# PARAMETRIC STUDY OF CO2/CH4 SEPARATION USING HOLLOW FIBER MEMBRANE: EFFECT OF IMPURITIES

By

Stephen Kiu Sie Kiong

Dissertation submitted in partial fulfillment of the requirements for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

May 2014

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CERTIFICATION OF APPROVAL

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Approved by,

____________________

(Dr. Lau Kok Keong)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2014

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.

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Stephen Kiu Sie Kiong

# ABSTRACT

This report shows my research study on hollow fiber membrane module in carbon dioxide separation from natural gas, methane with heavy hydrocarbon as impurities, Hexane. In gas exploration field, presence of carbon dioxide causes most gas reservoir are economically unfeasible and ends up undeveloped. It is because carbon dioxide is highly corrosive when dissolved in water, and it will rapidly destroy pipelines and other equipment. There are other current technologies used to separate carbon dioxide from natural gas such as amine adsorption technology. However, amine absorption technology is expensive and having large equipment size, which is inconvenient to install in offshore platform. Compared to amine absorption technology, hollow fiber membrane is a better option since it has high reliability, cheaper and space saving. However, there are limited studies done on hollow fiber membrane although it developed over 30 years ago. Researches are done for ternary and binary feed which will further discussed in later chapter. But, there is a gap which initiates my research that is studying parametric effect of impurities of heavy hydrocarbon, hexane on separation of carbon dioxide from methane gas. Limited literatures are found for this tittle. Therefore, this research will definitely contributes more understanding for hollow fiber membrane gas separation technologies. .

# ACKNOWLEDGEMENTS

First and foremost, the author desires to express his warmest gratitude to his Final Year Project supervisor, Prof Dr. Lau Kok Keong, for being very helpful and encouraging. From the start of the project, he has been giving guidance from the smallest detail. He has taught how to conduct proper research, starting from the inception through practical hands-on, on to the presentation of findings. He has instilled in the author the sense of team work, good communication skills and project management.

Besides, the author would like to express his appreciation to the graduate assistant, Mrs Serene Lock for her patience in explaining and guiding author in carrying out experiments. Besides, author also want to thank RCCO2 laboratory technician, Mr. Samad for all technical supports and efforts. They have been providing the author with assistance, technical knowledge and resources which are much needed to complete the project apart from providing apparatus. Their aids have been a good push to the project.

Last but not least, the author would like to appreciate the assistance from Dr. Yeong Yin Fong for her willingness to come to laboratory to demonstrate correct ways to pot hollow fiber membrane into module. She also provided consultations and ideas to further the cause.

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# CHAPTER 1

# INTRODUCTION

## 1.1 Background Study

### 1.11 Background of natural gas

Natural gas is one of the most efficient energy sources. The demand for natural gas is increasing rapidly every year. Normally, natural gas contains hydrocarbon and non-hydrocarbon gas. Hydrocarbon gas that present is methane, ethane, propane up to heptane and small amounts of higher molecular weight aromatic hydrocarbons. Besides, some inorganic gas present as well such as carbon dioxide, helium, water vapor and hydrogen sulfide. Presence of carbon dioxide and hydrogen sulfide will acidify the gas field. Removal of this acid gas is essential to prevent corrosion on pipelines and equipment.

### 1.12 Technologies for gas separation

Varieties process of carbon dioxide removal had been developed over years. Nowadays, available process to remove carbon dioxide in gas field are absorption and adsorption process, physical separation and hybrid solution separation (Maddox,R.N, 1982)

Selection among technologies is highly specific for each application. Factors to consider are reservoir conditions, feed gas rate and composition, operating pressure, operating temperature, cost of product gas, availability and cost of utilities and environmental regulations (Faudzi,M., Azhar, n.d.)

## 1.13 Membrane technology

Membrane is a thin, film-like structure act as barrier which allows selective particles to pass through (Wikipedia, n.d.).The concept of using membrane has been known since eighteenth century. However, membrane technology is first applied only in 1981. Synthetic membrane is widely used due to its high performance in separation. Synthetic membrane can be constructed from liquid (non-rigid materials), polymeric (general industrial use) and ceramic(inorganic materials which used in corrosive and high temperature area)

| Type | Hollow fiber | Tubular | Capillary | Spiral Wound |
|---|---|---|---|---|
| **Diameter(mm)** | <0.5 | 5-15 | 0.5-5.0 | 50-200 |
| **Packing density, m2/m3** | 500 to 900 | 30 to 200 | 600 to 1200 | 200 to 800 |
| **Ease of cleaning** | Poor | Excellent | Fair | Fair |
| **Relative cost** | Low | High | Low | Low |

Table 1.13 : Comparison of different types of membrane module (Tan,E.K., 2013)

## 1.14 Membrane performance

Membrane performance in gas separation will be affected by few factors including temperature, pressure drop, feed composition and concentration polarization.. As temperature increases, it will increase the membrane diffusivity but reduce solubility, and therefore it will affect the selectivity and rate of permeation. Higher pressure drop across the membrane will act as driving force for feed component to permeate through membrane, thus increases permeability. In membrane separation, large component will trapped at feed side boundary layer which inhibits the rate of permeation of more permeable species, hence affect selectivity.

