Performance Evaluation of Hollow Fiber Membrane for Carbon Dioxide Separation: An Experimental Approach

by

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CERTIFICATION OF APPROVAL

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Approved by,

____

(Dr. Lau Kok Keong)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
SEPT 2012

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.

29/8/12

(CHENG SYN YANG)

ABSTRACT

This report outlines the background of the project "Performance Evaluation of Hollow Fiber Membrane for Carbon Dioxide Separation: An Experimental Approach". Due to the fact that carbon dioxide (CO_2) is present in all natural gas sources worldwide, there is much attention placed on the efficiency in separating CO_2 from natural gas. Recent developments have discovered the benefits of utilizing membrane for this purpose.

The scope of this study is on the performance of hollow fiber in removing carbon dioxide with respect to the variations in pressure, composition, feed gas flow and membrane area or better represented as number of hollow fibers used in a membrane module. The procedures are conducted in the Research Centre for Carbon Dioxide Capture ($RCCO_2C$) with the Carbon Dioxide Separation Membrane Unit (CO_2SMU). The performance is evaluated based on experimental approach and the results are to be analyzed and discussed with reference to previous works as well as separation theories and principles.

In the first chapter, a general introduction is given on the separation techniques available in the current market which is followed by the problem statement on the drawbacks of the conventional separation techniques, of which supports the use of membrane for CO_2 capture. Next, the objectives and the scope of this study are defined.

In the following chapter, literature review is conducted to find out on membrane separation technology including general principles, key characteristics of membrane, its classifications, separation theories and principles, as well as the benefits of membrane for separation processes.

In Chapter 3, the general approach on the conduct of this study is illustrated, which generally summarizes the scope of work for this project. Also, the experimental procedures are illustrated in a detailed manner to ensure that the steps are that required to obtain satisfacatory results. Besides, all the chemicals required are also listed while each the equipments in the setup are defined for its function and purpose.

Next, reports on the findings of the experiments are illustrated. The experimental parameters conducted in the study includes pressure, feed composition, feed gas flow as well as membrane area. It was found that the stage cut and the selectivity of the membrane increases with pressure and feed composition while these two parameters shows a decrease when the feed gas flow rate is increased. Generally, the number of fibers used does not affect the selectivity of the membrane but the stage cut of the membrane is largely increased when the number of fibers used is increased. In terms of permeance, it was found that the gas permeance increases with increasing feed composition, feed gas flow rate as well as number of fibers or membrane area but decreases with increasing feed pressure.

Last but not least, the conclusion of this report summarizes the document as a whole together with the obtained results of this experimental work, in terms of the focus of this study, which is the effect of pressure, feed gas flow, CO_2 composition, and number of fibers in a module on the performance of the hollow fiber membrane.

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CHAPTER 1

INTRODUCTION

1.1 Background

The hike in the emission of carbon dioxide (CO_2) and the resulting environmental impacts has driven the development of sustainability as well as Carbon Capture and Storage (CCS). Fossil fuels are with 86% the dominant energy source utilized in the world where more than one-third of the CO_2 emissions come from the combustion of fossil fuels in power plants worldwide while the emission of CO_2 associated with the use of CH_4 is more than significant.

Natural gas (NG) is a vital component of the world's supply of energy. It is one of the cleanest, safest, and most useful types of energy sources. In recent years, due to the sharp increase in the price of oil, natural gas and processes related to its purification have attracted more attention than before. The increasing world-wide demand for energy has resulted in the exploration of NG with higher contamination of CO_2 as the number of easily accessible and NG sources containing low percentage of CO_2 is very limited.

The main sources of commercial CO_2 emissions include synthetic ammonia and hydrogen plants, flue gas from fossil fuel combustion processes, fermentation, lime-kiln operations, sodium phosphate manufacture, and natural gas wells (Othmer, 1999). There are in fact three incentives in removing carbon dioxide from a process stream. Firstly, it can be removed from a valuable product gas, such as hydrogen where it is emitted to the atmosphere as a waste by-product. Secondly, the recovered CO_2 from process gas, such as in ethanol production, can be marketed as a saleable product. Thirdly, CO_2 is recovered simply to prevent it from being released to the atmophere but this necessarily requires sequestration of the recovered CO_2 (Ritter & Ebner, 2004).

Besides its environmental impacts of global warming, CO_2 poses drawbacks such as reducing the heating value of natural gas. CO_2 by nature displays acidic characteristic that can lead to severe corrosions in equipments and pipelines in processing plants. The allowable CO_2 content in pipelines is marked at 2-5% while for Liquefied Natural Gas (LNG) applications, the content must be reduced to 50-100ppm, as they will freeze under cryogenic temperatures. This makes the removal of CO_2 from natural gas of crucial importance. Hence, development efforts are underway to improve the efficiency of separation processes to mitigate CO_2 emissions.

CO_2 separation technologies varies from techniques such as reversible solvent absorption using alkanoamines, molecular sieve adsorption, cryogenic distillation and more recently, membrane separation. The most widely used technique for removing carbon dioxide is absorption into alkanolamines solutions in classical contactor equipments. However, besides occupying huge spaces, these processes require high initial investment costs. Membrane technology is considered as an alternative technology which can potentially overcome the operational shortcomings encountered in traditional processes such as entrainment and flooding at high flowrates and reduces considerably the foaming effects (Noureddine Boucif 2011).

The current market depicts that the membrane market devoted to CO_2 separation from natural gas is about 20% which is only 2% of the total separation markets for natural gas. Amine-based absorption processes dominates the market. However, membranes are used in situations where the produced gas contains high levels of CO_2. In spite of this, the membrane must be protected from heavier heavy hydrocarbon such as C_{5+}, which can immediately degrade the performance and can cause irreversible damage to the membranes. Currently, membrane units has seen a number of applications such as for H_2 purification in reforming processes by UOP and Air Products and Chemicals and is seen to be a promising alternative to the other technologies with acticipated improvements and breakthroughs in membrane development which will lead to significant reductions in energy consumption, environmental impacts and feedstock requirements, and therefore considerable improvements in the process economics (Ritter & Ebner, 2004).

1.2 Problem Statement

With the limited sources of natural gas with low CO_2 content, it is then inevitable that exploration of lower quality sources must be done to keep up with the energy demand. It is then of crucial importance to remove the carbon dioxide content in the natural gas efficiently to an acceptable range so as to protect plant equipments, at a minimal cost. Also, the U.S. Department of Energy has shown that the separation of CO_2 represents 75% of the overall cost associated with its separation, storage, transport, and sequestration. Hence, more energy-efficient CO_2 separation processes are needed.

Current carbon dioxide capture techniques suffer from problems such as flooding, foaming, entraining, channeling, as well as high capital and operating costs (Sutrasno Kartohardjono 2009; Moradi 2010; Majid Mahdavian 2012). The most prominent capture technology is based on liquid-gas column absorption involving chemisorption of the CO_2 into aqueous alkanolamine solutions, such as monoethanolamine (MEA) and monodiethanolamine (MDEA). However, in this technology, there is an intensive energy requirement for solvent regeneration using steam to strip off the carbon dioxide, often using a kettle reboiler (Ryan P. Lively 2009). Besides, losses of the absorbent is also inevitable due to the evaporation at high temperatures which adds to the operating cost for solvent make-up.

Therefore, there is an urge for the development of separation technologies that could overcome the drawbacks in the conventional techniques and one of the promising techniques is the use of polymeric hollow fiber membrane which offers several obvious advantages compared to the earlier technologies.

1.3 Objective

- To evaluate the performance of the polyimide hollow fiber membrane under different experimental conditions of pressure, feed composition (CO_2-CH_4), feed gas flow and number of fibers in a module.

1.4 Scope of Study

For this experimental study, the scope includes the fabrication of the hollow fiber membrane module whereas the focus is to study the effects of pressure, feed gas composition, feed gas flow as well as the membrane area on the performance of the hollow fiber. A binary system of CO_2/CH_4 is studied where carbon dioxide is to be removed from methane. The effects on the performance of the polyimide hollow fiber membrane is studied by varying the experimental parameters of pressure, feed composition, feed gas flow and number of fibers in a module. The results are presented in terms of quantitative measure (stage cut) as well as qualitative measure (selectivity and permeance) as an evaluation of the performance of the hollow fiber membrane.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction on Membrane

Membrane-based CO_2 removal systems have become an established technology in the oil and natural gas industry (David R. Koch). The first use of membrane for carbon dioxide removal is in 1981 which initially had slow acceptance and is limited to smaller streams due to the economic risks. However, the multiple benefits of membrane technology have been proven in a wide variety of installations in locations around the world (David Dortmumdt 1999).

Membranes are thin semipermeable barriers that selectively separate some compounds from others. There are basically two types of polymeric membranes, namely glassy and rubbery. The glassy polymeric membranes have a glass transition temperature that is higher than room temperature. On the other hand, the rubbery polymeric membranes have a glass transition temperature that is well below room temperature. In most industrial applications, the glassy polymeric materials are preferred as opposed to their rubbery counterparts because of their superior mechanical properties and overall permeability-selectivity tradeoffs. Common glassy polymeric materials are such as cellulose acetate, polysulfones, polyimides, polyaramides, polyetherimide and polycarbonates.

The most widely used and tested material is cellulose acetate. Polyimide has some potential in certain CO_2 removal applications due to its thermal, chemical and plasticization resistance and considerably high mechanical strength, but it has not received sufficient testing to be used in large applications. Both permeability and selectivity are important considerations when selecting a membrane. The higher the permeability, the less membrane area is required for a given separation and therefore the lower the system cost. The higher the selectivity, the lower the losses of hydrocarbons as CO_2 is removed and therefore the higher the volume of salable product. Highly permeable materials are often low in selectivity, and vice versa due to the permeability-selectivity tradeoff, though achieving this combination is a constant goal for membrane scientists (David Dortmumdt 1999).

The membrane used in gas separation are manufactured either in the form of flat sheet or hollow fibers (85% of the market). The flat sheets are typically combined into a spiral-wound element, and the hollow fibers are combined into a bundle similar to a shell and tube heat exchanger (David Dortmumdt 1999). In this document however, the focus would be on the hollow fiber membrane as the scope of the study involves investigation on the performance of hollow fiber membrane under different experimental parameters. The element types of membranes are illustrated in the figures that follows.

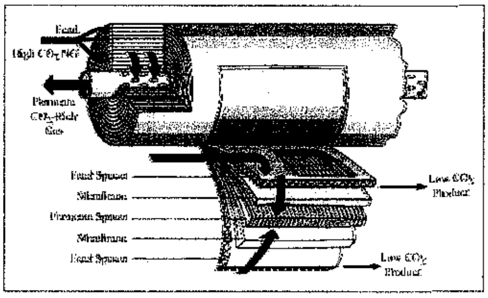


Figure 1: Spiral Wound Membrane Element

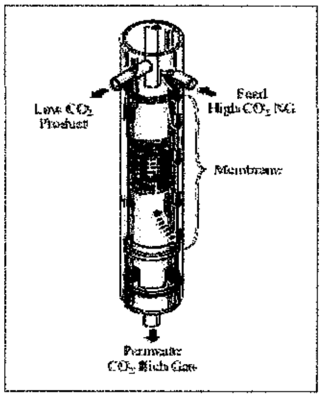


Figure 2: Hollow-Fiber Membrane Element

Adopted from "Recent Developments in CO2 Removal Membrane Technology" by Dortmundt & Doshi (1999).

2.2 Classification of Membrane Processes

In membrane separation, separation occurs by the membrane controlling the rate of movement of various molecules between two liquid phases, two gas phases, or a liquid and a gas phase. The two fluid phases are usually miscible and the membrane barrier prevents actual, ordinary hydrodynamic flow. The table on the following page summarizes the main types of membrane separation processes.

Table 1: Membrane Processes Classification

Membrane Processes	**Description**
Gas diffusion in porous solid	- A gas phase is present on both sides of the membrane, which is a microporous solid. - The rate of molecular diffusion depends on the pore size and molecular weight.
Liquid permeation or dialysis	- The small solutes in one liquid phase diffuse readily because of concentration differences through a porous membrane to the second liquid/vapour phase. - Passage of large molecules through the membrane is more difficuly.
Gas permeation in a membrane	- The membrane is usually a polymer such as rubber, polyamide and so on, and is not a porous solid. - The solute gas first dissolves in the membrane and then diffuses in the solid to the other gas phase and separation occurs as each type of molecule diffuses at a different rate through the membrane.
Reverse osmosis	- A membrane, which impedes the passage of a low-molecular-weight solute, is place between a solute-solvent solution and a pure solvent. - The solvent diffusses into the solution by osmosis and in reverse osmosis, a reverse pressure difference is imposed which causes the flow of solvent to reverse, as in the desalination of seawater.
Ultrafiltration membrane process	- Pressure is used to obtain a separation of molecules by means of a semipermeable polymeric membrane. - The membrane discriminates based on the molecular size, shape, or chemical structure and separates relatively high-molecular-weight solutes such as proteins, polymers, colloidal materials such as minerals, etc.

Microfiltration membrane process	- Pressure-driven flow through the membrane is used to separate micron-size particles from fluids which are typically larger than those in ultrafiltration.
Gel permeation chromotography	- The porous gel retards the diffusion of the high-molecular-weight solutes whereby the driving force is concentration. - This process is useful in analyzing complex chemical solutions and in the purification of very specialized and/or valuable components.

