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Separation of Nitrogen from Natural Gas using Inorganic Membrane

by

Mohamad Izwan Bin Ramly

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Supervisor: AP Dr. Hilmi Mukhtar

Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh

Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Approved by,

____

(AP Dr. Hilmi Mukhtar)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
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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.

(Mohamad Izwan Bin Ramly)

ABSTRACT

The presence of nitrogen as an inert in natural gas created a main issue of low heating value (calorific value) of the natural gas itself. The current technology applied for the nitrogen removal which is the cryogenic distillation requires high capital and operating cost due to feed pretreatment and high energy usage for that extremely low temperature cryogenic process. Membrane technology is offering a better alternative with low operating cost and most importantly meets the product specification of 4 mole% nitrogen maximum in natural gas. The objectives of the project are to study the separation process of nitrogen from natural gas (in this case methane) using inorganic membrane, to perform experiment of gas membrane separation based on nitrogen and methane gas permeability and selectivity, and to investigate factors influencing the separation of nitrogen from methane. The experiments were conducted on individual gases (nitrogen and methane) and their binary mixture using inorganic membrane coupled with online analysis. The gas permeability and selectivity were determined to evaluate the separation efficiency. The effect of pressure and feed concentration were also investigated. The experimental results show that the separation process is possible as the membrane is able to remove about 5 mole% of nitrogen from the feed with the highest selectivity of 1.39. However, this selectivity is considered low and further research and experiment need to be made to enhance the separation efficiency and to get desired minimum selectivity of 3 to 5. The experiment also has proven that efficient separation occurs at low nitrogen feed concentration and the membrane separates gases less efficiently at higher pressure.

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CHAPTER 1

INTRODUCTION

1.1 PROJECT BACKGROUND

Natural gas is widely used all over the world as the combustion fuel after crude oil and its products and the demand for the gas has been steadily increasing. Natural gas contains light hydrocarbon mainly methane with some amount of ethane, propane, butane and pentane as well as considerable amount of contaminants or non-hydrocarbons such as nitrogen, carbon dioxide, hydrogen sulfide, water vapour and also trace amount of mercury and helium.

The presence of nitrogen reduces the quality of natural gas in a sense that it lowers the heating value of natural gas which then reducing the heat (energy) produced during the combustion of the gas. In addition, excess amount of nitrogen in natural gas makes it unsuitable for pipeline transportation as the limit is 4 mole% of nitrogen maximum.

The cryogenic distillation process has been commercially used worldwide for removal of nitrogen from natural gas in various Gas Processing Plants but this technique requires high capital and operating costs. Furthermore, cryogenic process also associated with mechanical and operational complexity.

The implementation of inorganic membrane for separation of nitrogen from natural gas is looked up as very suitable as it is very cost effective for both capital and operating cost. The most important thing is that the membrane will allow the low quality natural gas to meet the pipeline specification of 4 mol% of nitrogen in natural gas. In addition, the inorganic membrane has high thermal and chemical stability where it is resistant to corrosive liquid and gas as well as can operate at elevated temperature and pressure.

For the membrane separation process, semipermeable membrane will be used where low quality natural gas will flow through the unit and the membrane will selectively separates the feed into permeate and retentate streams. The main concern for this membrane separation process is the selectivity and permeability of the inorganic membrane.

1.2 PROBLEM STATEMENT

Raw natural gas from gas field is categorized as low quality due to the presence of nitrogen. The typical amount of nitrogen in natural gas is 0.5-15 mole% but the exact composition will vary among different fields. There are fields of natural gas that contain extremely high nitrogen and in certain oil and gas fields, nitrogen (inert gas) has been utilized for enhanced oil recovery that leads to high nitrogen content in the natural gas.

Nitrogen gas causes the natural gas to have low heating value. Heating value is the amount of heat released during the combustion of specified amount of natural gas. In addition, nitrogen as an inert gas takes up capacity in pipeline that could be used for valuable methane. Excess amount of nitrogen in natural gas making them unsuitable for pipeline transportation and commercial gas regulatory as the most typical specification is 4 mole% nitrogen maximum.

The process that is widely used and proven commercially for removal of nitrogen from natural gas is cryogenic distillation where condensation and separation occur at very low temperature. The cryogenic process takes the advantage on the fact that methane and nitrogen change phase (from gas to liquid) at different temperatures. By controlling the pressure and temperature in the system, the methane is liquefied and collected as it drops out of the gas. However, because of the temperature involved (-150 to -250 ^{o}C), the long cool down time, and extensive equipment required, this conventional technique requires high capital and operating costs. It requires huge amount of energy cost for the separation process. In addition, this cryogenic process also associated with operational and mechanical complexity.

1.3 OBJECTIVE AND SCOPE OF STUDY

1.3.1 Objective

1) To study the separation process of nitrogen from natural gas (in this case methane) using inorganic membrane
2) To perform experiment of gas membrane separation based on nitrogen and methane gas permeability and selectivity
3) To investigate factors influencing the separation of nitrogen from methane

1.3.2 Scope of Study

This project will be focusing on experimental work for the separation of nitrogen from natural gas (in this case methane) across inorganic membrane. In order to achieve the desired objectives, the work is divided into two parts. The first part is to study the permeability of pure gases which are nitrogen and methane separately. The second part involved the study of permeability of binary gas mixture of nitrogen and methane as well as their selectivity (separation factor). The work scope of this research project can be summarised as follow:

1) To conduct study and literature review on membrane separation technique focusing on inorganic membrane and N_2/CH_4 gas stream.
2) To apply appropriate methodology for the development of N_2/CH_4 gas separation using inorganic membrane.
3) To conduct experiment on ideal permeability of nitrogen and methane individually.
4) To conduct experiment on permeability of nitrogen-methane mixture and asses the separation efficiency of the inorganic membrane using the Gas Membrane Separation Pilot Plant.

1.4 THE FEASIBILITY AND RELEVANCY OF PROJECT

Due to heating value specifications and pipeline transportation requirement for natural gas, plus with the expensive operation as well as the mechanical and operational complexity of cryogenic process, it is foresee that the use of inorganic membrane for separation of nitrogen from natural gas can present the economically viable scenario in the potential development of processing natural gas containing high nitrogen concentrations.

The commercial application of inorganic membrane is expected because of some qualifications. It requires low production cost for separation unit including fabrication and installation. The materials for construction are available at high quality and at reasonable price. It is easy to scale up from laboratory to production installation. Thus, it is economically efficient and cost effective in both capital and operating cost.

In addition, the inorganic membrane is very reliable as it has long term stability structure because it is resistant to corrosive fluid and can operate at elevated temperature above 500^{o}C. It is also strongly believed that the membrane system can meet the nitrogen limit specification of pipeline which is 4 mole% nitrogen maximum in natural gas as the membrane has high selectivity (separation) and also high permeability (production). Furthermore, membrane separation unit are smaller than other types of commercial separation unit and it requires lack of mechanical and operational complexity.

The approach to the nitrogen separation from natural gas is to use inorganic semipermeable membrane. The incoming feed which is the low quality natural gas will flow through the membrane separation unit and the membrane will selectively separates the feed into permeate and retentate streams. The stream with high purity methane will be the sales gas and the revenue producing product. The nitrogen-rich can be used as a fuel gas in the process unit. The inorganic materials that can be considered for fabrication of inorganic membrane include ceramic, silica, metal, alloy, zeolite, silicon, etc.

CHAPTER 2

LITERATURE REVIEW

2.1 NATURAL GAS

Natural gas is a mixture of gaseous hydrocarbons with varying quantities of non-hydrocarbons, which are normally considered impurities. It is obtained from a natural underground reservoir.

Natural gas contains a large quantity of methane along with heavier hydrocarbons such as ethane, propane, isobutene, normal butane, etc. In addition, it often contains a considerable amount of non-hydrocarbons, such as nitrogen, carbon dioxide, and hydrogen sulfide. There are traces amount of compounds like helium and mercury. In addition, it is also generally saturated with water. [1]

Table 2. 1: Typical composition range of natural gas [2]

Compound	Range (mol%)
Hydrocarbon	
Methane	75-99
Ethane	1-15
Propane	1-10
n-Butane	0-2
Isobutane	0-1
n-Pentane	0-1
Isopentane	0-1
Hexane	0-1
Heptane+	0-0.1

Compound	Range (mol%)
Non-hydrocarbons	
Nitrogen	0-15
Carbon dioxide	0-30
Hydrogen Sulfide	0-30
Helium	0-5
Mercury	Trace

Natural gas is combustible, and when burned it gives off a great deal of energy. Unlike other fossil fuels, however, natural gas is clean burning and emits lower levels of potentially harmful byproducts into the air. [3]

As it is a gas, it frequently contains impurities. Nitrogen itself in natural gas is unwanted in commercial sale gas. Nitrogen is inert and lowers the BTU value (Calorific value) of natural gas. It takes up capacity in pipeline that could be used for valuable methane. [4] Calorific value or heating value or BTU value is defined by the number of heat units released when a unit volume of the gas burns. It is measured in units of energy per unit of the substance, usually mass or volume, such as: kcal/kg, kJ/kg, J/mol, Btu/m³. [5]

According to McKetta's Encyclopedia of chemical processing and design, high nitrogen content in natural gas comes from two main sources. There are fields of gas that have extremely high nitrogen contents with corresponding to lower BTU contents in which the highest could be 25 mol%. Besides, nitrogen (inert gas) has been utilized for enhaned oil recovery in certain oil field. The dilution effect of nitrogen has caused the natural gas to have low heating value, thereby making them unsuitable for pipeline transportation. [6] The most typical specifications for commercial sale gas and pipeline specification limit is 4 mole% of nitrogen maximum in natural gas.

Table 2. 2: Physical properties of CH_4 and N_2 [7]

	CH_4	N_2
Molecular weight	16.04	28.01
Kinetic diameter, A	3.80	3.64
Specific volume at 70 °F, 1 atm, ml/g	1479.5	861.5
Sublimation point at 1 atm, °C	-161.5	-195.8
Triple point pressure, atm	0.115	0.121
Triple point temperature, °C	-182.5	-210.0
Density, gas at 0 °C, 1 atm, g/l	0.72	1.250^a
Specific gravity, gas at 0 °C, 1 atm (Air=1)	0.5549^b	
Critical temperature, °C	-82.1	-147.1
Critical pressure, atm	45.8	33.5
Critical density, g/ml	0.162	0.311
Viscosity, gas at 70 °F, 1 atm, cp	0.0106^c 0.0116^d	0.0170^e 0.0174^f
Solubility in water at 25 °C, 1 atm, ml/l water		23

^{a} At 20 °C; ^{b} 60 °F; ^{c} 4.4 °C; ^{d} 37.8 °C; ^{e} 0 °C; ^{f} 15 °C

2.2 CURRENT TECHNOLOGY

Nitrogen Rejection Unit is currently an expensive operation which can present uneconomic scenarios for processing natural gas containing high nitrogen content. The most reliable, well-known, and widely used process for nitrogen rejection from natural gas is cryogenic distillation. It consists of liquefying the feed stream using temperatures in the order of -150^{o}C to -250^{o}C and separating nitrogen via fractionation. In order to reduce the gas temperature to this level, the gas is compressed, cooled by heat exchanger, and then expanded to low pressure for recovery. Significant energy for compression and expensive materials of construction are required. Water and carbon dioxide concentration must be reduced to levels required to prevent freezing. [8]

Cryogenic processes are typically used to treat gas containing more than about 10% nitrogen but however, cryogenic process also requires extensive pretreatment. The pretreatment generally consists of amine scrubbing to remove carbon dioxide followed by glycol dehydration to remove most of the water vapour. Molecular sieve (PSA) then removes any remaining water vapour and carbon dioxide, after which the gas is cooled in a final polishing step to remove heavy hydrocarbons and aromatics. All of these components must be removed to avoid freeze-up in the cryo-section of the plant, which operates at -250^{o}C. Both pretreatment and cryogenic process involve with mechanical and operational complexity. [9]

In the pressure swing adsorption (PSA) process, methane and other hydrocarbons are adsorbed onto molecular sieves, leaving a nitrogen-rich gas stream. The PSA process is most competitive, therefore, for feed streams containing a high concentration of nitrogen. Multiple beds are typically used, with complicated switching controls between beds. The capital and operating cost of these systems are relatively high. In general, PSA processes are suited to low to medium gas flows. [9]

Lean oil absorption processes, such as the Mehra process, have been under development for about 10 years. These processes use chilled oil to absorb methane and other hydrocarbons. The oil is then heated and flashed at lower pressure in a series of vessels, and the liberated hydrocarbon gases are collected and recompressed to pipeline pressures. High recovery of hydrocarbons is achieved, but the process is capital intensive. As of with PSA, the process is best suited for throughputs of less than about 10 MMscfd with relatively high nitrogen concentrations. [9]

Table 2. 3: Processes currently used or under development for removal of nitrogen from natural gas [10,11,12]

Process (Status)	Method of Separation	Application	Comments
Cryogenic Distillation (Proven commercially)	Condensation and distillation at cryogenic temperatures	Typically high flow rate application	• High methane recovery • Significant pretreatment and compression costs • High capital cost
Pressure Swing Adsorption (PSA) (Limited commercial success)	Adsorption of methane	Generally small to medium flow rates	• Pretreatment required • High capital and compression costs • High operating costs • Moderate methane recovery
Lean Oil Absorption (New Process)	Absorption of methane in chilled hydrocarbon oil	Suitable for high nitrogen content streams	• High capital costs • Processing costs significant • Need to absorb bulk of methane increases equipment size and compression requirements
Nitrogen Absorption (Research stage)	Selective absorption of nitrogen in chelating solvent		• No methane recompression needed • Stability of solvent suspect

2.3 LATEST MEMBRANE TECHNOLOGY FOR NITROGEN REMOVAL

NitroSep™ from Membrane Technology & Research[13]

MTR's NitroSep™ system of polymeric membrane produces pipeline-quality or pipeline-acceptable gas and a nitrogen-rich fuel from raw natural gas. The proprietary membranes are significantly more permeable to methane, ethane, and other hydrocarbons than to nitrogen.

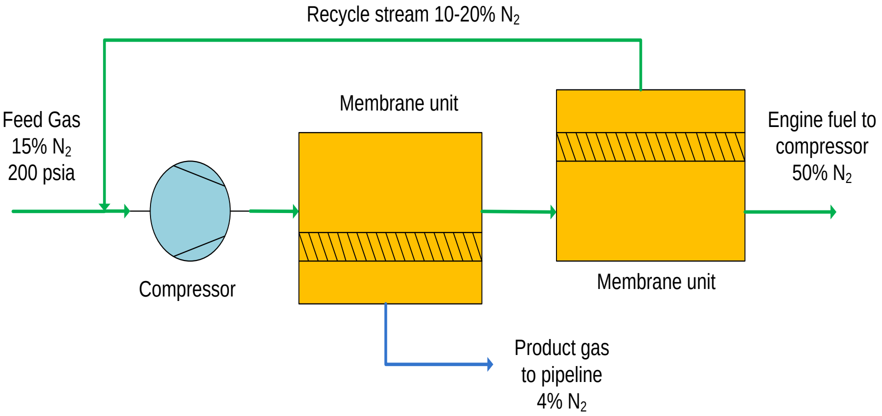


Figure 2. 1: NitroSep™ Process

Figure 2. 2: NitroSep™ units upgrading high nitrogen gas in California

Gas containing 8% to 15% nitrogen is compressed and passed across a first set of membrane modules. The permeate, which contains 4% nitrogen, is sent to the pipeline; the nitrogen-rich residue gas is passed to a second set of membrane modules. These modules produce a residue gas containing 50% nitrogen and a nitrogen-depleted permeate containing 10% to 20% nitrogen. The residue gas is used as fuel; the permeate is mixed with the incoming feed gas for further recovery.

Table 2. 4: Nitro-Sep Performance and benefits

System Performance	**Benefits**
• Feed rate: 0.4 to > 100 MMscfd • Feed N_2 content: 4% to 50% • Target N_2% or BTU value • Product gas pressure: 35 to 350 psia • Product BTU recovery: 90%+ • Heavy hydrocarbon recovery : >95%	• Nitrogen content reduced to pipeline specification (3% or higher) • Heavy hydrocarbons captured in the product gas • Easy, low-cost installation; system can be installed in 1-2 days • Membrane unit requires no maintenance; robust long-lasting membranes • System is easily moved from one location to another

2.4 PRINCIPLE OF MEMBRANE SEPARATION

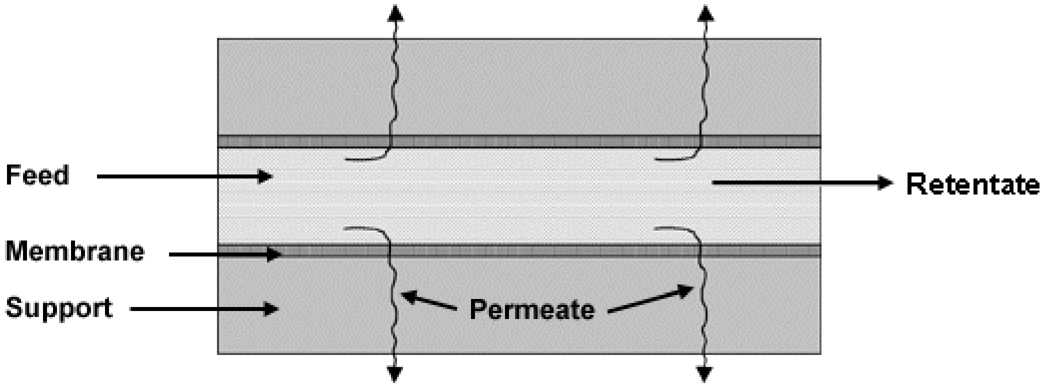


Figure 2. 3: Membrane

Membrane is a device that selectively permits the separation of one or more materials from a liquid or gas. According to Richard D. Noble (Professor at University of Colorado), membrane is a selective semipermeable barrier that allows different gases, vapours or liquids to move through it at different rate. [14]

Membrane restricts the motion of molecules passing across it so some molecules move slowly than the other or are excluded.