## 1.2 Problem statement

Due to the issue of corrosiveness after carbon dioxide mixed with water, it is very crucial to remove carbon dioxide to reduce maintenance cost. Also, gas field will generally located at offshore. Therefore is important to develop an optimal technology for carbon dioxide removal. Among different technologies, hollow fiber membrane is the cheapest and convenient due to its small size.

Hollow fiber technology is introduced to gas field since 1981, but limited research are done to study its performance under different conditions.

Before natural gas can be used as a fuel, it must undergo processing to remove impurities in order to meet the pipeline specifications. The removal of water and $CO_2$ is attempted in the present study under real life process conditions using hollow fiber membrane.

## 1.3 Objectives

The objectives of this project are listed below

1 To develop a hollow fiber membrane from commercialized hollow fiber

2 To study the separation performance of fabricated hollow fiber membrane with and without hexane

## 1.4 Scope of study

This work is focus on the fabrication of hollow fiber membrane and $CO_2$ removal with and without hexane under difference pressure. The focus of this project is on the permeance of the $CO_2$ in the hollow fiber membrane. Graphical analysis for both volume concentration and pressure is done to study the effect of adding hexane. The target of these experiment works is to study the effect of permeance and selectivity of carbon dioxide with or without hexane. The proposed research is expected to benefit the natural gas industries as well as all bulk carbon dioxide producing industries to mitigate environmental pollution, corrosion, and produce purified carbon dioxide for industrial use.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Composition of Carbon dioxide in gas field

Natural gas field can be categorized into sweet and sour gas field. Sour gas field are those which contains significant amount of hydrogen sulphide and carbon dioxide. In Brazil, there are lot of gas field contains carbon dioxide more than 10%. On the other side, carbon dioxide concentration at Australia and South East Asia generally greater than 20%. Highest carbon dioxide concentration recorded is at Platong and Erawa field, Thailand, up to 90%. Malaysian natural gas consist high impurities and the gas composition before its treatment is given below:

Table 1.1 Chemical Composition at Bergading Platform offshore of Terengganu, Malaysia(Karen, 2013)

| **Chemical Name** | **Chemical Formula** | **Percentage (%)** |
|---|---|---|
| Methane | $CH_4$ | 40 – 50 % |
| Ethane | $C_2H_6$ | 5 – 10 % |
| Propane | $C_3H_8$ | 1 – 5 % |
| Carbon Dioxide | $CO_2$ | 2 – 20 % |

## 2.2 HIGH $CO_2$ CONTENT MAKES MOST GAS FIELD UNECONOMICAL

Development of these high $CO_2$ gas fields will requires prudent management of $CO_2$ capture, transportation, and storage to enable commercialization of these gas fields (Faudzi,M., Azhar, n.d.). In Malaysia, there are 13 trillion cubic feet of natural gas remained undeveloped (Tan,E.K,2013).

## 2.3 DISADVANTAGE OF CARBON DIOXIDE PRESENT IN GAS FIELD

Presence of $CO_2$ within gas field will cause (Dortmundt, D., Doshi,K., 1999)

- Reduces heating value of natural gas stream
- Wastes pipelines capacity
- Decreases compression power
- Causes freezing in low temperature equipment

## 2.4 RESEARCH DONE ON TERNARY FEED COMPONENTS IN GAS SEPARATION

TABLE 2.2: Research Done on Ternary Feed Components in Gas Separation

| **Author** | **Finding** |
|---|---|
| *Al-Falahy et al (1998)* | • Develop a scheme for separating SO2, N2 and Co2 using series of liquid membrane cells<br>• Separation factors as high as 244 and 140 for CO2 and SO2 respectively when PEG400 and DEA/PEG400 liquid membrane is used<br>• Found that efficiency of separation declines at high pressure values |
| *M.Hedayat et al (2011)* | • Separation of hydrogen sufide, carbon dioxide from methane with hollow fiber membrane using mixture of alkanoamines<br>• Found that presence of Co2 in feed gas will decrease the efficiency of hydrogen sufide separation from methane.<br>• Found that increase in operating temperature will reduce co2 separation, hence decrease co2 selectivity |
| *Faiz et al (2011)* | • 2D mathematical model developed for separation of co2 and H2S from natural gas using hollow fiber membrane contactors with aqueous carbonate solution<br>• Lower concentrations of carbonate solution result in higher absorption rates for H2S and CO2<br>• Results shows that by using 2 carbonate solution membrane modules in series, C02 and H2S can be completely removed from gas mixture containing 5% CO2, 5% H2S and 90% CH4 |