2.3 General Hollow Fiber Membrane Characteristics

Hollow fibes are similar to a tube with very small diameter and often, the inside diameter of the fibers is in the range of 100 – 500 μm and the outside diameter is in the range of 200 – 1000 μm, with length up to 3 – 5 m. Thousands of fine tubes are bound together at each end into a tube sheet that is surrounded by a metal shell having a diameter of 0.1 – 0.2 m, so that the membrane area per unit volume is up to 10,000m^2/m^3. A typical large industrial permeator has fibers of 200 μm ID and 400 μm OD in a shell of 6 inch inner diameter and with length of 10 ft.

Typically, the high-pressure feed enters the shell side at one end and leaves at the other end. The hollow fibers are closed at one end of the tube bundles, commonly sealed with epoxy. The permeate gas inside the fibers flows countercurrent to shell-side flow and is collected in a chamber where the open ends of the fibers terminate. Then, the permeate exits the device. In some lower pressure operations, such as separation of air to produce nitrogen, the feed enters inside the tubes (Geankoplis, 2003).

2.4 Advantages of CO_2 Separation using Hollow Fiber Membrane

Unlike conventional CO_2 separation techniques using columns such as packed and tray towers, bubble columns, venture scrubbers and spray towers, membrane contactor has several obvious advantages. These include higher mass transfer rates, known and constant interfacial area, easy scale-up due to its modular design, no operational problems such as foaming, flooding and entrainment, as well as lower capital and operating cost.

By forming the membranes as hollow fibers, a very compact unit can be made with a specific area 1600–6500 (m^2/m^3), which is higher than 30–330 (m^2/m^3) offered by packed/tray towers and 160–500 (m^2/m^3) for mechanically agitated columns. Although the membrane wall introduces an additional resistance, which does not exist in conventional towers, the higher and fixed surface area in this type of modules offers much higher mass transfer rates (P. Keshavarz 2007), which all leads to significant operational and capital cost savings.

Besides, the membrane separation of CO_2 requires relative low energy consumption comparing to absorption or distillation type. Khoo and Tan (2006) stated that membrane technology uses 70-75 kWh per ton of recovered CO_2 compared to significantly higher values for pressure swing adsorption (160-180 kWh), cryogenic distillation (600-800 kWh) or amine absorption (330-340 kWh), making membrane technology an attractive alternative. It can be operated easily and inexpensively since it is a physical separation mechanism. Also, it is suitable to apply in a small-to-medium scale of CO_2 separation process (Dae-Hwan Lee 2004). These figures can be better compared in Table 2 and Table 3.

Table 2: Separation Techniques and their Range of Specific Area

Separation Techniques	**Specific Area (m^2/m^3)**
Hollow Fiber	1600–6500
Packed/tray towers	30–330
Mechanically agitated columns	160–500

Table 3: Separation Techniques and their Range of Energy requirement

Separation Techniques	**Energy requirement/ton of recovered CO_2 (kWh)**
Membrane	70-75
Pressure swing adsorption	160-180
Cryogenic distillation	600-800
Amine absorption	330-340

2.5 Membrane Separation Principles

Membranes act as filters to separate one or more gases from a feed mixture and generate a specific gas rich permeate. Two characteristics which dictates membrane performance is permeability and selectivity. Permeability refers to the flux of a specific gas through the membrane while selectivity is the membrane's preference to pass one gas species and not the other (Colin A. Scholes 2007). A schematic diagram of membrane gas separation is shown below:

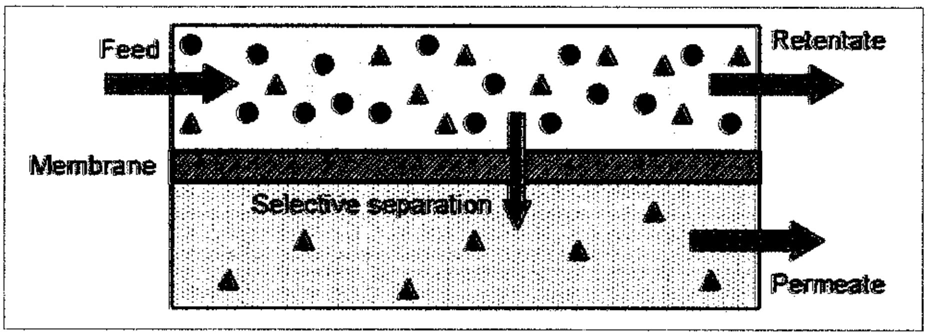


Figure 3: Schematic of Membrane Gas Separation

Adopted from "Carbon Dioxide Separation through Polymeric Membrane Systems for Flue Gas Applications" by Scholes et al. (2008).

Fick's Law, as shown below, is widely used to approximate the solution-diffusion process:

$$J = \frac{k \times D \times \Delta p}{l}$$

where,

J = membrane flux of CO_2, that is, the molar flow of CO_2 through the membrane per unit area of membrane.

k = solubility of CO_2 through the membrane

D = diffusion coefficient of CO_2 through the membrane

Δp = partial pressure difference of CO_2 between the feed (high pressure) and permeate (low pressure) side of the membrane

l = membrane thickness

2.5.1 Permeability

In general, the relationship between permeability, diffusivity and solubility in an ideal case can be described by:

$$P = D.S$$

where,

P = the permeability coefficient [cm^3(STP)$cm^{-2}s^{-1}$ $cmHg^{-1}$].

D = the diffusivity coefficient [cm^2 s^{-1}]

S = the solubility coefficient [cm3(STP)$cmHg^{-1}$]

The solubility and diffusion coefficients are usually combined into a new variable called *permeability* (P). Fick's law can therefore be split into two portions; a membrane-dependent portion, (P/*l*) and a process-dependent portion, (Δp). Hence, in order to achieve a high flux, the correct membrane material and the correct process conditions are needed. Note that P/*l* is not a constant; it is sensitive to a variety of operating conditions such as temperature and pressure (Dortmumdt, 1999).

For ideal gases, the permeability is related to the gas permeation rate though the membrane (Q), the surface area of the membrane (A), the thickness of the membrane, *l* and the driving force for separation, the pressure difference across the membrane (Δp).

$$\frac{P}{l} = \frac{Q}{A.\Delta p}$$

In our binary gas system, CO_2 is more condensable and more polar than CH_4 and a higher CO_2 solubility in the polymer membrane can be expected. As for diffusivity, it is influenced by the size of the gas molecules and the diffusion coefficient decreases with increasing kinetic diameter of the gas. As can be seen from the figure below, the diameter of CO_2 is smaller compared to CH_4 which aids the diffusivity across the membrane. In fact, the linear orientation of the carbon dioxide molecule also aids in the diffusion through the membrane.

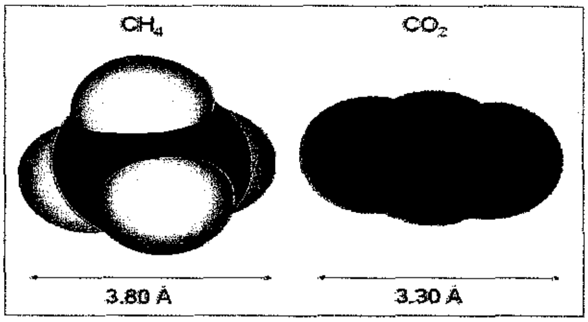


Figure 4: Schematic Representation of CH_4 and CO_2

Adopted from "Membrane Technologies for CO2 Capture", by Simon (2010).

Membranes are an attractive option for carbon dioxide separation, mainly because of the inherent permeating properties of this species. Besides being a fast gas, CO_2 also has a relatively high molecular weight and a large quadraple moment, enabling it to absorb more stongly to or dissolve at much higher concentrations in these membrane materials compared to other gas species. These properties give rise to very high CO_2 permeation rates and selectivities over many other gas species.

The accurate prediction of permeabilities of gases in membrane is generally not possible, and experimental values are needed. For the effect of temperature T in K, ln P_A' for gas A is approximately a function of 1/T and increases with T. However, operation at high temperatures can often degrade the membranes. When a mixture of gases is present, reductions of permeability of an individual component up to 10% or so can occur and in certain cases, much larger reductions have been observed. Hence, when using a mixture of gases, experimental data should be obtained to determine if there is any interactions between the gases. The presence of water vapour can have similar effects on the permeabilities and can also possibly damage the membranes (Geankoplis, 2003).

2.5.2 Selectivity

As for selectivity, the ideal selectivity for pure gases is given by the ratio of permeability as shown, permeability of carbon dioxide over that of methane as follows. We can also relate it to the previous equation of permeability to obtain the equation below.

$$\alpha_{ideal} = \frac{P_{CO_2}}{P_{CH_4}} = \frac{D_{CO_2}}{D_{CH_4}} \cdot \frac{S_{CO_2}}{S_{CH_4}}$$

However, for a mixture of gases, the feed composition has to be accounted for so the selectivity is given by the equation below, where Y_i is the concentration of component i in the permeate stream and X_i the concentration of component i in the feed stream. Normally, the range of selectivity for natural gas applications ranges from 15-25 which means that the carbon dioxide permeates through the membrane 15 to 25 times faster than methane.

$$\alpha = \frac{y_{CO_2}/y_{CH_4}}{x_{CO_2}/x_{CH_4}}$$

2.5.3 Stage Cut

Stage cut is a measure of the performance of the membrane from the perspective of quantity as it is the ratio of the permeate flow rate to the feed flow rate.

$$Stage\ Cut\ (\%) = \frac{Permeate\ Flow\ Rate}{Feed\ Flow\ Rate} \times 100\%$$

2.5.4 Mass Transfer Rate of CO_2

As proposed by Kumar et al. (2002), and Yeon et al. (2005), the mass transfer rate of carbon dioxide (J_{CO_2}) can be described by the following equations:

$$Mass\ Transfer\ rate\ of\ CO_2,\ J_{CO_2} = \frac{(Q_{in} \times C_{in} - Q_{out} \times C_{out}) \times 273.15}{0.0224 \times T_g \times S}$$

where,

η = CO_2 removal efficiency, %

J_{CO_2} = CO_2 mass transfer rate, mol/(m^2.h)

Q_{in} = inlet gas flow rate, m^3/h

Q_{out} = outlet gas flow rate, m^3/h

C_{in} = CO_2 volumetric fraction in the gas inlet, %

C_{out} = CO_2 volumetric fraction in the gas outlet, %

T_g = gas temperature, K

S = gas-liquid mass transfer area/effective membrane area, m^2

2.5.5 Methane Recovery

The efficiency of the membrane separation can also be measure from another perspective which is the methane recovery. It measures the methane that remains in the retentate stream upon exiting the system, relative to the inlet methane content.

$$Methane\ Recovery(\%) = \frac{Q_{out} \times C_{out}}{Q_{in} \times C_{in}} \times 100\%$$

where,

Q_{in} = inlet gas flow rate, m^3/h
Q_{out} = outlet gas flow rate, m^3/h
C_{in} = CH_4 volumetric fraction in the gas inlet, %
C_{out} = CH_4 volumetric fraction in the gas outlet, %

CHAPTER 3

METHODOLOGY

3.1 Scope

This project aims to study the performance of the polyimide hollow fiber membrane under different parametric conditions such as pressure, composition, feed gas flow rate and membrane area.

3.2 Research Methodology

Problem Statement	• Problem identification based on the current issue with regards to the scope of the project.
Literature Review	• Study and review of journals and books related to the proposed project to gain a general understanding.
Conduct of Experiment	• Experimental procedures carried out using specific equipments, apparatus and chemicals.
Data Analysis and Interpretation	• Evaluation of the results obtained and provide explanations based on scientific theories and principles.
Documentation	• Report findings in proper documentation and suggest possible recommendations to improve the project and to conclude on the overall results.

3.3 Experimental Parameters

The range of the experimentals parameters to be studied are summarized in the table below:

Table 4: Experimental Parameters with its range

Parameters	Range
Pressure [bar]	5-10
CO_2 composition [%]	20-70
Feed gas flow [SLPM]	5-20
Number of fibers	5-50

