Separation of Rhodium Catalyst in a Homogeneous System by using Nanofiltration

by

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Dissertation submitted in partial fulfillment of

the requirements for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

JULY 2010

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the

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Approved by,

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TRONOH, PERAK

July 2010

CERTIFICATION OF ORIGINALITY

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

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NUR SYAFINA BINTI ABDUL RAZAK

ABSTRACT

Nanofiltration process can assist the industry in recovering the catalyst in homogeneous system. The objective of this dissertation is to report the findings on the research of the separation for rhodium catalyst in a homogeneous system by using nanofiltration. The objectives of the research are to determine the solvent-membrane compatibility in a system, to study the solvent flux and rejection of catalyst in different solvent-membrane combination and to assess the effect of pressure and concentration towards the catalyst rejection.

The research starts with the compatibility test which is conducted by soaking the membrane in solvent for 24 hours to observe any physical changes that occur. The solvents used are ethyl acetate and toluene while the membranes used are STARMEM^{TM} 122 and STARMEM^{TM} 240. The membranes used for soaking are taken for analysis using Field Emission Scanning Electron Microscopy (FESEM) to observe the changes on the surface and the cross section of the membrane after immersing into the solvents. Stable combinations are used for permeability and rejection study. METcell separation unit is used to conduct the research for the solvent flux. Three reading for pressure is used for flux and rejection study which are 10 bar, 15bar, and 25 bar while the concentration is varied at 0.5 mM, 0.8 mM and 1.0 mM. UV-Visible spectrophotometer is used to determine the concentration of permeate and the retentate solution for this research.

STARMEM^{TM} 122 and STARMEM^{TM} 240 changed physically after 24 hours in Ethyl acetate and no changes observed for both membranes in toluene solvent. From the FESEM images, changes are observed as well as for all membranes with respect to the original membranes. During the flux study for pure solvent, it is proven that the flux declines with time to a steady level which is due to membrane compaction. As the pressure increases, the solvent flux also increases for both membrane but it is higher for STARMEM^{TM} 240. Flux reduces with the increase of catalyst concentration which can be explained by the phenomenon of pore fouling. Rejection is reported high for both membranes which are around 0.8 to 0.95.

In conclusion, nanofiltration is useful in producing green chemical process in industry especially in optimizing the purity of the products. Pressure and concentration affects the value of flux and rejection of a system. Prior to conducting the catalyst filtration, the membrane should be at steady flux condition to avoid unstable permeation. In obtaining, the right steady flux of system, the process should be done in continuous mode for more consistent result. The concentration of the rhodium catalyst in the permeate and retentate solution can be determined using various methods in order to obtain more accurate result.

ACKNOWLEDGEMENT

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CHAPTER 1

INTRODUCTION

1.0   PROJECT BACKGROUND

The separation of products from catalyst is a major problem in homogenous catalytic processes, including phase transfer catalytic and transition metal catalysed reactions. However, the ability of the catalytic organic synthesis to produce cleaner processes which supports the awareness of green chemistry has made the process more favorable to become a great focus nowadays (Schafer *et al.*, 2005). The development in designing the efficient catalyst and also the right separation method needs to be taken into account in order to produce a cost effective and cleaner process for industry.

One of the commercial homogenous catalytic processes is the rhodium-catalysed hydroformylation process. Hydroformylation process is a process of producing aldehydes from the reaction of alkene with the synthesis gas catalysed by transition metal catalyst. The catalyst and all other reactants are in solution form and the catalytic reaction takes place in the liquid phase. The overview of the hydroformylation process is shown below in Figure 1.
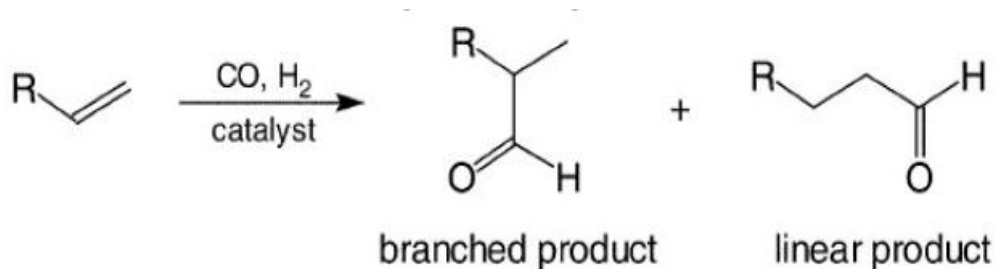
Figure 1: Hydroformylation process

The high selectivity and mild condition of rhodium-based process have made the manufacturers of n-butyraldehyde favor this process. The high cost of rhodium catalyst makes the near-complete catalyst recovery a must for the process to be commercially viable and the development of an efficient separation method based on

water-soluble phosphines has assisted the process to be more feasible (Bhaduri *et al.*, 2000).