## 2.5 RESEARCH DONE ON BINARY FEED COMPONENTS IN GAS SEPARATION

TABLE 2.2: Research Done on Binary Feed Components in Gas Separation

| | |
|---|---|
| *Himeno .S (2007)* | • Using DDR-Zeolite membrane for carbon dioxide and methane separation<br>• Results shows high selectivity for carbon dioxide and high carbon dioxide permeance |
| *Ainul(2010)* | • Polysulfone membrane used 100% ethanol as the non-solvent showed that the polymer and the non-solvent pair controlled the morphology of the membrane and eventually affect the performance of the polysulfone membrane |
| *Norwahyu(2011)* | • Duo mode model to study gas sorption, permeability and selectivity of membrane separation for $CO_2/CH_4$ separation<br>• Results shows that sorption, permeability and diffusion property of carbon dioxide-methane exhibit classical duo model behavior |
| *Ghasem(2011)* | • Co2 separation from natural gas by investigate the effect of quenching temperature on characteristics and gas absorption performance of polyvinvylidene fluoride micro porous hollow fiber membranes<br>• Results shows that high quench bath temperature improves the percentage of $CO_2$ removal |
| *Changhai et al (2013)* | • Research on the performance of ester-cross linked hollow fiber membrane for natural gas separations<br>• Successfully enhance $CO_2$ permeance and $CO_2/CH_4$ selectivity significantly by operate at lower temperature |
| *F. Ahmad et al (2012)* | • Study on process simulation and optimal design of membrane separation system for $CO_2$ separation from $CH_4$ by incorporating a 2D cross flow |

| | |
|---|---|
| | mathematical model using Aspen HYSYS |

| | |
|---|---|
| *Yoshimune, M., Haraya,K, 2013* | • Study of permeation properties of single and binary CO2/CH4 mixture using carbon hollow fiber membrane module made from sulfonated poly(phenylene oxide) (SPPO)<br>• SPPO has sharp pore size distribution, 0.35-0.4nm<br>• By using SPPO membrane, CO2/CH4 ideal selectivity is recorded at 25 $^oC$ in single gas system. |
| *Tan,E.K. , 2013* | • Study of Pressure and composition effect on asymmetric hollow fiber membrane for carbon dioxide-methane-pentane separation. |

**2.6 RESEARCH GAP**

Compared to binary gas feed, only limited research has done on ternary feed for gas separation in gas field. Although membrane separation technology industrial application started on 1981. there is still adequate for huge potential to study for different parameter that can increase effectiveness of gas separation. Therefore, my FYP project will be focus on this gap, effect of impurities-Heptane on CO2/CH4 separation (Ternary feed).

# CHAPTER 3

# METHODOLOGY

## 3.1 RESEARCH METHODOLOGIES AND PROJECT ACTIVITIES

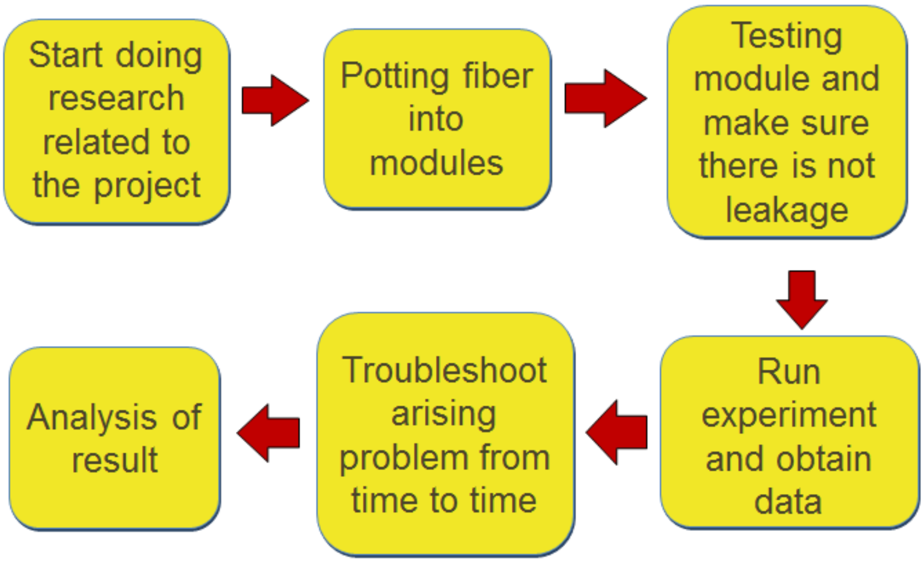


**Figure 3.1 Process flow chart of experiment**

## 3.2 EXPERIMENT WORK

TABLE 3.2: Overview of experimental activities

| **Phase** | **Activities** |
|---|---|
| Pre-experiment | Identifying the problems and purpose of experiments |
| | Study related journal to understand the theory and concept how impurities will affect effectiveness of $CO_2/CH_4$ separation |
| | Study of parameters and variables that will be used in the experiments |
| | Checking availability of equipment and chemicals |
| | Prepare laboratory documents such as material safety data sheet, job safety analysis and experiment procedures |
| Experiment | Potting Fibers into Membrane Module |
| | Membrane Characterization |
| | Run experiment |
| Post-experiment | Review experimental result |
| | Plot graph to analyse the result |
| | Repeat the experiment to get constant trend |
| | Conclude findings of the experiment and recommendations |
| | Compare experimental findings with theory based on literature review |
| | Documentation and reporting |

## 3.21 MODULE PREPARATION

The module does not need to be developed as it is already available subject to prior research conducted by previous students. However, it needs to be prepared before potting the fibers. The epoxy holding the membrane needed to be removed by means of drilling. This was done by drilling using a hand drill.

