College of Engineering

Chemical Engineering Department

Unit Opreation Lab

Expermint #1

Student Name: Mohammad Ahmad

ID # 435145267

Supervisor: Dr.Ahmad

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1) Abstract:

The aim of this experiment is to study the characteristic of four different types of membrane silicon in terms of separation process. Membrane separation process can be described by a feed stream that is divided into two which are retentate and permeate, where retentate is the part of feed that do not pass through the membrane but permeate can passing through. In this experiment, the model used to run the separation process is Membrane Test Unit model TR14. The experiment is carried out to study the characteristic of four (4) different types of membrane silicone used that undergoes different driving force which are reverse osmosis, nanofiltration process, ultrafiltration process and microfiltration process. The permeate mass flux of membrane 1, membrane 2, membrane 3 and membrane 4 are 30.07 g/min, 103.67 g/min, 6.621 g/min and 177.32 g/min, respectively. The result shows that microfiltration process has been identified to being the fastest separation process and most permeable, while nanofiltration process was the slowest process and less permeable. Membrane with hydrophilic nature allows water molecules passes through it, whereas membrane with hydrophobic nature inhibit the water molecules from passes through it. The objective is successfully obtained; thus, the experiment is successfully conducted

2) Introduction and Therotical Background:

Separations by using membranes are starting to become more relevant in process industries Membrane separation processes operate without heating and therefore use lesser energy when compared to conventional separation processes such as distillation, sublimation or crystallization. In membrane separation, the feed stream is separated into two, permeate and retentate. Permeate is the fraction that able to diffuse through the membrane while the retentate is the remaining fraction that have not been transported through the membrane. The membrane acts as a semi-permeable barrier and separation occurs depends on the membrane ability to allow rate of movement of molecules whether between two liquids phases, two gas phases or even liquid-gas phases. Usually, the two fluid phases are miscible and the barrier prevents natural dynamic flow from occurring.

Size of components to be separated and the degree of driving force provide criteria that were used to classified membrane separation processes, as shown in table below.

Table 1: Classification of Membrane Separation Process

Name of process	Driving force	Separation size range
Microfiltration	Pressure gradient	10 − 0.1 μm
Ultrafiltration	Pressure gradient	< 0.1 μm − 5 nm
Reverse osmosis (hyperfiltration)	Pressure gradient	< 5 nm
Electrodialysis	Electric field gradient	< 5 nm
Dialysis	Concentration gradient	< 5 nm

2.1) Microfiltration:

In micro filtration, pressure-driven flow through the membrane is used to separate micro-sized particles from fluids. Usually, the particles are larger than those in ultrafiltration. Examples of this type of membrane separation is separation of bacteria, paint pigment, yeast cells and many more.

2.2) Ultrafiltration:

This process use pressure to obtain molecules separation by using semipermeable polymeric membrane (M2). This membrane differentiates based on molecular size, shape, or chemical structure and separates high-molecular weight solutes, and colloidal materials.

2.3) Reverse Osmosis:

In this process, a membrane is placed between a solute-solvent solution and a pure solvent that prevents the passage of a low molecular weight solute. The solvent diffuses into the solution by osmosis. In reverse osmosis, reverse pressure difference is imposed which caused the flow of solvent to be in reverse manner.

2.4) Nanofiltration:

This process is a recent improvement in the membrane separation processes. It handles material that are dissolved in a liquid. The separation between solute and solvent occurs by diffusion of solvent molecules through membrane material, driven by transmembrane pressure.

This experiment was carried out to study the characteristic on 4 different types of membranes. The membrane unit consist of 4 membranes, which are:

Membrane 1: AFC 99 (Polyamide Film)
Membrane 2: AFC 40 (Polyamide Film)
Membrane 3: CA 202 (Cellulose Acetate)
Membrane 4: FP 100 (Polyvinylidene Fluoride)

Membrane separation processes is very essential in the separation industry. The processes is conducted without heating and thus use less energy than conventional thermal separation process which are distillation, sublimation or crystallization. Membrane is a selective barricade that allows the separation of some species in a fluid by combination of filtering and adsorption diffusion method. Separation is completed by selectively permeating one or more components of a stream through the membrane whilst hindering the channel of one or other components. Membrane separation also detach components over a huge range of particle sizes and molecular weight which is from macromolecular material (starch and protein) to monovalent ions.