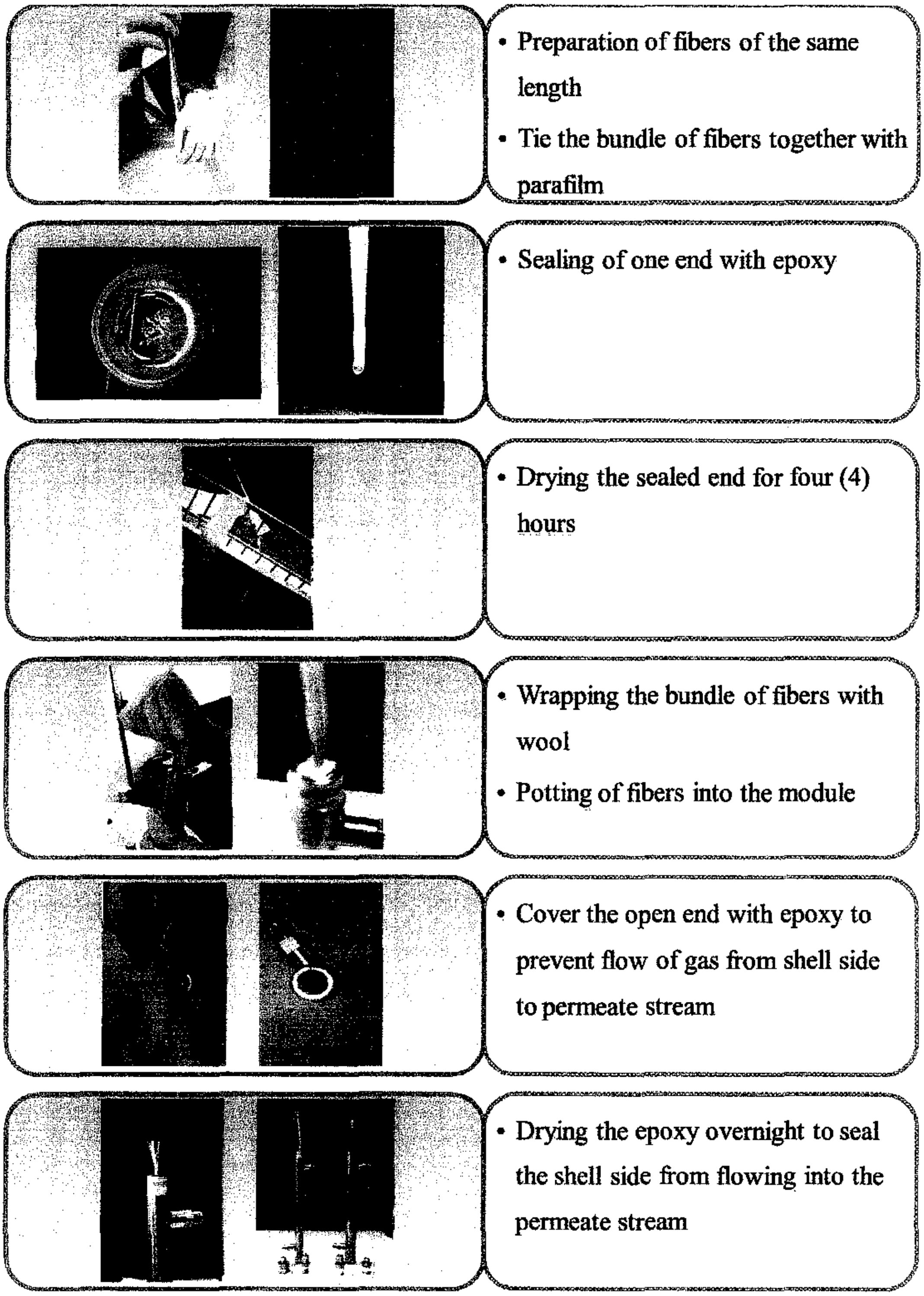
3.4 Specifications of the Hollow Fiber & Membrane Module

Table 5: Specifications of the Hollow Fiber & Membrane Module

Parameter	Value
Module material	Stainless Steel 316
Module inner diameter (inch) (2 modules)	1. 0.75 2. 1.0
Module length (mm)	300
Fiber inner diameter (μm)	180
Fiber outer diameter (μm)	400
Fiber length (mm)	250-280
Number of fibers	10-15 (0.5 inch module) 30-50 (1.0 inch module)
Pure gas separation at 25^0C (GPU)	40
CO_2/CH_4 Selectivity	35

3.5 Preparation of Hollow Fibers and Membrane Module

The preparation of the hollow fibers are done before the testing in the test rig so as to prepare the fibers to be fitted into the membrane module. The ultimate aim of the fiber preparation is to seal one end of the bundle of hollow fibers so as to prevent the permeate from flowing back into the shell side of the module. The other end of the fibers would be left open to allow the permeate flow for composition measurement as well as flow measurement using the bubble flowmeter.



- Preparation of fibers of the same length
- Tie the bundle of fibers together with parafilm

- Sealing of one end with epoxy

- Drying the sealed end for four (4) hours

- Wrapping the bundle of fibers with wool
- Potting of fibers into the module

- Cover the open end with epoxy to prevent flow of gas from shell side to permeate stream

- Drying the epoxy overnight to seal the shell side from flowing into the permeate stream

Figure 5: Hollow Fiber and Membrane Module Preparation Steps

3.6 Experimental Setup

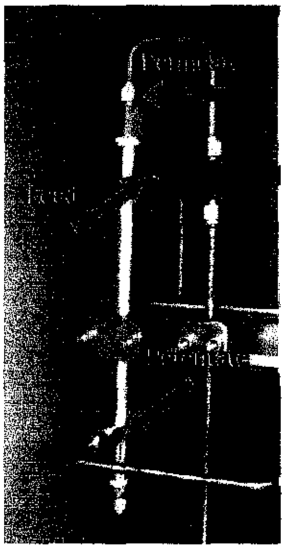


Figure 7: Membrane module with Feed, Permeate and Retentate Streams

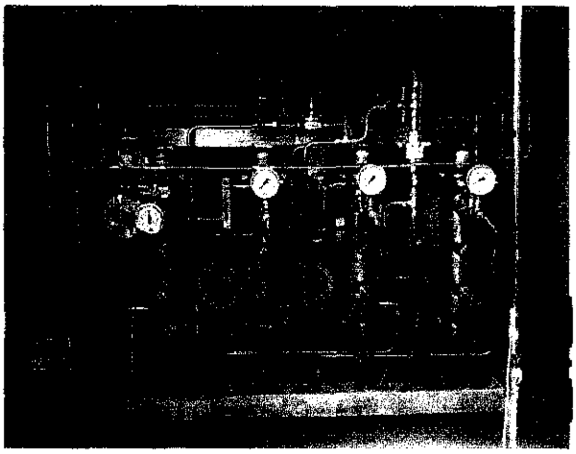


Figure 6: Carbon Dioxide Separation Membrane Unit (CO2SMU)

3.7 Experimental Procedure

1.0 Start-up of CO_2SMU System

1.1 The main power supply is switched on (inside the control panel - MCCB).

1.2 The main power supply to the computer is turned on.

1.3 The National Instrument (NI) lab view is activated and allowed to completely load in the system.

1.4 The analyzer switch is turned ON.

2.0 Heating-up of Hot Water System

2.1 The main power is switched on.

2.2 The heater is set to 80^0C.

2.3 The water pump is run to circulate the hot water inside the heat exchanger.

3.0 Setup of Feed Gases

3.1 The type of gases to be used for experimental purpose is determined.

3.2 The valve at the cylinder is opened.

3.3 The valves at the heat exchanger associated with the CO_2SMU test rig is opened.

4.0 Setup of Feed Gases at Feed Panel

4.1 The inlet and outlet valves for CO_2 and CH_4 are opened.

4.2 The feed regulator is set at 7 bar.

4.3 The flow rate for both type of gases is set at the mass flow controller (MFC).

5.0 Setup of MFC and Data Acquisition

5.1 The values of both mass flow controller is set according to the composition of intended feed gas percentage.

5.2 The toggle is tapped ON on the monitoring system to start collecting data for analysis.

5.3 Valves are opened according the intended experiment which are whether:

- *i. Through saturation vessel or bypass*
- *ii. Permeate line or retentate line*
- *iii. Manual back-pressure regulator (BPR) or Auto back-pressure regulator (BPR)*

6.0 Setup of Manual Back-Pressure Regulator (BPR)

6.1 The high pressure regulator is used to regulate.

6.2 The knob is turned clockwise to set the pressure, which is the system set pressure.

NOTE: Make sure that the feed gas pressure is always higher than the set value.

7.0 Start-up of Compressor

7.1 The compressor switch at the control panel is switched ON.

7.2 The "START" button at the compressor is pressed.

7.3 The inlet COMP1 valve is opened immediately.

7.4 The inlet pressure is set to 0.4 bar.

8.0 Monitoring of Readings

8.1 The monitoring is done through the National Instrument (NI) Interface.

8.2 The monitoring can also be done via the instrument indicator at the test rig.

9.0 Sampling

9.1 Needle valve at the manifold to be tested is opened slowly.

9.2 The ball valve at the Infrared Analyzer is opened.

9.3 The sampling pressure is set at 1 psig while the flow is set at 0.4 liter/min.

10.0 Shutdown of Compressor

10.1 The inlet ball valve to Compressor 1 is closed.

10.2 The "STOP" button at the compressor is pressed immediately.

10.3 "COMP1" switch is turned OFF to stop compressor 1.

11.0 Special Conditions

CASE 1: *If the conditions of the intended experiment is higher or lower than the compressor capacity, proceed to "spill-back" procedure.*

CASE 2: *Controlling the system pressure via automatic back-pressure regulator (Bronkhost BPR).*

11.1 Steps 1 to 5 are repeated.

11.2 The valve prior to the Bronkhost BPR is opened.

11.3 The pressure is set at the NI interface.

11.4 Step 7 is followed.

11.5 The inlet and outlet valves to the spill-back regulator are opened to start regulating.

11.6 Steps 8 to 10 are followed.

NOTE: f the spill back of mass is not enough, the value at MFC will be unstable.

12.0 Control of Environment Temperature

12.1 To maintain the environment temperature of the test rig, the temperature of the oven (Hot Box) is set within the range of ambient temperature up to 70^0C.

3.8 Tools

3.8.1 Chemicals

Table 6: Chemicals Required and its Specifications

NO.	Chemical	Specifications
1.	Carbon dioxide	25kg
2.	Natural gas	50L
3.	Methane	50L
4.	Nitrogen	50L

3.8.2 List of Equipment and their Functions

Table 7: List of Equipments

No	Equipment / Item	Quantity	Function
1.	Feed gas storage unit	8 (3x25kg)CO2 (3x50L) NG (1x50L)CH4 (1X50L) N2	1. Gas supply line (CH4, CO2, N2 and natural gas and one additional point for other gases) 2. Minimum amount of tank for each type of gas
2.	Pressure regulating system		1. Regulate pressure coming out from gas tank/before reaching mass flow controller 2. Pressure regulating system can be operated with inlet pressure up to 200barg 3. Outlet pressure from pressure regulating system = 10barg-80barg
3.	Automatic mass flow controller	3	1. Measure mass of gas volume under STANDARD conditions of pressure and temperature 2. Range of mass flow controller: 10-100 SLPM
4.	Feed vessel	1	1. Mix multiple gas streams before entering static mixer 2. Able to withstand operating pressure of 80bar
5.	Gas mixer	1	1. Mix multiple gas streams homogenously before entering saturation vessel or membrane test cell or passing through gas compressor 2. Able to withstand operating pressure of 80bar

No	Equipment / Item	Quantity	Function
6.	Compressor	1	1. Increase the mixed gas pressure for operating condition up until 80 barg 2. Can be used for natural gas application with the high content of carbon dioxide up to 70%. 3. SMOOTH AND CONTINUOUS flow of outlet gas. There shall be NO reciprocating motion or pulsation of outlet gas flow. 4. Able to operate under these condition: fixed flowrate, fixed pressure, fixed flowrate with varied pressure and vice versa 5. Flowrate of compressor shall be= 10-100 SLPM 6. A by-pass line so that it is optional to operate CO2SMU without compressor
7.	Mixed gas flow meter	2	1. Measure and indicate the flow rate of mixed gas before entering and after leaving the membrane test cell 2. Coriolis type. 3. Flowrate range= 10-100 SLPM 4. Resistant to corrosion
8.	Saturation vessel	1	1. Saturate mixed gases with water or heavy hydrocarbon (pentane as a basis) 2. Able to withstand operating pressure of 80 barg 3. Equipped with heater to heat process fluid until 100°C which can be controlled and adjusted at control panel. 4. Have opening for easy cleaning and maintenance purpose as well as draining system for change of solution.
9.	Membrane Test Cell	1	1. Milipore pressure filter holder (XX45 047 00). 2. Resistant to corrosion. 3. Equipped with pressure relief valve 4. Operating pressure = ambient pressure to 80 barg 5. Operating temperature = ambient temperature to 70°C
10.	Back Pressure Regulator	2	1. Maintain system pressure at particular set point. 2. ONE automatic BPR and ONE manual BPR 3. Able to regulate pressure from ambient to

No	Equipment / Item	Quantity	Function
			80 barg 4. Able to withstand operating temperature between ambient to 70°C
11.	Steam Trap	2	1. Collect any liquid carry over from the membrane test cell 2. All gases leaving from the steam trap shall be vented out to safe location and follows UTP's Health, Safety and the Environment (HSE) requirement.
12.	Online Infrared Gas Analyzer	1	1. Measure concentration of CO_2 and methane (CH_4) 2. Detection range : CH4: 0-100%, C02:0-100% and CO2-CH4 mixture at any combination between 0-100% 3. Minimal lag time (3 minute)
13.	Oven	1	1. Able to control and set by the users from ambient to 100°C. 2. Accommodate the process piping and equipment except compressor, gas analyzer, gas feeding and gas storage system. 3. The front door of oven should be transparent 4. Additional space of 25% from the oven size should be reversed for future expansion.
14.	Toxicap Fume Hood	1	1. Accommodate the process piping and equipment including oven except compressor, gas analyzer, gas feeding and gas storage system. 2. 2 levels of molecular filters that can filter impurities 3. Equipped with fan failure alarm to alarm system notification in case of ventilation failure.
15.	Data Acquisition System		1. Include interface and software for the temperature, gas concentration and pressure readings. 2. Measurement data can be viewed on computer's monitor via data acquisition software, and can be stored and exported into spreadsheet 3. Minimum Requirement for Data Collection = temperature (T), pressure (P), time (t), flow rate (F) and gas concentration from

No	Equipment / Item	Quantity	Function
			analytical equipment (C)
16.	Control Panel		1. Show the measurement of all instruments covering pressure, temperature, flowrate and concentration.
17.	Pressure transducer		1. Measure the gas pressure for particular gas stream line and send electrical signals to Data Acquisition System
18.	Pressure gauge		1. Measure and indicate the gas pressure for particular gas stream line
19.	Thermocouple		1. Measure and indicate the temperature for particular gas/liquid stream line and send electrical signals to Data Acquisition System
20.	Temperature gauge		1. Measure and indicate the temperature for particular gas/liquid stream line
21.	Pressure Relief Valve	1	1. Installed at necessary equipment/gas line as mechanical protection against the event of excessive pressure 2. Pressure relief valve with 10% of maximum pressure rating
22	Computer	1	1. Intel i5 processor, or better 2. 2 GB of RAM, or better 3. HDD 500GB, or better 4. Drives = Internal hard disk and DVD-ROM (read, write, rewrite) 5. Monochrome LaserJet Printer

