

Absorption Performance Study of STONVENT for CO₂-Natural Gas System

by

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

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CERTIFICATION OF APPROVAL

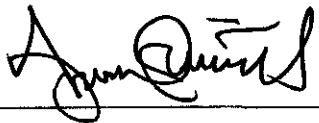
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A project dissertation submitted to the
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Approved by,



A.P. Dr. Azmi Mohd Shariff

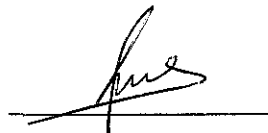
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TRONOH, PERAK

July 2010

CERTIFICATION OF ORIGINALITY

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



Anas Bin Ibrahim

ABSTRACT

As sweet gas in Malaysia has gradually decreased and most of the remaining natural gas is acidic gas where CO₂ is found highly contained (approximately 80% volumetric), CO₂ separation technique has become essential in which to remove as many CO₂ as possible. Numerous of separation techniques are available such as absorption into a liquid solvent, adsorption onto a solid, cryogenic separation, permeation through membranes, and chemical conversion but however, each of these techniques has limitations which cannot cater the problem mentioned above. In order to achieve natural gas sweetening, absorption is the most promising technique to remove the CO₂ gas. There are several liquid used as the solvent such as alkanolamines, potassium carbonate and sodium hydroxide. Alkanolamines however are the most commonly used solvent in the current industry but it still has the drawbacks which brings to the introduction of an enhanced amine-based solvent called STONVENT. Experimental work was done to evaluate the absorption performance of the solvent under atmospheric pressure, different temperature ranging from 30 to 50 °C and different liquid flowrates (0.5 and 1.0 L/min). 50 °C is found to be the best temperature while 1.0 L/min gives higher absorption efficiency. The recycle capacity of the solvent also has been studied where it is found that STONVENT has high recycle capacity as it can absorb as much even until six cycles.

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TABLE OF CONTENTS

CERTIFICATION OF APPROVAL	ii
CERTIFICATION OF ORIGINALITY	iii
ABSTRACT	iv
ACKNOWLEDGEMENT	v
TABLE OF CONTENTS	vi
LIST OF FIGURES	viii
ABBREVIATIONS AND NOMENCLATURES	ix
CHAPTER 1: INTRODUCTION	1
1.1 Background of Study	1
1.2 Problem Statement	2
1.3 Objective	3
1.4 Scope of study	3
CHAPTER 2: LITERATURE REVIEW	4
2.1 CO ₂ -Natural Gas Separation Techniques	4
2.1.1 Adsorption	4
2.1.2 Permeation through Membrane	4
2.1.3 Cryogenic Fractionation	5
2.1.1 Absorption	5
2.2 Alkanolamine Absorption	6
2.2.1 Alkanolamine Absorption Process	7
2.2.2 Experimental Work	9
CHAPTER 3: METHODOLOGY	11
3.1 Research Methodology	11
3.2 Experimental Procedure	14
3.2.1 Start up Procedure	14
3.2.2 CO ₂ Absorption Experiment	14
3.3 Milestone	15
CHAPTER 4: RESULTS & DISCUSSION	16
4.1 Temperature Effect	16

4.1.1 Experimental Results (Temperature Effect)	17
4.2 Liquid Load Effect	18
4.2.1 Experimental Results (Liquid Load Effect)	18
4.3 Recycle Capacity	20
4.3.1 Experimental Results (Recycle Capacity)	21
4.3.2 TGA Results	22
4.3.3 FTIR Results	25
CHAPTER 5: ERROR & RECOMMENDATION	28
5.1 Error	28
5.2 Recommendation	28
CHAPTER 6: CONCLUSION	30
REFERENCES	31
APPENDICES.....	33

LIST OF FIGURES

Figure 2.1 Typical Amine Gas Treating Process	7
Figure 2.2 Random packing materials	8
Figure 2.3 Flowsheet for the recovery of CO ₂ from flue gas using chemical absorption .	9
Figure 3.1 Schematic of the Gas Absorption Pilot Plant.....	12
Figure 3.2 Gas Absorption Pilot Plant	12
Figure 4.1 CO ₂ Concentration vs. Time	17
Figure 4.2 Absorption Efficiency vs. Temperature	17
Figure 4.3 CO ₂ Concentration vs. Time	18
Figure 4.4 Absorption Efficiency vs. Liquid Flowrate	19
Figure 4.5 CO ₂ Concentration vs. Time	21
Figure 4.6 Absorption Efficiency vs. Number of Cycle	21
Figure 4.7 Thermal gradation profile of Fresh STONVENT	22
Figure 4.8 Thermal gradation profile of STONVENT at cycle 2	22
Figure 4.9 Thermal gradation profile of STONVENT at cycle 3	23
Figure 4.10 Thermal gradation profile of STONVENT at cycle 4	23
Figure 4.11 Thermal gradation profile of STONVENT at cycle 5	24
Figure 4.12 Thermal gradation profile of STONVENT at cycle 6	24
Figure 4.13 IR Spectrum of Fresh STONVENT	25
Figure 4.14 IR Spectrum of STONVENT at cycle 2	25
Figure 4.15 IR Spectrum of STONVENT at cycle 3	26
Figure 4.16 IR Spectrum of STONVENT at cycle 4	26
Figure 4.17 IR Spectrum of STONVENT at cycle 5	27
Figure 4.18 IR Spectrum of STONVENT at cycle 6	27

ABBREVIATIONS AND NOMENCLATURES

DEA	diethanolamine
DGA	diglycolamine
DIPA	di-isopropylamine
GHG	green house gas
IR	infra-red
LNG	liquefied natural gas
MDEA	methyldiethanolamine
MEA	monoethanolamine
NG	natural gas
TEA	triethanolamine
TIPA	triisopropanolamine

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Natural gas that contains significant amount of hydrogen sulfide (H_2S) or carbon dioxide (CO_2); which is more than 5.7 milligrams of H_2S or CO_2 per cubic meter of natural gas or equivalent to approximately 4 ppm by volume can be classified as sour gas. Meanwhile, natural gas that free from or has insignificant amount of H_2S or CO_2 is referred to sweet gas. As for today, CO_2 contributes approximately 60% of the total greenhouse gas (GHG) emissions on a global warming potential basis due to high consumption of fossil fuel, chemical and petrochemical manufacturing, and natural gas purification (Aroonwilas et al., 2009). Moreover, in combination with water, it is highly corrosive and rapidly destroys pipelines and equipment. CO_2 also reduces the heating value of a natural gas stream and wastes pipeline capacity. In Liquefied Natural Gas (LNG) plants, CO_2 must be removed to prevent freezing in the low-temperature chillers. The gas sweetening is almost always required before the gas can meet sales gas specifications and before the sweet gas can be further processed for production of ethane, propane, butane, and higher hydrocarbon liquid products.

There are numerous of separation techniques in the removal of CO_2 from natural gas such as absorption into a liquid solvent, adsorption onto a solid, cryogenic separation, permeation through membranes, and chemical conversion. However, absorption into a liquid solvent is the most suitable process for removing CO_2 from high-volume flue gas streams (Aroonwilas et al., 2004). One of the most commonly used solvent is alkanolamine based solvents such as monoethanolamine (MEA), diethanolamine (DEA) and methyldiethanolamine (MDEA) but an enhanced amine solvent called STONVENT

is claimed to be highly effective in CO₂ removal. If STONVENT is proven to be highly effective in absorbing CO₂ from the natural gas stream, it can increase the value of the natural gas and the operating cost can be lower down as the process is believed to be carried out offshore without the need of pipeline and gas treating plant onshore.

1.2 Problem Statement

As sweet gas in Malaysia has gradually decreased and most of the remaining natural gas is acidic gas where CO₂ is found highly contained (approximately 80% volumetric), CO₂ separation technique has become essential in which to remove as many CO₂ as possible. Numerous of separation techniques are available such as absorption into a liquid solvent, adsorption onto a solid, cryogenic separation, permeation through membranes, and chemical conversion but however, all of has limitations which cannot cater the problem mentioned above.

As in example, for adsorption process such as pressure swing adsorption (PSA), thermal swing adsorption (TSA) and iron sponge; the heat produced during the process affects the adsorption capacity which limits the amount of the CO₂ that it can adsorb. In permeation through membranes technique, the same problem occurs where the membrane has minimal permeability which limits the amount of CO₂ to dissolve and diffuse later. Cryogenic fractionation is known for its expensive process which eliminates the process to be as an option.

As absorption is most likely to be the best option for this matter, the current solvent which have commonly used in the industry has its own drawbacks. For instance, primary amine solvent (MEA) and secondary amine (DEA) reacts rapidly with CO₂ and forms carbamate ions where associated to higher enthalpy of absorption. For tertiary amine (MDEA), it is slower in reactivity and provides lower capacity due to its large equivalent weight. The primary and secondary amine also demand higher cost of regenerating (due to high energy requirement for solvent regeneration) as compared to tertiary amine since it is unable to form carbamate ions. Moreover, primary is only suitable at low pressure

condition whilst secondary is normally operated at the medium pressure and tertiary amine is said to have the endurance in operating at high pressure. Thus, STONVENT is introduced and believed to possibly encounter the problems caused by the other alkanolamine based solvent used nowadays and it is claimed to be highly effective in CO₂ removal.

1.3 Objective

The main objective of this project is to evaluate the performance of STOVENT under several conditions, to see its efficiency in absorbing CO₂.

This project also has been designed to study the parameters which are essential in order to evaluate the performance of the solvent. The parameters include:

1. The effect of solvent's temperature on absorption performance of CO₂-CH₄ system.
2. The effect of liquid load (liquid flow rate per cross sectional area) on absorption performance of CO₂-CH₄ system.
3. The recycle capacity of the solvent in CO₂-CH₄ system.

1.4 Scope of Study

This project will be using absorption technique for the removal process of CO₂ from the mixture of CH₄-CO₂ where CH₄ is used to resemble natural gas as if in the real scenario. A novel solvent, STONVENT is used to absorb the CO₂ in the CO₂-CH₄ system. The experiment is conducted under atmospheric pressure (1 atm) by varying the temperature at a range of 25 – 50 °C while other parameters are varied as stated in section 1.3.