A membrane process is defined as a process which a feed stream is divided into two streams which are retentate and permeate streams as shown in Figure 2. Any of the two can be considered as products but for the case of hydroformylation process, permeate stream is the desired product. The performance of the membrane is classified by three parameters which are the selectivity, the flux through membrane and the recovery (Katleen Boussu, 2007).
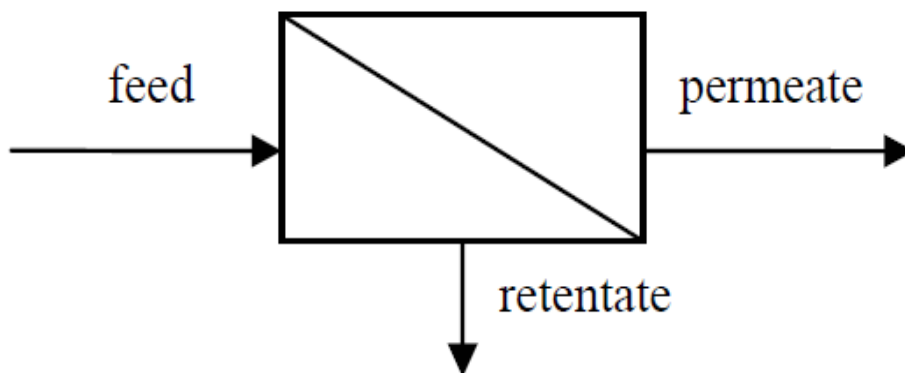
Figure 2: Representation of membrane separation

Nanofiltration membrane technology has become a great method for separation nowadays. Nanofiltration is a process between ultrafiltration (UF) and reverse osmosis (RO). Nanofiltration requires lower pressure than RO which saves more energy. Due to charged interaction with the membranes, multivalent ions are also well retained (Katleen Boussu, 2007).
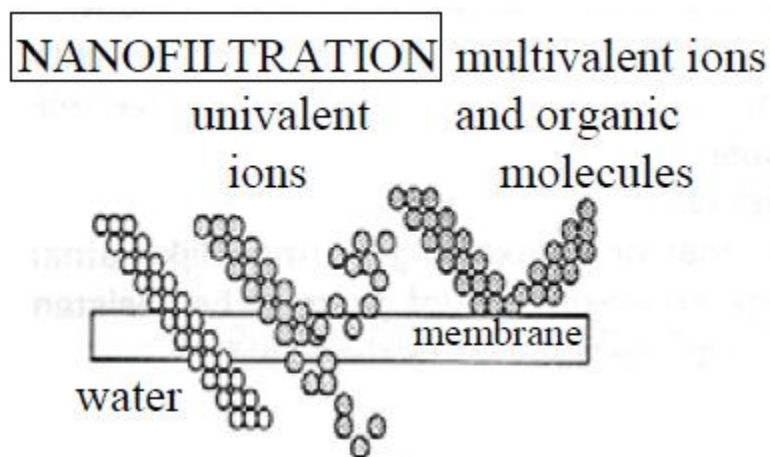
Figure 3: Nanofiltration process

This method has found to be feasible in the separation of homogenous catalysts which have relatively very large molecular structure compared to the reaction products which are smaller. However, only solvent stable nanofiltration membrane can be applied due to the nature of catalysis and reaction environment. The solvent-membrane interaction gives a significant impact in obtaining the optimum separation of catalyst. There are a few other factors that influence the catalyst separation such as the pore size, pressure, temperature, and catalyst concentration.

1.1   PROBLEM STATEMENT

In the plasticizers, soaps and detergents industries, hydroformylation of olefins to produce long chain aldehydes using cobalt-based catalyst is one of the crucial industrial processes since the 1950s. However, cobalt-based catalyst has poor conversion of the feedstock and low selectivity of linear product. Even though this catalyst is inexpensive but it requires high pressure and temperature condition which leads to high capital and maintenance cost.