3.9 Process Flow Diagram

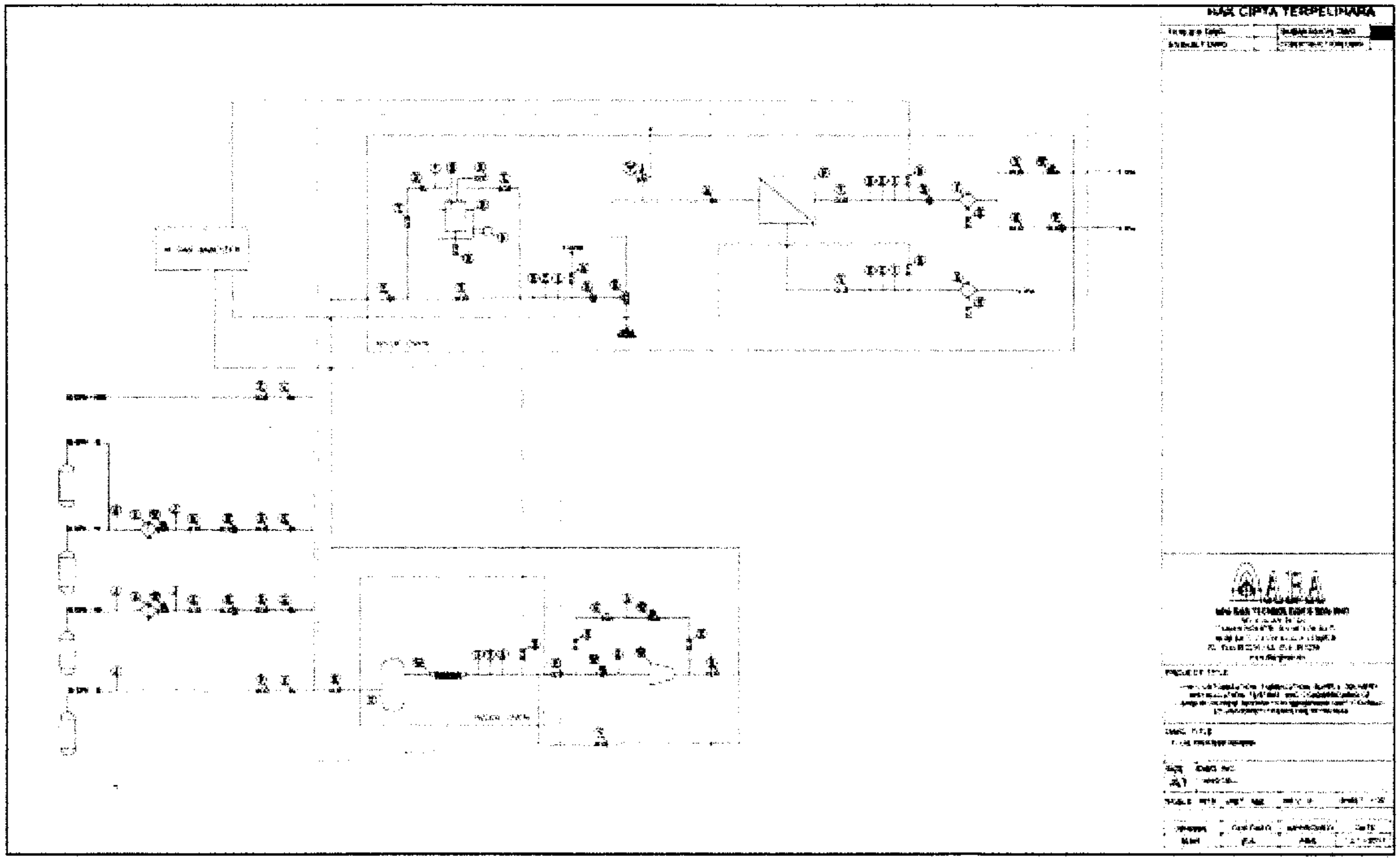


Figure 8: Process Flow Diagram of the Experimental Setup

3.10 Process Parameter Monitoring Tool for CO_2SMU

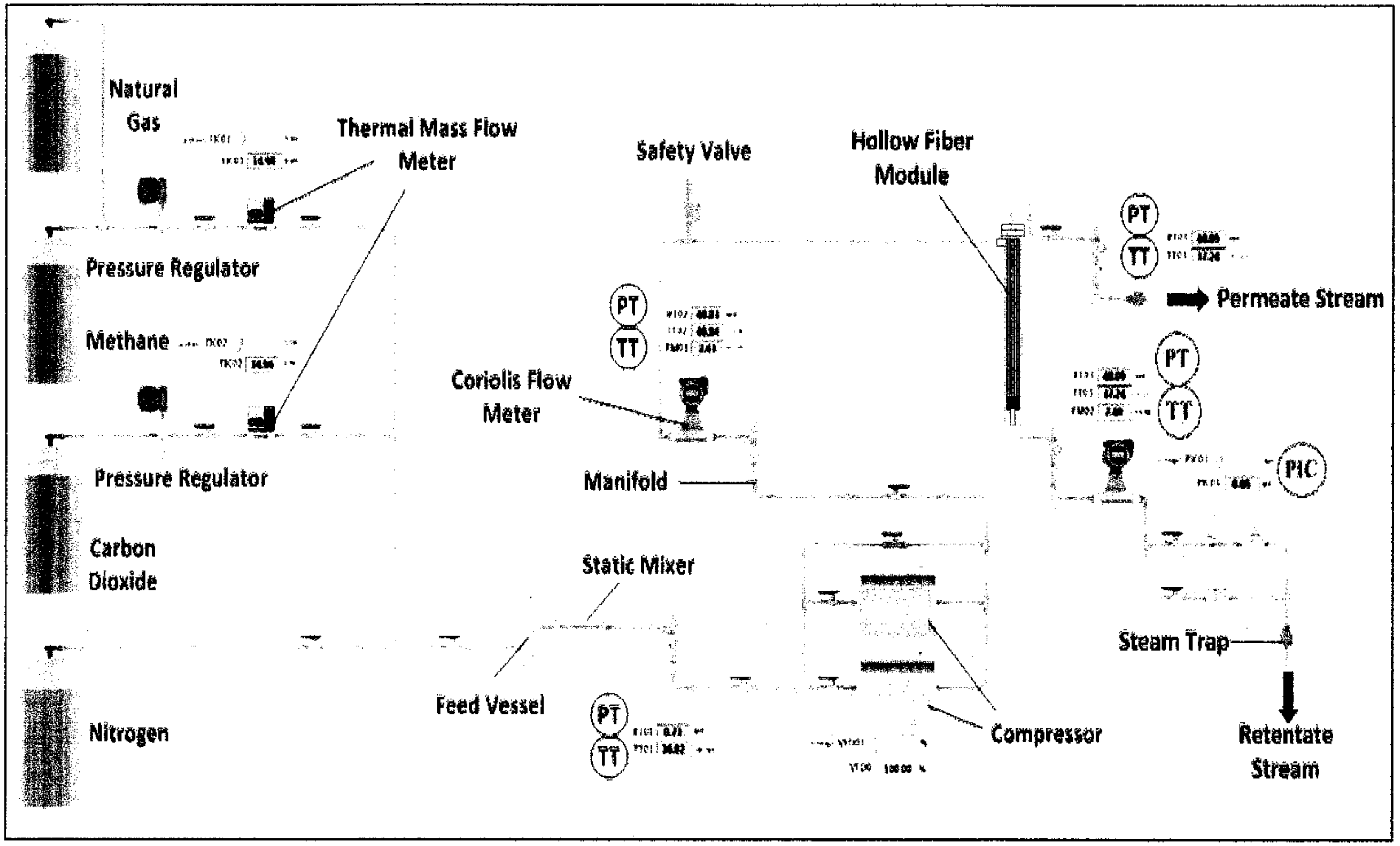


Figure 9: Process Parameter Monitoring Tool for CO_2SMU

3.11 Key Milestone & Gantt Chart

3.11.1 Key Milestone

Table 8: Key Milestone of FYP II

No	Action Item	Remarks
1.	Regular meeting with supervisor to discuss the project and prepare project proposal	Week 1-14
2.	FYP II Briefing	Week 2
3.	Submission of Progress Report	Week 8
4.	Pre-EDX	Week 11
5.	Submission of Draft Report	Week 12
6.	Submission of Dissertation (Soft-bound)	Week13
7.	Submission of Technical Paper	Week 13
8.	Final Oral Presentation	Week 14
9.	Submission of Project Dissertation (Hard-bound)	Week 15

3.11.2 Gantt Chart

NO.	ACTIVITY / WEEK	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1.	Continuation of Project Work	■	■	■	■	■	■	■	Mid-Semester Break							Raya Break	
2.	Submission of Progress Report									■							
3.	Continuation of Project Work									■	■	■	■	■			
4.	Pre-EDX												■				
5.	Submission of Draft Report													■			
6.	Submission of Dissertation (soft bound)														■		
7.	Submission of Technical Paper														■		
8.	Oral Presentation																■
9.	Submission of Dissertation (hard bound)																■

CHAPTER 4

RESULTS & DISCUSSION

The two main performance indicators are permeability and selectivity of the hollow fiber membrane. The gas permeance P/l (in GPU = $1 \cdot 10\text{-}6\ cm^3(STP)/cm^2 \cdot s \cdot cmHg$) is calculated as the flow rate of a specific gas of the permeate stream, Q divided by the partial pressure difference over the membrane (Δp) and the membrane area A of one membrane module.

$$\frac{P}{l} = \frac{Q}{A.\Delta p}$$

The selectivity α (CO_2/CH_4) is calculated as:

$$\alpha = \frac{Y_{CO_2}/Y_{CH_4}}{X_{CO_2}/X_{CH_4}}$$

Where Y_i is the concentration of component i in the permeate stream that leaves the desorber and X_i is the concentration of component i in the feed.

In fact, the results can also be represented in other forms such as stage cut, mass transfer rate of CO_2 as well as methane recovery. These representations can all illustrate the performance of the membrane in separating CO_2 from natural gas, as shown earlier in Chapter 2 of this report. In this report, the results will be reported in terms of permeance, selectivity as well as stage cut.

$$Stage\ Cut\ (\%) = \frac{Permeate\ Flow\ Rate}{Feed\ Flow\ Rate} \times 100\%$$

4.1 Effects of Feed Gas Pressure

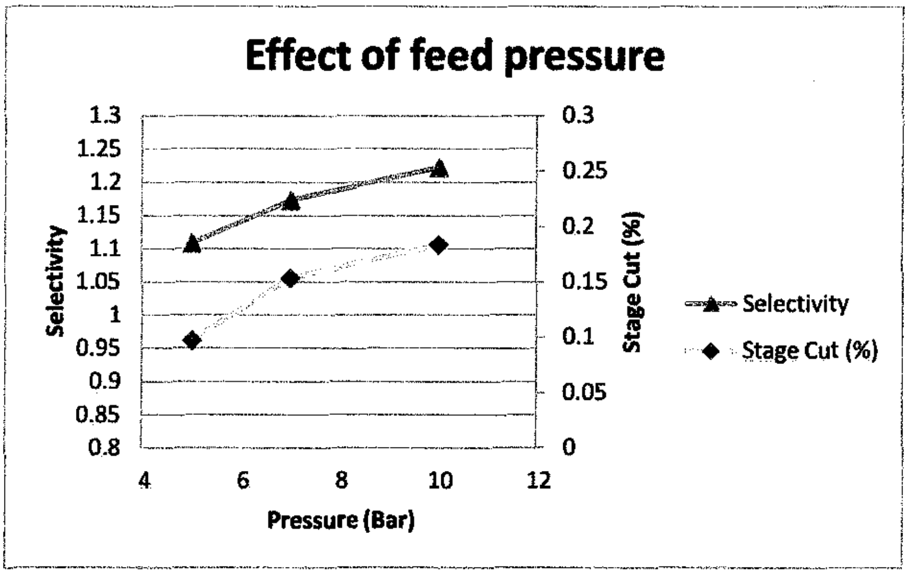


Figure 10: Effect of feed pressure on Selectivity and Stage Cut at 35^{0}C, 50% CO_2/50% CH_4 composition, flow of 6 SLPM with 15 fibers

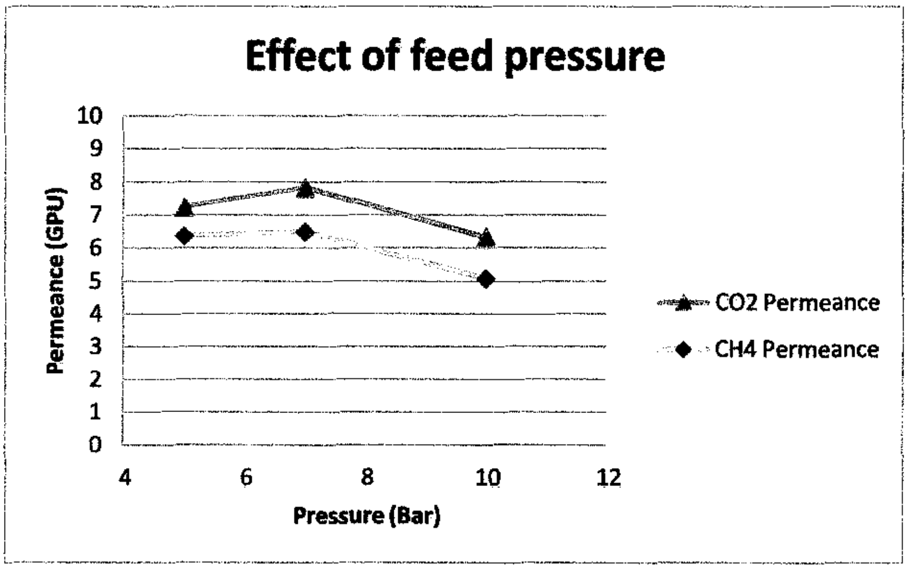


Figure 11: Effect of feed pressure on CO_2 and CH_4 Permeance at 35^{0}C, 50% CO_2/50% CH_4 composition, flow of 6 SLPM with 15 fibers

As can be seen from Fig. 10 and Fig.11 on the previous page, the effect of increasing the feed pressure has an positive impact on the selectivity and stage cut but decreases the permeance of methane and carbon dioxide. In studying the effect of pressure, the flow is fixed at 6 SLPM, composition at 50%CO_2 - 50%CH_4, at ambient temperature, with 15 fibers in the module.