CHAPTER 2

LITERATURE REVIEW

2.1 CO₂-Natural Gas Separation Techniques

There are numerous of separation techniques that have been applied in the industry for the removal of CO₂ gas from natural gas such as absorption into a liquid solvent, adsorption onto a solid, cryogenic separation, permeation through membranes, and chemical conversion.

2.1.1 Adsorption

For adsorption process such as pressure swing adsorption (PSA), thermal swing adsorption (TSA) and iron sponge; are commonly used where the natural gas will be passing through an adsorbent (solid) and the CO₂ particles will be captured on the surface of adsorbent. However, since the adsorption process is exothermic, a high $-\Delta H$ value will produce large quantities of heat within the adsorbent bed during an adsorption process cycle. This will cause an increase in the local column temperature. This increase in the column temperature will have an adverse effect on the adsorption capacities of the components. (Tezel et al., 2007)

2.1.2 Permeation through Membrane

In permeation through membranes technique, the membranes used for CO₂ removal do not operate as filters, where small molecules are separated from larger ones through a medium with pores. Instead, they operate on the principle of solution-diffusion through a nonporous membrane. The CO₂ first dissolves into the membrane and then

diffuses through it. Nevertheless, membrane systems were not a viable process because the membrane thickness required to provide the necessary mechanical strength was so high that the permeability was minimal. (Dortmundt et al., 1999) In recent years, a membrane gas-liquid contactor has been introduced where the membrane is wetted with liquid solvent for selective separation of gaseous components. However, when the membrane pores are filled with the liquid (wetted), the mass transfer resistance of the membranes becomes significant, resulting into economically unviable operation (Versteeg et al., 2004).

2.1.3 Cryogenic Fractionation

Other separation process technique that can be used for CO₂ removal from natural gas is cryogenic fractionation where methane and lighter components are separated from carbon dioxide and heavier components in a multi-stage tower or fractionator. The fractionator is operated at cryogenic temperature at the range of -60 to -130 °F which requiring high compressor horsepower to provide the external process refrigeration. Thus, the cost of utilizing the process for treatment of a high CO₂ content natural gas stream can be prohibitively expensive (Green et al., 1990).

2.1.4 Absorption

Absorption is probably the most important gas purification technique and is common to a great number of processes. It involves the transfer of substance from the gaseous to the liquid phase through the phase boundary. The absorbed material may dissolve physically in the liquid or react chemically with it (Kohl et al, 1997, p. 2). There are several solvents used in the industry to remove CO₂ from natural gas stream such as alkanolamines, ammonia, alkaline salt and water. However, aqueous solutions of alkanolamines are the chemical solvents that have been used extensively for the CO₂ capture as it often provides a greater absorption performance or meet unique needs when compared to the other solvents.

2.2 Alkanolamines Absorption

The idea of separating CO₂ from flue gas streams has started since 1970s. A wide variety of separation techniques involves the removal of vapor-phase impurities from gas stream including permeation through membrane, absorption into a liquid, condensation, chemical conversion to another compound and adsorption on a solid. One promising approach to remove CO₂ is via alkanolamines absorption. Alkanolamines absorption removes CO₂ from the gas stream by exothermic reaction of CO₂ with the amines. Credit for the development of alkanolamines as absorbents for acidic gases goes to R. R. Bottoms (1930). Triethanolamine (TEA) which was the first alkanolamine to become commercially available was used in the early gas-treating plants (Kohl et al, 1997, p. 41).

The amines that have proved to be of principal commercial interest for gas purification are primary amine whose members include monoethanolamine (MEA) and diglycolamine (DGA); secondary amine whose members include diethanolamine (DEA) and di-isopropylamine (DIPA); and tertiary amines whose members include triethanolamine (TEA) and methyldiethanolamine (MDEA) (Kohl et al, 1997, p. 41). The chemistry of acid gas reaction with sterically hindered amines such as 2-amino-2-methyl-1-propanol (AMP) was discussed by Sartori and Savage (1983) and by Weinberg et al. (1983). Based on their findings, sterically hindered amines proved better selectivity compared to the tertiary or secondary alkanolamines.

2.2.1 Alkanolamine Absorption Process

In the industry, a typical amine gas treating process (as shown in Figure 2.1) includes an absorber unit and a regenerator unit as well as accessory equipment. In the absorber, the downflowing amine solution absorbs H_2S and CO_2 from the upflowing sour gas to produce a sweetened gas stream (i.e., an H_2S -free gas) as a product and an amine solution rich in the absorbed acid gases. The resultant "rich" amine is then routed into the regenerator to produce regenerated or "lean" amine that is recycled for reuse in the absorber.

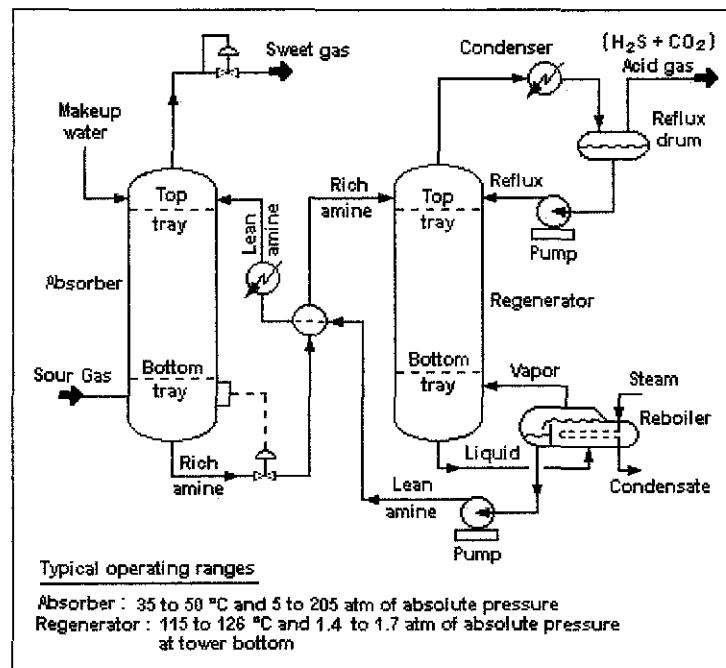


Figure 2.1: Typical Amine Gas Treating Process

The absorption efficiency is an important factor since it measures the mass-transfer rate of acid gas into the absorption solvent that is required to achieve a removal target. In most cases, the efficiency is theoretically evaluated by using classical laboratory reactors, such as stirred cell and laminar jet absorber where the interfacial area for the mass transfer is known and fixed. However, these reactors do not take into account the hydrodynamic features of the contacting devices, which may vary with type of absorption solvents during the plant operation. Therefore, it is necessary to use a column

(packed or tray) as a gas-liquid contacting device for evaluating the absorption efficiency of any solvent of interest. (Veawab et al., 2002)

A packed column is a vertical cylindrical pressure vessel containing one or more sections of a packing material over whose surface the liquid flows downward by gravity, as a film or as droplets between packing elements. Vapor flows upward through the wetted packing, contacting the liquid. The sections of packing are contained between a lower gas-injection support plate, which holds the packing, and an upper grid or mesh hold-down plate, which prevents packing moving. A liquid distributor, placed above the hold-down plate, ensures uniform distribution of liquid as it enters the packed section. If the depth of packing is more than about 20ft, liquid channeling may occur, causing the liquid to flow down the column mainly near the wall, and gas to flow mainly up the center of the column, thus greatly reducing the extent of vapor-liquid contact. In that case, a liquid redistributor should be installed. (Seader et al., 1998)

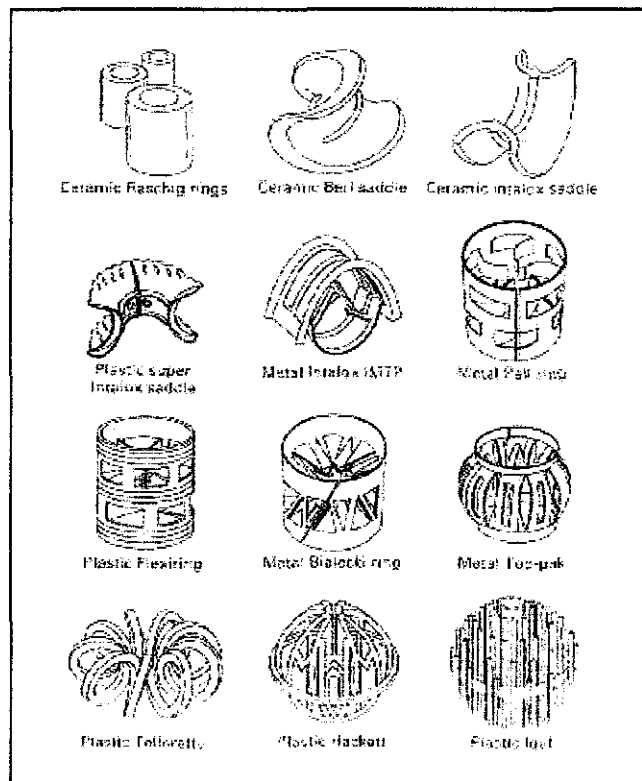


Figure 2.2: Random packing materials

2.2.2 Experimental Work

Numerous of experimental works have been carried out by using packed-column in order to evaluate the absorption performance of any solvent. The example of experimental setup is as shown in Figure 2.3 where a glass column with packing is used. The solvent is electrically preheated to the column control temperature in the stainless steel inlet line and the liquid flow rate is measured by a mass flow meter and controlled by a metering pump. The flue gas enters from the bottom of the column and the gas flow rate is controlled by a mass flow meter as well. The concentration of the CO₂ at the inlet and outlet is measured by the IR analyzer. (Yeh et al., 2001)

