewing several literatures on foaming behavior in amine solvent, the ranges of temperature of 40°C - 90°C and gas flowrate of 1-5 SCFH to be experimented on was obtained. The type of foam initiator selected was Bicine and its concentration of 20,000 ppm was also decided upon from literature review.

An initial study was done on the theory of foam to grasp the basic principles in foaming behavior such as the different types of physical properties that has a direct effect on foaming behavior. A detailed literature review was then conducted to study the works related to foaming behavior in alkanolamine solvents that is being carried out by other researchers. Then, a research gap was produced to determine the problem statement and objectives. Next, the type of alkanolamine used and type of foaming agent and its concentration were determined as previously mentioned. The ranges of temperature and gas flowrate were also determined to study its effect on foaming behavior. The experimental procedure was then developed by referring to past literature works which will be discussed further in the next part. After conducting the experiment, the foam height and foam stability was recorded and analyzed. Lastly, the conclusion was developed. Figure 3.1 shows the project flowchart as explained.

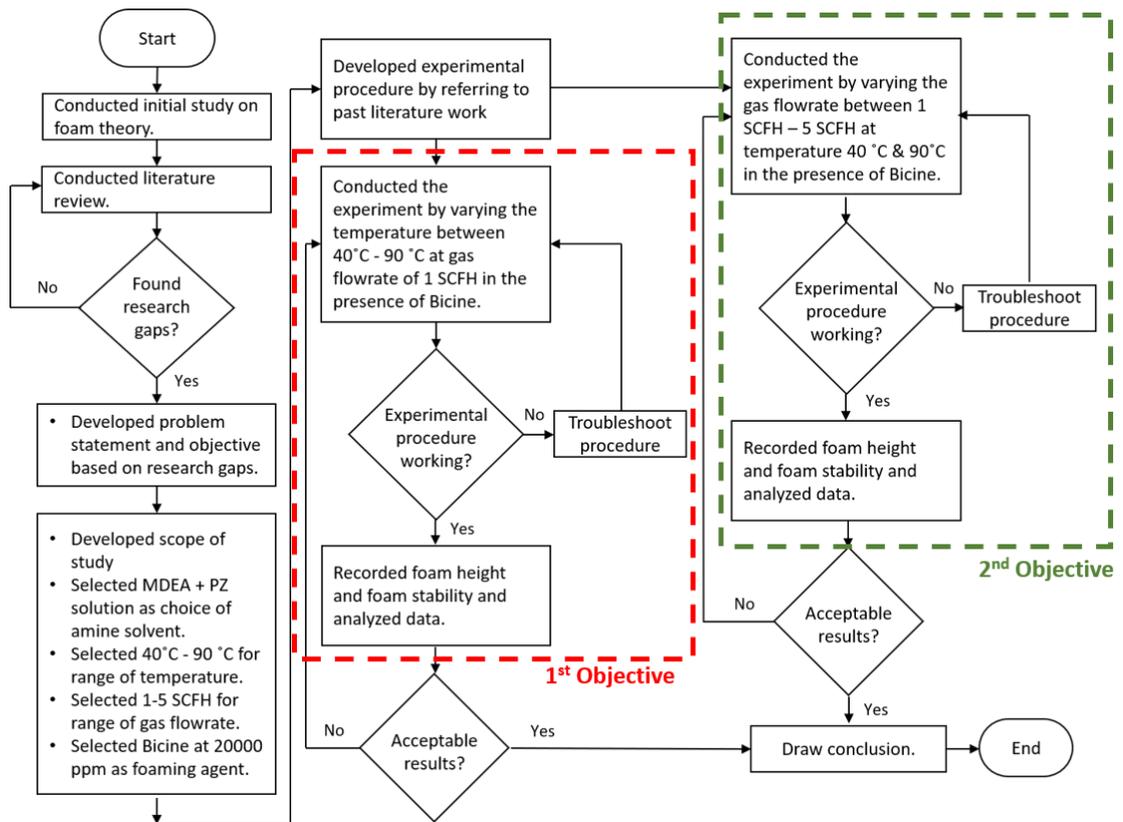


Figure 3.1: Project Flowchart

3.2 Experimental Procedure

3.2.1 CO₂ Loading Calibration Curve

Prior to the experiment, a sample of alkanolamine solution was loaded with CO₂ gas to develop a calibration curve for CO₂ loading. The calibration curve was developed to predict the concentration of CO₂ in the alkanolamine solution at different absorption time. 400 mL of the alkanolamine solution is bubbled by CO₂ at ambient temperature and pressure, for 150 mins where every 30 mins, the sample was taken to determine the CO₂ loading. Figure 3.2 shows the experimental set-up for CO₂ loading.