Both selectivity and stage cut increases with the increase in pressure. The increase in pressure provides a greater driving force to facilitate the diffusion of the gases through the membrane. Therefore, more CO_2 can permeate through the membrane to reach the permeate stream and create a higher flow of permeate stream and therefore a higher stage cut. As the pressure increases, the stage cut will increase proportionally as higher flow is measured at the permeate stream. As for the selectivity of the membrane, the selectivity is generally not affectly much in terms of magnitude, but a trend of increasing selectivity with increasing feed gas pressure can be observed. In terms of permeance, the permeance of both gases decreases with increasing pressure. This can be explained mathematically by the equation below:

$$\frac{P}{l} = \frac{Q}{A.\Delta p}$$

As the pressure is increased, the pressure drop increases as well as the permeate side pressure is always atmospheric. As the pressure is increased from 5 to 7 then to 10 bar, the pressure drop increases and caused a decreasing permeance, although the flow of the permeate stream increases, with the membrane surface area being constant. It can also be seen that the permeance of CO_2 is consistently above that of CH_4, due to the fact that CO_2 is a “fast” gas and therefore diffuses faster through the membrane compared to CH_4.

4.2 Effects of Feed Gas Composition

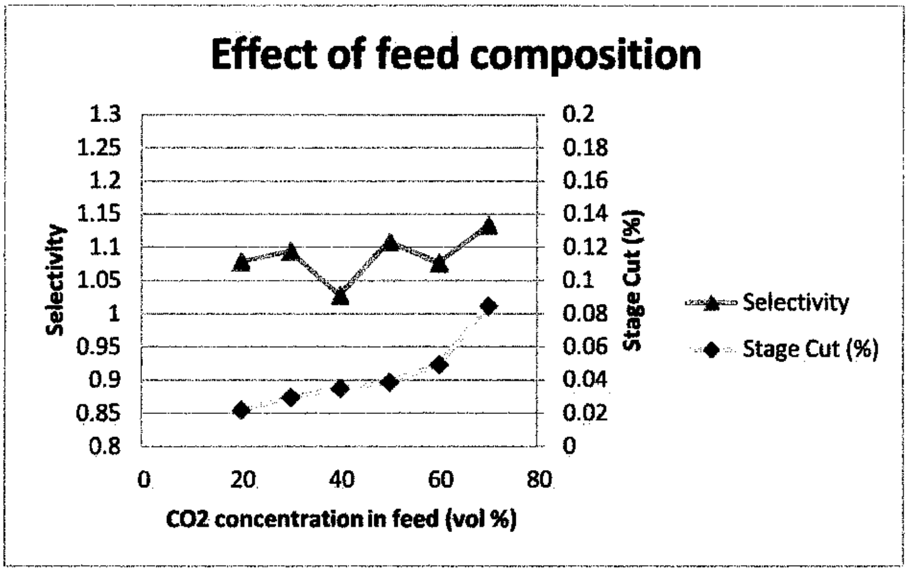


Figure 12: Effect of feed composition on Selectivity and Stage Cut at 35^{0}C, pressure of 5 bar, flow of 6 SLPM with 15 fibers

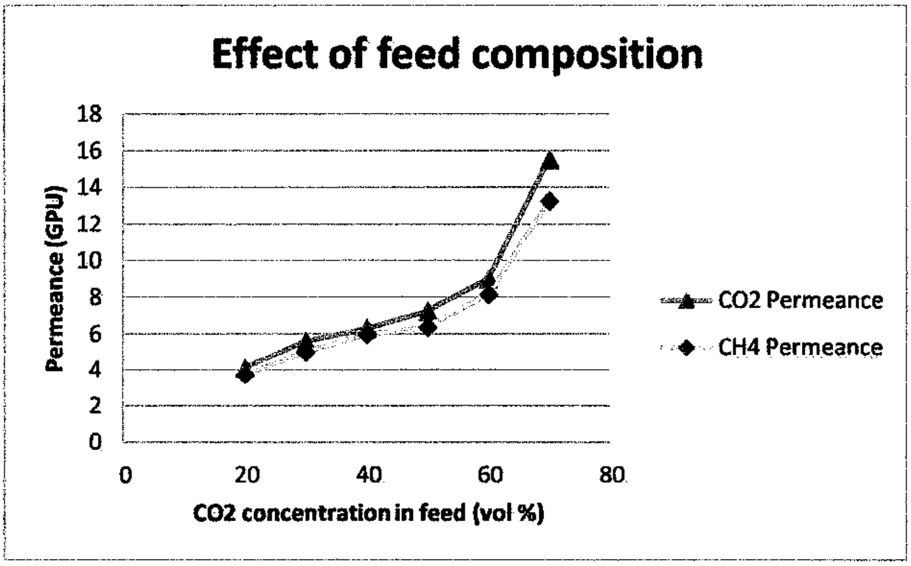


Figure 13: Effect of feed composition on CO_2 and CH_4 Permeance at 35^{0}C, pressure of 5 bar, flow of 6 SLPM with 15 fibers

In studying the effects of feed composition, or better represented as the percentage of carbon dioxide in the feed gas, the system is maintained at a flow of 15 SLPM, pressure of 5 bar and at ambient temperature, with a total of 15 fibers for the experiment.

When the content of CO_2 increases in the feed gas,there is an increase in all three parameters of selectivity, stage cut and permeance, whereby the CO_2 composition ranges from 20-70 vol %. The trends can be clearly observed in Fig. 12 and Fig. 13 on the previous page and this effect is attributed to the fact that as when the content of CO_2 increases, there are more gas for permeation and therefore results in the increase in stage cut, or better said as the permeate flow. The selectivity is generally not affected as the increase in selectivity is minimal from the experimental results.

When more CO_2 is present in the feed gas, the feed stream contains more gas molecules which can permeate through the membrane owing to the fact that it is a "fast" gas, as explained earlier. As a result, the permeate stream has a higher flow, and therefore increasing the stage cut and permeance. This observation can also be explained by the fact that as the CO2 percentage increases, there is a larger concentration gradient between the feed and permeate stream and therefore creating a larger driving force, which leads to the increase of CO_2 diffusion mass transfer rate.

The increasing permeance as the feed composition increases can be explained mathematically. Permeance in the unit of GPU, equals the permeate flow divided by the multiplication of area and pressure drop. As CO_2 composition increases, the flow in permeate stream increases while the active surface area of the membrane and the pressure drop across the membrane remains constant. Therefore, the increase in permeance can be expected.

4.3 Effects of Feed Gas Flow Rate

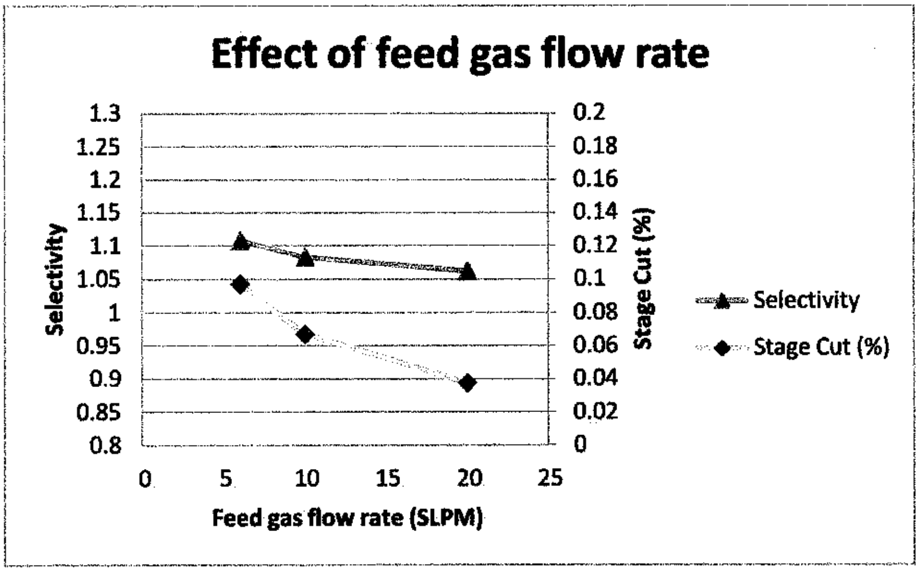


Figure 14: Effect of feed gas flow rate on Selectivity and Stage Cut at 35^{0}C, pressure of 5 bar, 50% CO_2/50% CH_4 composition with 15 fibers

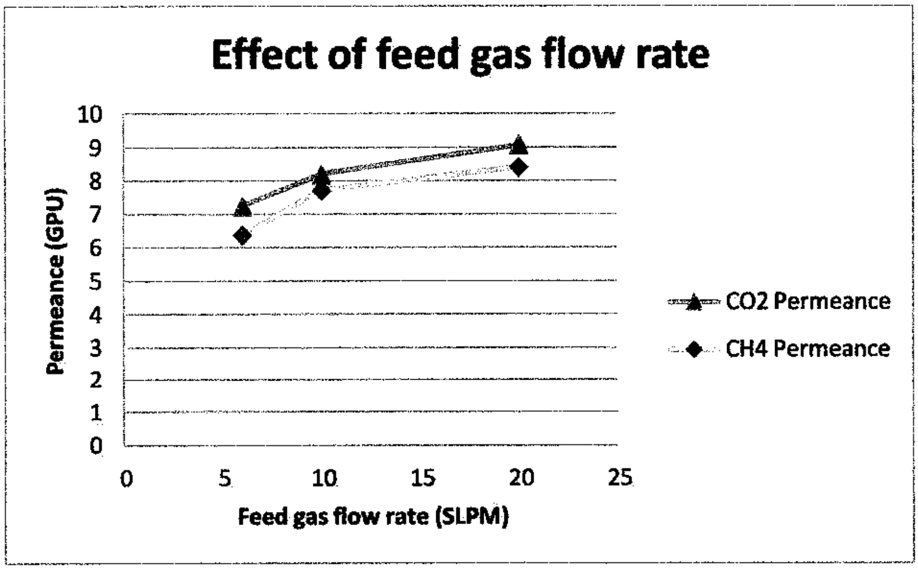


Figure 15: Effect of feed gas flow rate on CO_2 and CH_4 Permeance at 35^{0}C, pressure of 5 bar, 50% CO_2/50% CH_4 composition with 15 fibers

While the flow rate of the feed gas in increases from 6 SLPM to 10 SLPM and then to 20 SLPM, the other parameters of composition is fixed at 50%CO_2 - 50%CH_4, pressure at 5 bar, with a 15-fibers module at ambient temperature.

As the flow rate of the feed gas is increased, it can be observed that the selectivity of the membrane as well as the stage cut decreases proportionally. In other words, it can be said that the CO_2 removal efficiency decreases with increasing feed gas flow rate but this on the other hand will lead to an increase in the mass transfer rate across the membrane, or better known as permeance, which is represented by Fig. 15 on the previous page.

The effect on flow rate on the decreasing selectivity and stage cut can be explained to the fact that when the flow rate is incresed, the contact time for the gases to permeate through the membrane decreases and therefore causes such observations. The amount of CO_2 that permeates through the membrane decreases and hence, a decrease in the stage cut of the system. On the other hand, the permeance of both the gases increases due to the increase in the mass transfer rate. Also, the permeance of CO_2 is consistently higher than that of CH_4, similar to the previous observations on effects of feed pressure and feed composition. This is due to the characteristics of carbon dioxide, being smaller in kinetic diameter, in addition to being more condensable and more polar than CH_4.

Mathematically, the observation on the increase in permeance can be explained, again with the same equation of P/l. An increase in the feed flow rate will cause an increase in the mass transfer rate across the membrane and hence higher permeate flow. With the pressure and membrane surface area remain constant, the flow (Q) of each of the gases increases and this in turn increases the permeance of both methane and carbon dioxide across the membrane.