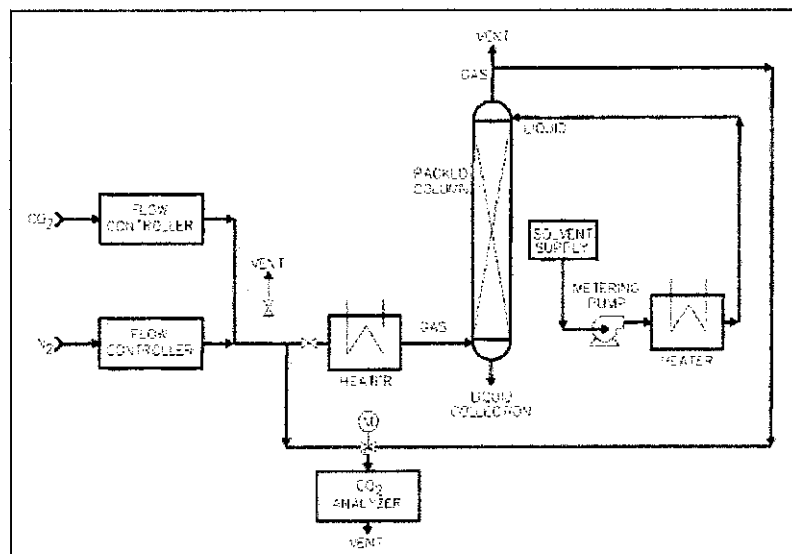


Figure 2.3: Flowsheet for the recovery of CO₂ from flue gas using chemical absorption

Later, once the experiment has been conducted, the CO₂ absorption or removal efficiency (η) needs to be calculated. Yeh and Pennline (2001) had suggested a way to calculate the efficiency. First, the inlet CO₂ is calculated using the following equation:

$$G_i = [(F \times D/M)/(1 - y_i)]y_i$$

where,

G_i = CO₂ inlet flow rate (lb-mol/h)

F = nitrogen gas flow rate (ft³/h)

D = density of nitrogen (lb/ft³) at standard conditions

M = molecular weight of nitrogen

y_i = CO₂ mole fraction at inlet of absorber (vol%)

Then, the outlet CO₂ concentration is calculated using the following equation:

$$G_o = [(F - D/M)/(1 - y_o)]y_o$$

where,

G_o = CO₂ outlet flow rate (lb-mol/h)

y_o = CO₂ mole fraction at outlet of absorber (vol %).

The CO₂ absorption rate is the difference between the inlet and outlet CO₂ flow rates. Total CO₂ absorbed in the column can be obtained by integrating the instantaneous rates over a selected time period. At steady state, efficiency of CO₂ absorption is defined as:

$$[(y_i - y_o)/y_i] \times 100\%$$

On the other hand, another simpler method to calculate the efficiency has been suggested by Veewab, Aroonwilas and Tontiwachwuthikul (2002). The efficiency can be determined by using the following equation:

$$\eta = \left[1 - \left(\frac{y_{out}}{1 - y_{out}} \right) \times \left(\frac{1 - y_{in}}{y_{in}} \right) \right] \times 100$$

where, y_{out} and y_{in} denote the mole fraction of CO₂ in gas phase at the top and bottom of the absorption column, respectively.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

This research will be based on the absorption performance study of STONVENT for CO₂-natural gas system. All academic journals and chemical textbooks that have been published from year 1991 until 2009 will be used as the reference. All those references have been selected within that range of years to cope with the changes of technology trend and taking account the extensive problem figures.

The solvent which is STONVENT was gotten from the supplier and 4 drums of the solvent were used throughout the experiment. The first 2 drums were used for the temperature study, the third drum was used for liquid loading study and the last drum was used for the recycle capacity study.

In meeting the objectives, this research will be conducted via experimental work and later, the data will be analyzed and CO₂ removal efficiency will be calculated by using the following formula:

$$\eta = \left[1 - \left(\frac{y_{out}}{1 - y_{out}} \right) \times \left(\frac{1 - y_{in}}{y_{in}} \right) \right] \times 100$$

The experiments were carried out in a bench-scale absorption unit; namely Gas Absorption Pilot Plant (as shown in Figure 3.1 & 3.2) of which the main component was the Borosilicate glass absorption column with an internal diameter of 100mm and height of 2.97m. The column was packed with raschig rings (10 x 10 mm glass) packing and with the packing height of approximately 1.5m.

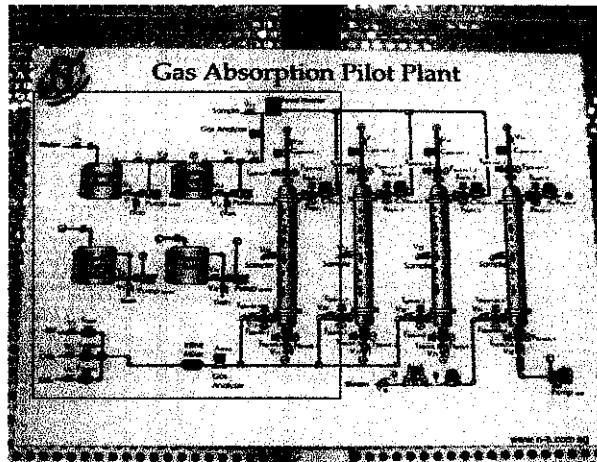


Figure 3.1: Schematic of the Gas Absorption Pilot Plant

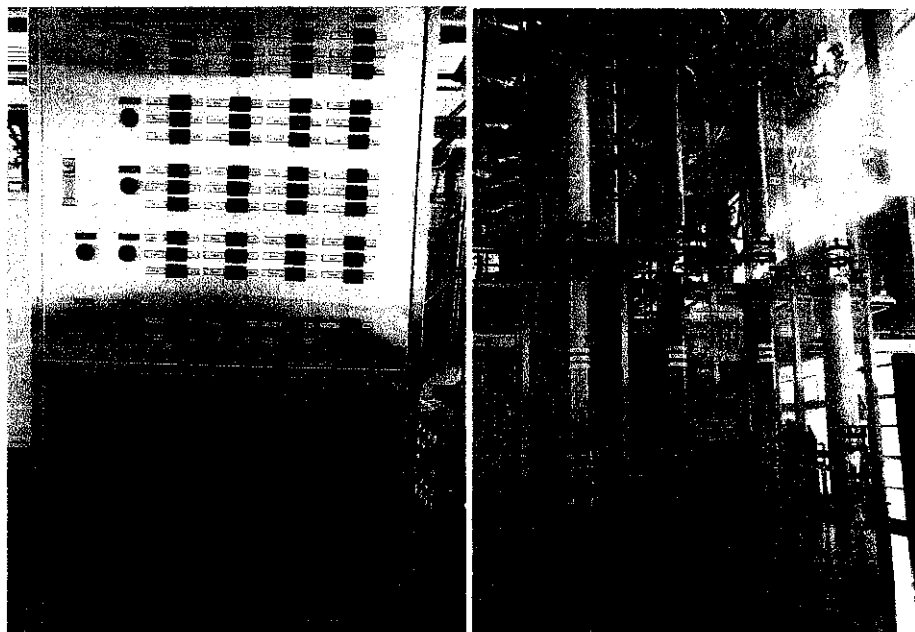


Figure 3.2: Gas Absorption Pilot Plant

It has been designed as a gas-liquid contacting device for evaluating the absorption efficiency of any solvent of interest. Effects of temperature, liquid load (flow rate per cross sectional area) and recycle capacity on the absorption efficiency were studied.

The absorption experiments began by pumping the STONVENT into the column, heated up to the desired temperatures and set the desired flow rates. Then a CO₂-CH₄ gas mixture is introduced and set at desired flow rates. The solvent and gas mixture is flowed simultaneously and co-currently. The column operation continued until a steady-state condition was reached for both gas- and liquid-sides (approximately 15 minutes). CO₂ concentrations in the gas stream at the top and bottom of the column were measured by the IR analyzer.

The experiment was started by studying the effects of solvent's temperature where the temperature of interest will be ranging between 25 – 50 °C whilst the other parameter will remain constant; pure solvent will be used, liquid loading at 1.0 L/min, gas flowrate at 5.0 L/min and approximately 45% of CO₂ content in the gas mixture. At the end of the experiment, the best temperature is identified.

Then, the effect of liquid flowrate was studied where the flowrate were 0.5 L/min and 1.0 L/min while other parameters are remain constant; temperature at 50 °C, gas flowrate at 5.0 L/min and approximately 45% of CO₂ content in the gas mixture.

The experiment is then continued with recycle capacity of the solvent. A fresh solvent is used as a start with temperature of 50 °C, liquid, flowrate at 1.0 L/min and gas flowrate at 2.5 L/min were set. The experiment is run until the process reached steady state and the process was repeated until 6 cycles. The absorption efficiency of each cycle is then determined.

3.2 Experimental Procedure

3.2.1 Start-up Procedure

1. Switch on the main power supply for the pilot plant.
2. Fill up the Water Tank with water.
3. Ensure that the necessary valves are opened and the rest is closed.
4. Turn on Pump 3 to circulate the water into the column and removes the remaining inside.
5. Allow the water circulation for 10 – 15 minutes and turn off the pump.

3.2.2 CO₂ Absorption Experiment

1. Fill up the Feed Tank with the desired solvent (e.g: STONVENT, MEA, etc).
2. Ensure that valves necessary are opened and the rest is closed.
3. Turn on Pump 1 (for Feed Tank) to pump in the solvent into the column and set at desired the flowrate.
4. Once the solvent starts flowing into the column, turn on the heater and set it up to the desired temperature.
5. Once the desired is reached, open valve V6 and close valve V9.
6. Open the valve for CO₂ and CH₄ gas line to allow the flow and set the flowrate of the gas accordingly.
7. Open valve connected to the IR Analyzer to measure the CO₂ concentration in the gas mixture via IR Analyzer (wait until the reading is stable).
8. Once the initial CO₂ concentration is known, close the *From Gas Inlet* valve and open the *From Gas Outlet* valve.
9. Wait around 15 minutes for the process to take place and then take the readings every 5 minutes until the process is reached steady-state condition.
10. Turn off the heater first and followed by the pump.
11. Switch off the main power supply.