Mechanism of restrictions:

1. Size variability of the molecules
2. Affinity for the membrane material
3. Permeation driving forces (concentration, pressure difference)

Key desirable properties for membranes [14] :

- a. Permeability
- b. Selectivity
- c. Processibility
- d. Stability
- e. Cost

2.4.1 Membrane Structure

Membrane can be classified into three, porous membranes classify according to size of particle or molecules, non porous membranes classify according to chemical affinities between components and membrane materials and also carrier membrane classify according to carrier transport. [15]

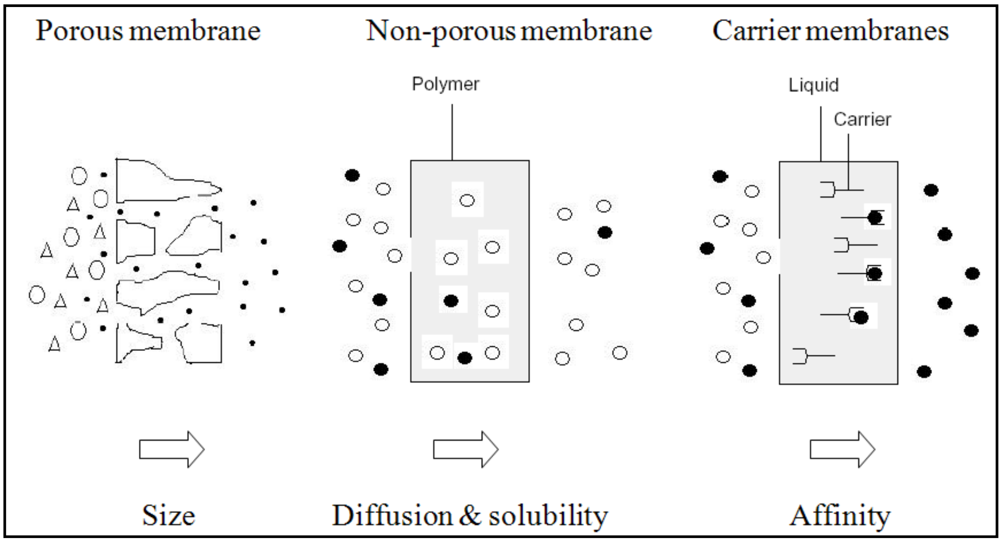


Figure 2. 4: Membrane Structure

For porous membrane, separation is mainly determined by the pore size. High selectivity can be obtained when the size of the solute is large relative to the pore size in the membrane. The non-porous (or dense) membrane is suitable for gas separation. It is capable to separate molecules of the same size (liquid or gas). The transport is determined by the diffusion mechanism, which means that the components must first dissolve into the membrane and then diffuse through the membrane due to a driving force. The separation is due to differences in diffusivity and/or solubility. On the other hand, carrier membrane allows separation to occur by a carrier molecule transporting the desired component across the membrane. The carrier molecule shows a very specific affinity to one component or class of components in the feed, which means that high selectivity can be obtained.

2.4.2 Fick's Law [16]

Mass flux, $J_i = -D_{ij} \frac{dc_i}{d_x}$ 2.1

J_i = flux of component i (mol/m^2s)

D_{ij} = diffusive coefficient (m^2s)

$\frac{dc_i}{d_x}$ = concentration gradient for component i over length, x (mol/m^3·m)

Fick's Law integrated and applied for membrane:

dx = *l* (membrane thickness)

dc_i = concentration difference over membrane (partial pressure of gas)

D_{ij} = vary according to the dominating transport mechanism

Therefore,

$$J_i = \frac{D_i(C_0 - C_1)_i}{l} \qquad 2.2$$

In a system where a gas or a vapor diffuses through a membrane, the concentration may be replaced by gas partial vapor pressure, *p*0 and *p*1 on either side of the membrane, then *J* is usually described by

$$J_i = \frac{P_i(p_0 - p_1)_i}{l} \qquad 2.3$$

where

P = permeability

p_0 = partial pressure of feed gas

p_1 = partial pressure of permeate gas

and

$$\Delta p_i = (p_0 - p_1)_i \qquad 2.4$$

Therefore,

Permeability, P for gas i:

$$P_i = \frac{J_i \cdot l}{\Delta \mathrm{p}_i} \qquad 2.5$$

P is also known in barrer unit.

Δp_i = partial pressure difference of i across membrane (Pa or Bar)

The flux of component i, J_i also can be substituted by flow rate, Q_i which will be divided by area of the membrane, A as of in the following equation:

$$P_i = \frac{Q_i \cdot l}{A \cdot \Delta p_i} \qquad (\mathrm{cm^3 \cdot cm/cm^2 \cdot Pa \cdot s}) \qquad 2.6$$

Thus, flux through membrane is proportional to the pressure difference across membrane. Flux is inversely proportional to membrane thickness.

Selectivity:

Ideal separation factor α* is the ratio of the pure gas permeability for individual component i and j,

$$\alpha^*{}_{ij} = \frac{P_i}{P_j} \qquad 2.7$$

Separation factor for gas mixture, α_{ij}:

$$\alpha^*{}_{ij} = \frac{y_i/y_j}{x_i/x_j} \qquad 2.8$$

x = mol fraction of feed

y = mol fraction of permeate

In addition,

Permeability, $P = D \times S$ 2.9

D= Diffusion,

S= Solubility,

The solubility/sorptivity (thermodynamic parameter) coefficient is a measurement of the amount of gas sorbed by the membrane when equilibrated with a given pressure of gas at a given temperature. The diffusion (kinetic parameter) coefficient indicates how fast a penetrant is transported through the membrane in the absence of obstructivesorption.

2.5 TRANSPORT MECHANISM FOR MEMBRANE GAS SEPARATION

Transport Mechanism of gas through membranes [18] :

- Solution Diffusion
- Knudsen Diffusion
- Selective Surface Flow
- Molecular Sieving
- Ion-Conductive Transport
- Facilitated Transport

Most common types of membrane used today are dense polymeric materials. The transport mechanism applied to polymeric membrane is Solution-diffusion based on Fick's Law.

For microporous membrane (inorganic or hybrid), the transport mechanism involves one of the mechanisms or combination:

a. Knudsen Diffusion – based on molecular weight
b. Molecular Sieving - based on size of molecules
c. Selective Surface Flow/Surface diffusion – surface interaction

Average pore size and pore size distribution is important as indication of which transport mechanism will be dominant for given gas mixture in a defined material and at given process condition

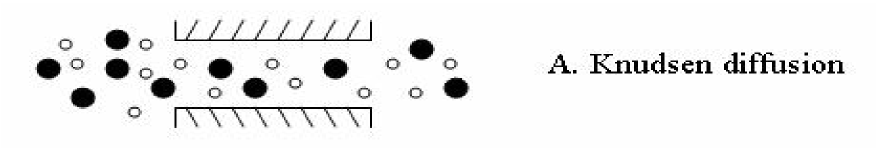


Figure 2. 5: Knudsen diffusion

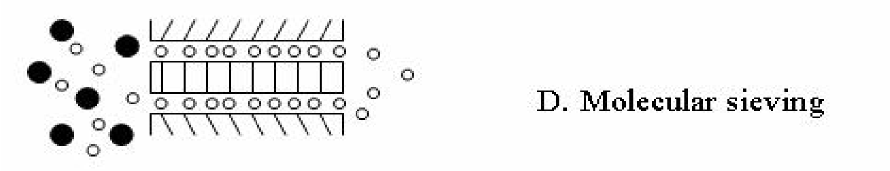


Figure 2. 6: Molecular sieving

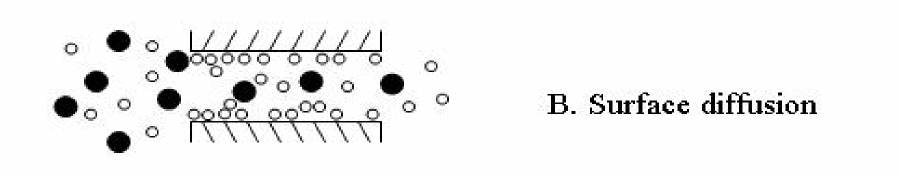


Figure 2. 7: Surface diffusion

2.6 INORGANIC MEMBRANE

Inorganic membranes are versatile. They can operate at elevated temperatures, with metal membranes stable at temperatures ranging from 500-800º C and with many ceramic membranes usable at over 1000º C. They are also much more resistant to chemical attack. Because of the wide variety of materials that may be used in the fabrication of our inorganic membranes, resistance to corrosive liquids and gases, even at elevated temperatures, can be realized. Inorganic membranes compete with organic membranes for commercial use. In many of the harsh operational environments listed above, organic membranes will not perform well, or will not survive at all. For these environments, only inorganic membranes offer needed solutions. [19]

2.7 MEMBRANE MODULE [22]

The various membrane materials describe in the previous section are available in one or more of the following modules: plate and frame, spiral-wound, tubular, and hollow-fiber.

Table 2. 5: Comparison of membrane modules

Characteristics	**Plate and frame**	**Spiral-wound**	**Tubular**	**Hollow-fiber**
Resistance to fouling	Good	Moderate	Very good	Poor
Ease of cleaning	Good	Fair	Excellent	Poor
Relative cost	High	Low	High	Low
Main application	• Dialysis • Reverse Osmosis • Pervaporation • Ultrafiltration • Microfiltration	• Gas Separation • Dialysis • Reverse Osmosis • Ultrafiltration • Microfiltration	• Reverse osmosis • Ultrafiltration	• Gas Separation • Dialysis • Reverse osmosis • Ultrafiltration

The project will be focusing on tubular membrane which is the most common module for commercial membrane used nowadays. Tubular membrane has high resistance to fouling, quite easy to construct for inorganic-based membrane, and easy to clean. It has a self-supporting structure, which provides a higher-pressure operation capability.

CHAPTER 3

METHODOLOGY

This chapter explains the laboratory work scopes, tools, chemicals, and experimental procedures used throughout this research project.

3.1 LABORATORY WORK SCOPE

Types of experiment:

1. Ideal permeability test for methane and nitrogen
2. Permeability of nitrogen-methane mixture and the selectivity. The experiment on tubular membrane module:
    a. Effect of feed pressure on gas membrane separation efficiency
    b. Effect of N_2 concentration in the feed on gas membrane separation efficiency

3.2 TOOLS

1. Gas Membrane Separation Pilot Plant
    - Membrane module: Tubular Membrane
    - Type of membrane: Proprietary membrane
    - Material: Ceramic

3.3 CHEMICALS

1. Methane – Flammable gas
    - Purity: 99.5 %
    - Supplier: Mox-Linde Sdn. Bhd
2. Nitrogen – Inert gas
    - Purity: 100%
    - Supplier: Mox-Linde Sdn. Bhd

3.4 EXPERIMENTAL STEPS [22]

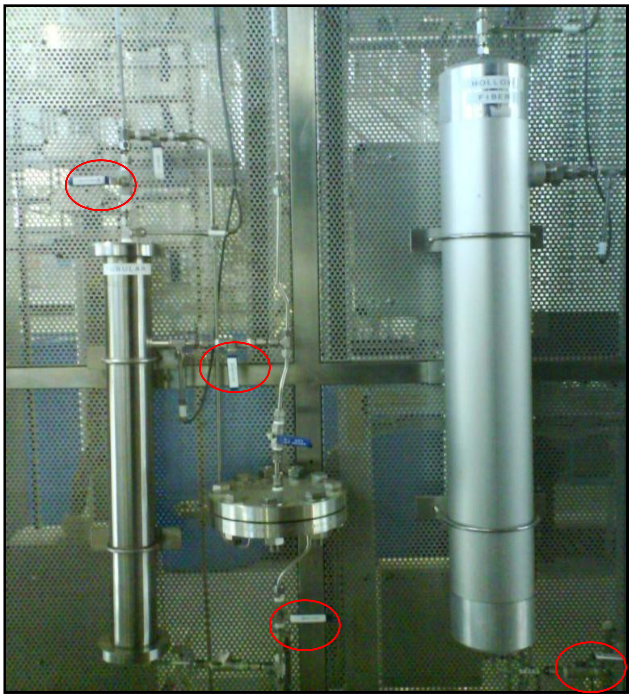


Figure 3. 1: Tubular and Hollow-Fiber Module

Equipment Start-up

- Do line tracing and check valve positioning
- Adjust desired pressure of feed gas cylinder

Figure 3. 2: CH_4 and N_2 gas cylinders

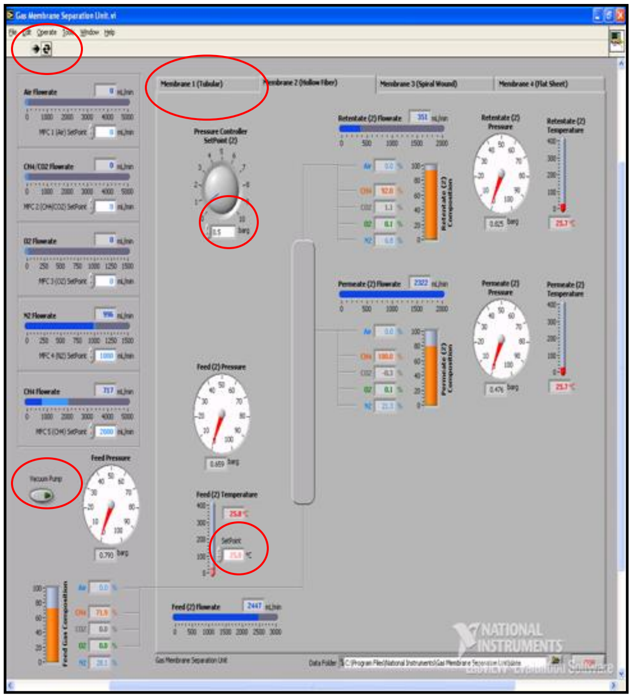


Figure 3. 3: Gas Membrane Separation Interface

Running the experiment

- Choose membrane module (eg. Tubular)
- Purge the system using vacuum pump
- Set the flow of each gases
- Set the desired temperature and pressure
- Start the separation process through membrane

Equipment Shut-down

- Set flow of all gases to zero
- Close gas cylinder valves
- Purge the system
- Close all valves

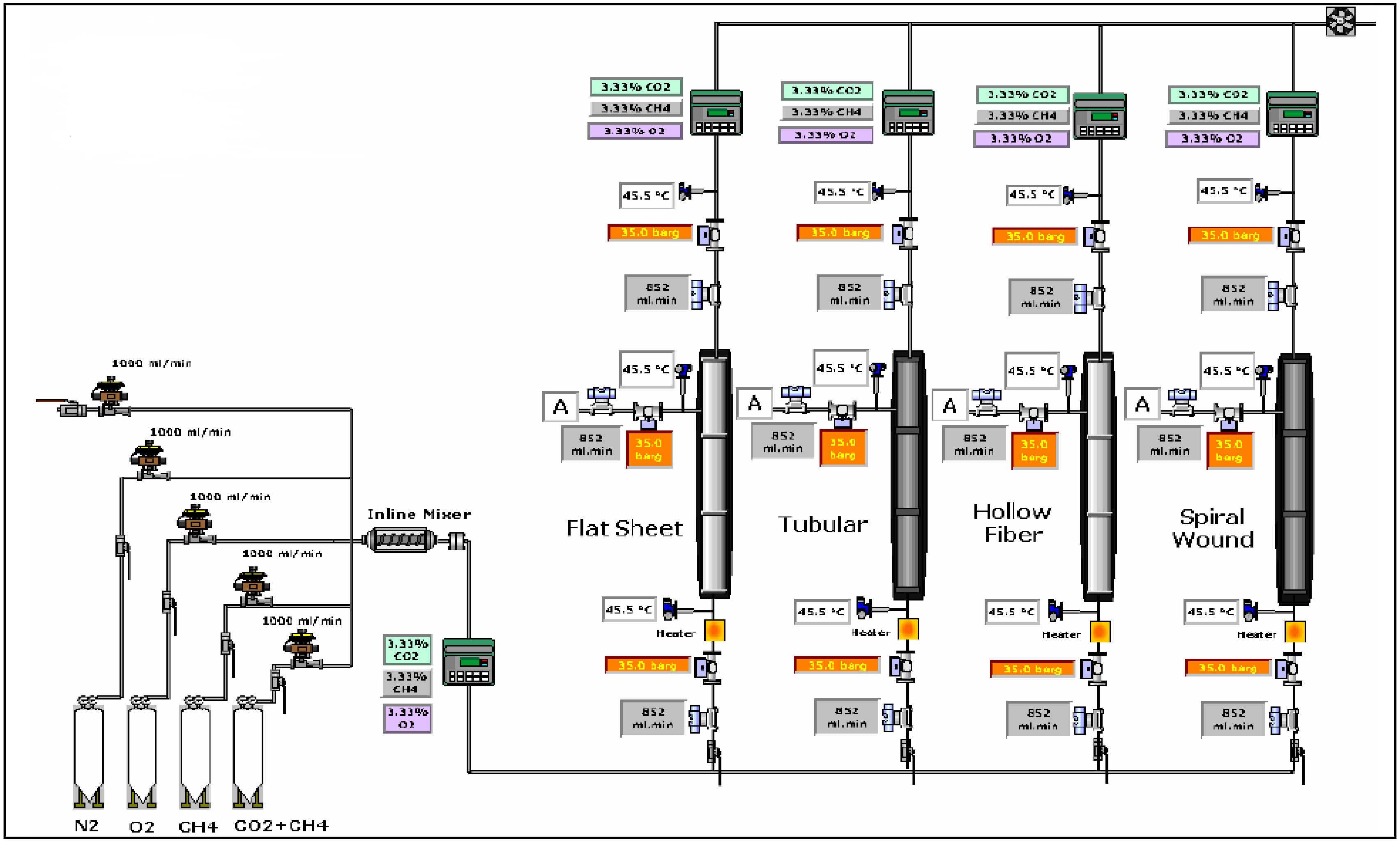


Figure 3. 4: Gas Membrane Separation Pilot Plant

3.5 DETAILED PROCEDURES FOR HANDLING EQUIPMENT

A. Equipment Start-up

1. Turn on the computer that is linked to the system.
2. Open software ‘National Instrument (NI) Labview’ in the start-up menu.
3. Open the application file called “Application” and starts the online data acquisition programme.
4. Choose membrane module (eg. tubular) and do line tracing including checking the valve positioning mode on the overall equipment condition before proceed to next stage.
5. Turn on vacuum pump to ensure all gas are cleared from gas analyzer and cooling chamber into vent.