On the other hand, rhodium-based catalyst provides higher selectivity and reactivity compared to cobalt-based catalyst besides having a "cleaner" product-mix after the reaction takes place unlike the cobalt-based catalyst that forms more byproducts. Rhodium-based catalyst requires much lower pressure and temperature to stabilize the catalyst.

However, cobalt-based catalyst is a better catalyst when dealing with branched olefins compared to rhodium-based catalyst. The other issue with the usage of rhodium-based catalyst is that it encounters difficulty in catalyst recovery for longer chain olefins (C6-C12) reaction as these olefins have high molecular weight that contributes to the high boiling points of the components. Thus, distillation process for catalyst recovery is not applicable since the catalyst will become unstable which will lead to catalyst degradation.

Most of the methods for separation of catalyst that are industrially employed are aimed to recover the catalyst in an active form to be used for another cycle of reaction without jeopardizing the products. Since rhodium-based catalyst is very expensive, a proper method in separating the catalyst from the product in active form is necessary in order to optimize the production which indirectly helps to minimize the cost. Thus for this research, nanofiltration process is utilized in regaining the catalyst in its active form from the product mixture.

1.2   OBJECTIVES

The objectives of the projects are:

i.   To assess the compatibility of the solvent-membrane combination in observing membrane stability in solvent plus non-zero solvent flux at 2.0 MPa (20 bar)

ii.   To determine the solvent flux and membrane rejection of catalyst for different types of solvent-membrane combination.

iii.   To assess the effect of pressure and catalyst concentration for different types of solvent-membrane combination.

1.3   SCOPE OF STUDY

The scope of study for this section is divided into three parts as listed below:

1.3.1   Compatibility of the solvent-membrane combination

The assessment of the compatibility of the solvent resistance nanofiltration (SRNF) membranes in the organic solvents is required prior to observing the other properties such as the flux, rejection and varying operating parameters. The condition of membrane in the reaction environment should be observed. The usage of polyimide membrane in organic solvent may cause the membrane to swell or dissolve during the reaction. Physical stability of the membrane disk after soaking in the solvent for 24 hour and non-zero flux achieved at 2.0 MPa are the method to inspect the stability (Scarpello *et al.*, 2002). By conducting the compatibility test, the separation of catalyst can be optimized using the best membrane-solvent combination.

1.3.2   Solvent flux and membrane rejection of catalyst

The study for this part is conducted for the membrane-solvent combinations that are stable for the reaction. Solvent flux is the amount of solvent that flows through the unit area per unit time. The amount of solvent flux indicates that the membranes function well with the organic solvents used. The flux study is conducted for both pure solvent and catalyst solution. Catalyst rejection by the membrane shows the efficiency of the membrane separation. The solvent-membrane combinations will be experimented at the same operating condition of pressure and temperature using the same catalyst concentration.

1.3.3   Effect of pressure and catalyst concentration

The pressure and catalyst concentration may influence the solvent flux and the rejection of the catalyst. The changes in the operating parameters lead to changes of the system itself. The fouling of the membrane may result from the manipulation of these parameters. The effect of these parameters will be studied for a few solvent-membrane combinations to observe the trend of flux and catalyst rejection.

1.4   ORGANIZATION OF DISSERTATION

This dissertation consists of six subsequent chapters in fulfilling the above mentioned objectives. Chapter 2 is basically about the literature review that is related to the research project which is more towards the technology of hydroformylation industry and also the previous outstanding research works done for membrane separation using nanofiltration process. Chapter 3 gives the detailed methodology that is required in completing the research starting from the study of the compatibility of the membrane until the study of various parameters towards catalyst rejection. Chapter 4 provides the result obtained from the research work and also the discussion of the respective result. Lastly, Chapter 5 concludes the research work and also some recommendations made for future tasks to be done by the author or other researchers or FYP students.

CHAPTER 2

LITERATURE REVIEW

2.0   HOMOGENEOUS CATALYSIS

Catalytic organic synthesis has become a great focus in industry today. Homogeneous and heterogeneous catalysts are used in the conversion of petrochemicals nowadays. However, the number of homogeneously catalysed processes has been steadily growing in the eighties and nineties (van Leeuwen, 2004). Homogeneous catalysis can be referred to as a catalytic process in which the catalyst and the substrates are in one phase, usually in the liquid phase. Most of the time, it involves organometallic compound as the catalyst. Organometallic catalyst consists of a metal element at the centre which is surrounded by organic or inorganic ligands. The properties of the catalyst are determined by the metal and the ligands. The performance of catalyst is observed at the relative ease of the catalyst modification by changing the ligands environment which directly affects the rate of reaction and the selectivity of the products.