3.3 Milestone

For the first semester, I was more focus on the literature research, gathering information on the theory of alkanolamine absorption and the absorption experimental work. Literature review is done by gather as much as possible through journal, online research and reference book on CO₂ absorption by using alkanolamine.

While the second semester, it is more on the implementation of the finding or in the other word, conducting the experiment. The data of the experiment are collected and analyzed in order to evaluate the absorption performance of the solvent. Graphs were constructed to represent the data collection and for the evaluation purposes.

CHAPTER 4

RESULTS & DISCUSSION

In each experiment that was being conducted, the CO₂ concentration in the gas stream at the gas outlet was measured and the CO₂ absorption efficiency is later calculated by using the formula below:

$$\eta = \left[1 - \left(\frac{y_{out}}{1 - y_{out}} \right) \times \left(\frac{1 - y_{in}}{y_{in}} \right) \right] \times 100$$

A graph of CO₂ concentration versus time is being plotted for each experiment and another graph CO₂ absorption concentration versus the respective measured parameter (temperature, liquid flowrate or number of cycle) is then being plotted to show a clearer comparison between the studied parameter.

4.1 Temperature Effect

In this experiment, 5 different range of temperatures of solvent have been studied which are 30°C, 35 °C, 40 °C, 45 °C and 50 °C. As shown in figure 4.1 and 4.2, it shows that 50 °C gives the highest absorption efficiency which is 57% as compared to the others where the CO₂ concentration in the gas stream is reduced from 44.6% down to 26.5%. As the solvent's temperature increases, it provides higher kinetic energy to favor the absorption of CO₂ into the solvent. That is why at 50 °C, it gives the highest absorption efficiency. However, due to the absorption rig limitation, the studied range of temperature can be only up to 50 °C. Thus, the temperature can be claimed to be optimum for this specific equipment and process only.

4.1.1 Experimental Results (Temperature Effect)

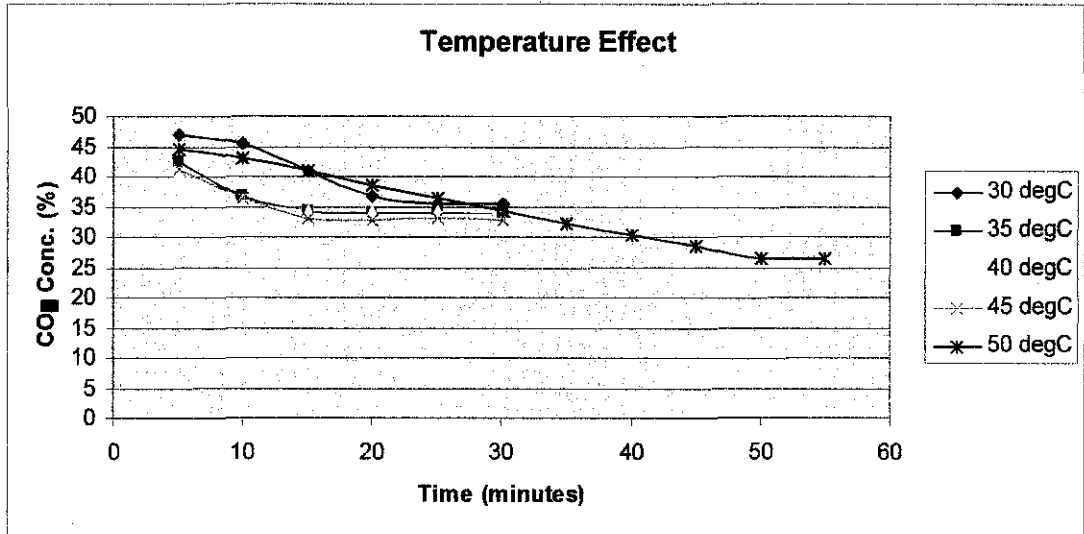


Figure 4.1: CO₂ Concentration vs. Time

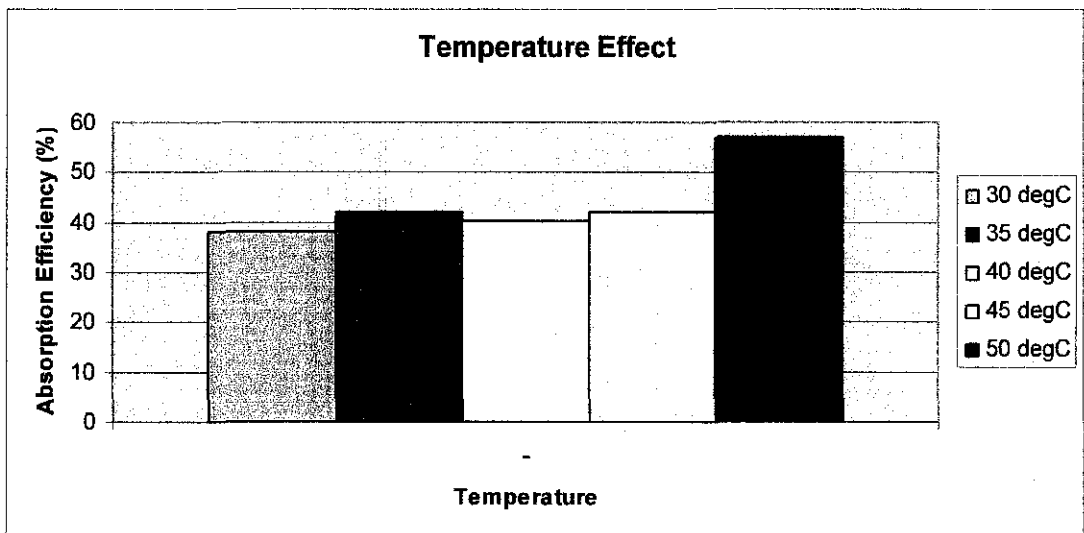


Figure 4.2: Absorption Efficiency vs. Temperature

4.2 Liquid Load Effect

For this experiment, only two different flowrates that have been studied which are 0.5 L/min and 1.0 L/min. The result as shown in figure 4.3 and 4.4 indicate that 1.0 L/min has slightly higher absorption efficiency which is 49.5% as compared to 0.5 L/min flowrate. This is because, as the flowrate increases, the contact between the solvent and the gas is more and thus gives the solvent more ability to absorb the CO₂ in the gas stream.

Due to the limitation of the absorption rig, the experiment can only be carried out to only two flowrates. Even though only two flowrates have been studied, it still provides an essential data to evaluate the absorption performance of the solvent. For future work, this parameter still need to be taken into account since it still affects the absorption performance of the solvent.

4.2.1 Experimental Results (Liquid Load Effect)

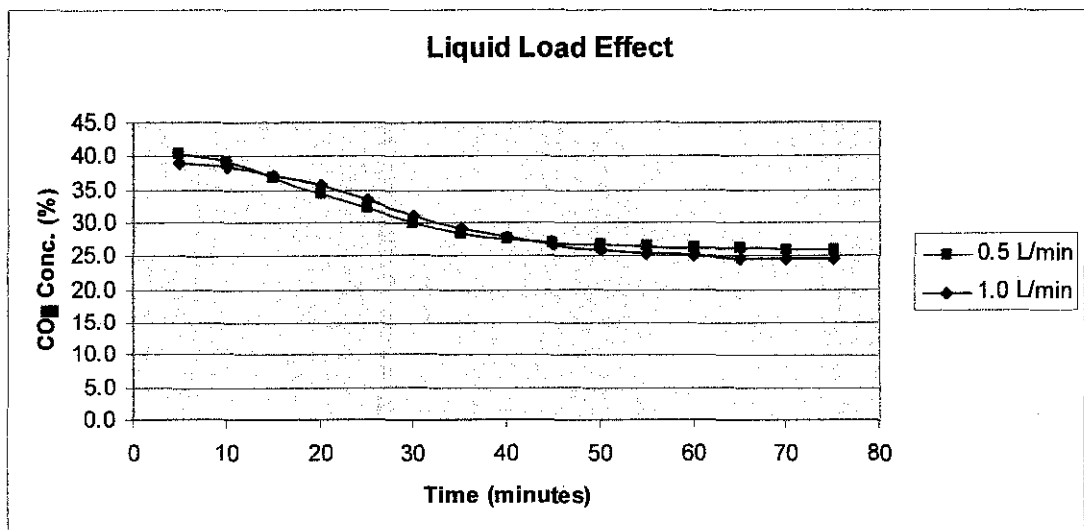


Figure 4.3: CO₂ Concentration vs. Time

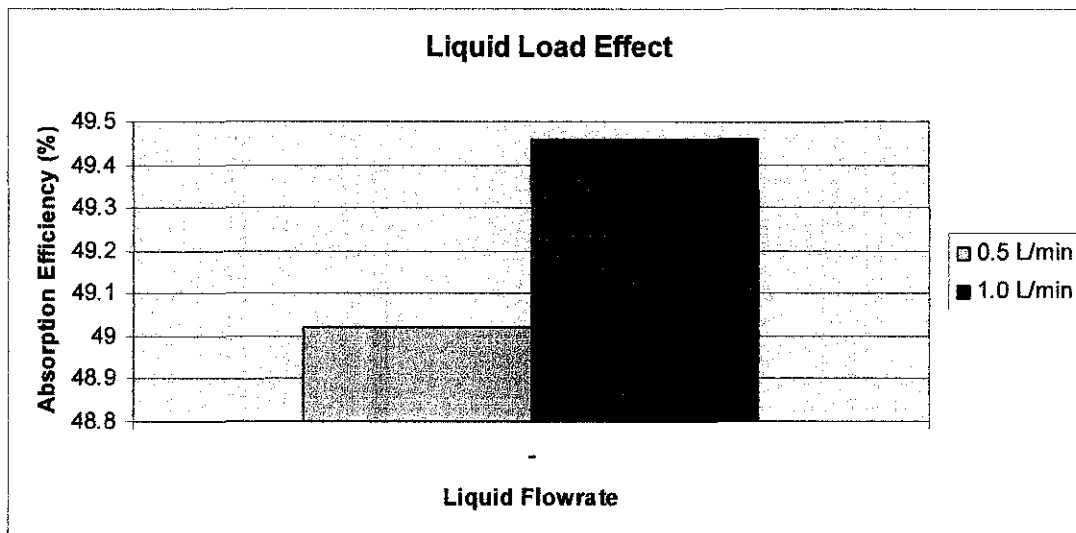


Figure 4.4: Absorption Efficiency vs. Liquid Flowrate

4.3 Recycle Capacity

The experiment is later conducted to measure the capability of the solvent to absorb CO₂ in a recycle process (where no fresh solvent is used). The cycle has been set to be until at the 6th cycle. From the result obtained (see figure 4.5 and 4.6), it shows that the solvent is capable to absorb about the same amount of CO₂ in each cycle which is in the range of 49% – 51%.