Membrane processes can be described by a feed stream that is divided into two which are retentate and permeate. The most universal process can be portrayed by the following Figure, Figure 1.

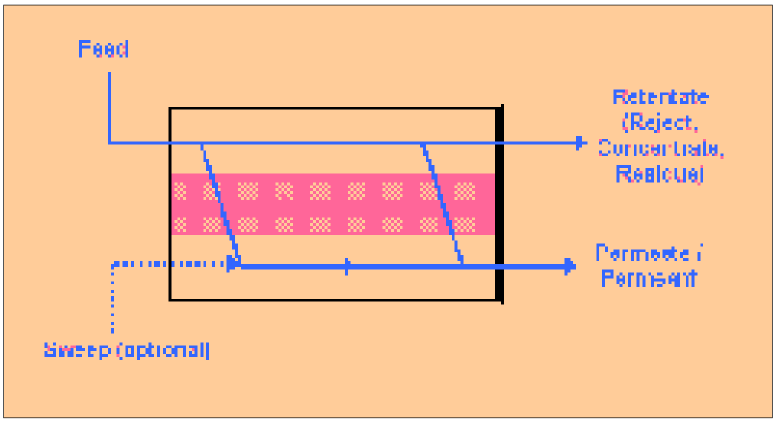

Figure 1 - Typical Membrane Separation Processes

The retentate is the part of the feed that does not passage through the membrane, whilst the permeate is that part of the feed that can pass through the membrane. The elective "sweep" is a gas or liquid that is used to assist eliminate the permeate. The significance of the component in membrane separation is known as the solute. The solute can be held on the membrane and eliminate in the retentate or passed through the membrane in the permeate. Membrane can perform separations by three different mechanisms:

1. By using pores that some species can pass but cannot by other species. This mechanism is also known as size exclusion.
2. By selective retardation by the pores when the pore diameters are close to molecular sizes. This mechanism is also known as pore flow.
3. By dissolution into the membrane, migration by molecular diffusion across the membrane, and reappearance from the other side. This is also known as solution diffusion.

The separation process is merely physical and the permeate and retentate can be used. Cold separation is one of a membrane technology that is extensively used in the biotechnology and pharmaceutical industries. Moreover, the advantage of using membrane is it allows separation to occur which is impractical by thermal separation processes. For instance, it is unachievable to separate the components of azeotropic liquids or solutes which form isomorphic crystals by distillation or recrystallization. However, it is possible with the used of membrane technology. Influenced by the different types of membranes, the selective separation of certain individual component or mixture components is practical. Example of essential applications in our daily life is the manufacture of drinking water by reverse osmosis, filtration in the food industry and many more.

The choice of synthetic membranes for the aimed separation process is basically based on few conditions. Membranes have to deliver optimum mass transfer area to process huge amounts of feed stream. The selected membrane must contain high selectivity (rejection) properties for certain particles; it has to repel fouling and with high mechanical stability. Besides, it is also can be recycled and have low production costs.

3) Exprmint Procedure:

- **Gnreal setup:**

1) All the valves are ensured to be initially closed.
2) Sodium Chloride, NaCl solution is prepared by adding 100 gram of sodium chloride into 20 L of water. The feed tank is then filled up with the salt solution.
3) The power for the control panel in turned on. All the sensors and indicators are checked to ensure they functioning properly.

- **Experiment A**

4) The experiment was started with Membrane 1. Valves V2, V5, V7, V11 and V15 were opened.
5) Plunger pump (P1) were switched on and valve V5 were slowly closed. Pressure value is then observed at pressure gauge and pressure regulator were adjusted to be maintained at 18 kPa.
6) Valve 5 were opened and maximum membrane inlet pressure were maintained at 18 kPa for Membrane 1`by adjusting the retentate control valve (V15).
7) The system was allowed to run for about 5 minutes. Valve V19 were opened and valve V11 were closed simultaneously before the first sample from permeate sampling port were collected. The sample is then weighed using digital weighing balance.
8) The weight of permeates were recorded and tabulated for 10 minutes with 1-minute interval.
9) Step 1 to 6 were repeated for Membrane 2, 3 and 4. Sets of valves were opened and closed according to their respective sets.