Hydroformylation process is one of the examples of the industry nowadays which has shifted from using cobalt-catalysed system to rhodium-catalysed system which is found to be more efficient and economic. Since this transition metal catalyst is very expensive, recycling the catalyst can help to reduce the cost of the operation.

Davy Process Technology Limited in collaboration with The Dow Chemical Company has developed the 'Low Pressure Oxo' process (LP Oxo^{SM} Process) a hydridocarbonyl coordination complex of rhodium, modified with triphenylphosphine (TPP) ligand as catalyst and becomes the world leading position in low pressure of hydroformylation process which comprises two thirds of the world's production of butyraldehydes (Tudor and Ashley, 2007). van Leeuwen (2004) also agrees that rhodium based catalyst does well in the 'Low Pressure Oxo' process as it is much faster and the feedstock utilization is much better compared to cobalt based catalyst.

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The comparison between rhodium based and cobalt based hydroformylation process is shown in Table 1. The development of rhodium based catalyst is favored by manufacturer due to the high selectivity and mild condition of the process.

Table 1: Process parameters of hydroformylation processes (Bhaduri *et.al*, 2000)

Process parameters	Cobalt	Cobalt + phosphine	Rhodium + phosphine
Temperature (^{o}C)	140-180	160-200	90-110
Pressure (atm)	200-300	50-100	10-20
Alkane formation	Low	Considerable	Low
Selectivity to n-butyraldehyde (%)	75-80	85-90	92-95
Isolation of catalyst	Difficult; $HCo(CO)_4$ is volatile	Less difficult	Less difficult; water soluble phosphine a major advancement

2.1   MEMBRANE SEPARATION FOR CATALYST RECOVERY

Current development of catalyst recovery can be achieved by combining the membrane separation with the chemical processing in the industry using solvent resistance nanofiltration membrane (SRNF) which the range of nominal molecular weight cut off (MWCO) is between 200 to 1000 Da (Luthra *et al.*, 2002). The membranes should be effective in separating the organic synthesis catalyst with the product by the exploitation of the size difference between the catalyst (> 600 Da) and the products (< 400 Da) as stated by Scarpello *et al.* (2002) in their study.

As for homogeneous catalyst system, the crucial problem needs to be overcome for a sensitive catalyst system is the catalyst degradation due to thermal separation process. Thermal separation might cause the irreversible destruction of catalyst as it can precipitate onto the apparatus walls as a metallic rhodium. Priske *et al.* (2010) proves that the solvent resistant nanofiltration (SRNF) membrane is possible to separate and recycle the homogeneous catalyst for hydroformylation process.

2.2   MEMBRANE-SOLVENT COMPATIBILITY

Most of nanofiltration membranes are designed for aqueous systems. Thus, the membranes typically lose their structural integrity and separation performance upon exposure to organic solvent (Yang *et al.*, 2001). Prior to the catalyst separation through nanofiltration, the right solvent-membrane combination is crucial part that needs to be taken into consideration. The compatibility of the membrane can be seen by observing the ability of the membrane to produce solvent flux, J_s at moderate pressure (Scarpello *et al.*, 2002) and also the physical stability.

The solvent flux produced can be calculated using this equation:

$$J_S = \frac{V_p}{A_m\, t} \qquad\qquad (1)$$

where A_m is the active membrane surface area and t is the time taken for a certain volume of permeate to be achieved. The usage of polymeric membrane in organic system may cause an aggressive effect. The 'like dissolve like' rule of thumb is applicable since the polymeric membranes tend to swell or even dissolve in an organic fluid environment. The stability of the membrane inside the reaction is important, so that the structure of the membrane and also the transport properties are not affected due to swelling and plasticization (Desrocher, 2004).

A few studies are reported to observe the stability of membrane in the solvent system. The solvent properties affect the solvent flux. The experiment using MPF-50 shows that non polar solvent has higher permeability than polar solvent (Machado *et al.*, 1999). However, Scarpello *et al.* (2002) reported the level of flux of a given solvent through a given membrane cannot be simply predicted from physico-chemical properties of the solvent such as the viscosity, molar volume and solvent hydrophobicity and air-liquid surface tension. This statement contradicts with the assumption of Machado *et al.* (1999) in their study saying that the decrease in viscosity leads to the increase in solvent flux and same trend observed for surface tension.