For this experiment, in each cycle (including fresh solvent), a sample of the solvent was taken into a bottle after the steady state is reached. The results were then been further analyzed by using Thermo Gravimetric Analysis (TGA) and Fourier Transform Infrared Spectroscopy (FTIR). As in section 4.3.2, the TGA results showed that even up until the 6th cycle, the solvent still has almost the same thermal gradation profile as compared to the fresh one. This shows that the solvent is still has the capacity in absorbing CO₂ even though it has been used six times. If the solvent is saturated with CO₂, the thermal gradation profile will be different since the composition of the solvent will certainly different from the fresh one.

In the FTIR results (see section 4.3.3), the first peek which has 3313.48 cm⁻¹ wave length indicates that the solvent contains of N-H and O-H bonds since the solvent is categorized under alkanolamine family. In comparison between the fresh STONVENT and the STONVENT after cycle 6; in both graph, the same peek is still exist which means even up until 6th cycle, the solvent still has the same composition and thus, it still capable to absorb more CO₂. Once the solvent is saturated with CO₂, the peek will be different as the O-H bond will react with CO₂ to form –COOH bond.

4.3.1 Experimental Results (Recycle Capacity)

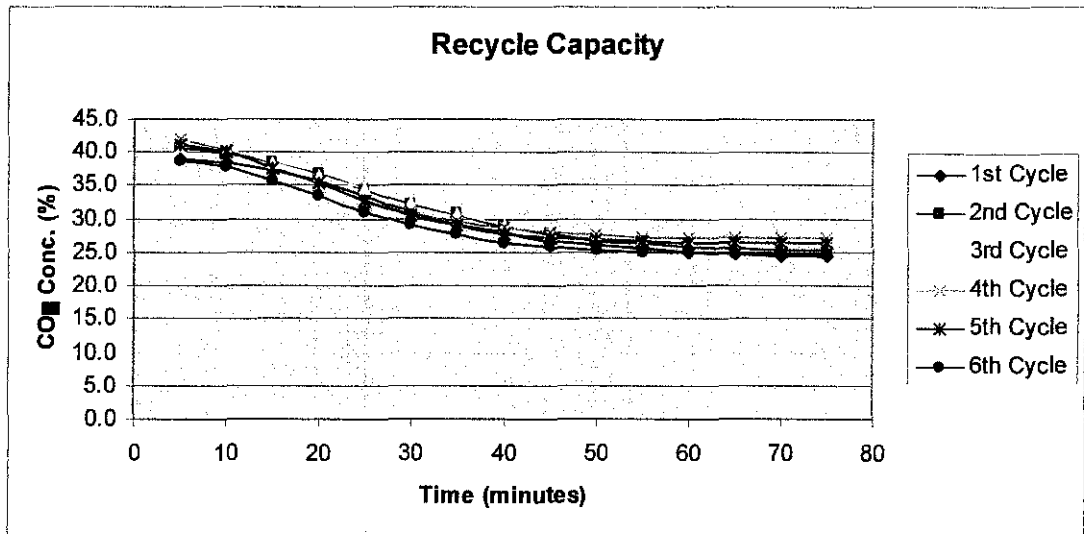


Figure 4.5: CO₂ Concentration vs. Time

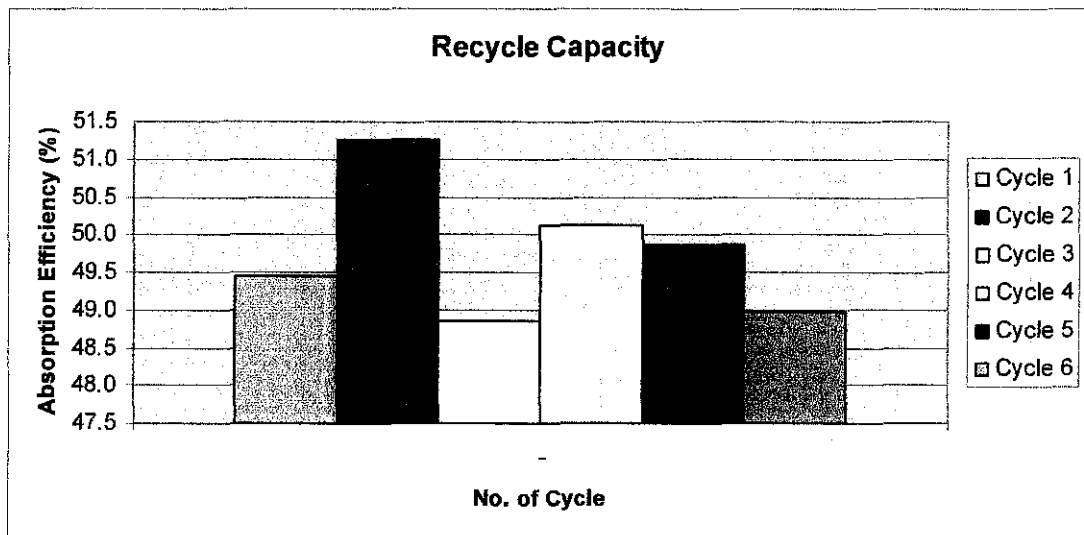


Figure 4.6: Absorption Efficiency vs. Number of Cycle

4.3.1 TGA Results

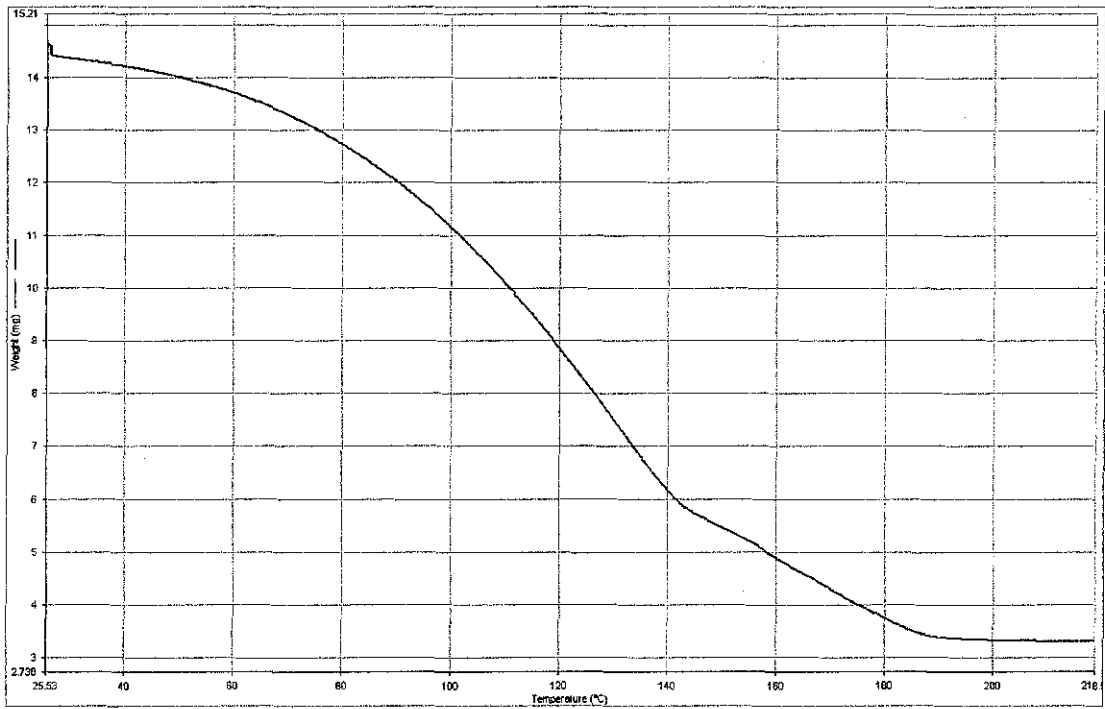


Figure 4.7: Thermal gradation profile of Fresh STONVENT

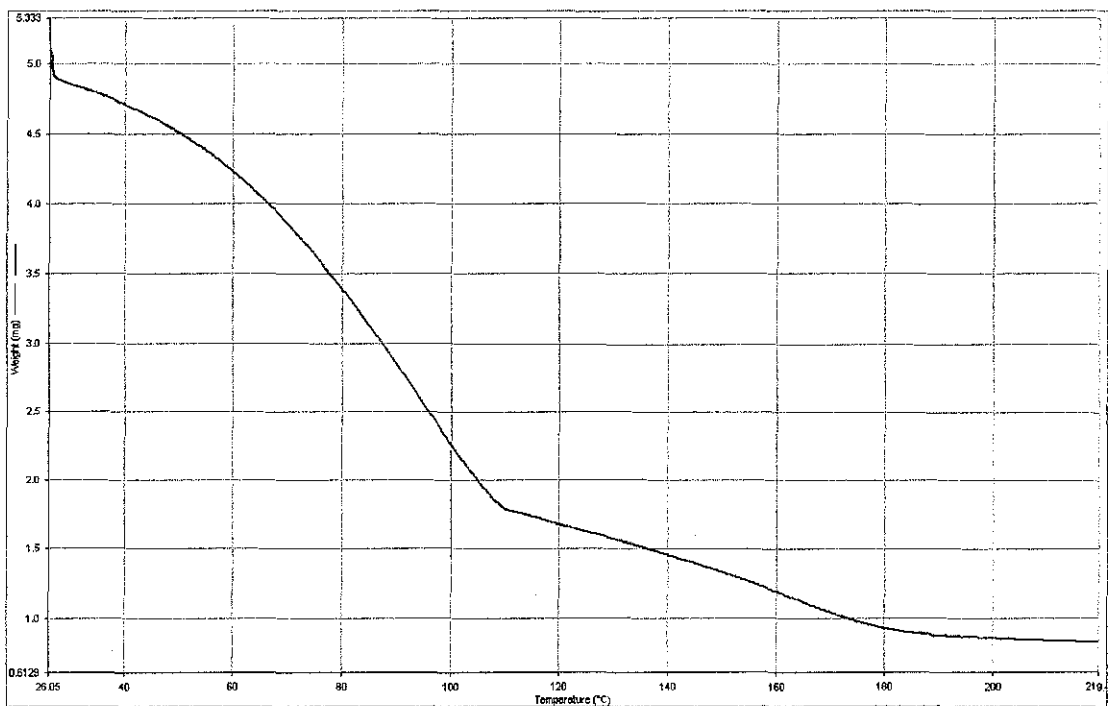


Figure 4.8: Thermal gradation profile of STONVENT at cycle 2

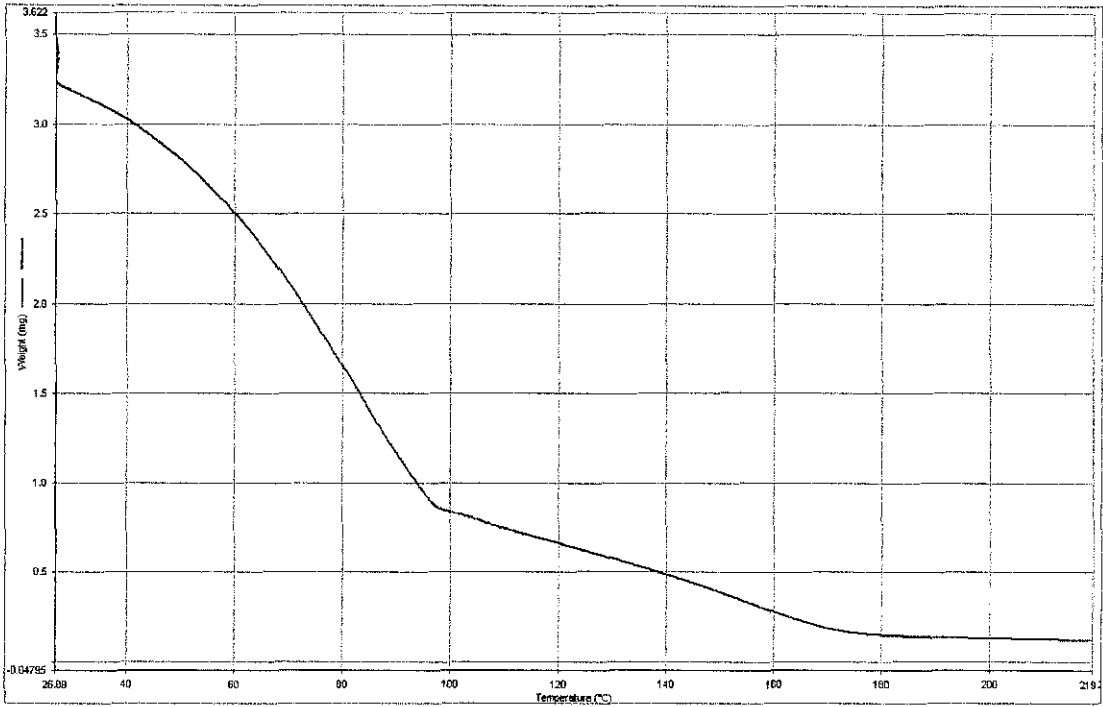


Figure 4.9: Thermal gradation profile of STONVENT at cycle 3

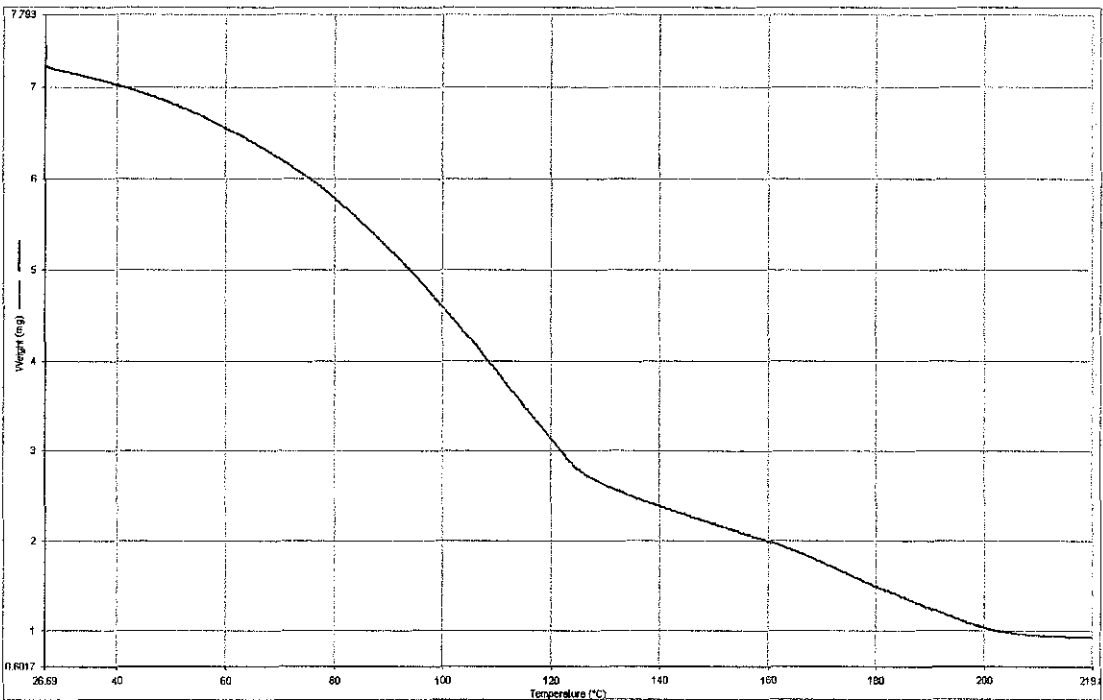


Figure 4.10: Thermal gradation profile of STONVENT at cycle 4

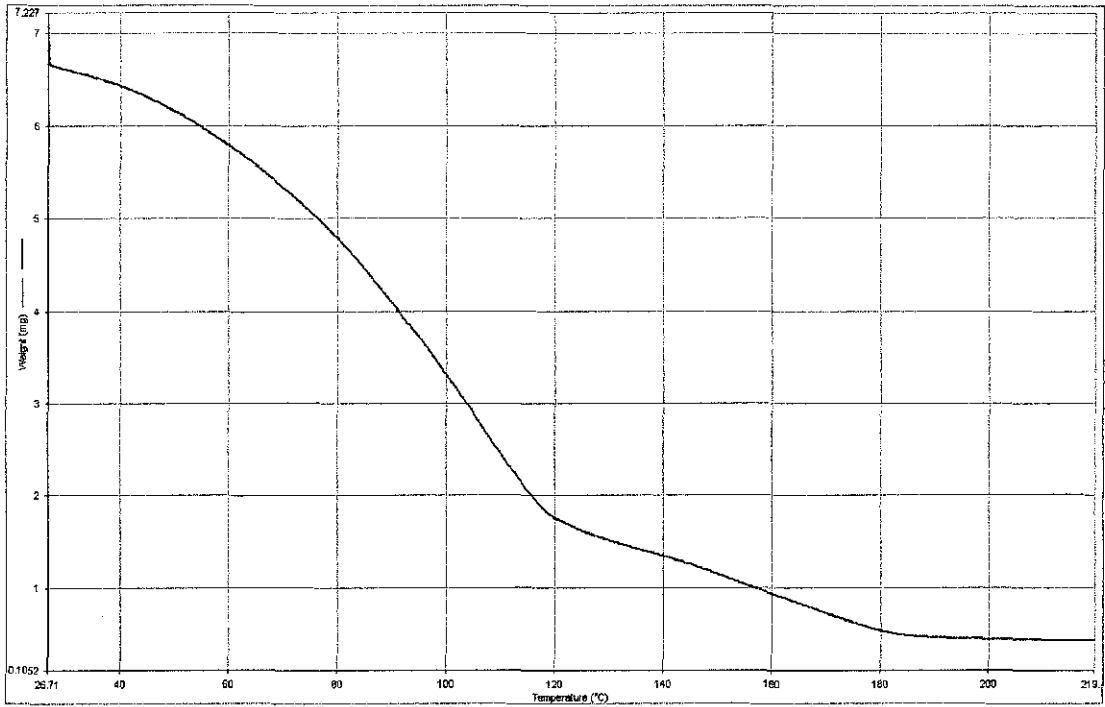


Figure 4.11: Thermal gradation profile of STONVENT at cycle 5