Table 3- Valves position.

Membrane	Opened valves	Sampling Valve	Retentate control valve	Membrane maximum inlet pressure (bar)
1	V2, V5, V7, V11 and V15	V19 opened and V11 closed	V15	18
2	V2, V5, V8, V12 and V16	V20 opened and V12 closed	V16	18
3	V2, V5, V9, V13 and V17	V21 opened and V13 closed	V17	18
4	V2, V5, V10, V14 and V18	V22 opened and V14 closed	V18	18

10) Graph of permeate weight versus time were plotted.

- **General Shut Down:**

11) The plunger pump is switched off.
12) Valve V2 is closed.
13) All liquid in the feed tank and product tank is drained by opening valves V3 and V4.
14) All the piping is flush with clean water. Then, valve V3 and V4 is closed.
15) Clean water is filled up to feed tank until 90% full.
16) The system is run with the clean water until the feed tank is nearly empty for cleaning purpose

4) Data and Result:

Table 3 – Data of the experiment:

Time (min)	Weight of Parameters (g)			
	Membrane 1	Membrane 2	Membrane 3	Membrane 4
1	31.54	63.31	84.57	95.58
2	48.70	114.26	157.00	172.93
3	66.51	163.56	221.65	252.06
4	84.63	218.64	311.13	330.10
5	103.19	263.18	399.64	407.54
6	122.65	312.40	487.67	485.72
7	140.65	362.49	575.61	563.42
8	157.42	412.56	661.39	642.15
9	173.66	463.84	747.50	715.95
10	189.77	512.64	834.44	789.26

Table 4 – Permeate Weight Reading For 4 Different Membranes

Time (min)	Weight of Samples (g)			
	Membrane 1	Membrane 2	Membrane 3	Membrane 4
1	48.84	105.65	19.96	167.03
2	67.80	200.90	23.09	323.40
3	97.64	296.45	30.75	482.65
4	128.73	388.90	36.24	630.48
5	155.40	495.85	41.50	823.06
6	184.49	600.89	46.38	999.68
7	215.60	708.20	51.77	1168.84
8	242.24	828.09	56.79	1323.06
9	271.65	929.03	61.76	1506.83
10	300.67	1036.72	66.21	1773.21

Table 5 – Permeate Mass Flux for four Different Membranes:

Membrane number	Membrane 1	Membrane 2	Membrane 3	Membrane 4
Permeate Mass Flux (g/min)	30.07	103.67	6.621	177.32