B. Running the Experiment

1. Before commencing experiment, turn on the water supply into outlet gas cooler by open the V_{water}.
2. Make sure valve positioning for tubular membrane as per table below. Be in mind that we are conducting Tubular Membrane module, so other modules valve positioning should be close.

Table 3. 1: Valve positioning

Line	Open	Close
Feed	V_{feed}, V_{1feed}, $V_{1(heater)in}$, $V_{1(heater)out}$	$V_{r\&d\ (feed)}$
Retentate	$V_{tubular\text{-}ret}$, $SV_{1A(retentate)}$, $V_{1(retentate)vent}$	$V_{r\&d\text{-}ret}$, $SV_{1B(retentate)}$,
Permeate	$V_{tubular\text{-}per}$, $V_{1(permeate)vent}$	$V_{r\&d\text{-}per}$, $SV_{1A(permeate)}$, $S_{V1B(permeate)}$,

3. Set the flow of each gases (nitrogen and methane) in accordance to the mass flow controller (MFC) setting.
4. Note the gas inlet temperature. Only turn on the heater if conducted the experiment under elevated temperature.

5. Gas pressure can be set up and detect manually from each gas vessel.
6. To run data, go to top left corner of application and click “Run Continuously” button. Be note that all data is record in an Excel file the minute “Run” mode is selected. Experiment data must be retrieved and save after each experiment set.
7. Repeat the procedure for other membrane module by changing membrane module operation and valve numbering.

C. Equipment Shut Down

1. Turn off heater if used.
2. Set flow of all gases to zero value in NI (Labview).
3. Close gas vessel valves.
4. Monitor temperature of the outlet temperature of gas leaving the equipment. If temperature is lower than 30°C, stop cold water circulation by close V_{water} manually and turn off vacuum pump.
5. Save and print all experiment data in pen drive. Make sure there is no data record in saved in the computer hard disk.
6. Shut down the computer.

D. Purging the System

1. Purging the system can be done before experiment start or after experiment done.
2. Make sure the valve positioning is same as running the experiment procedure.
3. Turn on the vacuum pump.
4. Introduce Nitrogen gas (N_2) into the equipment for several minutes.
5. Stop nitrogen gas supply.
6. Stop vacuum pump after nitrogen supply stop a while.
7. Close all valves.

CHAPTER 4

RESULT AND DISCUSSION

4.1 IDEAL PERMEABILITY AND SELECTIVITY OF INDIVIDUAL GAS

Ideal permeability test was conducted on both nitrogen and methane separately in the gas membrane pilot plant as shown above using the tubular membrane module. The system was set to operate at 1 bar and 25^{o}C and the experiment was conducted for 30 minutes. The permeability study was first conducted on nitrogen at rate of 1000 mL/min based on Mass Flow Controller (MFC) setting. The gas is introduced from the purified nitrogen gas cylinder through the MFC to static inline mixer and then went through feed gas analyzer and Mass Flow Meter (MFM). The feed gas then enters the heater (not activated since the experiment was at room temperature) before entering the hollow fiber module. Separation occurs in the membrane to allow formation of two outlet streams of permeate and retentate. The concentration and flow rate of both streams are measured by the permeate / retentate analyzer and MFM respectively. The outlet streams are cooled in the cooler before vented out to the surrounding.

The permeability test was then repeated using methane gas with the same conditions. Further study was conducted on the individual gas at various pressures which are 1 bar, 3 bar, 5 bar, and 7 bar. The result of the ideal permeability and selectivity test is as follows:

Feed Pressure: 1 bar

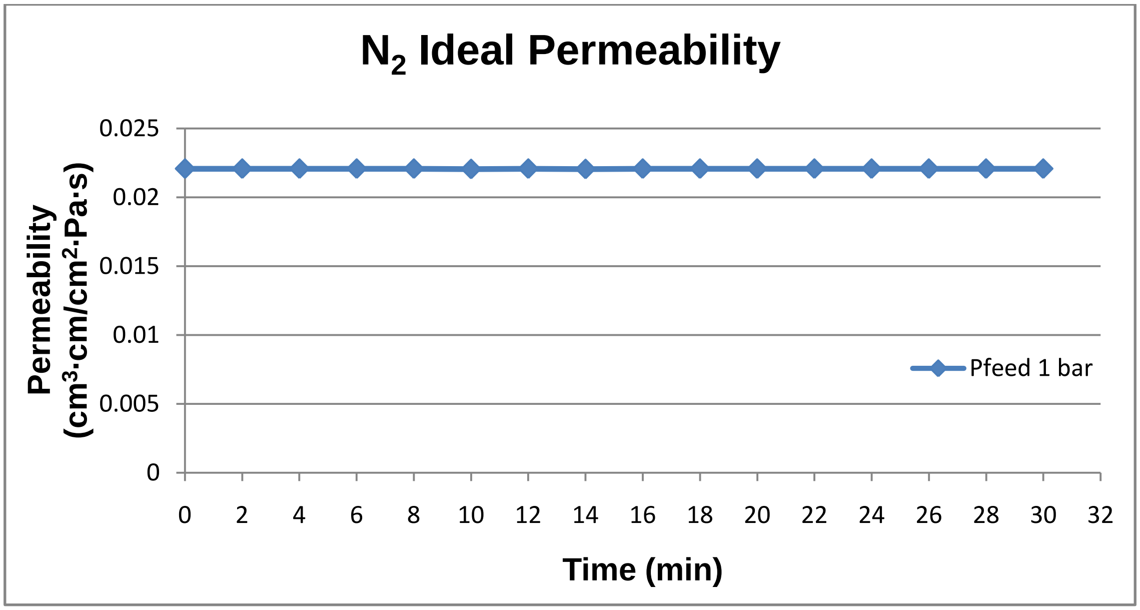


Figure 4. 1: Ideal permeability of nitrogen at feed pressure 1 bar

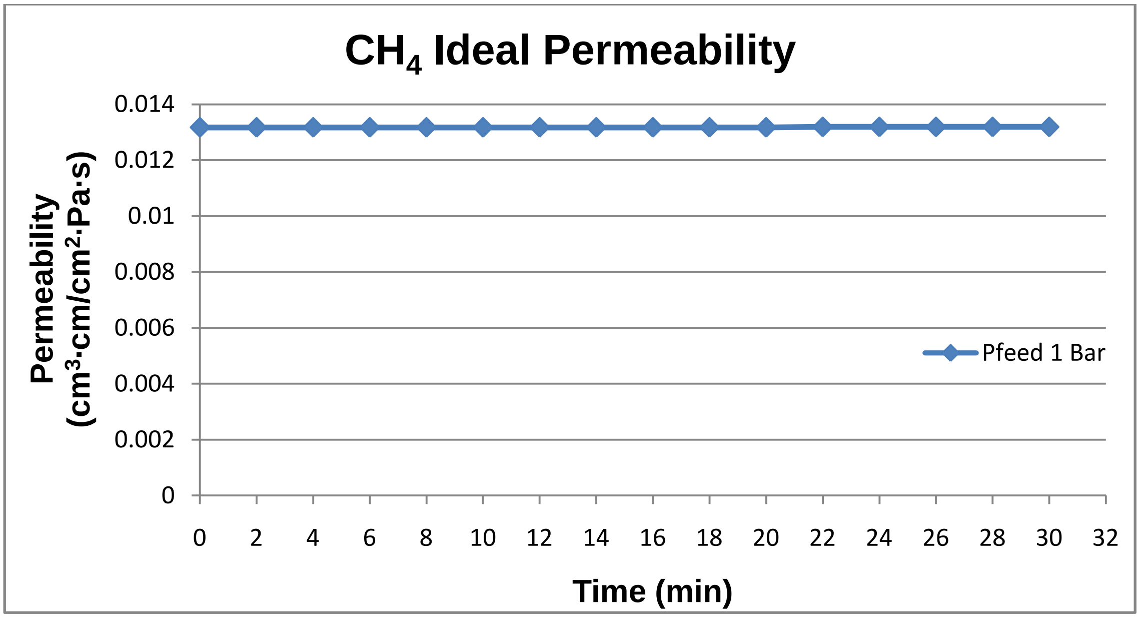


Figure 4. 2: Ideal permeability of methane at feed pressure 1 bar

Figure 4.1 and 4.2 shows the ideal permeability of nitrogen and methane respectively at 1 bar feed pressure. At 1 bar, nitrogen gas has permeability of 0.023 barrer ($cm^3 \cdot cm/cm^2 \cdot Pa \cdot s$) while methane 0.013 barrer. We can observe clearly from the plots that nitrogen has higher permeability than methane. Permeability in this case is the amount of flux that permeates through the membrane. The higher the permeability of certain gas, the more it permeates through membrane. From the observation, we can say that the membrane is nitrogen selective as it permeates more nitrogen than methane and the high permeability of nitrogen may caused by its kinetic diameter which is smaller than methane gas. Kinetic diameter of nitrogen is 3.64 Å while methane is 3.8 Å. Smaller diameter allows the nitrogen molecules to pass through membrane much easier than methane.

For feed pressure: 3 bar, 5 bar, and 7 bar, the plots are available in the appendix. The effect of pressure on individual gas permeability and selectivity can be observed from the graphs below:

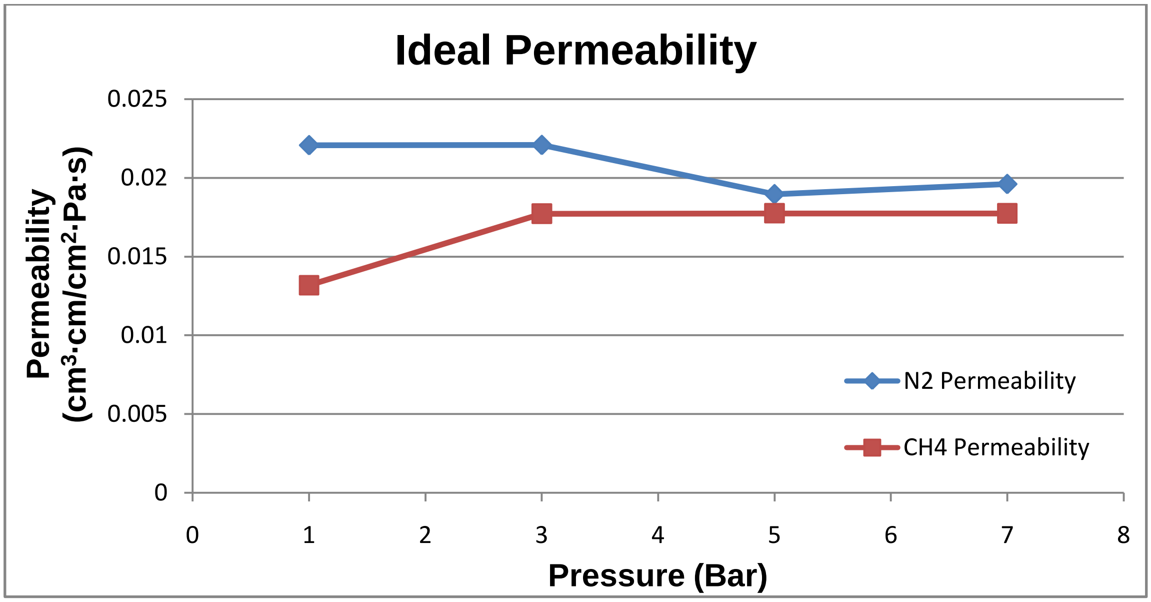


Figure 4. 3: Ideal permeability of nitrogen and methane at various pressures

Figure 4.3 shows the ideal permeability of nitrogen and methane at feed pressure 1 bar, 3 bar, 5 bar, and 7 bar. The trend indicates that the ideal permeability of nitrogen slightly decreases with pressure while the methane permeability increases with pressure. Although methane is a larger molecule thane nitrogen, this could be happened because methane has higher solubility or slightly more condensable than nitrogen at higher pressure. As if more methane is 'absorbed' into the membrane, the more it permeates through membrane.

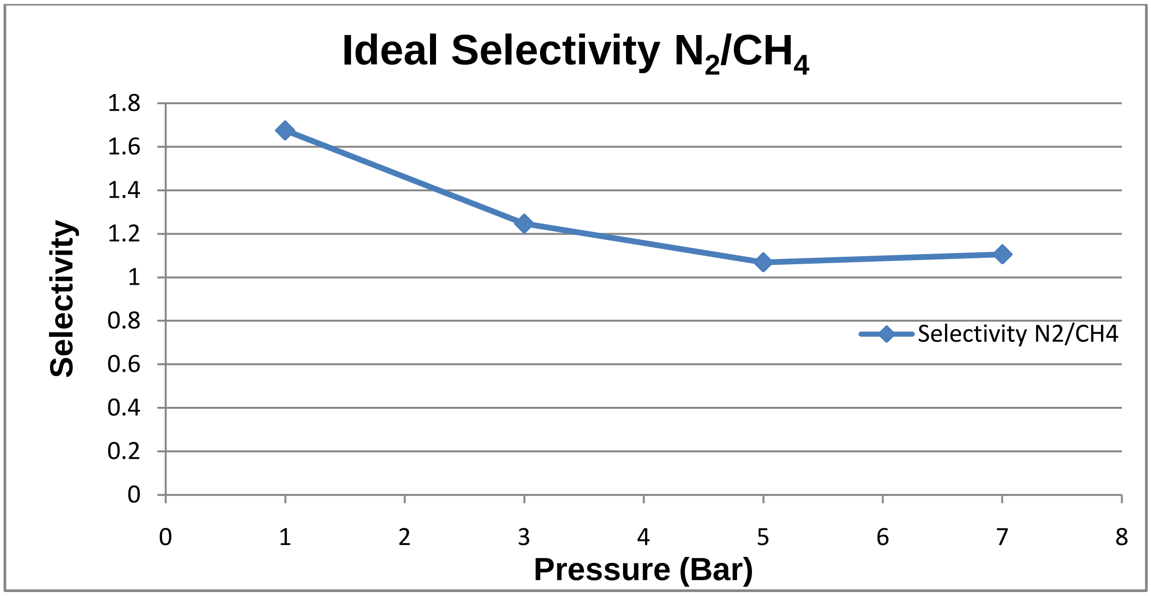


Figure 4. 4: Selectivity of nitrogen over methane at various pressures

Figure 4.4 shows the plot of ideal selectivity of nitrogen over methane at selected pressure. In addition to the permeability, the selectivity is also a function of pressure as the selectivity becomes lower when pressure is increased and it becomes quite stable or constant when operated at 5 to 7 bar pressure. The selectivity of nitrogen decreases simply because higher pressure leads to higher permeability of methane and the other way around for nitrogen. As we are already concerned, selectivity is the ratio of nitrogen permeability over methane permeability.