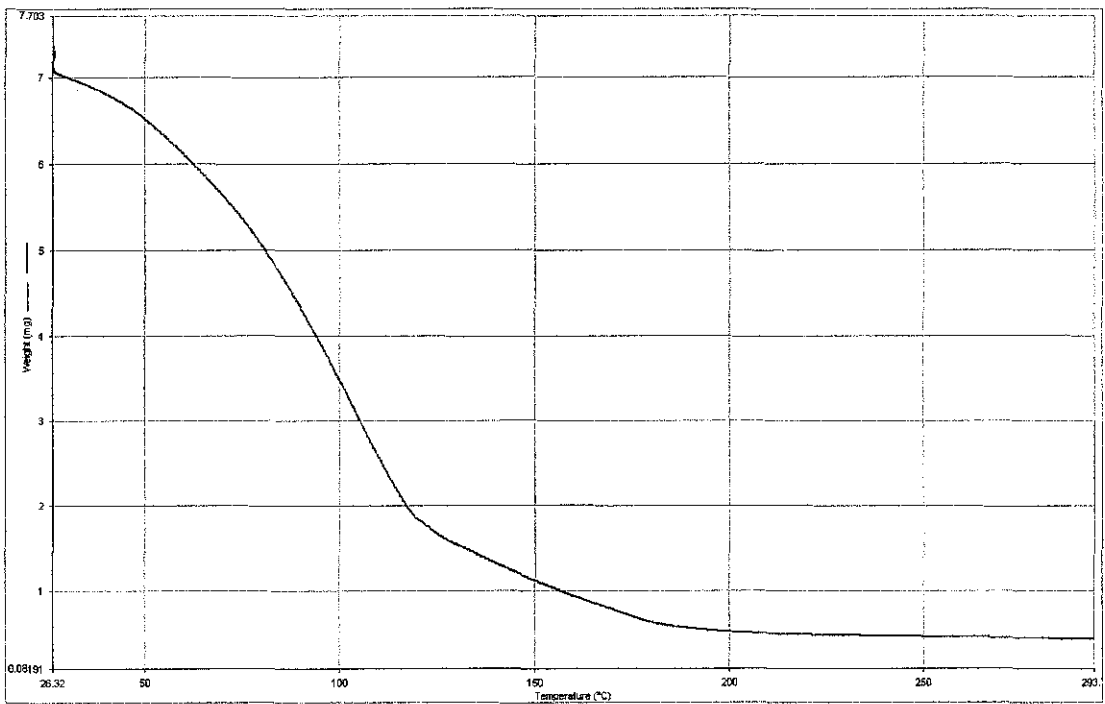


Figure 4.12: Thermal gradation profile of STONVENT at cycle 6

4.3.2 FTIR Results

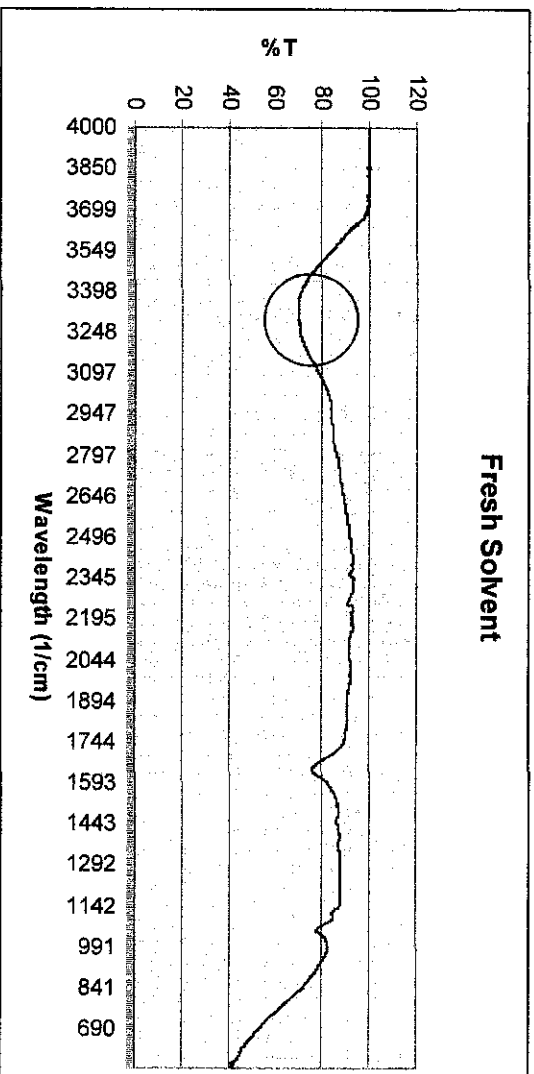


Figure 4.13: IR Spectrum of Fresh STONVENT

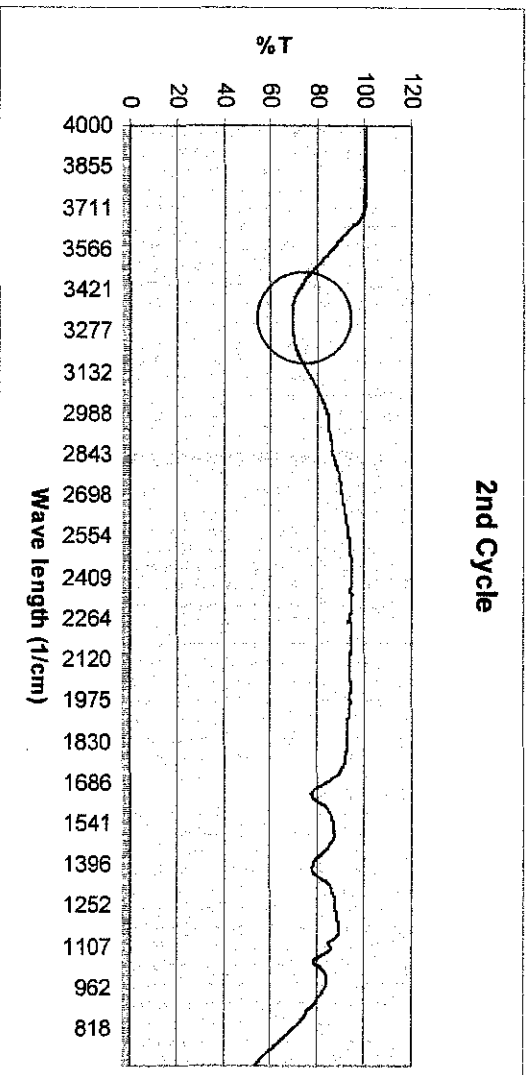


Figure 4.14: IR Spectrum of STONVENT at cycle 2

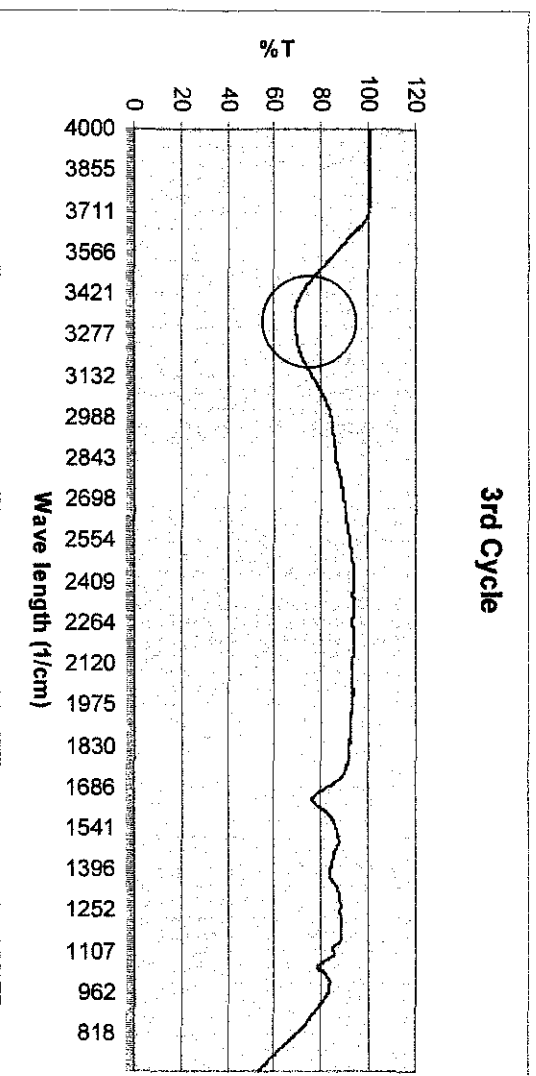


Figure 4.15: IR Spectrum of STONVENT at cycle 3

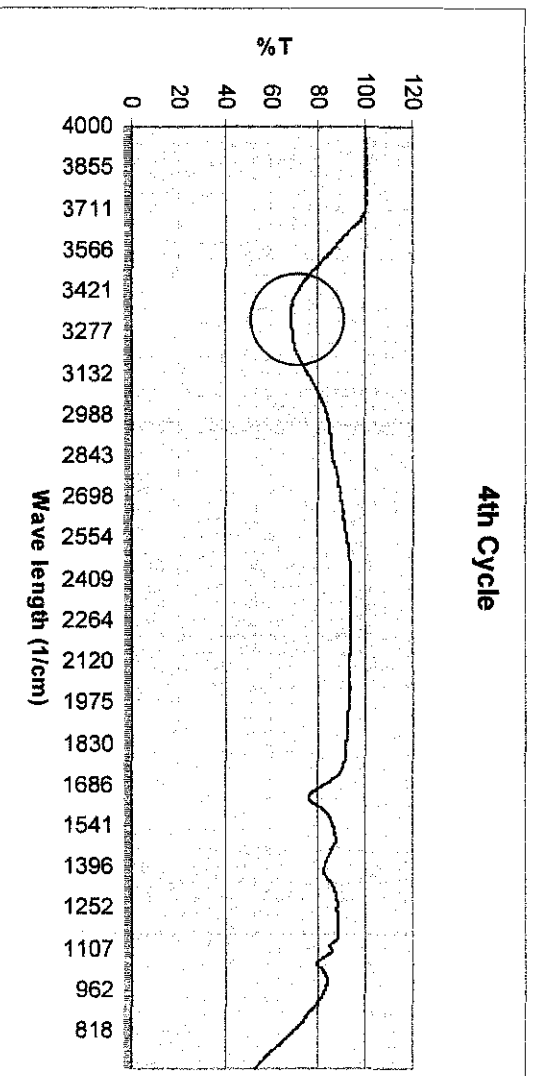


Figure 4.16: IR Spectrum of STONVENT at cycle 4

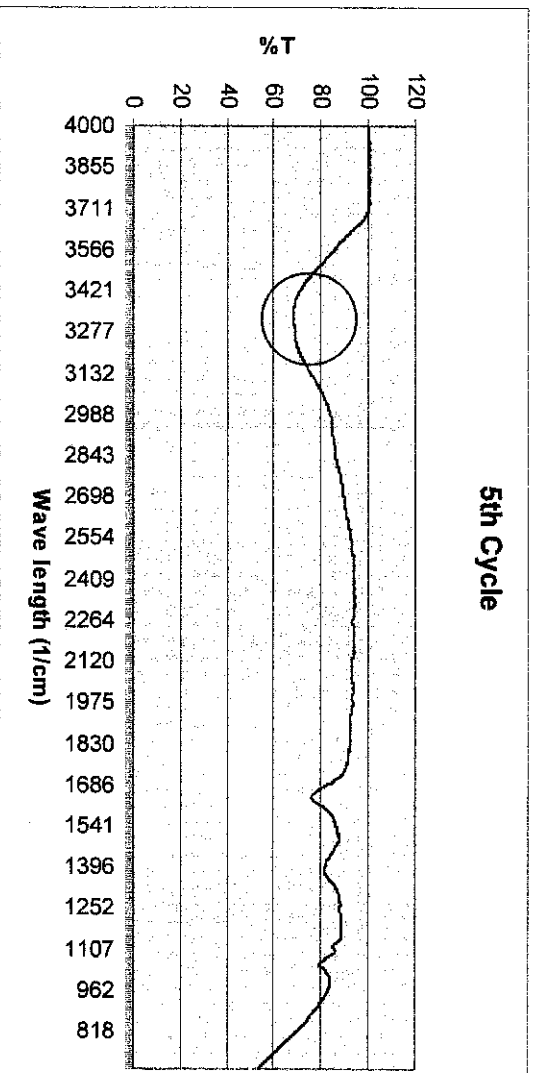


Figure 4.17: IR Spectrum of STONVENT at cycle 5

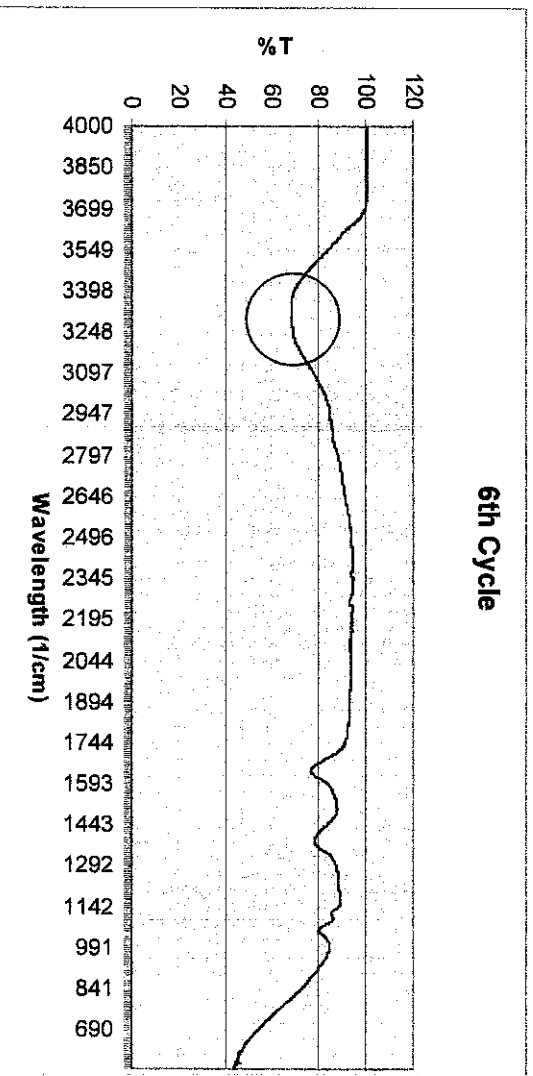


Figure 4.18: IR Spectrum of STONVENT at cycle 6

CHAPTER 5

ERROR & RECOMMENDATION

5.1 Error

1. Solvent might have exposed and thus oxidized and absorbs some CO₂ from the surrounding air.
2. Impurities in the solvents.
3. Some remaining solvent may be inside the line and the column which affect the absorption of CO₂.
4. The concentration of CO₂ in the gas mixture slightly varies (approximately +/- 2%) in one experiment to another.
5. The IR analyzer has inconsistency in the CO₂ measurement.
6. Only run once for each experiment.

5.2 Recommendation

1. Solvent must be kept properly and transported as quickly as possible to avoid oxidization or absorption of CO₂.
2. To ensure solvent is impurity-free before starting the experiment (either filter before the experiment or buy the solvent without any impurities).
3. All the line and column must be clearly washed out by water and purged with air to ensure it is clear from any remaining of solvent or any gases.
4. Buy gas cylinder which contains fixed ration of CO₂ and CH₄ inside.
5. The IR analyzer needs to be calibrated.
6. The experiment need to be repeated at least 3 times to verify the results or to reduce error.

7. To have more flexible absorption rig that can cater the limitation of the existing rig.
8. To compare the performance with other amine solvents which commonly used in the industry such as MEA, DEA and MDEA.

CHAPTER 6

CONCLUSION