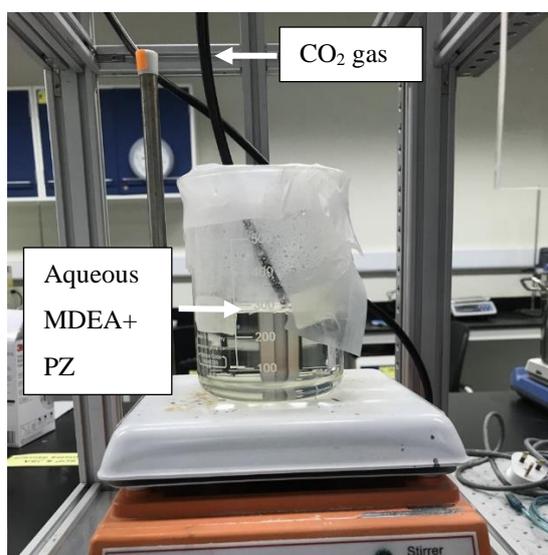


Figure 3.2: Experimental set-up for CO₂ loading

The alkanolamine solution used is a blend of 25 wt% MDEA and 5 wt% PZ, amounting to 30 wt% of aqueous MDEA+PZ solution (Khan, Halder & Saha, 2017). The method used to determine CO₂ content and CO₂ loading is titration. The steps used for titration follows the work done by Phak, Saleh & Muzammil (2018) as explained below.

Preparation of Test Solution

1. Prepare the 25 wt% MDEA and 5 wt% PZ of aqueous solution by diluting the reagent alkanolamine with deionized water.
2. Determine the solution concentration by using the titration method described as the following:

1. Preparation of Potassium Hydroxide (KOH) Solution

1. Dissolve 66g of KOH pellet completely using distilled water.
2. Diluted KOH solution in 2L volumetric flask.
3. Dissolve 1g of potassium hydrogen phthalate (KHP) in 40ml distilled water.
4. Add 6 drops of phenolphthalein into KHP solution.
5. Titrate the KHP solution with prepared KOH solution until the solution turns pink.
6. The normality of KOH will be calculated as shown in Equation (4).

$$N_{KOH} = \frac{W_{KHP}}{204.22} \times \frac{1000}{V_{KOH}} \quad (4)$$

Where,

W_{KHP} = Weight of KHP (g)

N_{KOH} = Normality of KOH Solution (mol/L)

V_{KOH} = Volume of KOH after titration (mL)

2. HCl Preparation

1. Dilute 84ml of concentration HCl (37%) in a 2L volumetric flask with distilled water.
2. Add 6 drops of phenolphthalein.
3. Titrate 25ml of KOH until solution turn colourless.
4. The normality of HCL will be calculated as shown in Equation (5).

$$N_{HCl} = \frac{V_{KOH} \times N_{KOH}}{V_{HCl}} \quad (5)$$

Where,

N_{HCL} = Normality of HCL (mol/L)

V_{KOH} = Volume of KOH solution (ml)

N_{KOH} = Normality of KOH Solution (mol/L)

V_{HCL} = Volume of HCL solution titrated (mL)

3. Determination of Amine content

1. Dilute 4.5g of amine sample in 100ml volume beaker with distilled water
2. Stir the solution at 400rpm with magnetic stirrer.
3. Titrate with HCl until pH is 4.5. (measure with pH meter probe)
4. The amine content is determined following Equation (6) and Equation (7).

$$\text{Amine content, wt\%} = \frac{V_{HCl} \times N_{HCl} \times A}{W_{\text{amine sample}}} \quad (6)$$

$$A = \frac{rMW_{MDEA}}{10(2-r)}, \quad (7)$$

Where,

r = Mole fraction of MDEA

MW_{MDEA} = Molecular weight of MDEA solution

4. CO₂ Content and CO₂ Loading

1. Titrate 120ml of methanol with prepared KOH until pH increase to 11.2.
2. Add 20g of amine solution and stir at 400rpm with magnetic stirrer while titrating with KOH until pH increase to 11.2.
3. The CO₂ content and CO₂ loading can be calculated as shown in Equation (8) and Equation (9).

$$CO_2 \text{ content, wt\%} = \left(\frac{V_{\text{amine sample with CO}_2}}{W_{\text{amine sample with CO}_2}} - \frac{V_{\text{amine sample without CO}_2}}{W_{\text{amine sample without CO}_2}} \right) \quad (8)$$

$$CO_2 \text{ loading, } \frac{\text{mol}}{\text{mol}} = \frac{CO_2 \text{ content, wt\%} \times B}{\text{Amine content, wt\%}}, \quad B = \frac{MW_{\text{Amine}}}{MW_{CO_2}(1+y)} \quad (9)$$

3.2.2 Study On Foaming In Aqueous MDEA + PZ Alkanolamine Solution

The temperature that was experimented on is between 40 - 90 °C as they represent the operating conditions in an absorber column (Thitakamol & Veawab, 2008) while the range of gas flowrate is between 1 - 5 SCFH. Bicine with concentration of 20,000 ppm was added prior to the experiment to initiate foaming. Figure 3.3 shows the experimental set up for foaming in aqueous MDEA+PZ amine solution.