4.2 SEPARATION OF NITROGEN-METHANE BINARY MIXTURE

The separation efficiency experiment of binary mixture of nitrogen and methane was conducted in the gas membrane pilot plant using the tubular membrane module for 30 minutes. The experiment was divided into two parts which are 20% and 30% concentration of nitrogen in the feed stream. The first experiment is 20% nitrogen feed concentration and the system was set to operate at 1 bar and 25°C. The experiment was conducted with a total flow rate of 2000 mL/min at nitrogen and methane feed concentration of 20% and 80% respectively. The gases are introduced from the gas cylinder through the Mass Flow Controller (MFC) to static inline mixer and then went through feed gas analyzer and Mass Flow Meter (MFM). The feed gas then entered the heater (not activated since the experiment was at room temperature) before entering the tubular module. Separation occurred in the membrane to allow formation of two outlet streams of permeate and retentate. The concentration and flow rate of both streams are measured by the permeate/retentate analyzer and MFM respectively. The outlet streams are cooled in the cooler before vented out to the surrounding. The process was then repeated with 30% nitrogen feed concentration. The results are shown in the following graph plots.

4.2.1 Feed: 20% N_2 and 80% CH_4

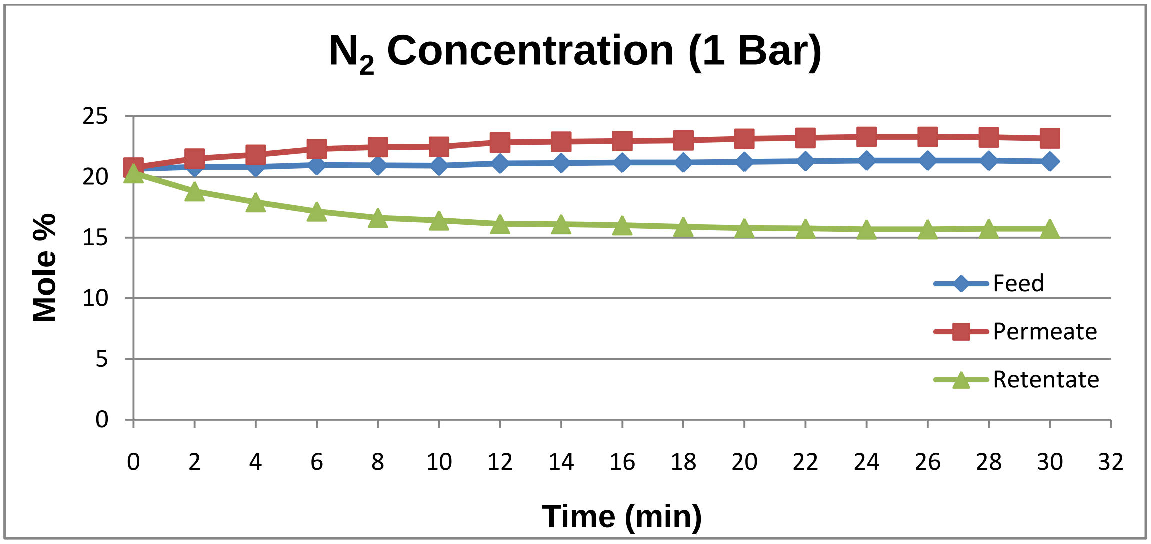


Figure 4. 5: Nitrogen concentration in feed, permeate, and retentate at 1 bar

Figure 4.5 clearly shows that throughout the 30 minutes period, some amount of nitrogen in feed (20 mole% nitrogen) permeate through the tubular membrane and collected in the permeate stream. It indicates that about 23 mole% of nitrogen is collected in the permeate stream while the retentate stream has much lesser nitrogen than the feed. The difference of nitrogen content in feed and retentate is about 5 mole%. This findings show that separation of nitrogen from natural gas (in this case methane) is possible. However, further improvement need to be done on membrane and the process itself to enhance the separation efficiency.

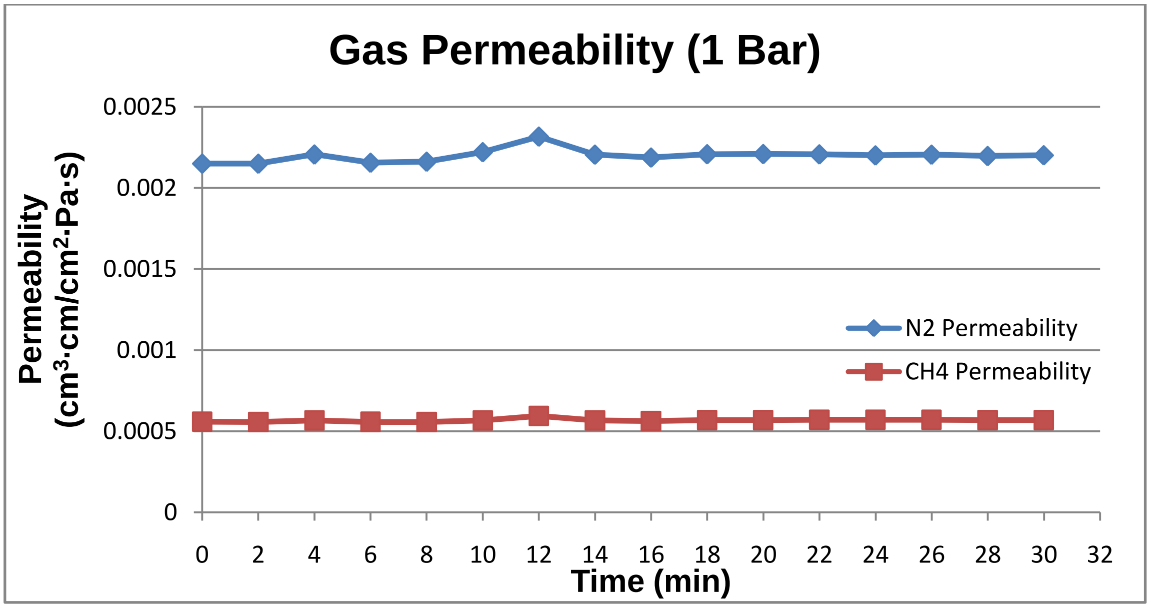


Figure 4. 6: Nitrogen and methane permeability in binary mixture at 1 bar

Based on the graph in Figure 4.6, nitrogen has higher permeability than methane. This outcome is exactly the same as the ideal permeability test for the individual gas. At 1 bar, nitrogen has a permeability of 0.023 barrer ($cm^3 \cdot cm/cm^2 \cdot Pa \cdot s$) while methane is 0.00058 barrer. However the permeability of both gases is lower than their ideal permeability which might be caused by the counter-reaction of each molecule of nitrogen and methane gases in the binary mixture. The experiment on the effect of pressure to the separation efficiency is further conducted at 3 bar, 5 bar, and 7 bar. The results are shown in the appendix.

Table 4. 1: N_2 Concentration (mole %) at Various Pressure

Mole %	1 bar		3 bar		5 bar		7 bar	
	Initial	**Final**	**Initial**	**Final**	**Initial**	**Final**	**Initial**	**Final**
Feed	20.65	21.27	20.55	21.09	20.71	21.15	21.14	22.47
Permeate	20.77	23.17	21.64	26.67	20.96	26.59	21.99	27.18
Retentate	20.31	15.74	19.49	15.77	20.47	15.93	20.35	16.76

The mole concentration of nitrogen in feed, permeate, and retentate at 1 bar, 3 bar, 5 bar, and 7 bar is tabulated in Table 4.1. Basically, we can say that nitrogen permeates through membrane at various pressures. However, increasing the pressure is making more nitrogen to be collected in the retentate stream. This is due to the slight increase of methane permeability compared to the slight decrease of nitrogen permeability.

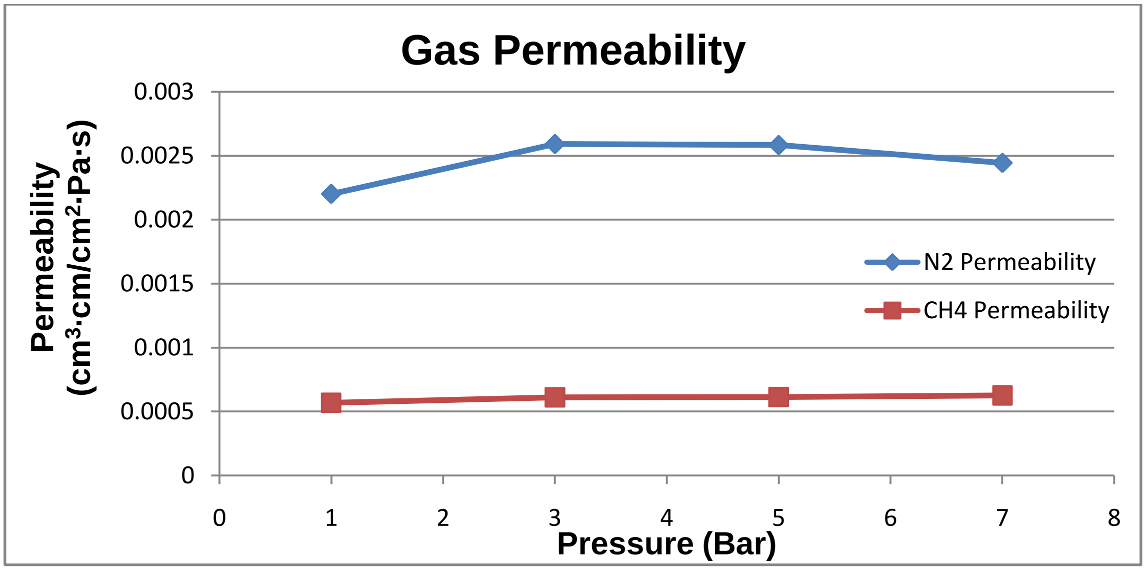


Figure 4. 7: Nitrogen and methane permeability in binary mixture at various pressures

The plots in Figure 4.7 are the permeability of both nitrogen and methane at 1 bar, 3 bar, 5 bar, and 7 bar. At various pressures, permeability of nitrogen is higher than methane for this binary mixture. Increasing pressure from 1 bar to 7 bar gives slight increment of permeability for both gases. Nitrogen permeability increases from 1 bar to 3 bar but slightly reduces from 3 bar to 7 bar while on the other hand, methane permeability slightly increases from 1bar to 3 bar and then becomes constant throughout 7 bar. The permeability of nitrogen is increasing at low feed pressure as the pressure difference is small between the feed and the permeate streams. As we know, permeability is inversely proportional to pressure difference. However, towards the higher feed pressure, the permeability is decreasing as the pressure difference between feed and permeate streams become higher. These findings are correlated with equation 2.6 derived in the Literature Review and Theory Section. On the other hand, the permeability of methane is slightly increasing and becomes constant throughout higher pressure because of its high solubility and slightly more condensable than nitrogen.

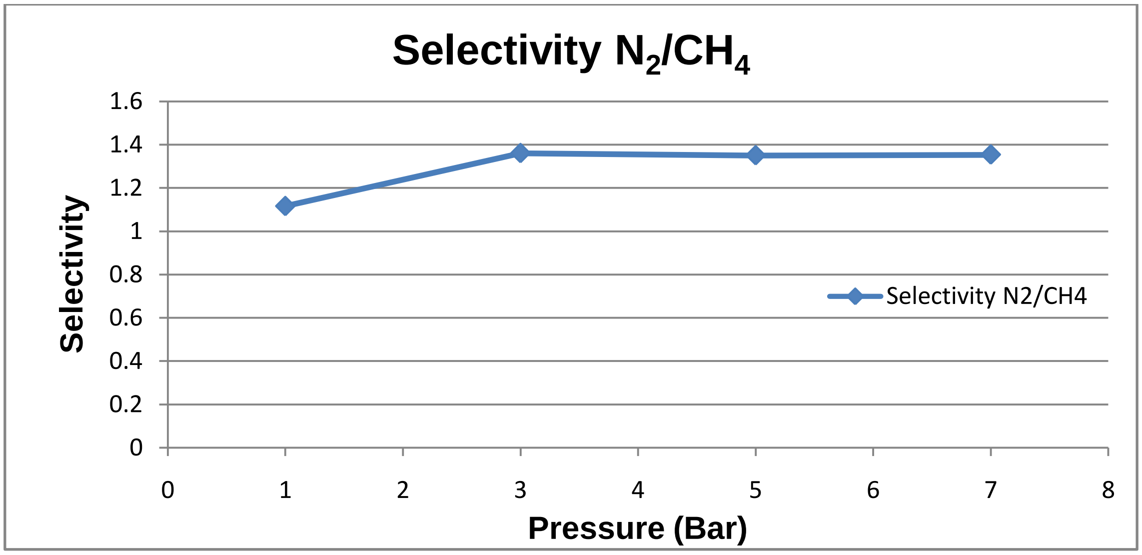


Figure 4. 8: Nitrogen selectivity in binary mixture at various pressures

One important feature of the plot in figure 4.8 is that the selectivity is highest at 5 bar pressure which is 1.39 after a quite increment in the selectivity from 1 bar pressure. In addition, the permeability shown in figure 4.7 also indicates high result at 5 bar. After highest selectivity at 5 bar, the selectivity decreases when operated at 7 bar. Generally, it shows that an increase in feed pressure at any given temperature results in a decrease in the nitrogen/methane selectivity. The most likely reasons for low selectivity are the Knudsen diffusion effects on the membrane which is a factor of molecular weight of gas species. Knudsen flow is characterized by the mean free path (λ) of the molecules, which is larger than the pore size and hence collisions between the molecules and the pore walls are more frequent than intermolecular collisions. Since the driving force for transport is the partial pressure of the gas species, Knudsen transport can occur either by concentration or by pressure gradients. The relative permeation rate of each component is inversely proportional to the square root of its molecular weight. Thus, the selectivity of nitrogen over methane is low because methane permeability increases during Knudsen transport effect as the molecular weight of methane is lesser than nitrogen gas. In addition, the low selectivity might be resulted from the non-ideality effects in the gas phase at higher pressures and perhaps some concentration polarization effects. Thus, this behavior implies that the membrane separates gases less efficiently at higher pressures.

4.2.2 Feed: 30% N_2 and 70% CH_4

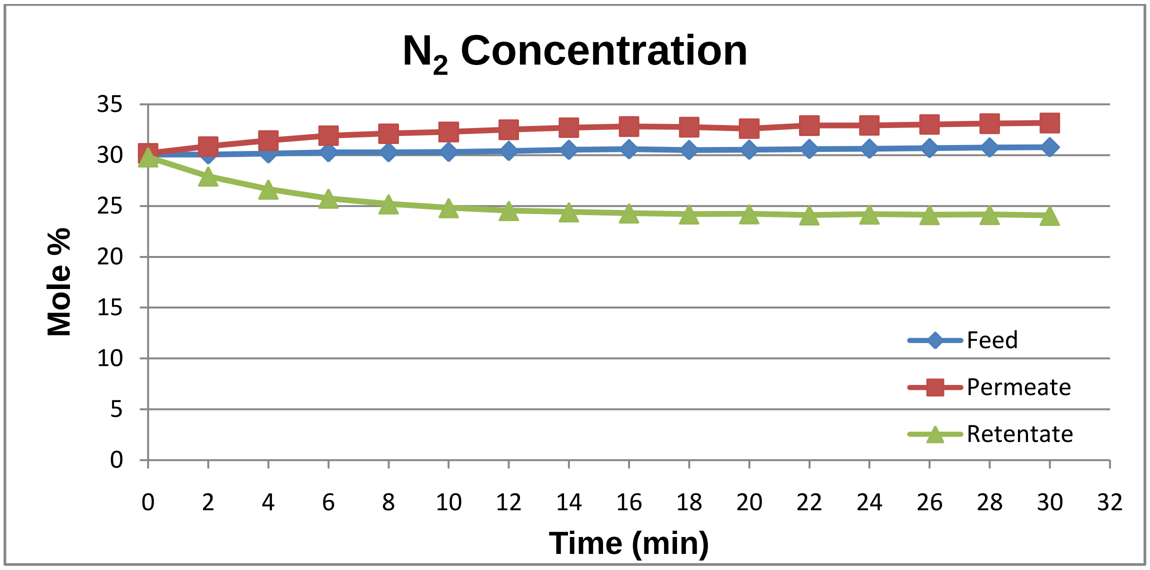


Figure 4. 9: Nitrogen concentration in feed, permeate, and retentate at 1 bar

Figure 4.9 displays the nitrogen concentration in permeate and retentate after being fed with 30% nitrogen in feed at 1 bar. The nitrogen content in permeate is 33 mole% while the retentate has 24 mole% nitrogen. It means that throughout the 30 minutes period, there are some nitrogen molecules permeates through the membrane and cause a reduction of nitrogen content in the retentate stream.