Most of nanofiltration membranes, designed for aqueous system lose their structural integrity and separation performance upon exposure to organic solvent which cause membrane instability such as cracking, swelling and shrinking of membrane matrix that affect the solvent flux (Yang *et al.*, 2001). Yang *et al.* (2001) has conducted an observation to see the stability of the membrane inside organic solvent for a few types of membrane and the result is as shown in Table 2 below.

Table 2: Observation of membrane compatibility in organic solvent by Yang *et al.*, 2001

Membrane type	Methanol	Ethyl acetate	Toluene
UTC-20	Curled	Immediately cracked	Immediately cracked
MPF-44	Flat	Curled	Rolled, translucent
MPF-50	Flat	Curled	Rolled, translucent
MPF-60	Flat	Curled	Curled, translucent
Desal-5	Flat	Flat	Flat
Desal-DK	Slightly curled	Immediately cracked	Immediately cracked

The observation for ethyl acetate by Yang *et al.* (2001) for MPF-50 does not tally with the manufacturer's claim regarding the stability of the membrane in a wide range of organic solvent including methanol, ethyl acetate and toluene. It also opposes the observation made by Scarpello *et al.* (2002) since they have concluded that MPF-50 appears to be most flexible membrane due to its compatibility with all the chosen solvents including ethyl acetate.

2.3   MEMBRANE PRE-TREATMENT

Volume of solvent permeated before obtaining steady flux is a function of the membrane as the universal pre-fluxing volume cannot be defined as it is dependent on the polymer from which it is made. Each membrane should be pre-conditioned with pure solvent until a steady flux is obtained prior to using it to investigate the separation properties of the membrane (Gibbins *et al.*, 2002).

Prior to reaction mixture separation, Luthra *et al.* (2002) also conduct the pre-conditioning by passing the pure toluene through membrane to allow benchmarking of the membrane status via the pure toluene flux. The flux was found to be decreased

due to membrane compaction. Membrane compaction effect is agreed by Yang *et al.* (2001) in their experiment of determining the flux of pure solvent at steady state using MPF membranes. Silva *et al.* (2005) claims that the membrane compaction is a slow process and flux data collected in a dead cell end testing with a limited collection period (typically 1-3 hours) may not be reliable to predict long term performance.

Yang *et al.* (2001) make comparison between the methanol fluxes from Machado et.al (1999) and manufacturer value and find significant difference in value of flux of Machado *et al.* (1999) compared to theirs and the manufacturer's value. Different solvent flux value for the same membrane may due to insufficient of washing out of pre-conditioning agent or insufficient time to allow steady state flux.

Zhao *et al.* (2006) claims that the membrane treatment with methanol and acetone has a considerable effect on solvent flux and membrane rejection properties in methanol solutions for polyamide and polyimide membranes (Desal-DK and STARMEM^{TM} 228) and insignificant effect on a PDMS-based membrane (MPF-50). Membrane pretreatment could result in reorganization of membrane structure, changes in membrane pore size (or free volume between the polymer chains) and hydrophobicity of the membrane.

Gryp *et al.* (2010) also pre-treat the STARMEM^{TM} membranes with toluene in order to remove the preserving agent from the membrane. The cell is then washed out with the desired solvent that is later used for steady state characterization. Gryp *et al.* (2010) also conduct membrane break-in procedure after the pre-treatment in order to establish steady state condition and to avoid irreversible membrane compaction which is previously done by Scarpello *et al.* (2002).

2.4 EFFECT OF MOLECULAR WEIGHT CUT-OFF TO CATALYST REJECTION

Molecular weight cut-off is the cut-off value, defined as that the lowest molecular weight value that 90% of the solute is retained by the membrane. In nanofiltration, the high MW of the catalyst compared to the MWCO of the membrane has assisted the catalyst separation.

The rejection of catalyst can be calculated using the formula below:

$$r = 1 - \frac{C_p}{C_r} \qquad (2)$$

where r is the rejection of the catalyst, C_p is the concentration of permeate and C_r is the concentration of the retentate. Besides, the mass balance of the catalyst can also be conducted to confirm that no catalyst was sticking to the membrane or absorbed into the pressure cell seal by using the formula below:

$$MB = \frac{C_p V_p - C_r V_r}{C_0 V_0} \; x \; 100\% \qquad (3)$$

where MB is the mass balance of the catalyst, C_p is the concentration of permeate, C_r is the concentration of the retentate and C_0 is the initial catalyst concentration. V_p, V_r, and V_0 are the final permeate, final retentate and initial solution volume respectively.