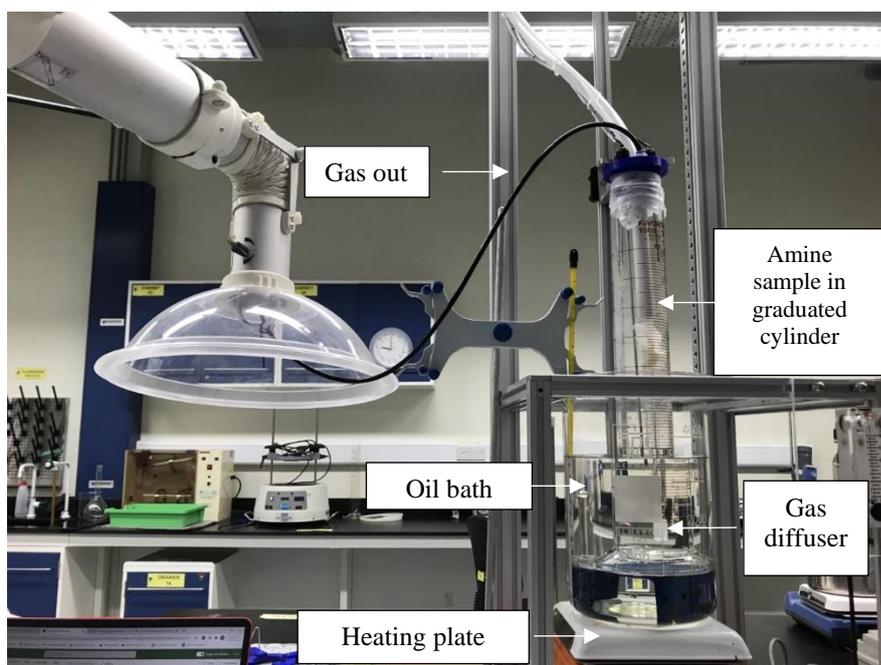


Figure 3.3: Experimental set-up for study on foaming behavior in aqueous MDEA+PZ amine solution.

Figure 3.4 shows the experimental procedure which was developed by referring to literature work by Thitakamol & Veawab (2008). Firstly, the MDEA solution was prepared by diluting the solution with deionized water to achieve 30 wt% of aqueous alkanolamine solution comprising of 25% MDEA and 5% PZ. The MDEA+PZ solution was then placed into test cells at a volume of 0.0004 m³. As stated by Thitakamol & Veawab (2008), the selected volume has shown a steady reading of foaminess coefficient (Σ), hence eliminating the effect of solution volume to the experimental data. Bicine with concentration of 20,000 ppm was added into the test cell to initiate foaming.

Prior to each experiment, the MDEA+PZ solution will be loaded with CO₂ to simulate real absorber conditions. After the preparation of the MDEA+PZ solution was done, bicine was added into the solution to initiate foaming. Then, the test cell was heated in a temperature bath to a set temperature for 20 minutes. Then, the test solution will be bubbled by N₂ gas at a set flowrate through the gas diffuser for 10 mins to reach steady volume. After 10 mins, the foam volume above the gas dispersion layer will be recorded to observe foam height. The time taken for the foam to rupture completely after gas supply is turned off was also recorded to observe foam stability. The steps will be repeated for each temperature and flowrate ranges.