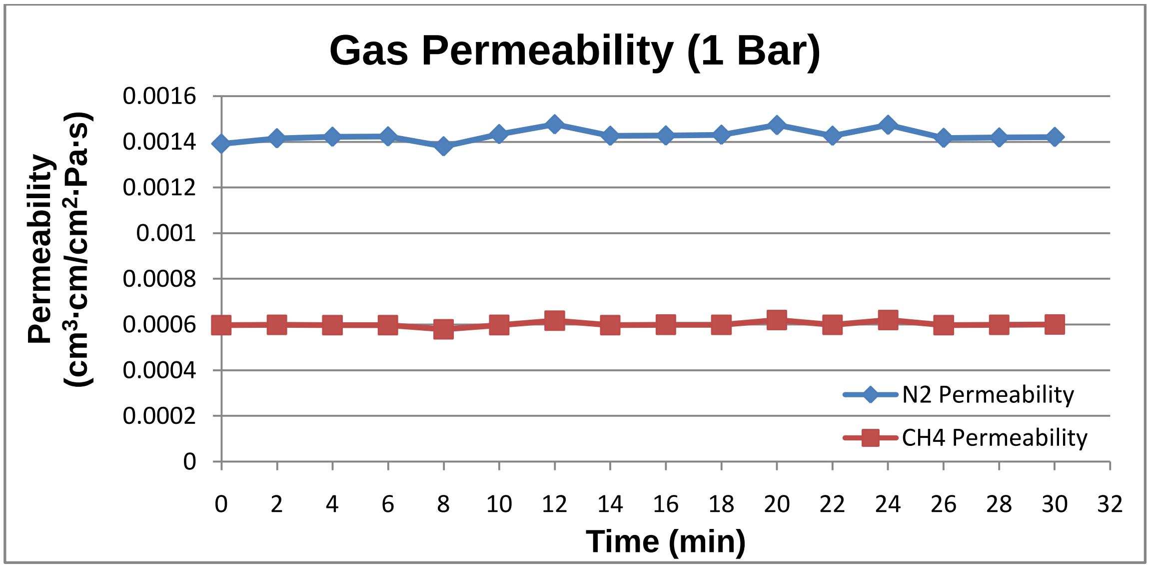


Figure 4. 10: Nitrogen and methane permeability in binary mixture at various pressures

It is clearly shown in the Figure 4.10 that nitrogen has higher permeability than methane which is 0.0014 barrer and 0.0006 barrer respectively. Thus, nitrogen has higher permeability than methane at both 20% and 30% nitrogen concentration in feed stream. However, if we recall the previous Figure 4.6, nitrogen has higher permeability at 20% nitrogen feed concentration compared to 30% nitrogen feed concentration. At 20%, nitrogen permeability is 0.0023 barrer while at 30%, nitrogen permeability is 0.0014 barrer. This might be happened because the diffusion of nitrogen through membrane is faster at lower concentration, as a result of less packed path through the membrane.

Table 4. 2: N_2 Concentration (mole %) at Various Pressure

Mole %	**1 bar**		**3 bar**		**5 bar**		**7 bar**	
	Initial	**Final**	**Initial**	**Final**	**Initial**	**Final**	**Initial**	**Final**
Feed	30.07	30.75	30.03	30.72	30.12	30.64	30.28	30.73
Permeate	30.17	33.14	31.15	36.49	32.93	36.36	33.30	36.85
Retentate	29.79	24.04	29.09	24.75	27.55	24.76	27.64	24.98

The nitrogen concentration in those three streams tabulated in Table 4.2 shows the same pattern as in 20% nitrogen feed concentration. Basically, nitrogen permeates through membrane at all pressures but the nitrogen content in retentate increases when pressure is added up from 1 bar to 7 bar. This is the result of reduction in nitrogen permeability at higher pressure.

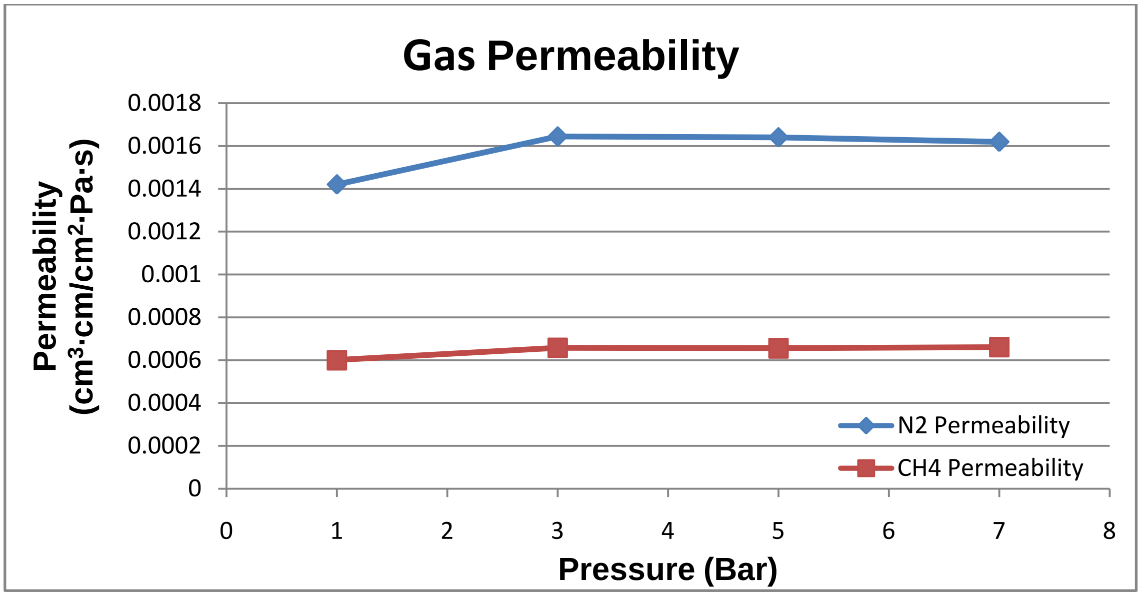


Figure 4. 11: Nitrogen and methane permeability in binary mixture at various pressures

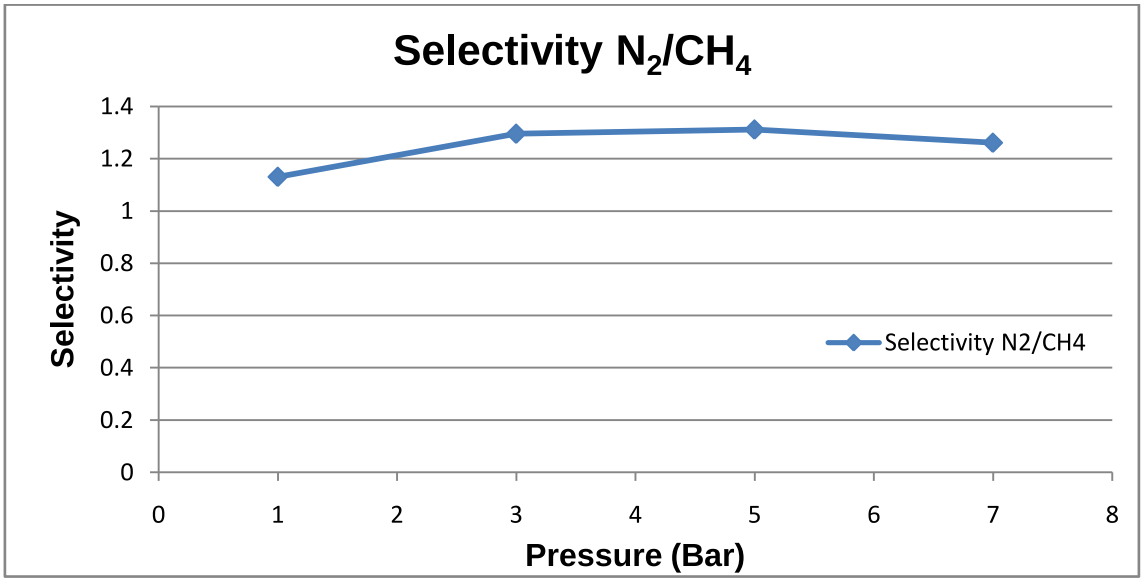


Figure 4. 12: Nitrogen selectivity in binary mixture at various pressures

Figure 4.11 shows that nitrogen has higher permeability than methane at all pressure. This result has the same pattern as the previous 20% nitrogen feed concentration where for nitrogen, it increases from 1 bar to 3 bar and then slightly decreases towards 7 bar; while for methane, slightly increases from 1 bar to 3 bar and constant towards 7 bar. However, in overall, nitrogen permeability in 20% nitrogen feed concentration is higher than the 30% nitrogen feed concentration. Generally, it shows that an increase in feed pressure results in a decrease in the permeability due to bigger pressure difference in feed and permeate stream.

In addition, the selectivity shown in Figure 4.12 for 30% nitrogen feed concentration also has the same trend as in the 20% nitrogen feed concentration. The important point is that it has the highest selectivity of 1.3 at 5 bar which however, is lower than the 20% nitrogen feed concentration, that is 1.39.

The selectivity range of 1 – 2 is considered low to be implemented in industry. The required minimum selectivity for efficient separation is between 3 to 5, and the higher the selectivity, the better the nitrogen-methane separation. For better results which in this case efficient separation and high capacity, the separation process through membrane can be done in a pattern of series and two-stage flow.

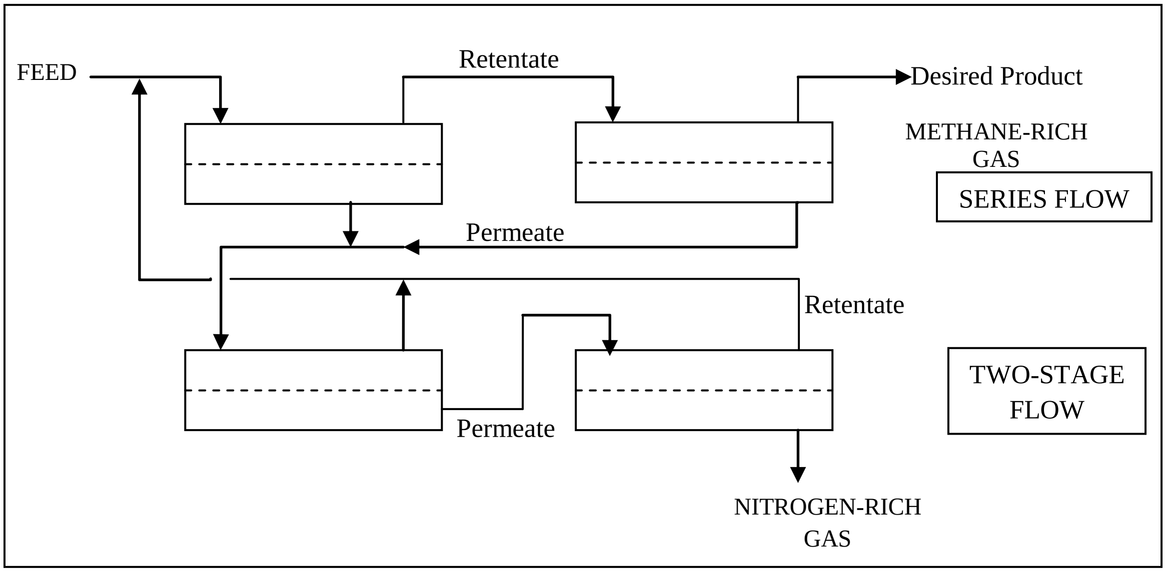


Figure 4. 13: Recommended flow pattern

CHAPTER 5

CONCLUSION & RECOMMENDATION

The separation of nitrogen from methane is very challenging in term of process and technology. Thorough research has been done in this particular area especially on process of nitrogen separation from natural gas as well as inorganic membrane technology. The experimental works have been conducted to complete the research project. The project has been very successful as the progress flow smoothly along with the key milestones. The experiment on factors affecting membrane separation efficiency has been completed which included the effects of pressure and feed concentration. The research project meets the objectives which are to study the separation process of nitrogen from natural gas (in this case methane) using inorganic membrane, to perform experiment of gas membrane separation based on nitrogen and methane gas permeability and selectivity, and to investigate factors influencing the separation of nitrogen from methane.

Based on the research and experiment, it shows that membrane process has a promising technology to be used for nitrogen-methane separation. The experimental works demonstrated that separation of nitrogen from natural gas using inorganic membrane is possible as the membrane is able to remove about 5 mole% of nitrogen from the feed with highest selectivity of 1.39. However, this selectivity is considered low and further research and experiment need to be done to enhance the separation efficiency and to get desired minimum selectivity of 3 to 5. The low selectivity of nitrogen over methane might be resulted from Knudsen diffusion effect that occurred either by concentration or by pressure gradients.

The experiment also has proven that efficient separation occurs at low nitrogen feed concentration and the membrane separates gases less efficiently at higher pressure. All in all, future works can be planned to enhance the separation efficiency and thus producing large scale membrane for nitrogen removal in oil and gas industry.

Here are few recommendations for future works in this project:

1. In a technical aspect, for smooth experimental works, it is suggested to implement manual operation of the Membrane Pilot Plant as currently; the operation is solely depended on the computer and software. Lesson was learnt from past experience that failure of those devices has hindered and slowing down the progress of the experimental works as well as the project generally.

2. Consideration for methane-selective membrane implementation to determine the effect of separation efficiency especially on permeability and selectivity. Conduct experimental work for nitrogen separation from natural gas using inorganic methane-selective membrane at low temperature. The selectivity might increases due to a potential decrease in the nitrogen permeation flux compared to the methane flux.

REFERENCES

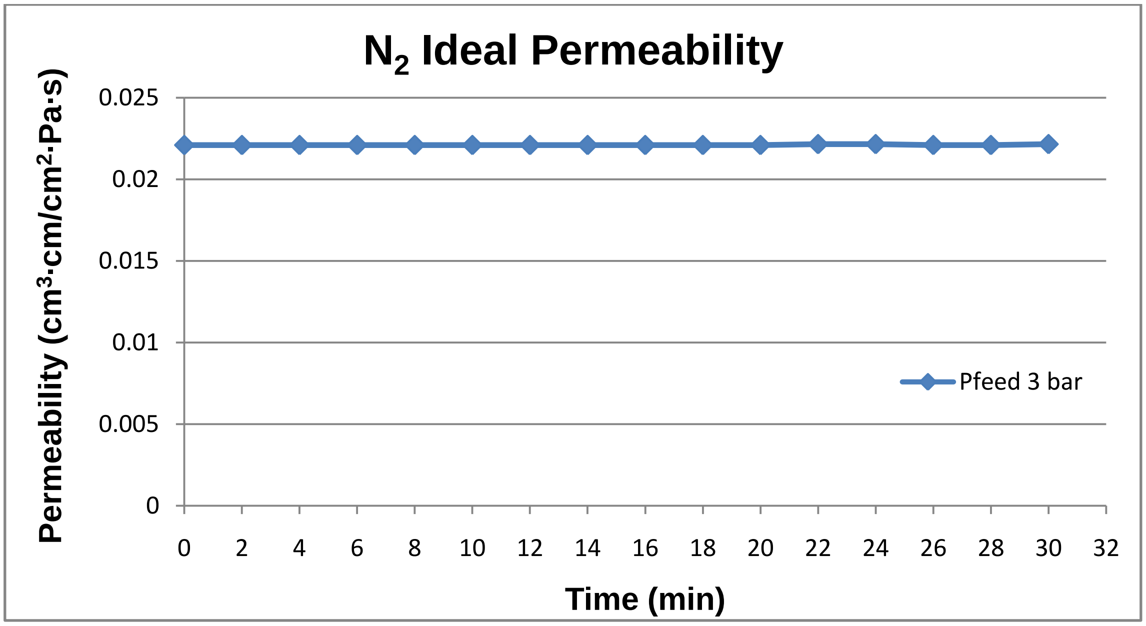
1. A.H Younger, (2004). *Natural Gas Processing Principles and Technology*, University of Calgary, Canada.
2. D. Ullman, (2003). *Ullman's Encyclopedia of Industrial Chemistry*. 6th Edition, Wiley-VCH, Weinheim, 243-322.
3. NaturalGas.org, (2004). *Natural Gas*, http://www.naturalgas.org/overview/background.asp, 5 February 2010.
4. A. Rojey, (1997). *Natural Gas Production Processing Transport*, Edition Technip, Paris.
5. V. Chandra, (2006). *What is Natural Gas?*, http://www.natgas.info/html/natgasunitsconversion.html, 5 February 2010.
6. J. McKetta, (1978), *Encyclopedia of Chemical Processing and Design*. 1st Edition, Marcell Dekker, Florida, 301-366.
7. D. Shekhawat, R. Luebke, and W. Pennline, (2003). *A Review of Carbon Dioxide Selective Membranes*. National Energy Technology Laboratory, United States Department of Energy.
8. B. Alvarado, F. Asaro, L. Bomben, S. Damle, S. Bhown, (1998). *Nitrogen removal from low quality natural gas*. U.S. Department of Energy, U.S.A.
9. A. Lokhandwala, M. Ringer, H. Wijmans, W. Baker, (1996), *Nitrogen removal from natural gas using membranes*. Membrane Technology and Research Inc., U.S.A.
10. J.C. Tannehill, J.F. Strickland, and H. Meyer, (1999). *High Nitrogen Gas-Snapshot of the Present Requirement for the Future*. Presented at GPA Meeting, Nashville, TN
11. Y.R. Mehra, G.C. Wood and M.M. Ross, (1993). *Noncryogenic Nitrogen-Rejection Process Gets Hugoton Field Test*, Oil and Gas Journal, 91, 21.
12. M.J Healy, A.J Finn, and L. Halford, (1999). *UK Nitrogen-Removal Plant Start UP*, Oil and Gas Journal, 97, 5.
13. Membrane Technology & Research (2007). *Nitrogen Removal from Natural Gas: NITROSEP™*, http://www.mtrinc.com/nitrogen_removal.html, 26 May 2010.
14. Chemical & Engineering News, (2005). *Membranes for Gas Separation*, http://pubs.acs.org/cen/coverstory/83/8340membranes.html, 25 May 2010.