4.4 Effects of Number of Fibers/Membrane Area

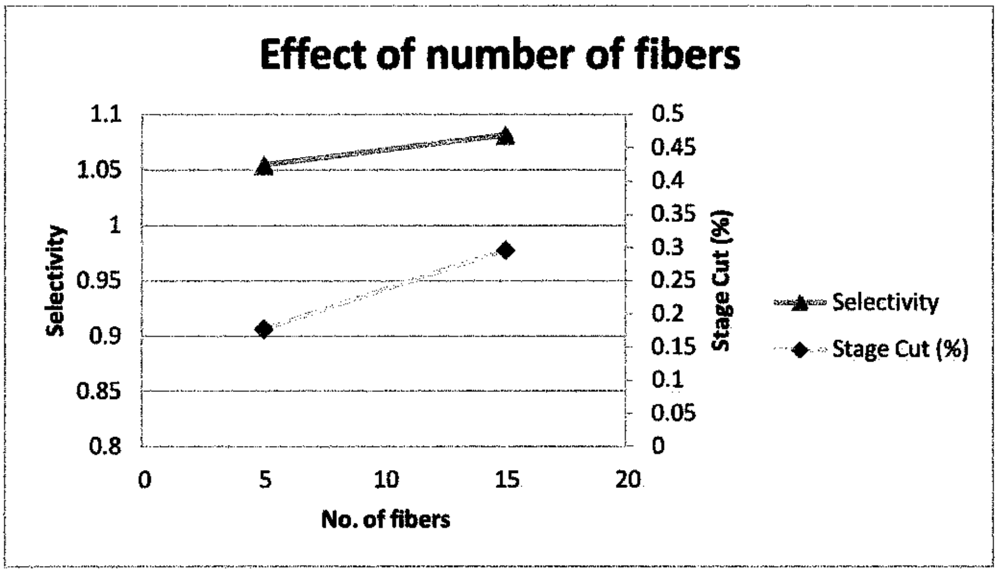


Figure 16: Effect of number of fibers on Selectivity and Stage Cut at 35^{0}C, pressure of 10 bar, 50% CO_2/50% CH_4 composition, at flow of 10 SLPM

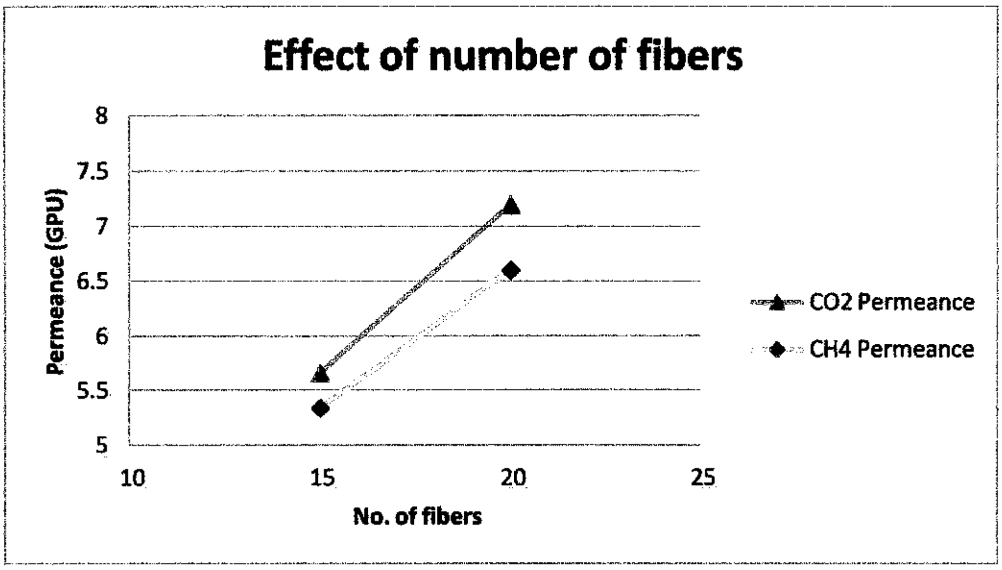


Figure 17: Effect of of number of fibers on CO_2 and CH_4 Permeance at 35^{0}C, pressure of 10 bar, 50% CO_2/50% CH_4 composition, at flow of 10 SLPM

Generally, variating the number of fibers equal changing the effective surface area for gas permeation. For this purpose, the number of fibers is changed from 5 to 50 fibers to observe the effects, while maintaining the pressure at 10 bar, total feed gas flow at 10 SLPM, 50% CO_2 and at ambient temperature.

From Fig. 16, it can be seen that the selectivity of the membrane is not affected much by the change in the number of fibers as selectivity is an intrinsic property of a membrane that is not affected by the number of fibers used. The number of fibers used will only affact the stage cut and permeance, which can be seen in Fig. 16 and Fig. 17. As the number of fibers/membrane surface area is increased, the stage cut and permeance increases proportionally to the rise.

When the number of fibers used increases, there is more surface area for gas permeation through the membrane and therefore, the permeate flow will increase resulting in an increase in the stage cut, which is the percentage of permeate stream in relation to the feed stream. Permeance can also be explained in a similar manner whereby when the number of fibers increase, more gas permeates through the larger membrane area resulting in a larger permeate flow. The increase in permeate flow corresponds to higher individual gas flow of CO_2 and CH_4 and hence, the permeance increases with pressure remain constant. From the increasing trend of permeance, we can justify that the increase in the permeate flow rate is larger then the increase in active surface area and as a result, the permeance increases as the number of fibers used is increased.

CHAPTER 5

CONCLUSIONS & RECOMMENDATIONS

This study is conducted to observe the performance of hollow fiber membrane when parameters such as pressure, composition, gas feed flow and the number of fibers used in a hollow fiber membrane module variates in a binary gas system through experimental methods.

A detailed literature review on membrane general characteristics and theories relating the principles of solution-diffusion across membrane has been conducted, together with the governing equations. Besides, a detailed methodology accompanies the report highlighting the steps in performing the experiments, the chemicals required as well as the equipments involved in the experimental setup.

The results obtained from this experiment is found to be in line with literatures researched and from this point onwards, more work can be conducted to bring the technology a step forward in this university. It was found that the stage cut and the selectivity of the membrane increases with pressure and feed composition while these two parameters showed a decrease when the feed gas flow rate is increased. Generally, the number of fibers used does not affect the selectivity of the membrane but the stage cut of the membrane is largely increased when the number of fibers used is increased. In terms of permeance, it was found that the gas permeance increases with increasing feed composition, feed gas flow rate as well as number of fibers or membrane area but decreases with increasing feed pressure.

To make the experimental work a more reliable and accurate one, some improvements can be made to the current equipment setup. It was realize through the conduct of the experiments that the IR analyzer do not give very accurate results and therefore can be replaced with a gas chromatography analyzer. Besides, the range of the measuring instrument such as the rotameter should also be improved for the measurement of the permeate flow.

Membrane gas separation is seen as a promising separation technology which offers several advantages compared to its earlier counterparts and is therefore given much attention recent years, with acticipated breakthroughs and improvements to enhance the CO_2 separation process. Despite the many advantages of membrane for CO2 capture, its potential drawbacks such as membrane fouling and wall-wetting should be addressed in the future work to make this technology a superior choice over the others, especially in offshore applications.

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APPENDIX

A. Results and Calculations

A.1 Data from National Instruments (NI) Data Logging

Date	Time	Time (s)	PT01(bar)	PT02(bar)	PT03(bar)	PT04(bar)	CO2 Conc (%)	CH4 Conc (%)	FIC01(slpm)	FIC02(slpm)	FM01(kg/hr)	FM02(kg/hr)	PIC01(bar)	SP, FIC01(slpm)	SP, FIC02(slpm)	SP, PIC01(bar)	SP, VFD01(	TT01(dC)	TT02(dC)	TT03(dC)	TT04(dC)	Remarks
											20% CO2											
24/07/2	9:24:01 PM	4362	5.978	6.158	5.313	0.028	25.559	75.983	12.002	2.62	0.907	0.926	0.033	0	0	0	0	32.848	34.498	34.187	34.778	Permeate
24/07/2	8:57:53 PM	2794	5.869	6.058	5.155	0.026	24.026	77.784	12.026	3.065	0.951	0.775	0.033	0	0	0	0	33.097	34.747	34.405	34.903	Retentate
24/07/2	8:44:10 PM	1971	5.679	5.854	5.156	0.025	24.207	77.619	11.77	3.048	0.759	0.769	0.033	0	0	0	0	33.191	34.809	34.498	34.903	Feed
NormalizedValue			CO2 (%)	CH4 (%)																		
		Feed	23.77291	76.22709																		
		Permeate	25.17087	74.82913																		
		Retentate	23.59886	76.40114																		
											30% CO2											
24/07/2	7:46:44 PM	4938	5.513	5.687	5.073	0.023	33.365	67.964	7.017	3.048	0.694	0.705	0.028	0	0	0	0	33.471	34.934	34.591	35.307	Permeate
24/07/2	6:48:07 PM	1421	5.347	5.532	4.923	0.02	31.292	70.135	7.159	3.047	0.669	0.491	0.029	0	0	0	0	33.969	35.743	35.463	36.023	Retentate
24/07/2	6:42:23 PM	1077	5.172	5.353	4.906	0.02	31.447	70.1	7.101	3.07	0.45	0.461	0.029	0	0	0	0	34.062	35.774	35.463	35.899	Feed
NormalizedValue			CO2 (%)	CH4 (%)																		
		Feed	30.96793	69.03207																		
		Permeate	32.92739	67.07261																		
		Retentate	30.85175	69.14825																		
											40% CO2											
24/07/2	6:16:31 PM	3475	4.902	5.241	4.863	0.056	40.849	59.151	0.056	0.056	0.357	0.41	0.029	0	0	0	0	34	35.899	35.588	36.241	Permeate
24/07/2	5:31:41 PM	785	6.911	7.102	0	0.015	39.058	60.942	0.044	0.049	0	0	0.031	0	0	0	0	34.311	36.272	34.996	36.335	Retentate
24/07/2	5:20:40 PM	124	6.589	6.784	4.267	0.015	39.814	60.186	2.504	0.054	0	0	0.029	0	0	0	0	34.125	36.335	35.774	1770	Feed
NormalizedValue			CO2 (%)	CH4 (%)																		
		Feed	39.814	60.186																		
		Permeate	40.849	59.151																		
		Retentate	39.058	60.942																		
											50% CO2											
24/07/2	4:05:57 PM	10769	5.043	5.23	4.848	0.018	49.686	50.303	3.058	3.084	0.356	0.367	0.024	0	0	0	0	34	36.054	35.65	1770	Permeate
24/07/2	2:51:37 PM	6309	4.953	5.133	4.761	0.015	47.069	53.728	3.058	3.052	0.355	0.16	0.024	0	0	0	0	33.066	35.525	35.058	1770	Retentate
24/07/2	2:49:20 PM	6172	4.978	5.16	4.804	0.015	47.363	53.154	3.089	3.075	0.294	0.299	0.024	0	0	0	0	33.44	35.525	35.089	1770	Feed
NormalizedValue			CO2 (%)	CH4 (%)																		
		Feed	47.11939	52.88061																		
		Permeate	49.69147	50.30853																		
		Retentate	46.69683	53.30317																		
											60% CO2											
24/07/2	11:18:24 PM	4612	5.274	5.452	4.923	0.021	58.142	42.753	3.047	4.551	0.623	0.634	0.031	0	0	0	0	31.79	33.284	33.035	33.595	Permeate
24/07/2	10:22:36 PM	1264	5.128	5.31	4.793	0.019	56.111	44.515	3.053	4.542	0.606	0.387	0.031	0	0	0	0	32.475	34.156	33.875	34.342	Retentate
24/07/2	10:29:08 PM	1656	5.141	5.323	4.791	0.019	56.173	44.652	3.058	4.542	0.611	0.389	0.031	0	0	0	0	32.381	34.062	33.813	34.311	Feed
NormalizedValue			CO2 (%)	CH4 (%)																		
		Feed	55.71336	44.28664																		
		Permeate	57.62625	42.37375																		
		Retentate	55.76193	44.23807																		
											70% CO2											
25/07/2	1:15:44 AM	5217	4.917	5.086	4.722	0.018	68.727	29.623	3.063	7.051	0.288	0.298	0.033	0	0	0	0	31.323	32.755	32.475	33.004	Permeate
25/07/2	12:22:57 AM	2050	4.899	5.073	4.717	0.018	66.459	33.048	3.058	7.079	0.251	0.256	0.033	0	0	0	0	31.728	33.284	32.973	33.44	Retentate
25/07/2	12:06:24 AM	1057	4.874	5.046	4.721	0.017	66.831	32.651	3.06	7.067	0.218	0.199	0.033	0	0	0	0	31.634	33.44	33.16	33.471	Feed
NormalizedValue			CO2 (%)	CH4 (%)																		
		Feed	67.17899	32.82101																		
		Permeate	69.88002	30.11998																		
		Retentate	66.78827	33.21173																		

Date	Time	Time (s)	PT01(bar)	PT02(bar)	PT03(bar)	PT04(bar)	CO2 Conc (%)	CH4 Conc (%)	FIC01(slpm)	FIC02(slpm)	FM01(kg/hr)	FM02(kg/hr)	PIC01(bar)	SP, FIC01(slpm)	SP, FIC02(slpm)	SP, PIC01(bar)	SP, VFD01(	TT01(dC)	TT02(dC)	TT03(dC)	TT04(dC)	Remarks
24/07/2	4:05:57 PM	10769	5.043	5.23	4.848	0.018	49.686	50.303	3.058	3.084	0.356	0.367	0.024	0	0	0	0	34	36.054	35.65	1770	Permeate
24/07/2	2:51:37 PM	6309	4.953	5.133	4.761	0.015	47.069	53.728	3.058	3.052	0.355	0.16	0.024	0	0	0	0	33.066	35.525	35.058	1770	Retentate
24/07/2	2:49:20 PM	6172	4.978	5.16	4.804	0.015	47.363	53.154	3.089	3.075	0.294	0.299	0.024	0	0	0	0	33.44	35.525	35.089	1770	Feed

NormalizedValue		CO2 (%)	CH4 (%)
	Feed	47.11939	52.88061
	Permeate	49.69147	50.30853
	Retentate	46.69683	53.30317