The foam height will be calculated from the equation derived by the Bikerman foaming model where the foaminess coefficient (Σ) will be used to characterize the foam height. It is given by, average steady foam volume (v_o) divided by the given gas flowrate (G) as expressed in equation (10) (Thitakamol & Veawab, 2009). The lower the Σ , the lower is the foam volume, indicating a lower foam height.

$$\Sigma = \frac{v_o(m^3)}{G(m^3/h)} \quad (10)$$

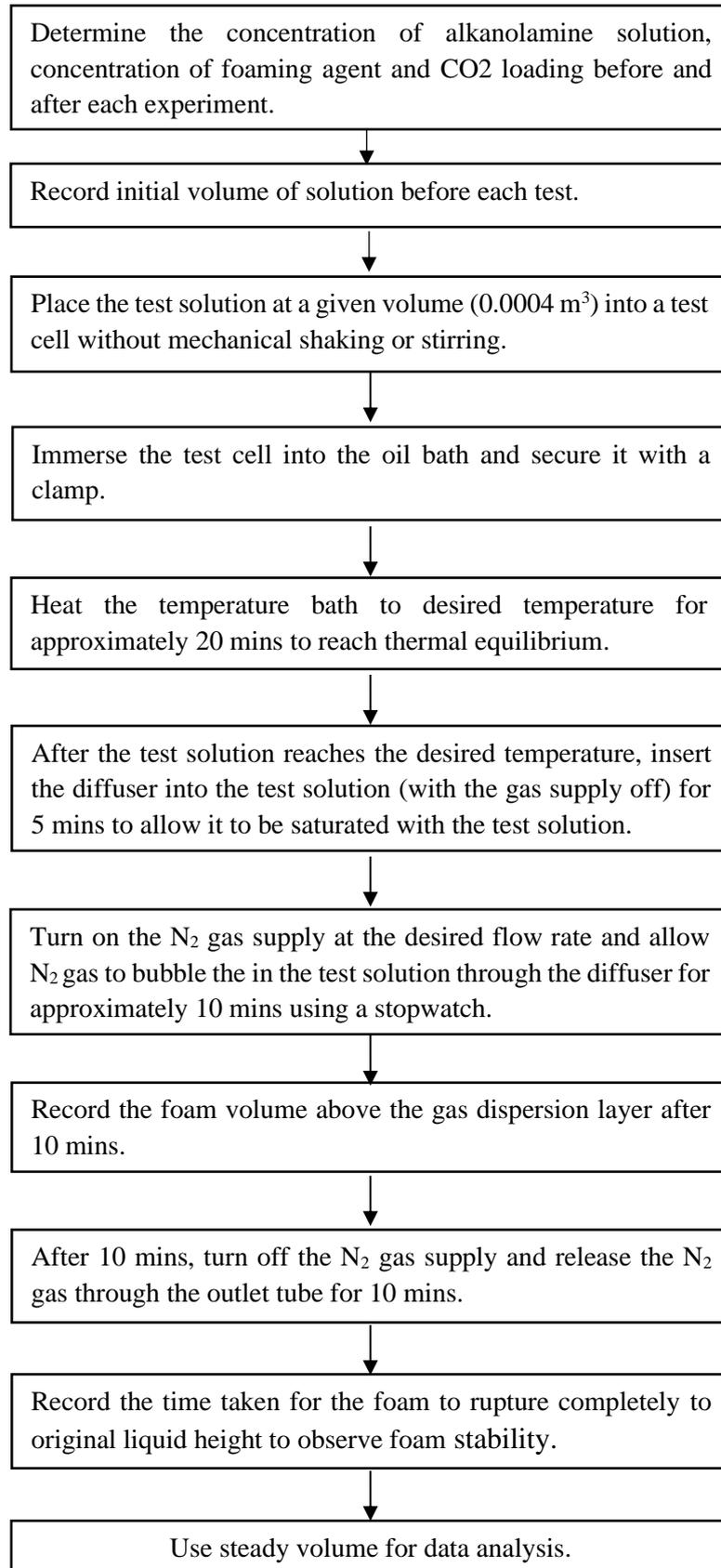


Figure 3.4: Experimental procedure to investigate the effect of temperature and pressure on foam height in MDEA solution.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 CO₂ Loading Calibration Curve

Prior to the experiment, 0.0004 m³ of amine sample was bubbled with CO₂. The sample was bubbled for approximately 2 hours and 30 minutes where every half an hour of bubbling time, 4.5 g of the sample were taken for CO₂ loading determination. The amine sample contains 25 wt% of MDEA and 5 wt% of PZ in distilled water. The method used to determine the CO₂ loading is titration, as stated in Chapter 3. Figure 4.1 shows the CO₂ loading calibration curve which exhibits the concentration of CO₂ in the sample at every 30 minutes interval of bubbling time.