Scarpello *et al.* (2002) claim that the Wilkinson which is the rhodium based catalyst, Pd-BINAP and Jacobsen catalyst rejection values for three STARMEM^{TM} membranes reflects the different MWCO of each membranes where MWCO STARMEM^{TM} 120 (200Da) < MWCO STARMEM^{TM} 122 (220 Da) < MWCO STARMEM^{TM} 240 (400). In their study, one or more of the membranes provide a separation system which gives high solvent flux (> 50 L m^{-2} h^{-1}) coupled with good catalyst rejection (> 0.95) for all types of catalyst solution except dichloromethane (DCM) solvent for separation of Jacobsen catalyst. The STARMEM^{TM} 120 and STARMEM^{TM} 122 become the best system in all cases.

However, the study done by Luthra *et al.* (2002) on the solvent flux and catalyst rejection has shown that using only the nominal MWCO as a guide is difficult. Luthra *et al.* (2002) use ammonium bromide salts (quats) as the solute in toluene solvent since the quats have similarity to common transition metal catalysts that are used in organic synthesis reaction as stated by Gibbins *et al.* (2002). The rejection of tetrabutylammonium bromide, TBABr (MW=322) and tetraoctylammonium bromide, TOABr (MW= 542) in STARMEM^{TM} series is > 99% except for STARMEM^{TM} 240 in TBABr (80%) since its MWCO of the membrane is bigger that the MW of TBABr. STARMEM^{TM} series still follow the trend the catalyst rejection based on the membrane MWCO. As for MPF-50 which has MWCO 700 Da, rejection of TBABr is higher than TOABr even though the MW of TBABr is lower. TBABr is more hydrophilic compared to TOABr and the better rejection of TBABr might be due charge effect to the membrane which is thought to be composite in structure with a silicon based top layer.

Yang *et al.* (2001) discuss about the charge effect on rejection due to different functional groups that result in different charges. So, when the molecular size is much smaller than the membrane pores, these charge effects can be the critical factor in determining the retention of highly charged membranes. However, the explanation is only valid for aqueous solvent while the charge effects might not apply for organic solvent or are negligible which is also agreed by Zhao et.al (2006). The solute-membrane-solvent interaction for aqueous and organic system is proven to be different.

2.5 EFFECT OF OPERATING PARAMETER TO SOLVENT FLUX AND CATALYST REJECTION

Scarpello *et al.* (2002) prove that solvent flux increases with the increase of pressure which is consistent with observation by Machado *et al.* (1999) and the catalyst existence has no effect to the solvent flux. The catalyst rejections appear to increase with the increase of pressure due to a partially reversible, increasing compression of active layer when membrane subjected to higher pressure. Thus, pores were tightened or sealed (cylinder pore-based permeability).

According to Scarpello *et al.* (2002) and Machado et.al (1999), the solvent flux increases with the increase of temperature. However, the existence of catalyst significantly slows down the permeate flux compared to pure solvent. The effect of temperature to catalyst rejection is found to be system dependent as the trend of rejection varies for some of the systems tested.

Increasing surface and/or pore fouling resulting from building up of a layer of catalyst at the membrane surface acts to partially block the pores of the membrane. With this explanation, the increase of catalyst concentration explains the reduction in solvent flux and also the increase of catalyst rejection reported by Scarpello *et al.* (2002) which is further confirmed by Luthra *et al.* (2002). Scarpello *et al.* (2002) find the effect of initial catalyst concentration to catalyst rejection improves substantially for STARMEM^{TM} 122-Wilkinson catalyst-tehrahydrofuran system from 0.971 to 0.995 by increasing concentration from 0.785 to 5.0 mM. However, the rejection does not give a significant effect to STARMEM^{TM} 122- Jacobsen catalyst-ethyl acetate system. Luthra *et al.* (2002) proves that, as the concentration of TBABr in toluene increase from 0.001M, 0.005M, and 0.01M, the rejection by Desal-5 membrane is measured as 71, 77, and 80% respectively. Yang *et al.* (2001) also find an inverse effect in increasing concentration of solute, Orange II dye on water flux.

Rhodium-based catalyst separation using a few compatible membrane-solvent combinations will be used to study the catalyst rejection and the solvent flux by varying the operating parameters.