Date	Time	Time (s)	PT01(bar)	PT02(bar)	PT03(bar)	PT04(bar)	CO2 Conc (%)	CH4 Conc (%)	FIC01(slpm)	FIC02(slpm)	FM01(kg/hr)	FM02(kg/hr)	PIC01(bar)	SP, FIC01(slpm)	SP, FIC02(slpm)	SP, PIC01(bar)	SP, VFD01(	TT01(dC)	TT02(dC)	TT03(dC)	TT04(dC)	Remarks
25/07/2	6:26:23 PM	5218	5.144	5.323	4.989	0.019	52.451	47.75	5.045	5.016	0.429	0.437	0.036	0	0	0	0	35.525	37.206	36.802	39.198	Permeate
25/07/2	5:45:11 PM	2746	5.021	5.208	4.953	0.017	49.979	50.136	5.048	5.166	0.388	0.193	0.036	0	0	0	0	35.588	37.019	36.708	39.012	Retentate
25/07/2	5:41:21 PM	2516	5.021	5.201	4.938	0.018	50.881	50.176	3.076	5.198	0.291	0.293	0.036	0	0	0	0	35.619	37.019	36.739	38.576	Feed

NormalizedValue		CO2 (%)	CH4 (%)
	Feed	50.34881	49.65119
	Permeate	52.34578	47.65422
	Retentate	49.92159	50.07841

Date	Time	Time (s)	PT01(bar)	PT02(bar)	PT03(bar)	PT04(bar)	CO2 Conc (%)	CH4 Conc (%)	FIC01(slpm)	FIC02(slpm)	FM01(kg/hr)	FM02(kg/hr)	PIC01(bar)	SP, FIC01(slpm)	SP, FIC02(slpm)	SP, PIC01(bar)	SP, VFD01(	TT01(dC)	TT02(dC)	TT03(dC)	TT04(dC)	Remarks
25/07/2	8:06:32 PM	3228	6.166	6.365	5.448	0.03	52.55	48.971	9.541	8.623	1.12	1.132	0.036	0	0	0	0	34.903	36.21	35.93	41.689	Permeate
25/07/2	7:45:46 PM	1982	6.125	6.321	5.345	0.028	46.219	55.652	9.828	9.269	1.18	0.98	0.036	0	0	0	0	35.089	36.615	36.335	49.907	Retentate
25/07/2	7:36:49 PM	1445	6.127	6.321	5.525	0.03	50.881	50.372	9.472	8.773	1.102	1.104	0.036	0	0	0	0	35.152	36.739	36.49	50.996	Feed

NormalizedValue		CO2 (%)	CH4 (%)
	Feed	50.25135	49.74865
	Permeate	51.76269	48.23731

Date	Time	Time (s)	PT01(bar)	PT02(bar)	PT03(bar)	PT04(bar)	CO2 Conc (%)	CH4 Conc (%)	FIC01(slpm)	FIC02(slpm)	FM01(kg/hr)	FM02(kg/hr)	PIC01(bar)	SP, FIC01(slpm)	SP, FIC02(slpm)	SP, PIC01(bar)	SP, VFD01(	TT01(dC)	TT02(dC)	TT03(dC)	TT04(dC)	Remarks
24/07/2	4:05:57 PM	10769	5.043	5.23	4.848	0.018	49.686	50.303	3.058	3.084	0.356	0.367	0.024	0	0	0	0	34	36.054	35.65	1770	Permeate
24/07/2	2:51:37 PM	6309	4.953	5.133	4.761	0.015	47.069	53.728	3.058	3.052	0.355	0.16	0.024	0	0	0	0	33.066	35.525	35.058	1770	Retentate
24/07/2	2:49:20 PM	6172	4.978	5.16	4.804	0.015	47.363	53.154	3.089	3.075	0.294	0.299	0.024	0	0	0	0	33.44	35.525	35.089	1770	Feed

NormalizedValue		CO2 (%)	CH4 (%)
	Feed	47.11939	52.88061
	Permeate	49.69147	50.30853
	Retentate	46.69683	53.30317

Date	Time	Time (s)	PT01(bar)	PT02(bar)	PT03(bar)	PT04(bar)	CO2 Conc (%)	CH4 Conc (%)	FIC01(slpm)	FIC02(slpm)	FM01(kg/hr)	FM02(kg/hr)	PIC01(bar)	SP, FIC01(slpm)	SP, FIC02(slpm)	SP, PIC01(bar)	SP, VFD01(	TT01(dC)	TT02(dC)	TT03(dC)	TT04(dC)	Remarks
25/07/2	9:35:59 PM	3492	7.06	7.258	6.832	0.02	53.279	48.059	3.053	3.326	0.484	0.49	0.036	0	0	0	0	33.44	34.747	34.529	1770	Permeate
25/07/2	8:58:38 PM	1251	6.962	7.166	6.764	0.018	49.022	52.465	3.135	3.347	0.441	0.219	0.036	0	0	0	0	34	35.588	35.245	1770	Retentate
25/07/2	8:53:17 PM	930	6.964	7.154	6.792	0.019	49.26	52.136	3.035	0.153	0.338	0.332	0.036	0	0	0	0	34.249	35.805	35.556	1770	Feed

NormalizedValue		CO2 (%)	CH4 (%)
	Feed	48.5818	51.4182
	Permeate	52.57554	47.42446
	Retentate	48.30372	51.69628

Date	Time	Time (s)	PT01(bar)	PT02(bar)	PT03(bar)	PT04(bar)	CO2 Conc (%)	CH4 Conc (%)	FIC01(slpm)	FIC02(slpm)	FM01(kg/hr)	FM02(kg/hr)	PIC01(bar)	SP, FIC01(slpm)	SP, FIC02(slpm)	SP, PIC01(bar)	SP, VFD01(	TT01(dC)	TT02(dC)	TT03(dC)	TT04(dC)	Remarks
26/07/2	10:40:05 AM	3913	5.814	8.808	8.466	0.015	54.818	46.503	0.061	0.054	0	0	0.031	0	0	0	100	29.922	31.696	31.198	1770	Permeate
26/07/2	9:57:56 AM	1384	4.317	9.95	9.587	0.015	49.012	51.684	3.075	3.721	0.386	0.168	0.031	0	0	0	100	27.494	29.642	29.393	1770	Retentate
26/07/2	9:53:37 AM	1125	4.334	9.893	9.576	0.015	50	51.845	3.065	2.808	0.105	0.101	0.031	0	0	0	100	27.37	29.518	29.3	1770	Feed

NormalizedValue		CO2 (%)	CH4 (%)
	Feed	49.09421	50.90579
	Permeate	54.1033	45.8967

Date	Time	Time (s)	PT01(bar)	PT02(bar)	PT03(bar)	PT04(bar)	CO2 Conc (%)	CH4 Conc (%)	FIC01(slpm)	FIC02(slpm)	FM01(kg/hr)	FM02(kg/hr)	PIC01(bar)	SP, FIC01(slpm)	SP, FIC02(slpm)	SP, PIC01(bar)	SP, VFD01(	TT01(dC)	TT02(dC)	TT03(dC)	TT04(dC)	Remarks
[illegible]																						
28/07/2	10:11:49 AM	5337	8.878	9.067	8.681	0.019	50.777	50.221	3.042	3.147	0.467	0.449	0.029	0	0	0	0	29.798	31.728	31.292	1770	Permeate
28/07/2	9:38:48 AM	3356	8.819	9.005	8.634	0.018	47.77	53.691	3.025	3.02	0.402	0.376	0.029	0	0	0	0	29.424	31.105	30.825	1770	Retentate
28/07/2	9:27:16 AM	2664	8.761	8.947	8.604	0.017	49.559	51.749	3.099	2.826	0.295	0.267	0.026	0	0	0	0	29.113	30.981	30.669	1770	Feed

NormalizedValue		CO2 (%)	CH4 (%)
	Feed	48.91914	51.08086
	Permeate	50.27525	49.72475
	Retentate	47.08213	52.91787

Date	Time	Time (s)	PT01(bar)	PT02(bar)	PT03(bar)	PT04(bar)	CO2 Conc (%)	CH4 Conc (%)	FIC01(slpm)	FIC02(slpm)	FM01(kg/hr)	FM02(kg/hr)	PIC01(bar)	SP, FIC01(slpm)	SP, FIC02(slpm)	SP, PIC01(bar)	SP, VFD01(	TT01(dC)	TT02(dC)	TT03(dC)	TT04(dC)	Remarks
[illegible]																						
27/07/2	9:18:07 PM	4223	8.99	9.195	8.71	0.019	51.071	49.27	3.053	3.035	0.413	0.416	0.033	0	0	0	0	35.058	36.553	36.335	1770	Permeate
27/07/2	8:35:39 PM	1675	8.875	9.092	8.685	0.016	47.059	47.201	3.082	0.031	0.27	0.065	0.033	0	0	0	0	35.899	37.798	37.518	1770	Retentate
27/07/2	8:26:36 PM	1132	8.62	8.831	8.468	0.015	49.809	50.669	3.005	2.267	0.001	0	0.033	0	0	0	0	36.366	38.296	38.389	1770	Feed

NormalizedValue		CO2 (%)	CH4 (%)
	Feed	49.57205	50.42795
	Permeate	50.89744	49.10256
	Retentate	49.92468	50.07532

Date	Time	Time (s)	PT01(bar)	PT02(bar)	PT03(bar)	PT04(bar)	CO2 Conc (%)	CH4 Conc (%)	FIC01(slpm)	FIC02(slpm)	FM01(kg/hr)	FM02(kg/hr)	PIC01(bar)	SP, FIC01(slpm)	SP, FIC02(slpm)	SP, PIC01(bar)	SP, VFD01(	TT01(dC)	TT02(dC)	TT03(dC)	TT04(dC)	Remarks
[illegible]																						
27/07/2	10:23:14 PM	2528	9.324	9.529	9.154	0.019	51.361	47.855	3.087	3.063	0.43	0.42	0.031	0	0	0	0	33.844	35.556	35.339	1770	Permeate
27/07/2	10:13:55 PM	1969	9.275	9.484	9.117	0.017	47.893	54.337	3.084	3.331	0.429	0.214	0.031	0	0	0	0	33.969	35.65	35.463	1770	Retentate
27/07/2	10:04:35 PM	1409	9.205	9.419	9.1	0.017	50.852	51.218	3.089	3.037	0.175	0.166	0.031	0	0	0	0	34.062	35.837	35.619	1770	Feed

NormalizedValue		CO2 (%)	CH4 (%)
	Feed	49.82071	50.17929
	Permeate	51.76685	48.23315
	Retentate	46.84828	53.15172

Date	Time	Time (s)	PT01(bar)	PT02(bar)	PT03(bar)	PT04(bar)	CO2 Conc (%)	CH4 Conc (%)	FIC01(slpm)	FIC02(slpm)	FM01(kg/hr)	FM02(kg/hr)	PIC01(bar)	SP, FIC01(slpm)	SP, FIC02(slpm)	SP, PIC01(bar)	SP, VFD01(	TT01(dC)	TT02(dC)	TT03(dC)	TT04(dC)	Remarks
[illegible]																						
28/07/2	8:11:01 AM	632	3.071	3.214	2.463	0.047	51.285	49.385	3.025	3.284	0.698	0	0.024	0	0	0	0	27.276	29.58	29.206	1770	Permeate
28/07/2	8:10:40 AM	611	3.224	3.369	2.601	0.049	49.958	48.122	3.008	3.17	0.724	0	0.026	0	0	0	0	27.245	29.611	29.268	1770	Retentate
28/07/2	8:07:37 AM	428	5.932	6.086	5.105	0.11	50.901	50.308	2.883	2.78	1.186	0	0.024	0	0	0	0	27.494	30.109	30.638	1770	Feed

NormalizedValue		CO2 (%)	CH4 (%)
	Feed	50.29296	49.70704
	Permeate	50.94368	49.05632
	Retentate	50.93597	49.06403

A.2 Sample Calculations

The set of data from effect of feed composition at 50% CO_2 is taken to illustrate the calculation steps required to obtain the values of selectivity, stage cut and permeance. Below are the composition of the feed, retentate and permeate streams required for the calculations:

Stream	**Composition (%)**	
	CO_2	**CH_4**
Feed	47.36	53.15
Retentate	47.07	53.73
Permeate	49.69	50.30

Step 1:

The first step is to normalize the values as the total percentage of the compositions is not 100% due to some limitations of the IR analyzer.