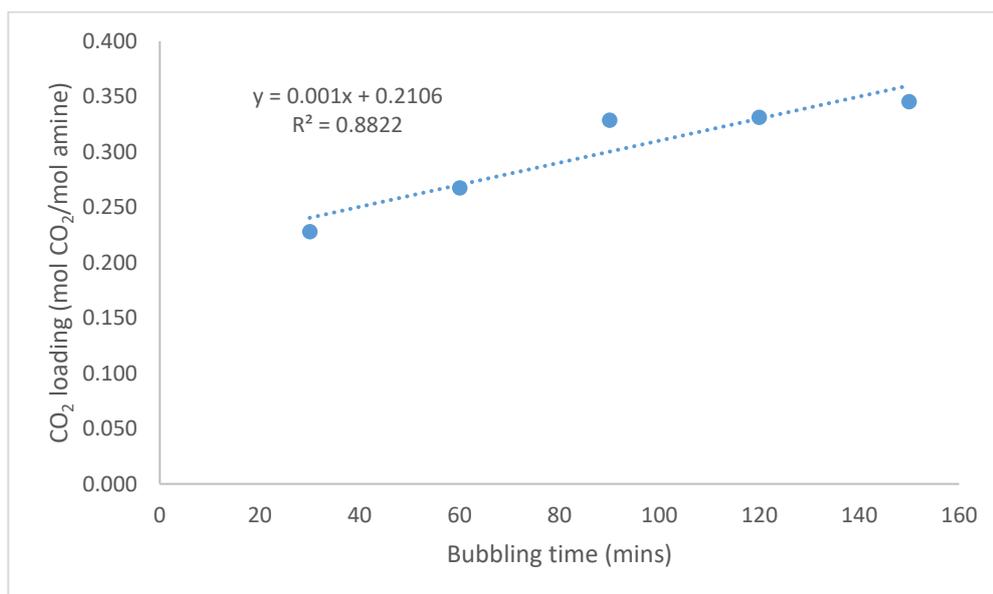


Figure 4.1: CO₂ loading calibration curve

Using the linear regression, we can see an increase in bubbling time, increases the CO₂ loading where, the lowest CO₂ loading achieved is 0.24 mol CO₂/mol amine at 30 minutes while the highest CO₂ loading achieved is 0.36 mol CO₂/mol amine at 150 minutes.

According to Thitakamol & Veawab (2008), at a higher CO₂ loading, the solution viscosity will increase, which in turn will reduce foam formation. Hence, to ensure foam is formed during the experiment, the sample used were bubbled with the lowest loading achieved which is 0.24 mol CO₂/mol amine, prior to each testing.

4.2 Effect Of Temperature On Foam Height

Prior to the experiment, a selected degradation product was added into the sample to initiate foaming since multiple studies show that clean amine solutions do not foam. According to Islam, Yusoff, & Ali (2010), bicine is a degradation product of MDEA. Since bicine was proven to show some foaming (Thitakamol & Veawab, 2008), bicine was used to initiate foam for each testing. From the study conducted by Iwan & AF (2011), most impurities used with the concentration of 20,000 ppm shows highest foam height. Hence, for this experiment, the concentration of bicine was set at 20,000 ppm. According to Keewan et. al (2018), a gas flowrate of 200 mL/min and above will start to show some foaming. However, the flowmeter used for the set up is in units of standard cubic feet per hour (SCFH) which only ranges from 1-5 SCFH. For 1 SCFH is equivalent to 471.95 mL/min, which is well above 200 mL/min. Therefore, the gas flowrate used for this experiment was set at 1 SCFH.

From this experiment, there was slight formation of foam. However, the increase in temperature from 40°C to 90°C did not affect the foam height of the amine sample where the foam volume was constant at 10 mL throughout the experiment. As previously mentioned, the foam height of the sample will be characterized by the foaminess coefficient (Σ). Figure 4.2 shows the foaminess coefficient plot, as calculated from the Bikerman index from equation 10, against temperature.