Evaluation on the absorption performance of STONVENT for CO₂ removal was performed to cater the problem that the current solvents has. Increasing the solvent's temperature has an impact on the absorption efficiency where the solvent can absorb higher CO₂ as the temperature increases. In this experiment, 50 °C is found to be the best temperature for the solvent since it has the highest absorption efficiency as compared to the other. The experiment also revealed that higher liquid flowrate improves the absorption efficiency and absorption rate of the solvent as compared to lower flowrate. The STONVENT also is proven to have high recycle capacity since it can absorb as much even up to six cycles in the recycle capacity experiment. The experimental result has been supported through TGA and FTIR experiment which confirmed that the solvent still can absorb more CO₂ even it has been recycled six times. Other parameters need to be evaluated as well in the future experiment to further support the claim.

REFERENCES

- Plaza, M.G., Pevida, C., Arenillas, A., Rubiera, F., Pis, J.J., (2007) "CO₂ Capture by Adsorption with Nitrogen Enriched Carbons", *Fuel*. **86**: 2204-2212
- Dindore, V.Y., Brillman, D.W.F., Geuzebroek, F.H., Versteeg, G.F., (2004) "Membrane-Solvent Selection for CO₂ Removal Using Membrane Gas-Liquid Contractors", *Separation and Purification Technology*. **40**: 133-145
- Dortmundt, D., Doshi, K., (1999). *Recent Developments in CO₂ Removal Membrane Technology*. Des Plaines, Illinois, UOP LLC
- Kohl, A., Richard, N., (1997), *Gas Purification*, Houston, Texas, Gulf Publishing Company
- Veawab, A., Aroonwilas, A., Tontiwachwuthikul P., (2002) "CO₂ Absorption Performance of Aqueous Alkanolamines in Packed Columns", *Fuel Chemistry Division Preprints*. **47(1)**: 49-50
- Veawab, A., Aroonwilas, (2004) "Characterization and Comparison of the CO₂ Absorption Performance into Single and Blended Alkanolamines in a Packed Column", *Ind. Eng. Chem. Res.* **43**: 2228-2237
- Yeh, J.T., Pennline, H.W., (2001) "Study of CO₂ Absorption and Desorption in a Packed Column", *Energy & Fuels*. **15**: 274-278