15. P. Tremblay, M. Savard, J. Vermette, R. Paquin, (2006). *Gas permeability, diffusivity and solubility of nitrogen, helium, methane, carbon dioxide and formaldehyde in dense polymeric membranes using a new on-line permeation apparatus,* Journal of Membrane Science, 282, 245–256.
16. M. Mulder, (1996). *Basic Principle of Membrane Technology,* 2nd Edition, Academic Publisher, W.B.
17. K. Pabby, S. Rizvi, A.M. Sastre, (2009). *Handbook of Membrane Separations*: *Chemical, Pharmaceuticals, Food, & Biotechnological Applications,* CRC Press, Florida.
18. Inorganic Membrane Technology Laboratory, (2002). *Membranes and Their Uses,* http://www.inorganicmembranes.info/uses.html, 23 April 2010.
19. Universiti Teknologi PETRONAS, (2005). *Gas Membrane Separation Pilot Plant – Instructor Manual*, Perak.
20. The Physical and Theoretical Chemistry laboratory, Oxford University (2010). *Chemical and Other Safety Information,* http://msds.chem.ox.ac.uk, 12 March 2010.
21. J. Koros, R. Mahajan, (2010). *Pushing the Limit on Possibilities for Large Scale Gas Separation: Which Strategies?*, Journal of Membrane Science, 175, 181-196.
22. A. Rojey, C. Jaffret, S. Cornot-Gandolphe, B. Durand, S. Jullian, M. Valais, (1997). *Natural Gas: Production, Processing, Transport*, Editions Technip, Paris.

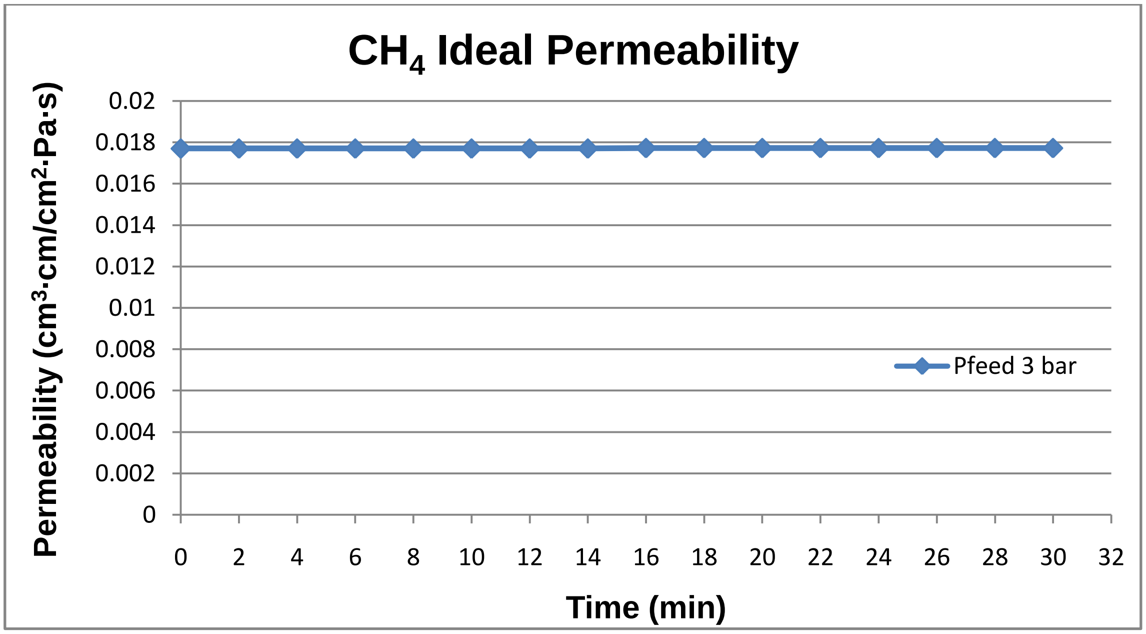
APPENDICES

Appendix A: Experimental Results

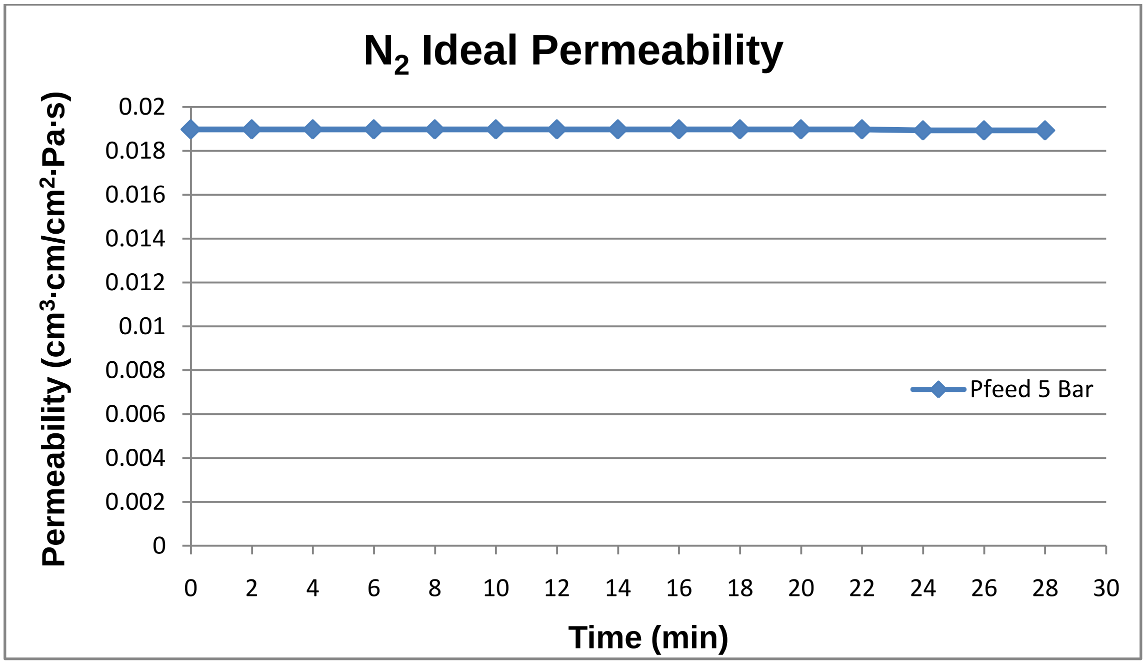
Ideal Permeability:



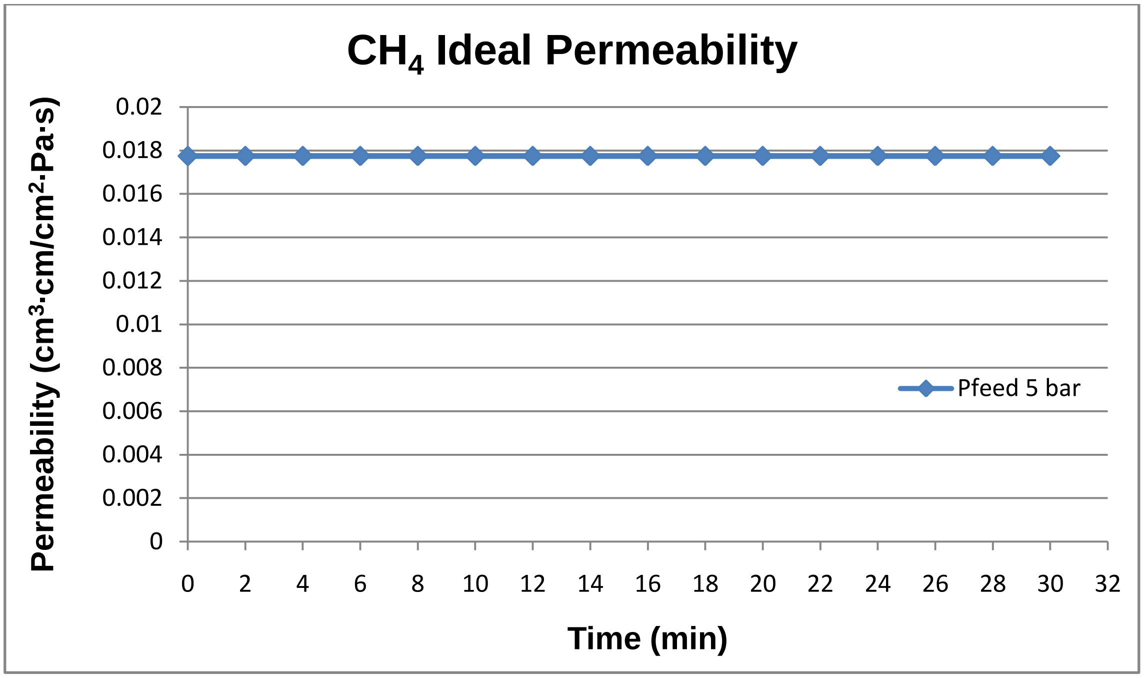
A-1: Nitrogen ideal permeability at feed pressure 3 Bar



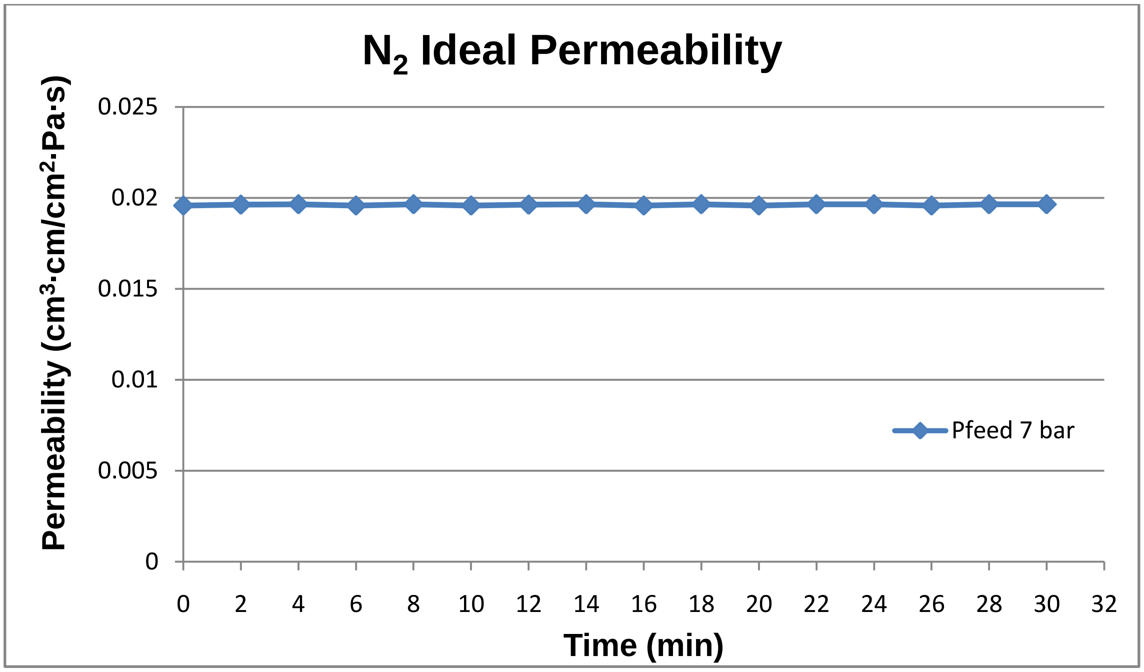
A-2: Methane ideal permeability at feed pressure 3 Bar



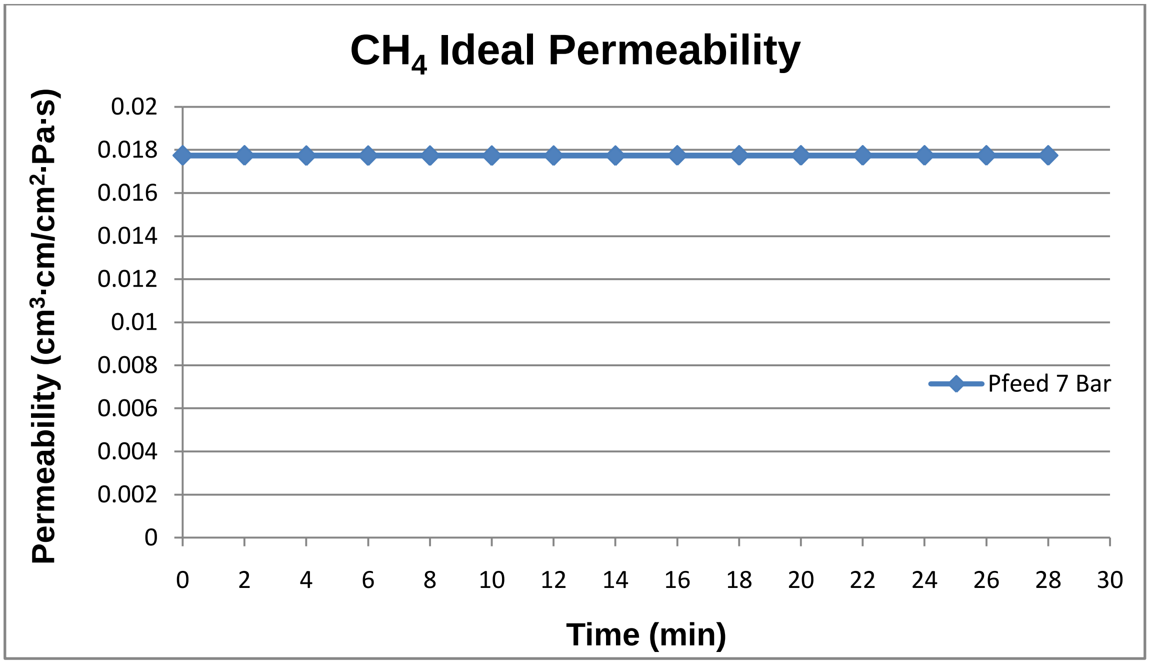
A-3: Nitrogen ideal permeability at feed pressure 5 Bar



A-4: Methane ideal permeability at feed pressure 5 Bar



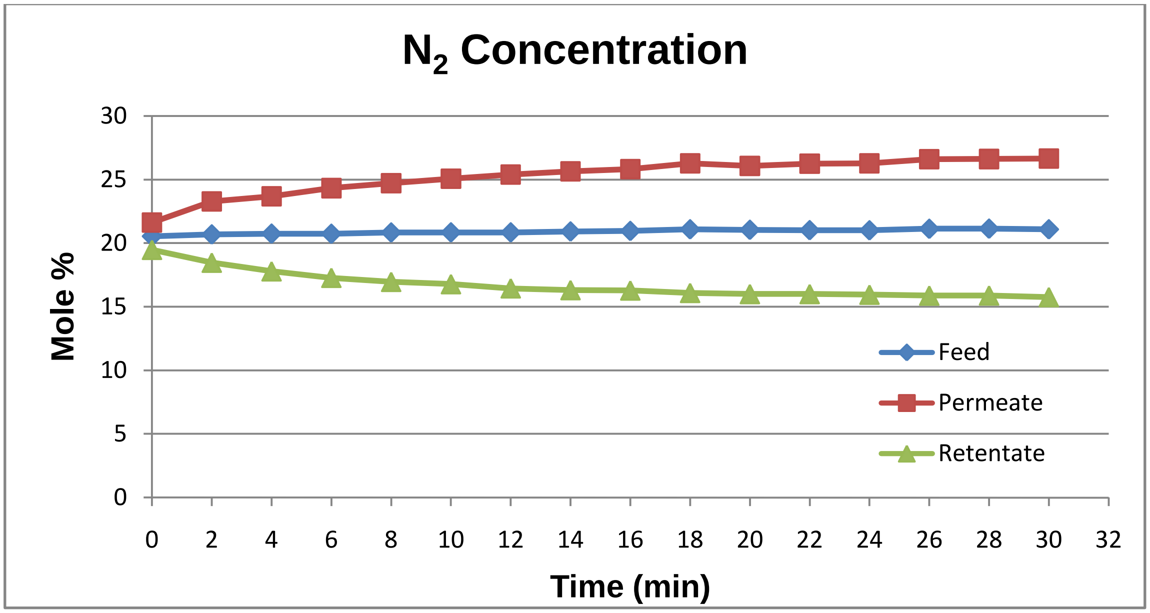
A-5: Nitrogen ideal permeability at feed pressure 7 Bar