CHAPTER 3

METHODOLOGY

3.0   CHEMICALS

The catalyst used is carbonyl triphenylphosphine rhodium hydride, $RhH(CO)(PPh_3)_3$ with a molecular weight of 918.79 g/gmol is supplied by ABCR GmBH & Co. KG, Germany. It is a yellow solid that is soluble in chloroform and methylene chloride. The structure of the catalyst is shown in Figure 4 below.
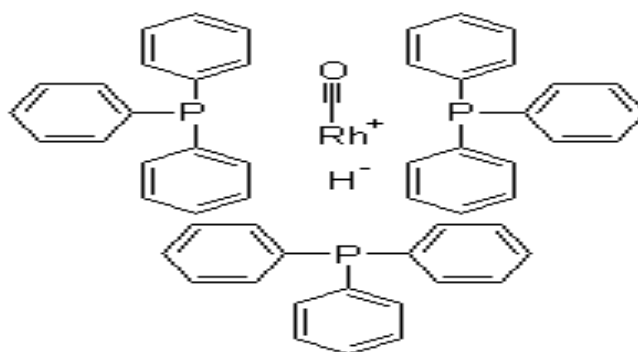
Figure 4: The structure of carbonyl triphenylphosphine rhodium hydride

Ethyl acetate and toluene are chosen since these solvents are able to dissolve the tested catalyst to produce homogeneous solution and these solvents are commonly employed in organic synthesis. The physical parameters of the solvents are shown in Table 3 below.

Table 3: Physical parameters of the solvents

Solvent	Supplier	Purity (%)	MW (g/mol)	Water miscibility /solubility (%,w/w)	Surface tension at 25^oC (mN.m)	Viscosity at 20° C (cP)	Dielectric constant at 20 ^{o}C
Ethyl acetate	Merck	>99.9	89	7.9	23.9	0.44	6.4
Toluene	Merck	>99.9	92	0.05	27.9	0.59	2.4

3.1 MEMBRANES

Two SRNF membranes are considered; STARMEM^{TM} 122 and STARMEM^{TM} 240 supplied by W.R. Grace, Columbia. The active surface of these membranes are manufactured from polyimide with an active skin layer less than 0.2 mm in thickness and pore sizes that are less than 50 ANG (Scarpello *et al.*, 2002). STARMEM^{TM} 122 has molecular weight cut off of 220 Da while STARMEM^{TM} 240 is 400 Da. From the supplier specification, it is found that the maximum pressure and temperature of these STARMEM^{TM} series are at 60 bar and 50°C, respectively. Both membranes are considered as hydrophobic membrane.

3.2 EXPERIMENTAL SETUP

The equipment that will be used for nanofiltration process is METcell, a 316 stainless steel high pressure stirred cell and it is capable of performing a wide range of membrane separations as shown in Figure 5. The *METcell* is utilized as dead-end filtration unit. The *METcell* has a maximum working pressure of 69 barg and is capable of conducting nanofiltration (NF) separations using aqueous and non-aqueous solvents.

Figure 5: METcell unit and the gas control unit

O-ring seals which are made from Flourinated Ethylene Propylene (FEP)-encapsulated Viton materials are used to allow high-pressure operation. The *METcell* is pressurized using inert gas, nitrogen from a cylinder and with the gas control unit