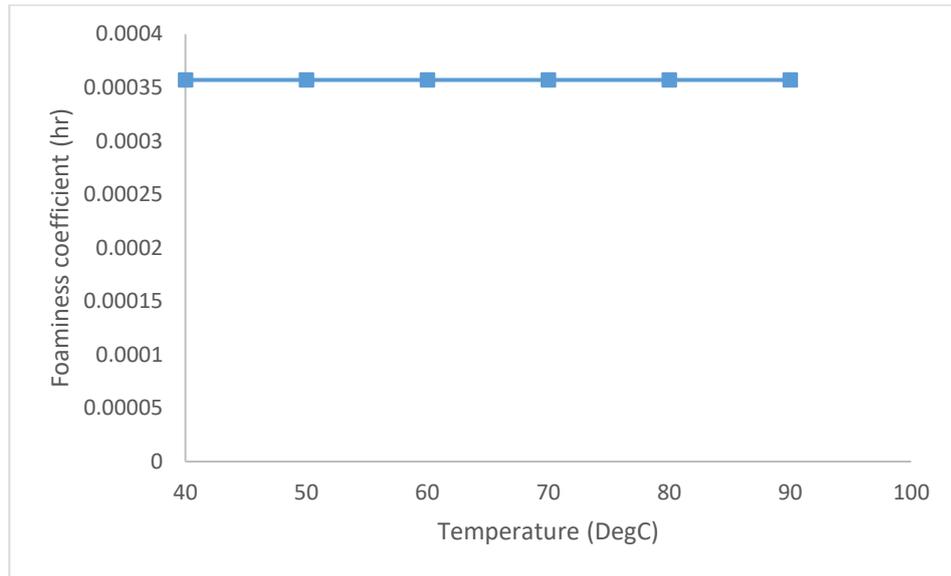


Figure 4.2: Effect of temperature on foaminess coefficient

The foaminess coefficient is found to be the same for each temperature which is 0.0036 hr which indicates that foam height did not change as temperature increases. This result contradicts further to both studies done by Thitakamol & Veawab (2008) and Keewan et. al (2018) where the former states that an increase in temperature decreases foaminess coefficient while the latter states the opposite.

The constant foam volume for each temperature could be due to several reasons. While conducting the experiment, the position of the gas diffuser was not taken into consideration. The gas diffuser was connected to the gas tube manually, by hand with no specific measurement of its position. The position of the gas diffuser might affect the formation of foam. As stated by Thitakamol & Veawab (2008), when the solution volume is too small, foam formation does not occur due to inadequate hydrostatic force to resist the buoyancy force of the gas bubble. As a result, the bubbles detached from the gas diffuser induces turbulent flow in the test cell which may destroy the foam. Since the effect of volume has been eliminated, where the volume of 0.0004 m³ should suffice to produce foam as stated by Thitakamol and Veawab (2008), the position of gas diffuser may be the reason behind the constant foam height. Similar to the effect of small volume, if the position of the gas diffuser is too close to the foam and liquid interface, the turbulent flow of the gas bubbles may retard the formation of foam.

Aside from that, the constant volume of foam with the increase in temperature may be affected by the pore size of the glass frit of the gas diffuser. According to Keewan et. al (2018), at small pore size (10-16 μm), no foaming occurred while for medium pore size (16-40 μm), foaming was the highest and foaming dropped when large pore size (40-100 μm) is used. The gas diffuser used for this experiment was the extra course designation which has a large pore size of 170-220 μm . When the pore size of the frit is large, larger bubbles will be formed where the buoyancy force of the bubble was less to overcome the hydrostatic force (Keewan et. al, 2018).

4.3 Effect of temperature on foam stability

The foam stability is analyzed through recording the foam break time. Break time is defined as the time taken for the foam to rupture completely after gas flow is turned off. Figure 4.3 shows the trend of foam break time against temperature.

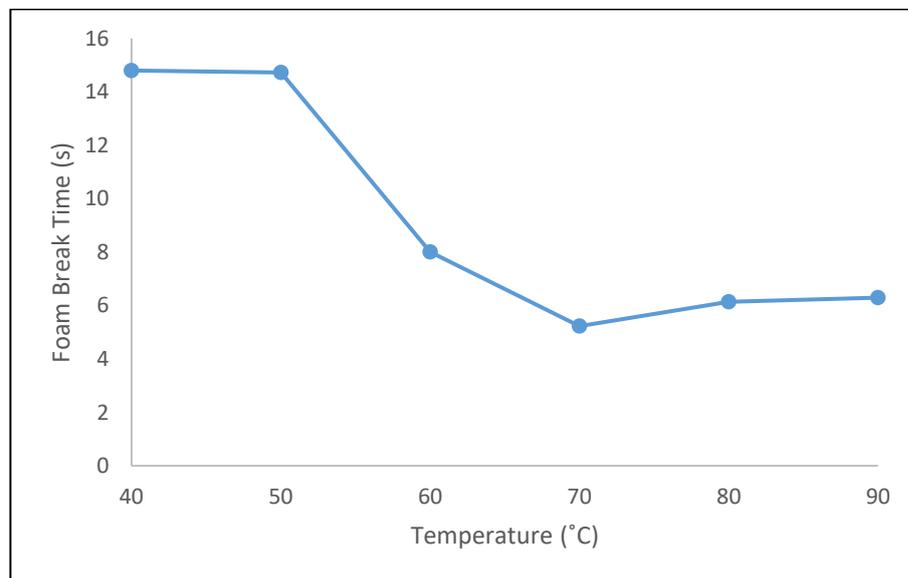


Figure 4.3: Effect of temperature on foam stability

From Figure 4.3, we can see that as temperature increases from 50 $^{\circ}\text{C}$ to 90 $^{\circ}\text{C}$, the time taken for foam to rupture decreases indicating that foam stability decreases. This is because, at elevated temperature, bulk viscosity reduces and the vigorous movement of the molecules causes the foam to be unstable (Thitakamol & Veawab, 2008).