- Rinker, E.B., Ashour, S.S., Sandall, O.C., (2000) "Absorption of Carbon Dioxide into Aqueous Blends of Diethanolamine and Methyl-diethanolamine", *Ind. Eng. Chem. Res.* **39**: 4346-4356
- Lin, C.C., Lin, Y.H., Tan, C.S., (2010) "Evaluation of Alkanolamine Solutions for Carbon Dioxide Removal in Cross-Flow Rotating Packed Beds", *Journal of Hazardous Materials*. **175**: 344-351
- Kuntz, J., Aroonwilas, A., (2009) "Mass-Transfer Efficiency of a Spray Column for CO₂ Capture by MEA", *Energy Procedia* **1**. 205-209
- Dey, A., Aroonwilas, A., (2009) "CO₂ Absorption Into MEA-AMP Blend: Mass Transfer and Absorber Height Index", *Energy Procedia* **1**. 211-215
- Hagewiesche, D.P., Ashour, S.S., Al-Ghawas, H.A., Sandall O.C., (1995) "Absorption of Carbon Dioxide into Aqueous Blends of Monoethanolamine and N-Methyl-diethanolamine", *Chemical Engineering Science*. **50**: 1071-1079

Aroonwilas, A., Veawab, A., Tontiwachwuthikul P., (1999) "Behavior of the Mass-Transfer Coefficient of Structured Packings in CO₂ Absorbers with Chemical Reactions", *Ind. Eng. Chem. Res.* **38**: 2044-2050

Aroonwilas, A., Tontiwachwuthikul P., (1998) "Mass Transfer Coefficients and Correlation for CO₂ Absorption into 2-Amino-2-methyl-1-propanol (AMP) Using Structured Packing", *Ind. Eng. Chem. Res.* **37**: 569-575

Bonenfant, D., Mimeault, M., Hausler, R., (2003) "Determination of the Structural Features of Distinct Amines Important for the Absorption of CO₂ and Regeneration in Aqueous Solution", *Ind. Eng. Chem. Res.* **42**: 3179-3184

Mandal, B. P., Guha, M., Biswas, A. K., Bandyopadhyay, S. S., (2001) "Removal of Carbon Dioxide by Absorption in Mixed Amines: Modeling of Absorption in Aqueous MDEA/MEA and AMP/MEA Solutions", *Chemical Engineering Science.* **56**: 6217-6224

Atalla, M.I., Puxty, G.D., Allport, A.W., Bown, M., Yang, Q., Rowland, R.C., (2009) *Improved Alkanolamines for CO₂ Removal from Gas Streams*

Seader, J.D and Henley, E.J, (1998), *Separation Process Principles*, John Wiley & Son Inc.,

APPENDICES