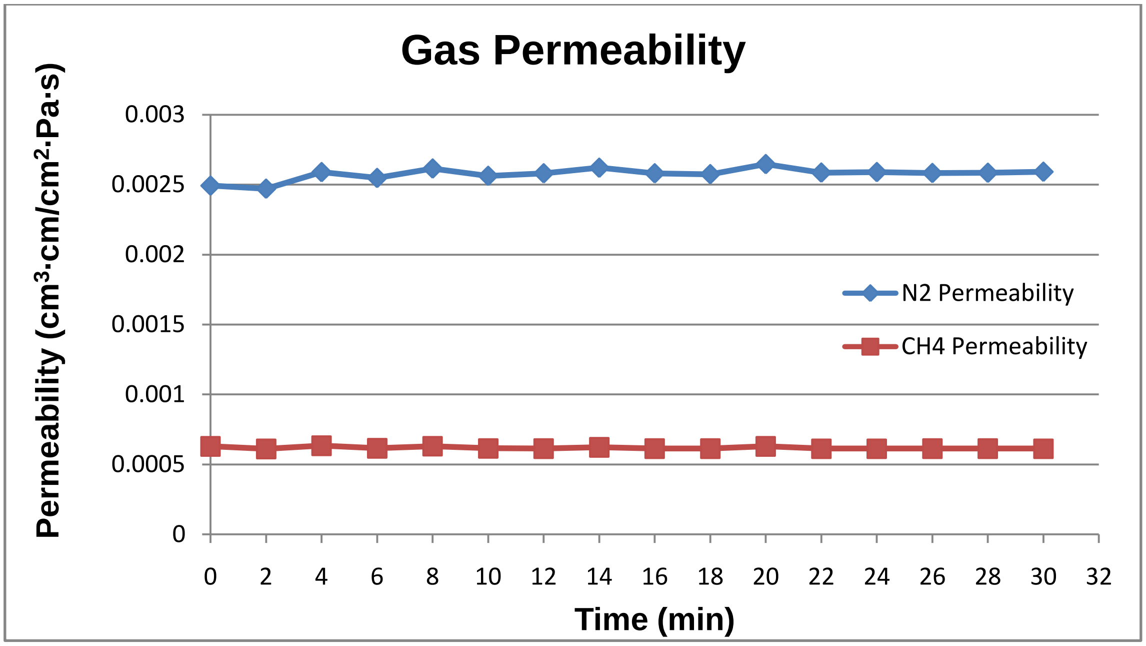
A-6: Methane ideal permeability at feed pressure 7 Bar

Binary Mixture [20% N_2 – 80% CH_4]:

Feed Pressure: 3 Bar

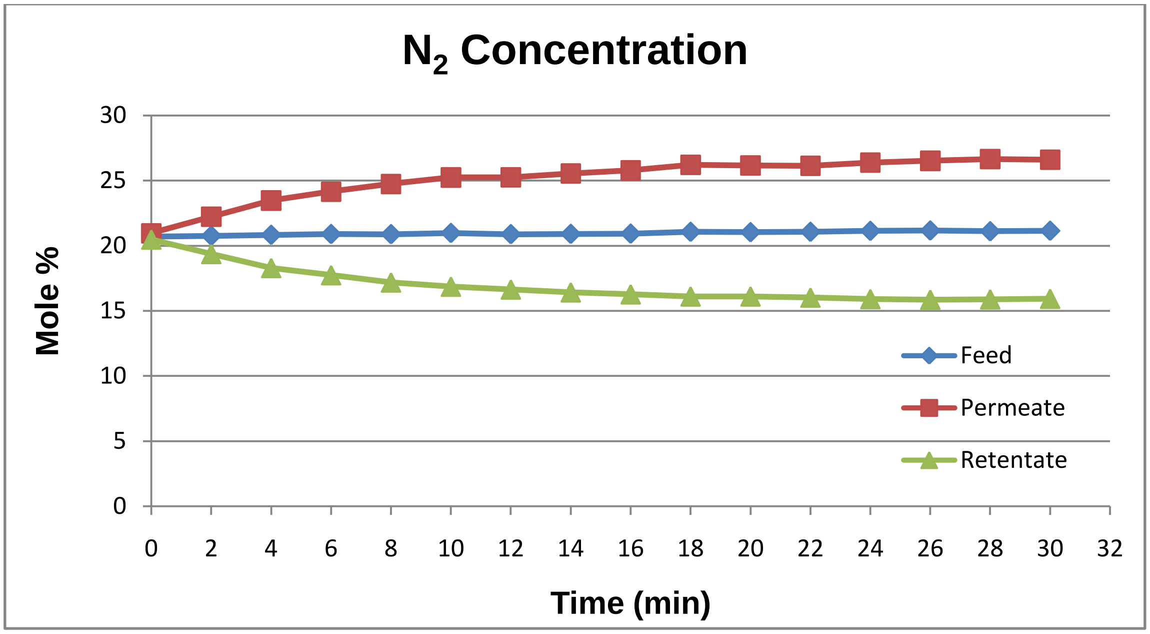


A-7: Nitrogen Concentration at 3 bar

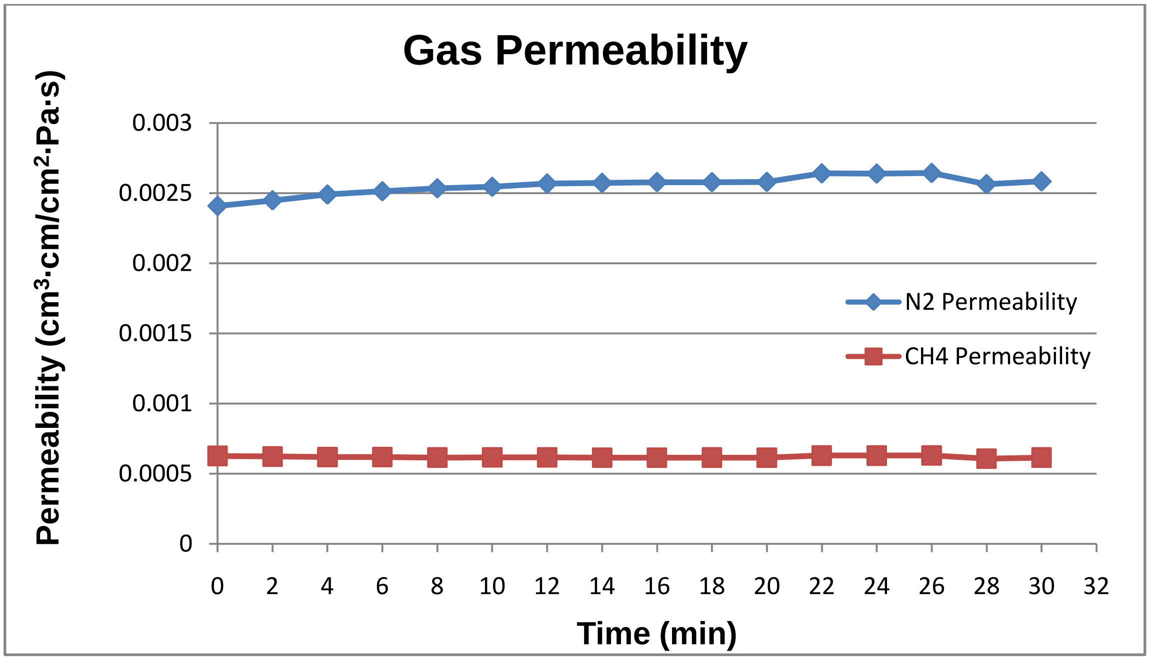


A-8: Gas Permeability at 3 bar

Feed Pressure: 5 Bar

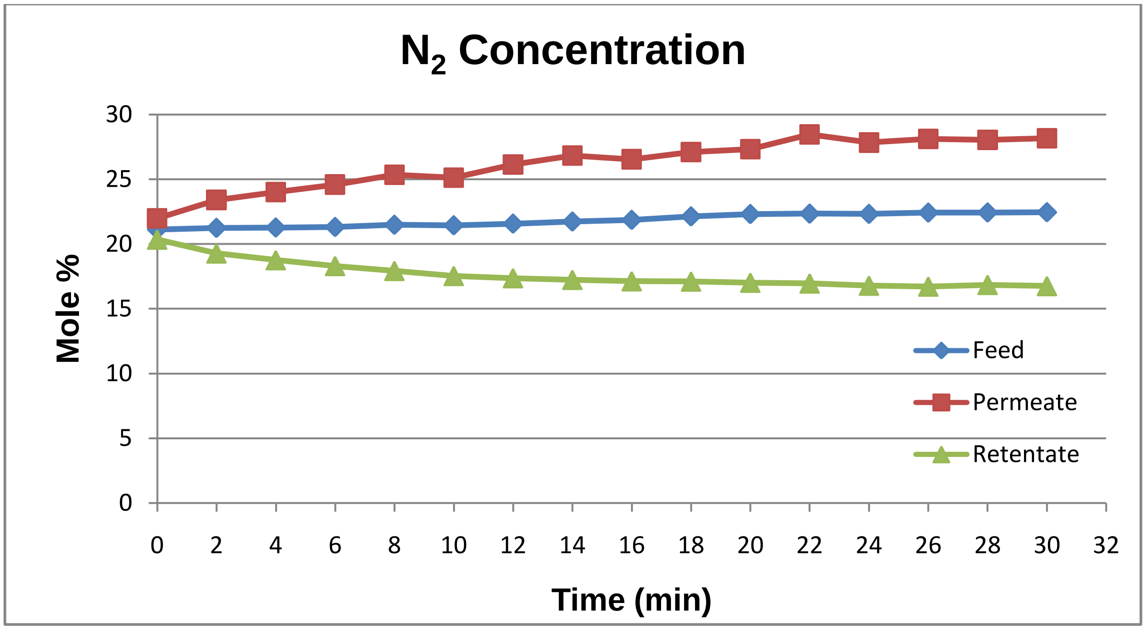


A-9: Nitrogen Concentration at 5 bar

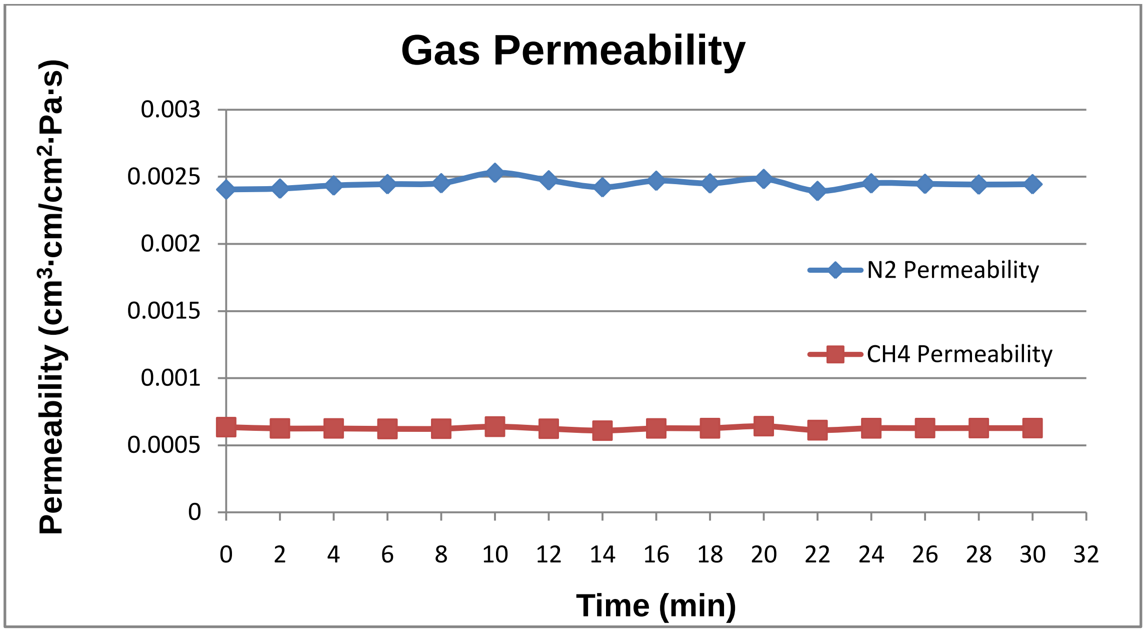


A-10: Gas Permeability at 5 bar

Feed Pressure: 7 Bar



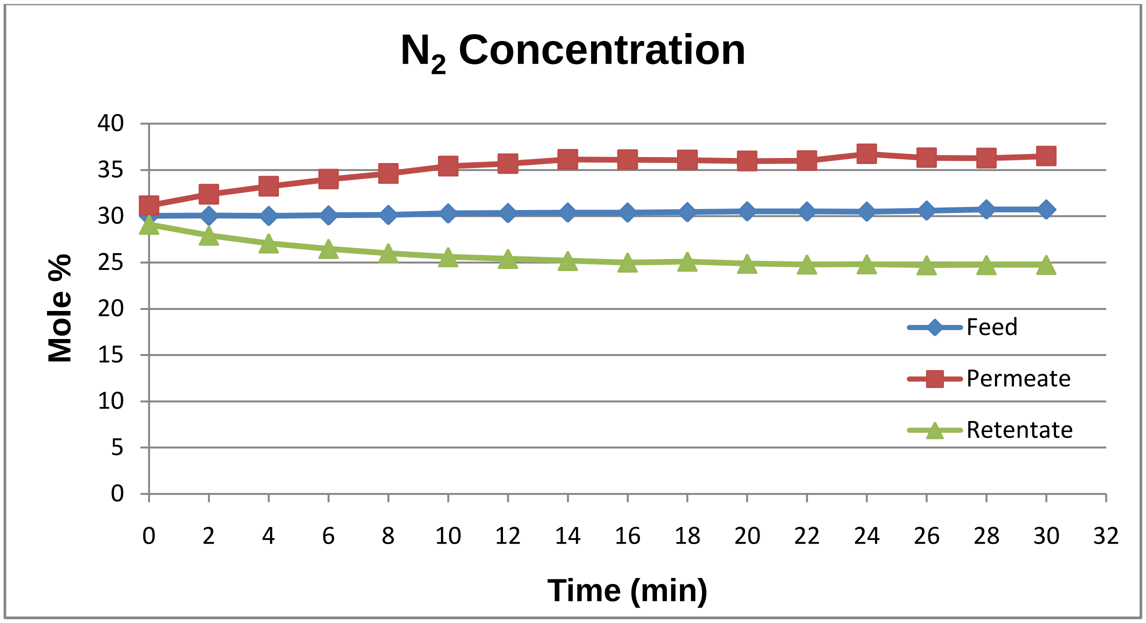
A-11: Nitrogen Concentration at 7 bar



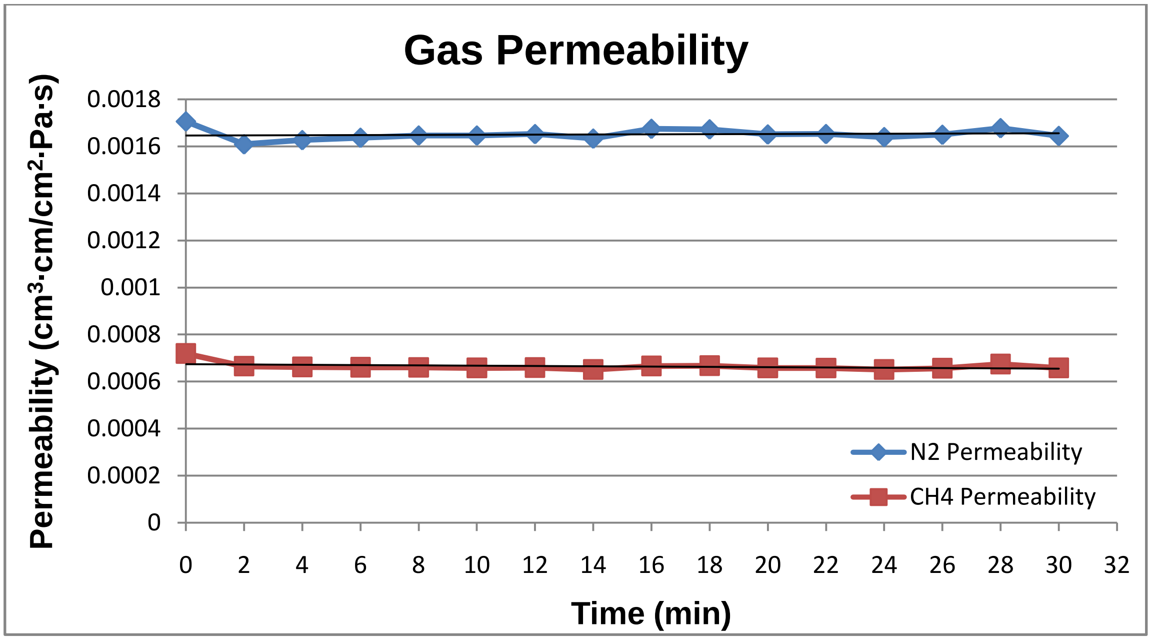
A-12: Gas Permeability at 7 bar

Binary Mixture [30% N_2 – 70% CH_4]:

Feed Pressure: 3 Bar

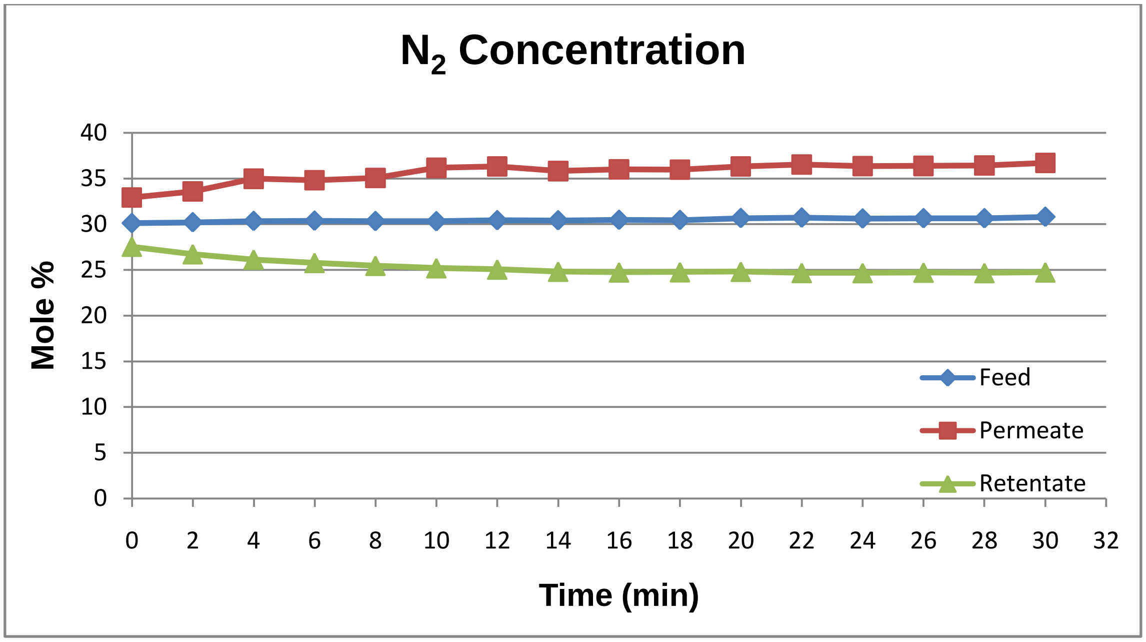


A-13: Nitrogen Concentration at 3 bar

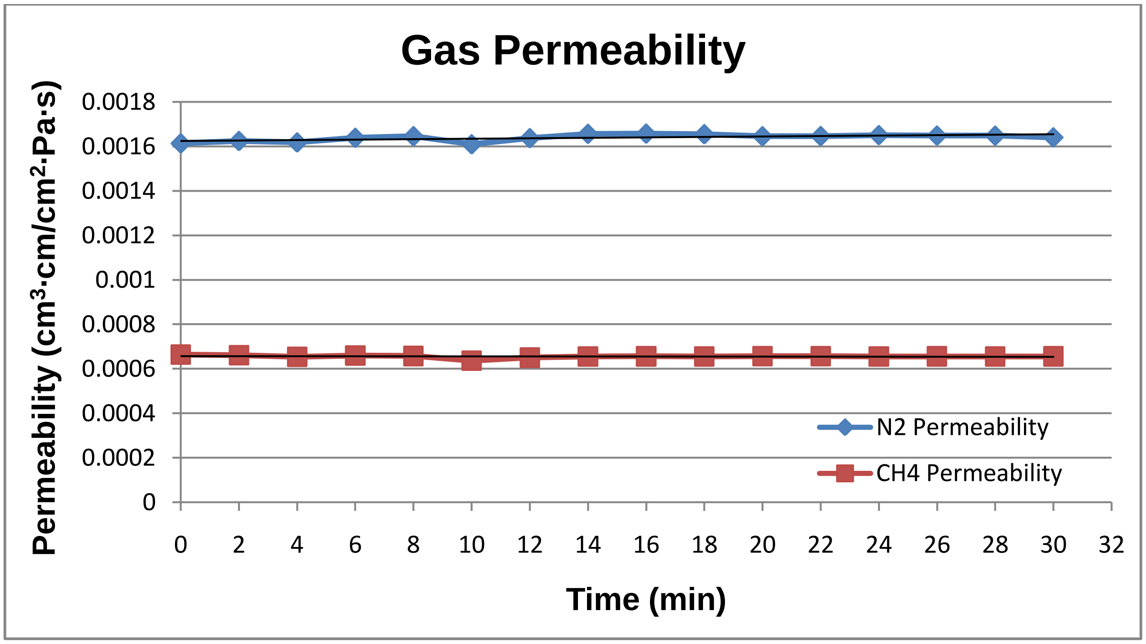


A-14: Gas Permeability at 3 bar

Feed Pressure: 5 Bar

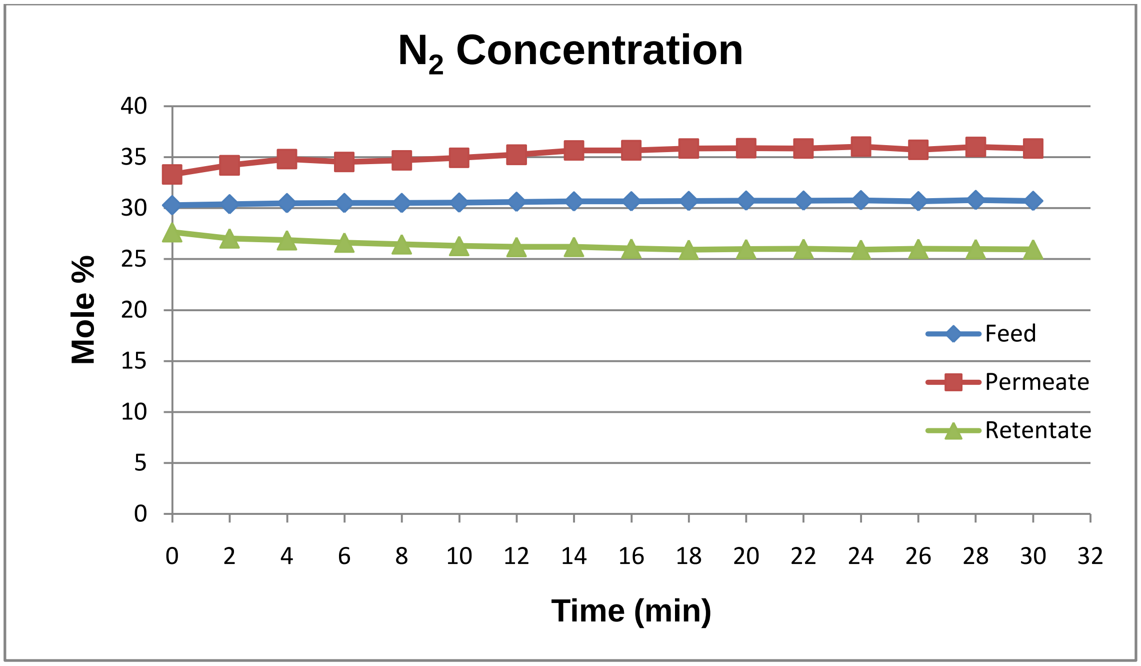


A-15: Nitrogen Concentration at 5 bar

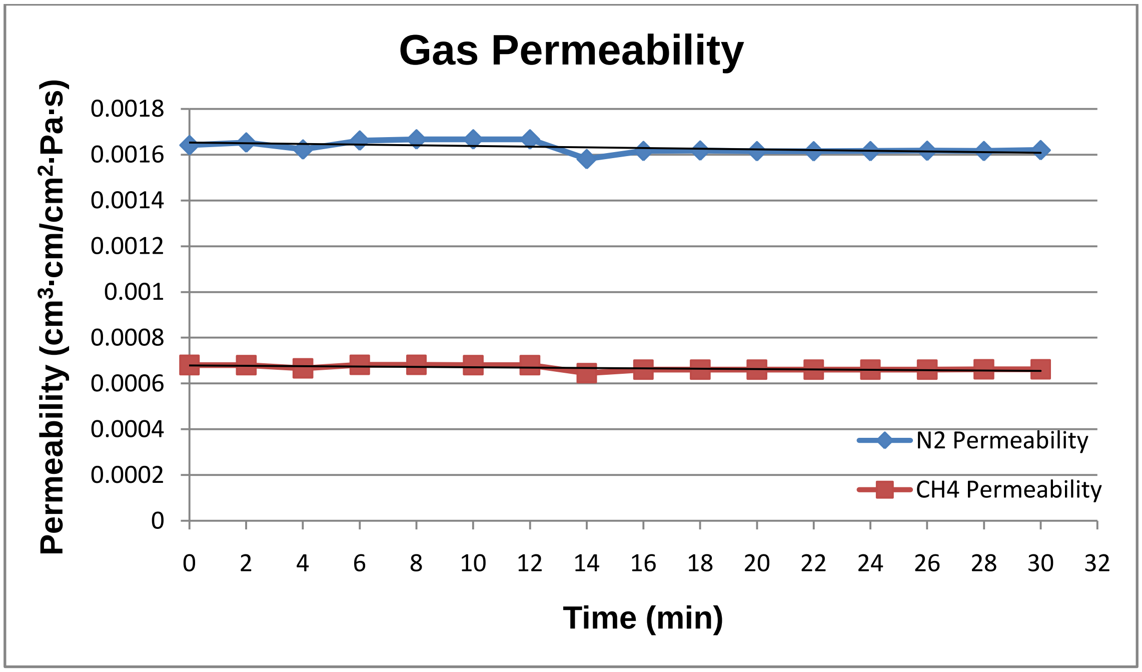


A-16: Gas Permeability at 5 bar

Feed Pressure: 7 Bar

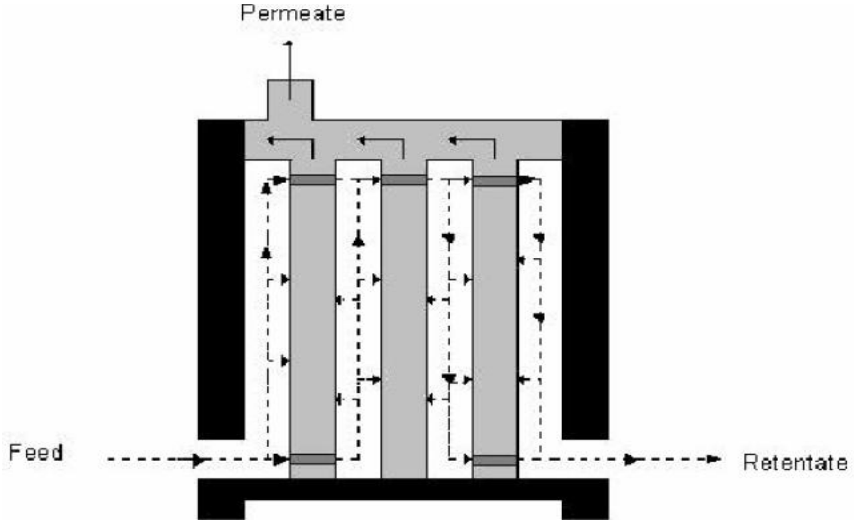


A-17: Nitrogen Concentration at 7 bar

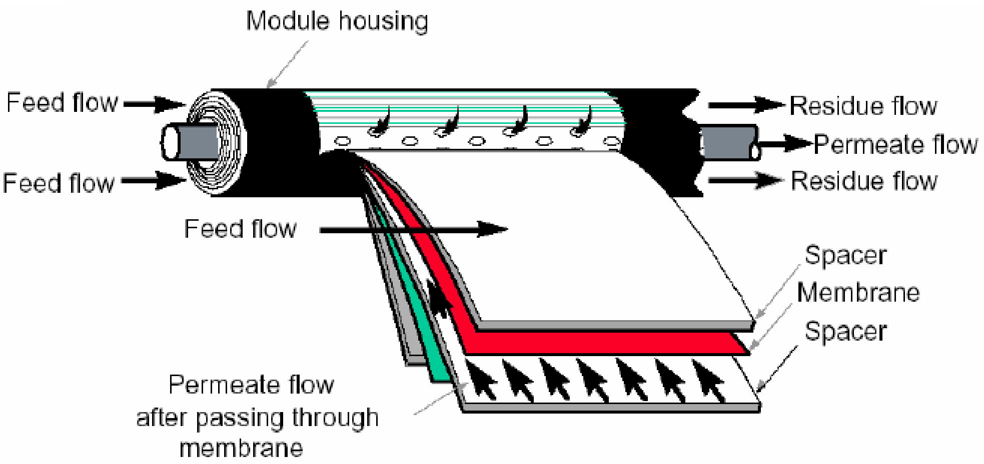


A-18: Gas Permeability at 7 bar

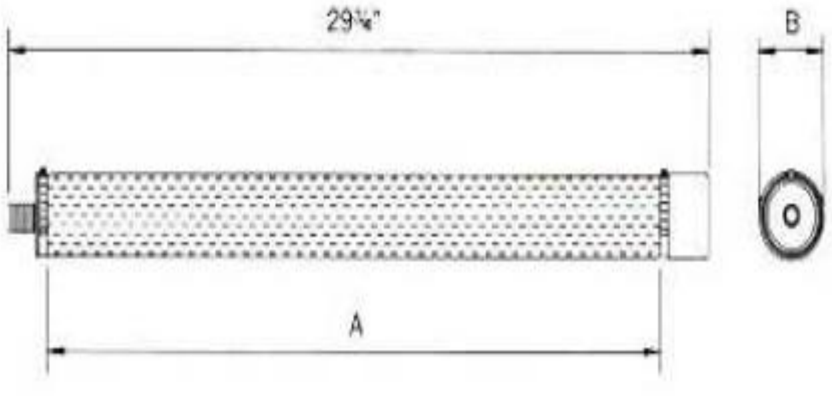
Appendix B: Membrane Modules Schematic



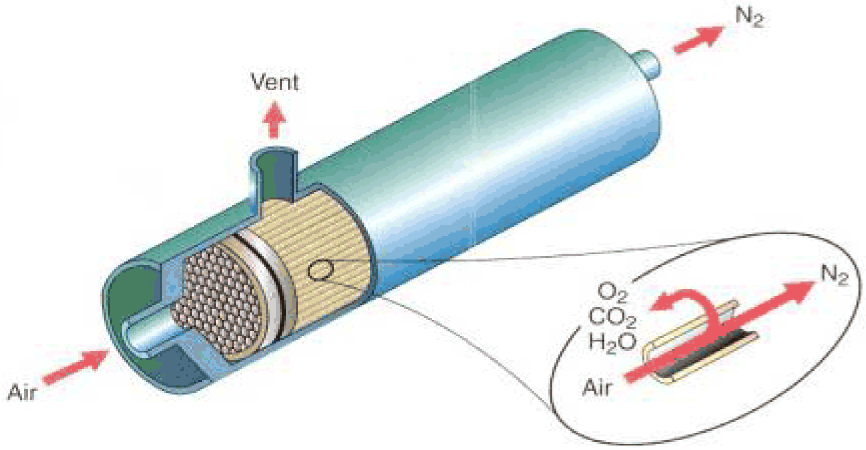
B-1: Plate and Frame



B-2: Spiral-wound



B-3: Tubular



B-4: Hollow-fiber

Appendix C: Material Safety Data Sheet [23]

The Physical and Theoretical Chemistry Laboratory, Oxford University.

C-1 : Chemical Safety Data: Methane

Common synonyms	marsh gas, fire damp
Formula	CH_4
Physical properties	Form: colourless, odourless gas Stability: Stable Melting point: -182 C Boiling point: -164 C Water solubility: slight Density 0.717 g/l at 20 C Explosion limits: 5 - 15%
Principal hazards	Methane is very flammable. Mixtures of methane with air are explosive within the range 5-15% by volume of methane. Methane can react violently or explosively with strong oxidizing agents, such as oxygen, halogens or interhalogen compounds. At high concentration methane acts as an asphyxiant.
Safe handling	Wear safety glasses. The primary danger is from fire and explosion, so ensure that you work in a well-ventilated area, preferably within a fume cupboard, and that there is no source of ignition present.
Emergency	Eye contact: Unlikely to occur. Skin contact: Unlikely to occur. If inhaled: Remove from the source of gas. If the amount inhaled is large or if breathing has ceased call for immediate medical help.
Disposal	Small amounts of methane can be allowed to disperse naturally. Be aware that any significant build-up of gas presents a danger or fire or explosion.
Protective equipment	Safety glasses.

C-2: Chemical Safety Data: Nitrogen

Common synonyms	None
Formula	N_2
Physical properties	Form: colourless gas Stability: Stable. Melting point: -210 C Boiling point: -195.9 C Water solubility: slight Liquid density: 0.808 g cm^{-3} Vapour density: 1.25 g/l
Principal hazards	Asphyxiant at high concentrations
Safe handling	High pressure gas cylinders contain a great deal of stored energy and can present a significant hazard. Ensure that those who fix and operate high pressure gas regulators are properly trained.
Emergency	Eye contact: - Skin contact: - If swallowed: -
Disposal	Excess nitrogen may be allowed to disperse in the atmosphere. If the volume of nitrogen is large, ensure that no one might be exposed to an atmosphere depleted in oxygen.
Protective equipment	-

Appendix D: Risk Assessment [22]

Sequence of basic job stage	Potential accidents or hazards	Recommended safe job procedures
Running the equipment	1.1 Machine tools / Hot surface area	1.1.1 Wear protective clothing including gloves and goggles. Be sure sleeves are not rolled up
	1.2 Electrical – Burns/shock	1.2.2 Care with electrical connections, particularly with grounding. Avoid using flayed electrical cords can reduce hazard
	1.3 High Pressure Air – Fluid / Gas / Cylinder / vacuum	1.3.1 Inspect before using any pressure / vacuum equipments
	1.4 Water / Slip hazard	1.4.1 Any spillage must be cleaned and reported to technician/ demonstrator

Appendix E: Gas Membrane Separation Pilot Plant