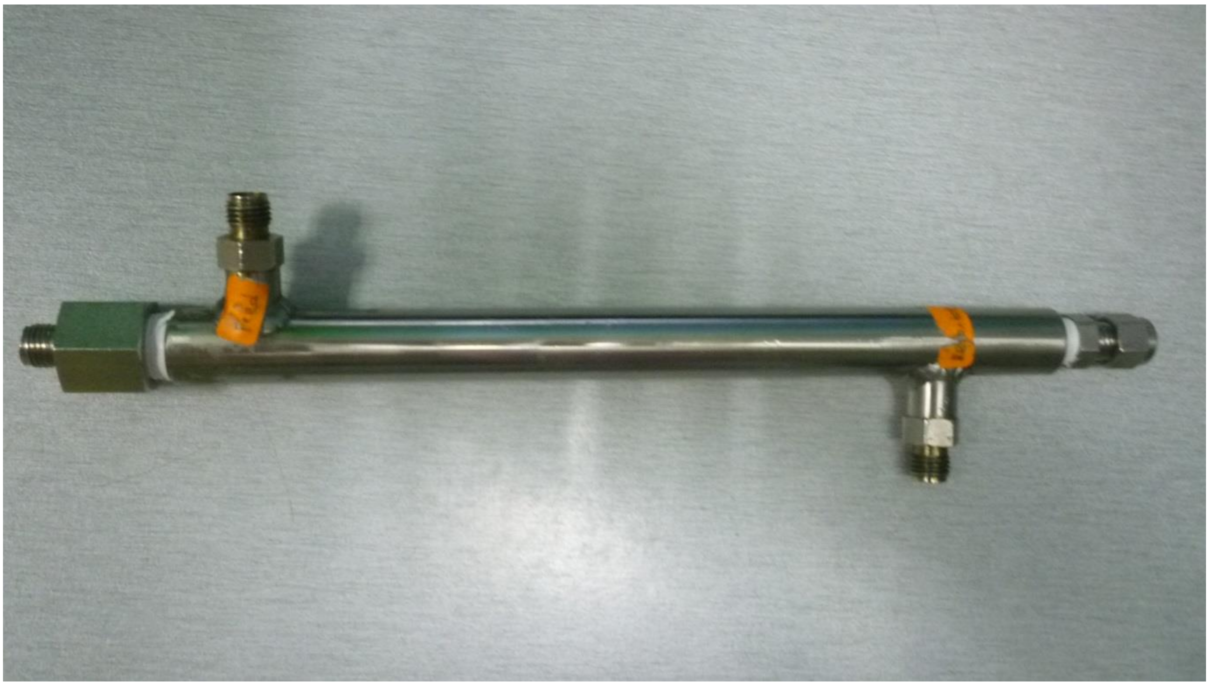

Figure 3.2a: A completed hollow fiber membrane module

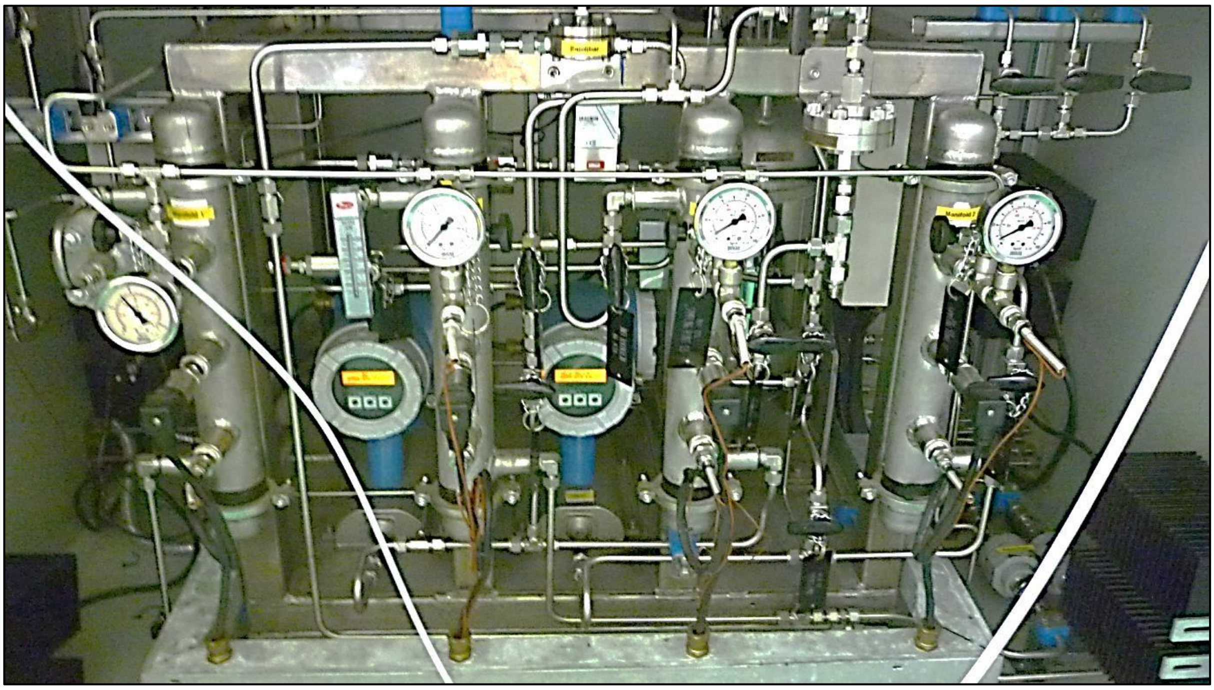

Figure 3.2b: Automated system to study used to study gas separation for membrane

## 3.3 POTTING OF FIBERS

Before potting of fibers into the module prepared, the packing density needs to be decided. The packing density can be calculated based on the formula given:

$$packing\ density\ (\emptyset) = no.of\ fibers\ (n) \times \frac{cross - section\ area\ of\ a\ fiber}{cross - section\ area\ of\ module}$$

**Equation 3.1: Membrane Packing Density**

For the purpose of this experiment, a constant number of fibers will be set as the packing density, but it is not one of the tests parameter. The number of fibers will be chose depending on the constraint set by the diameter of the module. The fiber potting can be divided into several categories, which are:

- Module bundle preparation
- Module assembly
- Epoxy resin casting

### 3.3.1 MODULE BUNDLE PREPARATION

1. Calculate the number of fibers and the length of the fibers base on the diameter of hollow fibers and the length of module.
2. Cut the fibers to a desired length. Remove the visibly defective fibers. Place the fiber in parallel order and put it together as a fiber bundle.
3. Cut a piece of paraffin film at each end, stretch it slowly without snapping to four or five times of its original length. Wrap on one end of the fiber bundle before it relax to a natural condition. Cut the wrapped end with a razor blade to yield a smooth cross-section.
4. Encircle the wrapped end with a thin string and make sure the diameter is smaller than the inner diameter of the shell.

### 3.3.2 MODULE ASSEMBLY

1. Place the shell vertically on a holder and leave enough space under the module shell so that it can accommodate the fiber bundle.
2. Lay a long string through the shell lumen and tie with the thin string that is roped upon the fiber bundle.
3. Pull the long string gently upwards so that the fiber bundle is housed in the shell at a designed position. The untied portion of the fiber bundle should be suspended freely and hang loosely; thus the fibers become ordered and packed naturally when being pulled into the shell.
4. Repeat step (3) in the Module bundle preparation and wrap the other end of the fiber bundle with a piece of paraffin film. Each end should emerge out of the module shell with a length of 10mm.

### 3.3.3 EPOXY RESIN CASTING

1. Apply a layer of Araldite 5 min curing adhesive on the cross-sections of the bundle ends to seal each hollow fiber and prevent the creeping of epoxy through the fiber lumens by the capillary flow.
2. Mix the suitable proportion of epoxy and hardener.
3. Fill a 50 ml syringe with the epoxy resin mixture slowly so that no air bubbles are generated. Put in the piston and manually push it forward to discharge any air trapped in the syringe. Then continuously push the piston till the liquid-like epoxy mixture completely covers the space.
4. Leave the module in a dry room overnight so that the epoxy can completely dry.

## 3.4 GAS SEPARATION TESTING UNIT

Install the hollow fiber membrane module in the experimental set up as shown in the diagram below:

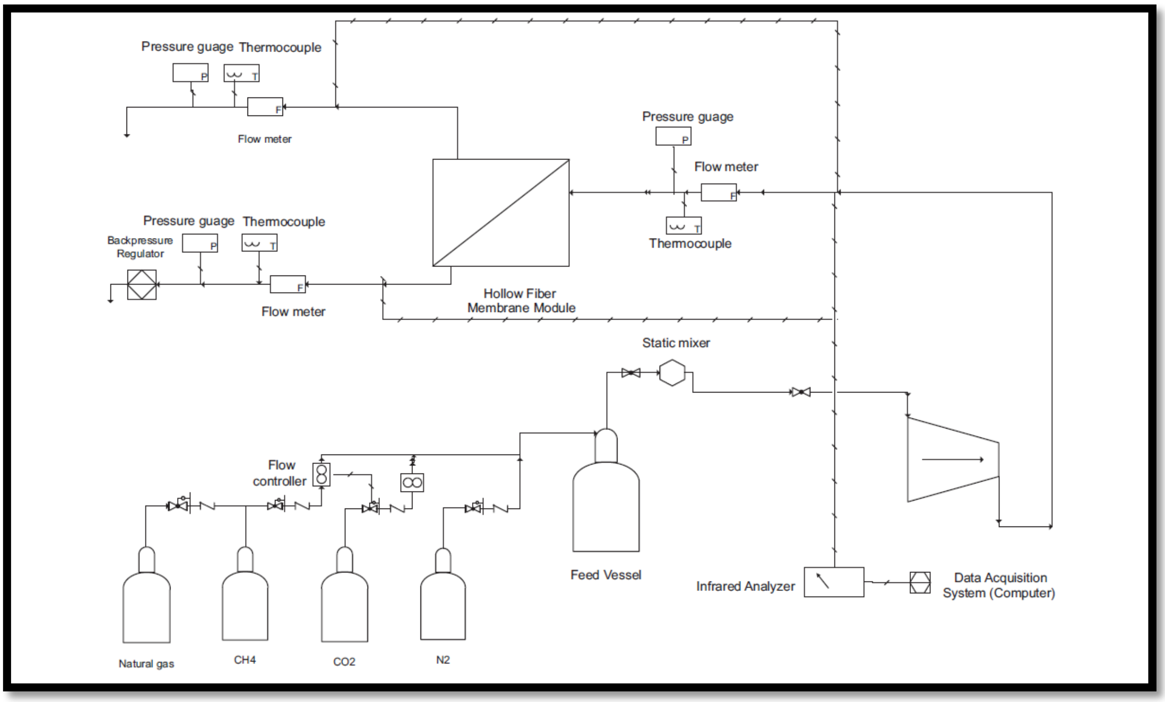


FIGURE 3.4: Flow sheet of gas separation testing unit for experimental validation

The testing unit mainly consists of gas cylinders, mass flow controllers, compressor, and infrared analyser. There are two experiments that will be conducted in this study, which are:

- The effect of feed pressure on relative permeance and permeance of gases
- The effect of membrane area on relative permeance and permeance of gases

### 3.4.1 STARTING THE SYSTEM

1. Turn on the main power supply inside the control panel.
2. Switch on the main power supply to the computer.
3. Activate NI lab view and allow the software to load completely.
4. Switch on the analyser switch at the control panel.
5. Set the operating temperature according to the experiment.

### 3.4.2 HEATING UP THE HOT WATER SYSTEM

1. Power up the main power of the control panel of the hot water system.
2. Set the heater temperature to 80 ºC.
3. Circulate the hot water inside the heat exchanger using pump.
4. Open the valve at the top of $CH_4$ and $CO_2$.

### 3.4.3 SETTING UP FEED GAS

1. Open the inlet and outlet valve for $CO_2$ and $CH_4$.
2. Set the feed pressure regulator according to the experiment.
3. Set the flow rate of $CO_2$ and $CH_4$ according to the experiment mass flow controller.

### 3.4.4 SETTING UP MANUAL BACK PRESSURE REGULATOR

1. Use the high pressure regulator to regulate the retentate side pressure.
2. Turn the knob clockwise up to set the pressure to I bar less than the feed pressure.

### 3.4.5 TAKING THE READING

1. Slowly open the needle valve at the top of manifold 1, 2, 3, or 4.
2. Open the inlet valve to the gas analyser.
3. Wait till the reading of gas analyser stabilizes.
4. Take the reading of the gas analyser.
5. Repeat the process using different membrane area.

### 3.5 GHANTT CHART ( -milestones achieved -Pending)

| Detail/Week | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| FYP Topic Familiarization | | | | | | | | | | | | | | |
| Identifying Problem Statement | | | | | | | | | | | | | | |
| Define FYP Objectives | | | | | | | | | | | | | | |
| Outline Scope of Study | | | | | | | | | | | | | | |
| Literatures Reviews | | | | | | | | | | | | | | |
| Lab Visit and Briefing by Lab Technician | | | | | | | | | | | | | | |
| Checking Availabilities of Equipments and Chemicals | | | | | | | | | | | | | | |
| Review Experimental Procedures | | | | | | | | | | | | | | |
| Fabricate Hollow Fiber Membrane | | | | | | | | | | | | | | |
| Potting New fiber into Membrane Module | | | | | | | | | | | | | | |
| Experiment Work on Membrane Separation of CO2 from Heptane | | | | | | | | | | | | | | |
| Data Gathering and Analysis | | | | | | | | | | | | | | |
| Documentation and Writing Report | | | | | | | | | | | | | | |

# CHAPTER 4

# RESULTS AND DISCUSSION

## 4.1 ANALYSIS OF RESULTS

The result that will be obtained through the experiments will be analysed mainly in terms of permeability and selectivity. The permeability for individual gases can be calculated using:

$$P_A = \frac{\dot{m}_A \times MW_A \times 22400}{A_m \times \Delta P}$$

**Equation 3.3a: Permeability of individual gas**

Where: $\dot{m}_A$ = Mass flow rate of A, g/s

$MW_A$ = Surface area of membrane, $cm^2$

$\Delta P$ = Pressure difference between feed and permeate side, cmHg

The selectivity of $CO_2$ over $CH_4$ can be calculated using:

$$A_{AB} = \frac{P_A}{P_B}$$

**Equation 3.3b: Selectivity of the gas**

Where: $P_A$ = Permeability of A, GPU

$P_B$ = Permeability of B, GPU

**4.2 RESULT EXPERIMENT 1 (WITHOUT HEXANE)**

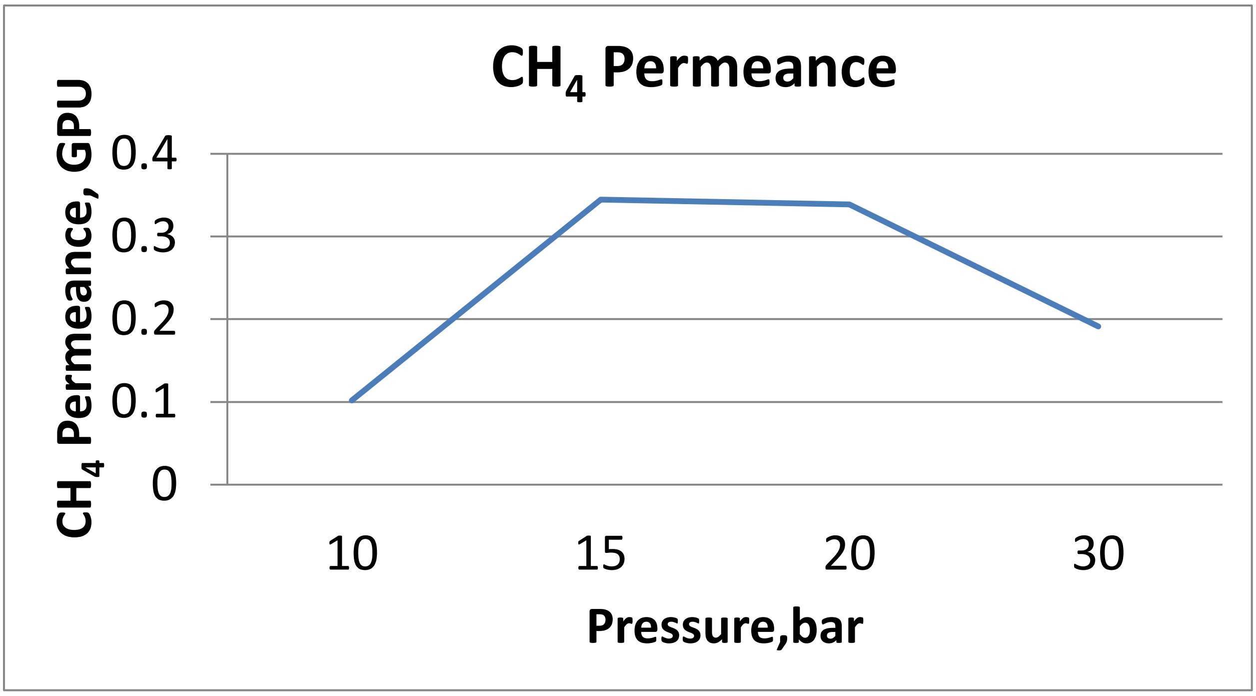


Figure 4.2a: Permeance of $CH_4$ againt pressure graph

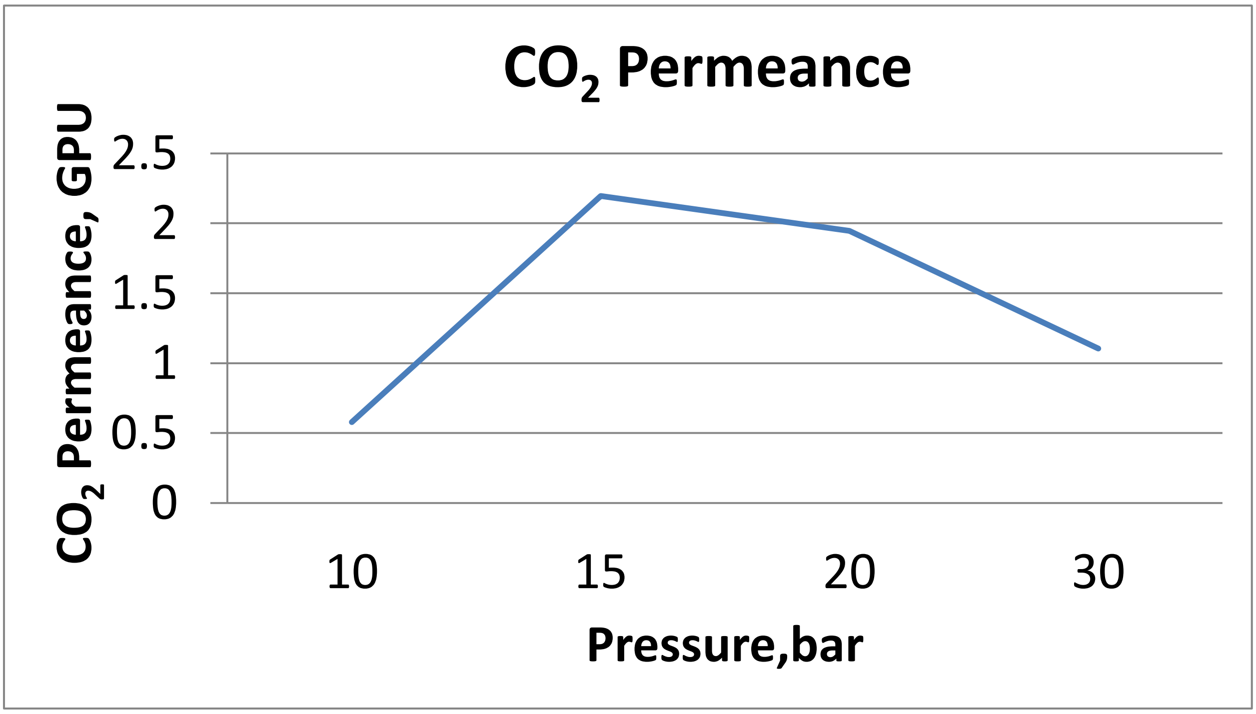


Figure 4.2b: Permeance of $CO_2$ against Pressure graph

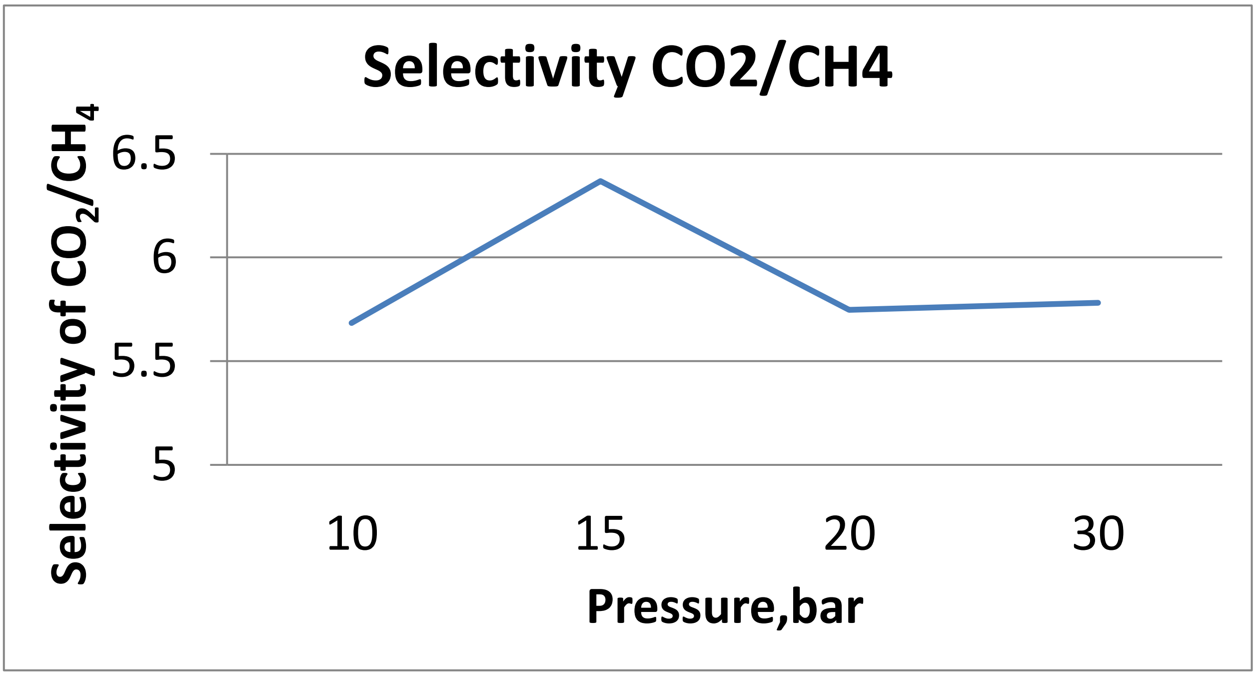


Figure 4.2c: Selectivity of $CO_2/CH_4$ against pressure graph

**Discussion**

From figure 4.2a, b and c it shows that CO2 and CH4 permeance increases at first and then decrease back after 15 bar. Permeance increases first due to plasticization phenomenon.( Wahyyu). It further decrease due to compaction effect.(Source). Sweeling occurs instantaneously below plasticization pressure, until it reaches an equilibrium.

### 4.3 EXPERIMENT 2 (WITH HEXANE)

Before doing experiment 2 by including hexane in feed as vapor, author calculate the required temperature to vaporize liquid hexane by using equation

$$xP *= yP$$

Followed by using Antoine equation to calculate respective temperature at the respective set pressure.

$$log10\ P = A - \frac{B}{C + T}$$

Unfortunately, the hot box which controls the overall system temperature broke down while carrying up experiment 2. No relevant data can be obtained or published for author to compare with experiment 1.

# CHAPTER 6

## 6.1 CONCLUSION

Due to equipment failure and time constraint, author is not able to achieve ultimate objective of study the effect of adding hexane as impurities. The experiment on heavy hydrocarbon has to be presumed after hot box heater is fixed. However, author can conclude from data of experiment 1 is increasing operating pressure will affect carbon dioxide and methane presence, and also its selectivity. In conclusion, permeance will increase until reaches its plasticization pressure, and decreases due to compaction**.**

## 6.2 RECOMMENDATION

Author recommend for next FYP student to continue study on effect of heavy hydrocarbon as it will give yields more understanding performance of hollow fiber membrane. It would be greatly beneficial in the future. Further optimization and intensification work to enable the use of this technology for offshore application. For future research, instead of ternary feed, mixture of other hydrocarbon can be include at once to study their effect on separation performance. In reality, gas field exploration will have more than 1 impurity Since the limited studies had done on ternary feed for separation of $CO_2$ using hollow fiber membrane, more research work needed to be done in order to assess the system. More analysis and sensitivity study is required to be undertaken.

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