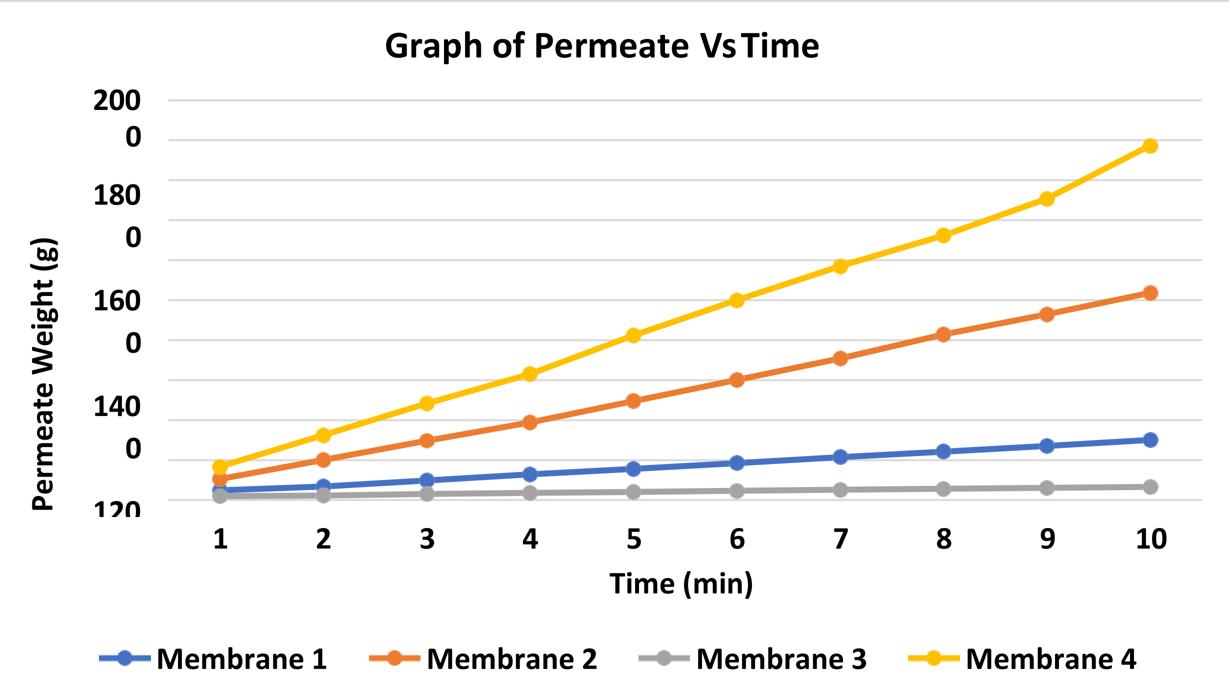

Figure 2 - Graph of Permeate Vs Time

The graph shows each membrane has the ability to separate sodium chloride from its solution as the graph show steady increase in permeate weight over time for each membrane. However, the permeability of the membrane is varied with membrane 4 as the most permeable followed with membrane 2, membrane 1 and membrane 3, respectively.

5) Calculations:

Sample Calculation for Permeate Mass Flux:

$$\text{Permeate Mass Flux}, \mathrm{P_m} = \frac{\text{Final Permeate Weight } (\mathrm{T} = 10 \text{ min})}{\text{Time } (\mathrm{T} = 10 \text{ min})}$$

$$\mathrm{P_m} = \frac{300.67 \text{ g}}{10 \text{ min}}$$

$$\mathrm{P_m} = 30.067 \text{ g/min}$$

6) Discussion:

The experiment is mainly to study the characteristic of 4 different types of membranes. The membranes are used as separation mediums to separate sodium chloride from its solution that have been prepared by adding 100g sodium chloride into 20L water. Figure 1 shows the graph of permeate weight versus time for the respective membrane; AFC 99 Polyamide Film (Membrane 1), AFC 40 Polyamide Film (Membrane 2), CA 202 Cellulose Acetate (Membrane 3) and FP 100 Polyvinylidene Fluoride (PVDF) (Membrane 4). From the graph, each membrane shows the ability to separate sodium chloride from its solution. However, the separation rate is difference with membrane 4 is the highest, followed by membrane 2, membrane 1 and membrane 3, respectively. Membrane 1, Membrane 2, Membrane 3, and membrane 4 used reverse osmosis, ultrafiltration process, nanofiltration process and microfiltration process, respectively as the separation mechanism which highly dependent on the pressure gradient. Higher pressure will resulting in a higher separation rate. As the pressure is set to constant at 18 kPa, the separation rate is mainly determine based on the permeability of the membrane, process the solution gone through and the nature of the material used for the membrane. Membrane 4 shows the fastest rate of separation compared to the others which resulting in maximum of 1773.21g of permeate weight passing through it. This could be due to this membrane is highly permeable or the filtration process is enhanced at 18kPa compared to the other three membranes. Undergoing microfiltration process, thus only allowing molecules that have smaller size than 0.1μm to pass through shows the membrane ability to separate a purer permeate but also in a faster rate. Besides, the nature of the Polyvinylidene Fluoride is hydrophilic. Since, the permeate mainly consist water molecule, it easily passes through the membrane making the mass of the permeate passing through the highest compared to other membranes. Membrane 3 shows the lowest separation rate which resulting in maximum of only 66.21g of permeate weight after 10 mins. The sodium chloride solution undergoes nanofiltration process which has the ability to separate nanosized molecules from the solution which is lower than 0.002μm up to 0.001μm. Blockage at the pore of the membrane could be the reason why the solution is unable to flow pass through the membrane since the porosity of the membrane is very small. This actually shows the positive site of the membrane since it allows a purer permeate to be produced since even small particle is trap at the pore of the membrane. However, cellulose acetate is naturally hydrophobic. Since the permeate mainly consist water molecule, this could also be the reason why the mass of permeate for the membrane is the smallest. Membrane 1 shows slightly higher separation rate compared to membrane 3. Reverse osmosis allows large molecules to pass through the membrane. Thus, the separation should be highest among the others. Besides, the nature of the Polyamide Film is hydrophilic allowing water molecules to easily passes through the membrane. However, this process requires high pressure to overcome the osmotic pressure. 18 kPa might be not high enough to overcome the osmotic pressure resulting in the solution not able to flow smoothly passing through the membrane. Thus, resulting in lower separation rate. Membrane 2 shows slightly lower separation rate compared to membrane 4. Ultrafiltration process allows molecules below 0.1μm up to 0.002μm to pass through the membrane. The lower separation rate could be due to, part of the pore of the membrane is blocked by the sodium chloride molecules which inhibit the solution flow passing through the membrane. In a bright side, the permeate is purer than that from membrane 4 separation. Besides, the nature of the Polyamide Film is hydrophilic allowing water molecule to easily pass through the membrane. Although membrane 1 and 2 comes from the same type of materials, however membrane 2 is more hydrophilic and have big nodular structured compared to membrane 1. In short, although membrane 4 shows significant permeate weight after 10 mins, the purity of the permeate is still questionable as compared to the other three membranes especially membrane 2 since the pore size of the membrane is much smaller than membrane 4. The smaller the pore size of the membrane will yield the purer permeate from the solution. The efficiency of the separation process also depends on the nature of the membrane either hydrophobic or hydrophilic. Higher separation rate does not necessarily result in a purer permeate. The limitation of the experiment is the students are not checking the purity of the permeate at the end of the experiment. Thus, although the rate of separation is different, the efficiency in gaining a pure water from the salt solution cannot be determine. Besides, the reading taken during the experiment is taken every 1 min. During this experiment, the equipment is vibrating due to the pump effect. This, resulting inaccuracy in the weighting scale reading as the weighing scale is connected to the separation unit.

7) Conclusion:

At the end of the experiment, the characteristic between 4 membrane silicon had been identified. The increasing and decreasing for each membrane almost similar. From the graph plotted, the value of weight of output for each membrane steadily increase when the time increase for each membrane. The value of weight collected for each membrane also increase along with the membrane used. Furthermore, each membrane shows the same pattern of the outlet with different value of weight of the outlet. Membrane 1, membrane 2, membrane 3 and membrane 4 used reverse osmosis, ultrafiltration process, nanofiltration process and microfiltration process, respectively which is highly dependent on the pressure gradient and pressure 18 KPa was set as constant in this experiment. The smaller the pore size of membrane will yield the purer permeate from the solution.

Based on the weight data for each membrane shows that the forth membrane was identified being fastest in separation process and most permeable followed with membrane 2, membrane 1 and membrane 3. In membrane 4, the process undergoing microfiltration process that allowing molecules that have smaller size than 0.1μm to pass through shows the membrane ability to separate a purer permeate. Besides, the nature of the Polyvinylidene Fluoride is hydrophilic that consist of water molecules easily passes through the membrane making the mass of the permeate passing through the highest compared to other membrane. Meanwhile, membrane 3 was the slowest separation process occur and less permeable. The nanofiltration process which has the ability to separate nanosized molecules from the solution which is lower than 0.002μm up to 0.001μm produces low permeate from the separation process. Other than that, blockage at the pore of the membrane could be the reason why the solution is unable to flow pass through the membrane. At the end, the objective of the experiment achieved in identifying the characteristic of the membrane in separation process.

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