For the feed stream,

$$Percentage\ of\ CO_2\ in\ feed\ stream = \frac{47.36}{47.36 + 53.15} \times 100\% = 47.12\%$$

$$Percentage\ of\ CH_4\ in\ feed\ stream = 100\% - 47.12\% = 52.88\%$$

The same steps are repeated for the retentate and permeate streams to obtain the following:

Stream	**Composition (%)**	
	CO_2	**CH_4**
Feed	47.11939	52.88061
Retentate	46.69683	53.30317
Permeate	49.69147	50.30853

Step 2:

After normalizing all the values, we can then calculate the selectivity with the following formula:

$$Selectivity, \alpha = \frac{Y_{CO_2}/Y_{CH_4}}{X_{CO_2}/X_{CH_4}} = \frac{49.69/50.31}{47.12/52.88} = 1.1084$$

Step 3:

For the calculation of the stage cut, the flow rate of the permeate stream is required. The time for the permeate stream to travel 2ml in a bubble flow meter is measured to find the flow rate. The average time taken for the permeate stream to travel 2ml is 18.251 seconds.

$$Flow\,rate\,of\,permeate\,stream = \frac{2ml}{18.251s} = \frac{0.1096ml}{s}$$

Then, the flow rate is converted to the flow rate at standard conditions:

$$Flow\,rate\,of\,permeate\,stream = \frac{0.1096ml}{s} \times \frac{273.15K}{308.15K} = \frac{0.09714ml}{s}$$

$$Flow\,rate\,of\,permeate\,stream, SLPM = \frac{0.09714ml}{s} \times \frac{1l}{1000ml} \times \frac{60s}{1min}$$

$$= 0.005828\,SLPM$$

The stage cut can then be calculated using the formula:

$$Stage\,Cut\,(\%) = \frac{Permeate\,Flow\,Rate}{Feed\,Flow\,Rate} \times 100\% = \frac{0.005828}{15} \times 100\% = 0.03885\%$$

Step 4:

To calculate the permeance of the gases, it is required to find the individual flow of the gases through the composition.

$$Flow\ rate\ of\ CO_2 in\ permeate\ stream = Composition \times Permeate\ flow\ rate$$

$$= \frac{49.69}{100} \times \frac{0.09714ml}{s} = \frac{0.04827ml}{s}$$

Then, it is required to calculate the pressure drop across the membrane in unit of cmHg.

$$Pressure\ drop, \Delta p$$

$$= \left[\frac{CO_2\ vol\%\ in\ feed}{100} \times P_{feed}\right]$$

$$- \left[\frac{CO_2\ vol\%\ in\ permeate}{100} \times P_{permeate}\right] = \left[\frac{47.12}{100} \times 5\right] - \left[\frac{49.69}{100} \times 1\right]$$

$$= 1.8591\ bar = 141.29\ cmHg$$

For the calculation of permeance, the total surface area of the membrane is also required. For this set of data, 15 fibers are used with inner and outer diameter of 180μm and 400μm, respectively. The length of the fiber is 25cm.

$$Total\ surface\ area = No. of\ fibers \times Surface\ area\ for\ 1\ fiber$$

$$= 15 \times [3.142 \times (400 \times 10^{-4}) \times 25] = 47.13cm^2$$

Finally, the permeance (GPU) can be calculated.

$$\frac{P}{l} = \frac{Q}{A.\Delta p} = \frac{0.04827ml/s}{47.13 \times 141.29} \times 10^6 = 7.25\ GPU$$

A.3 Summary of Calculated Results

Composition (% CO2)		Selectivity	Permeance (GPU)		Stage Cut (%)
			CO2	CH4	
	20	1.078585294	4.10901177	3.736493514	0.021913942
	30	1.094336779	5.595667784	4.996951578	0.029683347
	40	1.028020106	6.30718921	5.97670843	0.035001736
	50	1.108503063	7.248713056	6.372465274	0.038854592
	60	1.076543487	8.983510162	8.150814354	0.049348306
	70	1.133487867	15.51532983	13.27737426	0.084622334

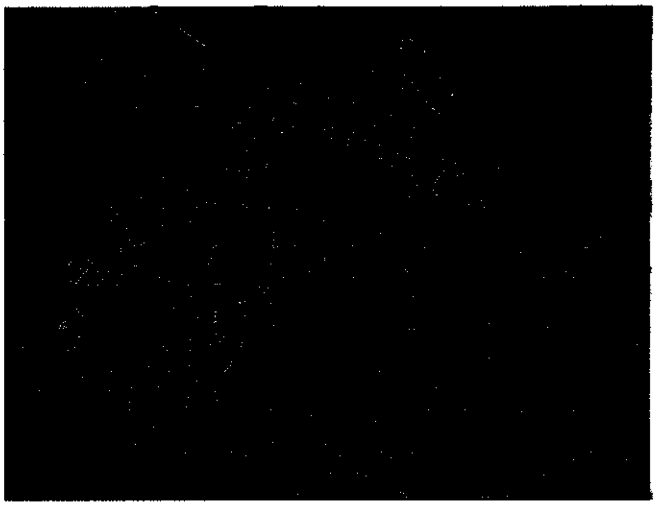
Flow (SLPM)		Selectivity	Permeance (GPU)		Stage Cut (%)
			CO2	CH4	
	6	1.108503063	7.248713056	6.372465274	0.09713648
	10	1.083230289	8.20281898	7.733886646	0.067152951
	20	1.062349222	9.075873073	8.41506367	0.037586669

Pressure (bar)		Selectivity	Permeance (GPU)		Stage Cut (%)
			CO2	CH4	
	5	1.108503063	7.248713056	6.372465274	0.09713648
	7	1.173342051	7.836576174	6.503157738	0.153492459
	10	1.222303598	6.352331799	5.082531011	0.183713772

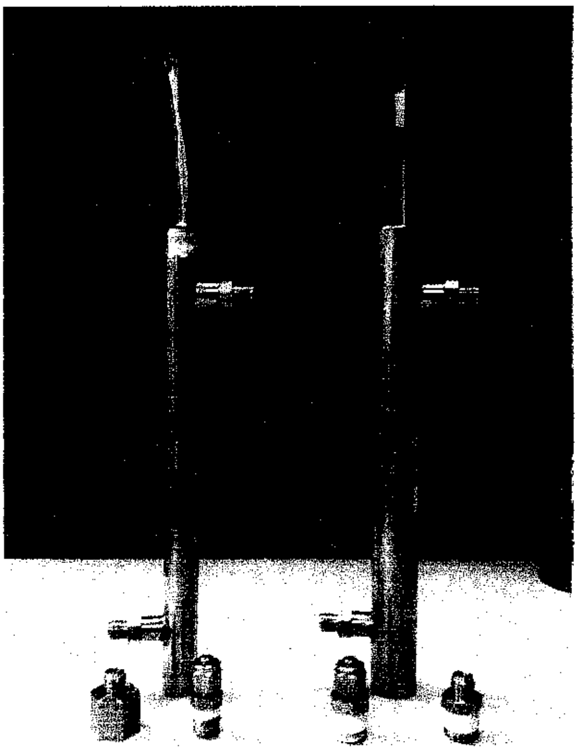
No. of Fibers		Selectivity	Permeance (GPU)		Stage Cut (%)
			CO2	CH4	
	5	1.055750047			
	15	1.054450774	5.663262371	5.339269475	0.17728379
	20	1.080987639	9.597214826	8.801727564	0.296461188
	50	1.026374939	283.6706176	275.5828991	9.01488074

B. Materials & Equipments

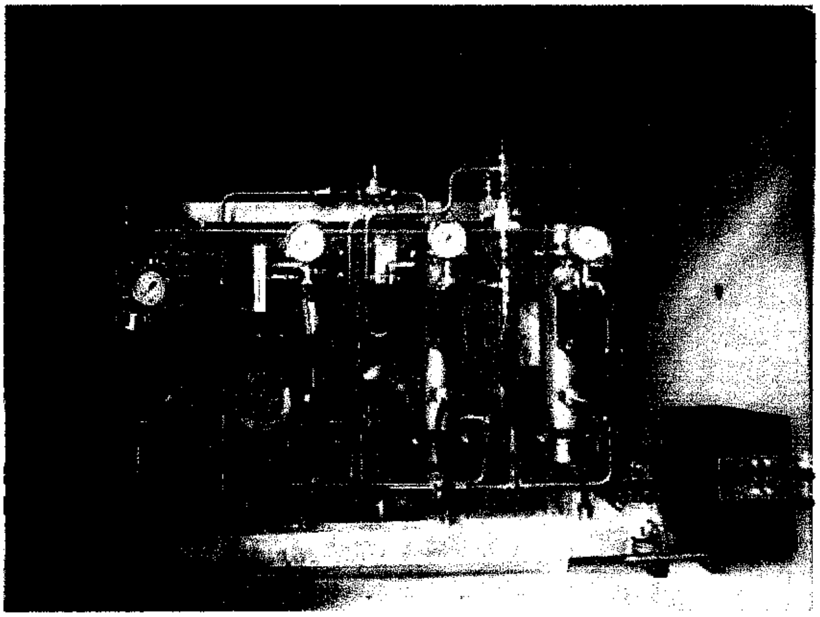
B.1 Polyimide Hollow Fibers



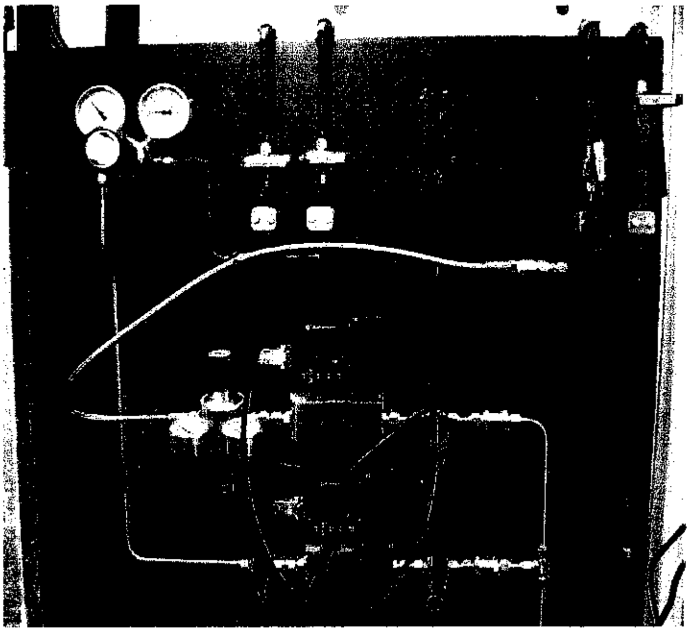
B.2 Membrane Modules



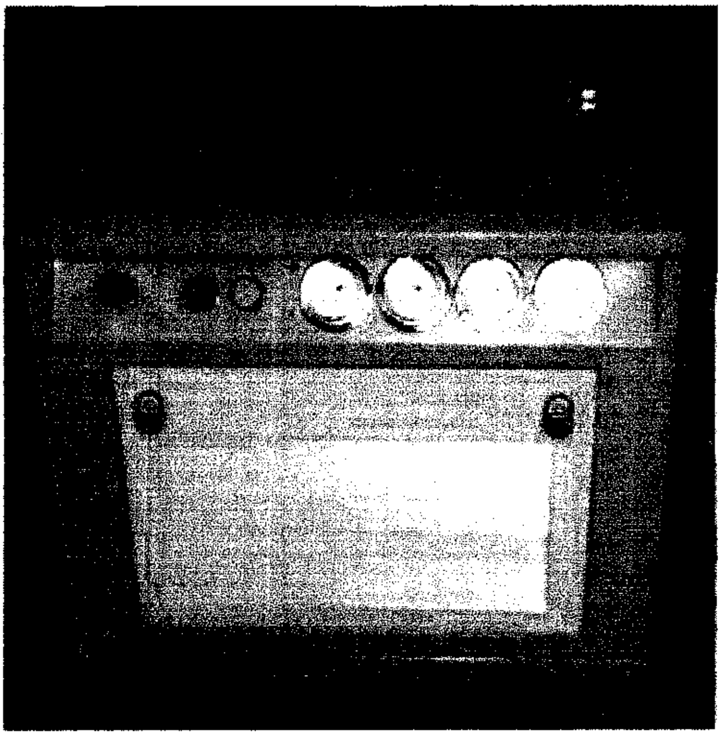
B.3 Test Rig



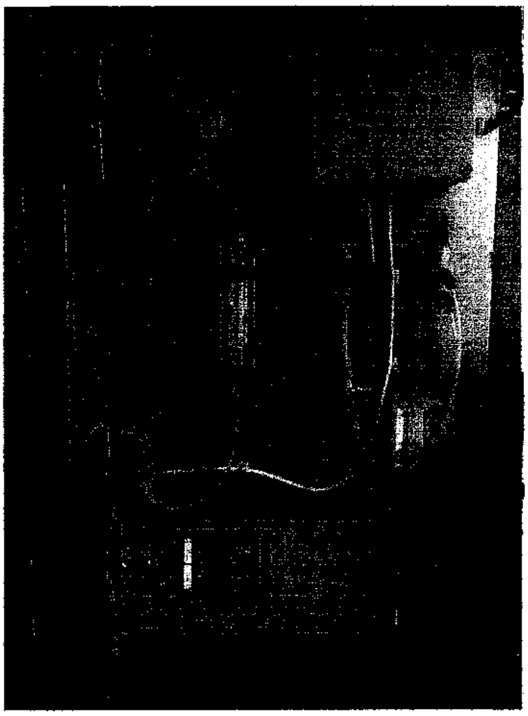
B.4 Mass Flow Controllers



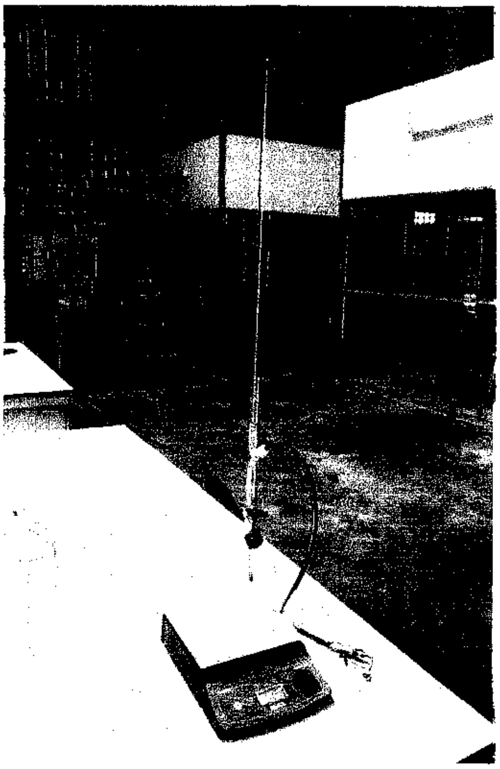
B.5 Compressor



B.6 Infrared (IR) Analyzer



B.7 Bubble Flow Meter



B.8 Hand drill to remove epoxy