4.4 Effect Of Gas Flowrate On Foam Height

The effect of gas flowrate on the foam height was studied by varying the gas flowrate from 1 SCFH to 5 SCFH at temperature 40°C and 90°C. Figure 4.4 shows the foaminess coefficient against gas flowrate for each temperature.

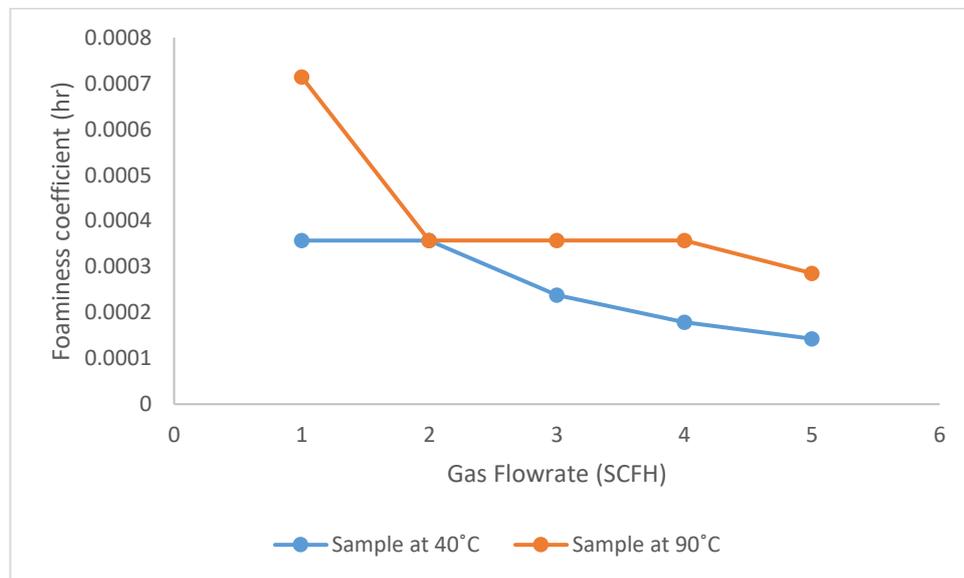


Figure 4.4: Effect of gas flowrate on foaminess coefficient

From the experiment, it was found that the foaminess coefficient decreases with increasing gas flowrate for both temperatures. This is due to the turbulence caused by the increasing gas flowrate which reduces foam stability and disrupts foam formation (Thitakamol & Veawab, 2008). From Figure 4.4 it is seen that the foaminess coefficient at 90°C is higher than the foaminess coefficient at 40 °C. This could be due to the lower surface tension at higher temperature which makes it easier for the bubbles to overcome the surface tension and increase foaming tendency.

4.5 Effect Of Gas Flowrate On Foam Stability

As gas flowrate increases, the foam stability increases for both temperatures as shown in Figure 4.5. The foam stability at temperature 40°C increases slightly as gas flowrate increases from 1 SCFH to 3 SCFH while at temperature 90°C, the foam stability has a larger incremental. This result contradicts to the previous statement

where, as gas flowrate increases, foam stability decreases. However, the bubbles that take more than 5 seconds to break appear as very small bubbles as shown in Figure 4.6. This could be due to the small leftover bubbles that was formed due to the turbulence caused from the increasing flowrate which took a longer time to overcome the surface tension.

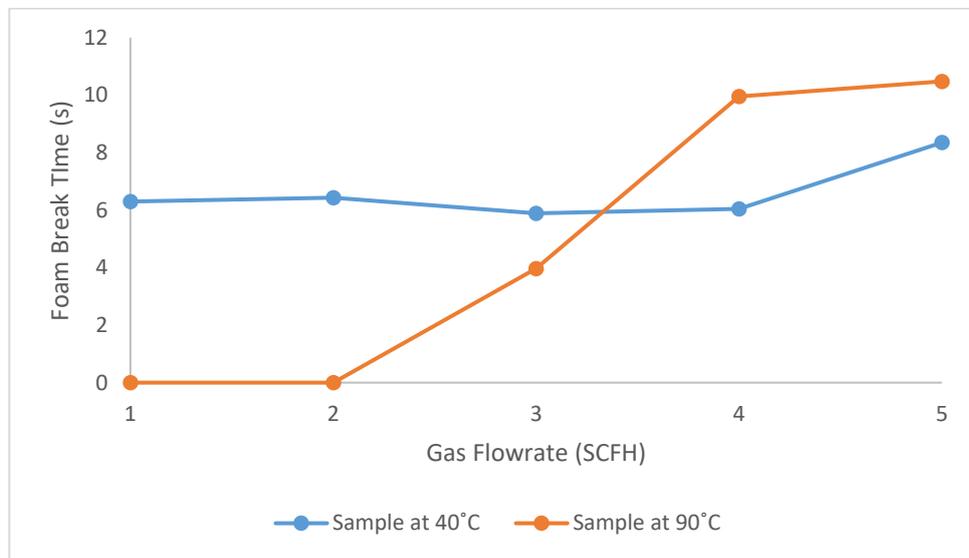


Figure 4.5: Effect of gas flowrate on foam stability

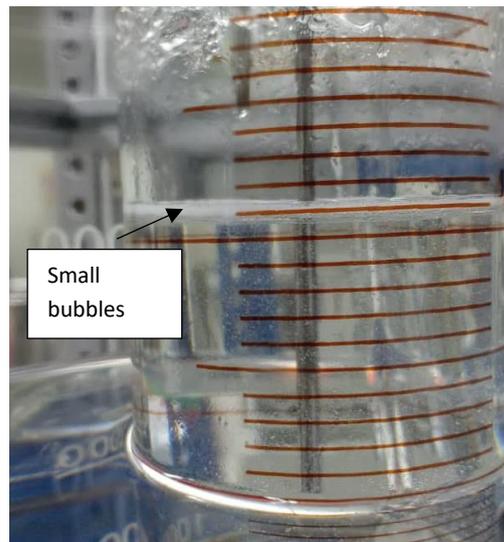


Figure 4.6: Foam stability at 90°C and 5 SCFH

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The objectives of the project include studying the effect of temperature and gas flowrate on the foaming behavior in MDEA+PZ solution blend in the presence of bicine. The foaming behavior was studied in terms of foam height which was characterized as foaminess coefficient and the foam stability.

The first objective was achieved by varying the temperature of the MDEA+PZ solvent from 40°C to 90°C in the presence of bicine. It was found that as temperature increases, the foaminess coefficient did not change. This could be due to several factors such as the position of the gas diffuser and the pore size of the glass frit. However, results on foam stability showed an increase in temperature will decrease the foam stability which could be due to the reduced bulk viscosity and the vigorous movements of the molecules at elevated temperature.

For the second objective, the effect of gas flowrate on the foaming behavior of the MDEA+PZ solvent in the presence of bicine was achieved by varying the gas flowrate from 1 to 5 SCFH. Results showed that when gas flowrate was increased, the foaminess coefficient decreased. The increasing gas flowrate caused turbulence which reduces the foam stability and disrupts the foam formation. In terms of foam stability, it was observed that as gas flowrate increases, the foam stability increases as well. This is because leftover small bubbles produced from the turbulence took a longer time to overcome the surface tension after gas flow was turned off.

5.2 Recommendations

In the study of effect of temperature and gas flowrate on the foam height of MDEA solvent, there were a few challenges faced while carrying out the experiment. Therefore, recommendations discussed here aims to improve the quality of results for any similar works related to the study of foaming in amine solvents.

Firstly, when studying the effect of temperature on the foam height of the MDEA solvent, the position of the gas diffuser should be taken into consideration. A set measurement for the position of the gas diffuser should be included in order to obtain a more stable foam and reduce the effect of turbulence. This would aid in obtaining a better reading for the volume of foam.

Secondly, to reduce parallax error while recording the time taken for foam to rupture (foam stability), a camera set up could be used in future works. The camera would be able to capture at which precise moment the bubbles would stop rupturing. It would also aid in better understanding about the characteristics of the bubble and how it will affect foaming behavior.

Thirdly, to ensure foaming occurs to study foaming behavior, the pore size of the glass frit should be considered. The medium pore size glass frit (16-40 μm) should be considered as the optimum pore size to induce foaming.

Lastly, average readings should be taken for each experimental run to ensure the readings are reliable. Due to time constraint, the reading of steady volume was taken instead of average readings during the experiment.

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APPENDICES

APPENDIX A: Gantt chart for FYP II

No.	Activities	Academic weeks											
		1	2	3	4	5	6	7	8	9	10	11	12
1	Literature review to find research gaps.												
2	Planning of the experiment.												
3	Conducting the experiment												
4	Submit draft of dissertation.												
5	Ammendment of draft												
6	Submission of soft bound report												