supplied with each *METcell*. A laboratory magnetic stirrer plate can be used to generate the stirring/mixing required in the cell to minimize concentration polarization effects (Instructions Manual for MET*cell*, 2006). The diagram of the equipment setup including the pressure source is shown in Figure 6 below.
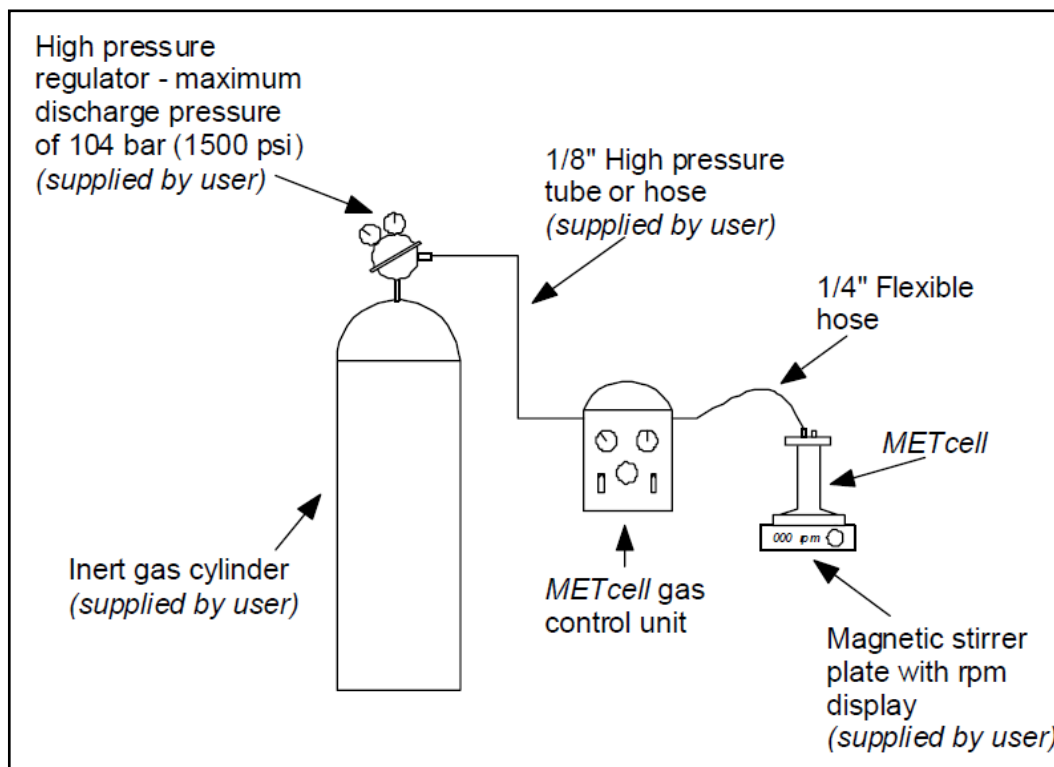
Figure 6: MET*cell* connection to pressure source

UV visible spectrophotometer is another tool that is used to measure the concentration of the catalyst in solution. The sample of solution of the compound to be tested is placed inside a glass cuvette, which is a small transparent container. Another glass cuvette is filled with the solution used for reference purpose. A beam of visible light and/or UV light source is separated into its components through diffraction grating. The monochromatic single wavelength will be divided into two equal intensity beams by a half-mirrored device. Each beam will pass through the sample in the glass cuvette and the intensities of the light beam for the sample of the solution of the compound, I and the reference sample, I_o will be detected by electronic detector as shown in Figure 7.
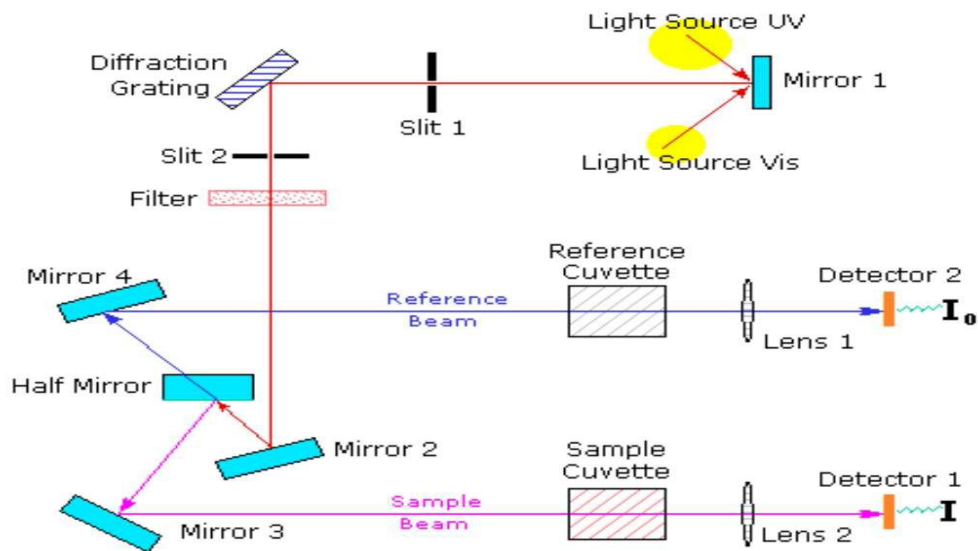
Figure 7: Diagram of typical spectrophotometer

Absorption may be presented as transmittance ($T = I/I_0$) or absorbance ($A = log\ I_0/I$). The concentration of the solution can be calculated since the absorbance is directly proportional to the sample concentration, molar absorptivity and pathlength through the glass cuvette.

Other equipment used during the experiment is Field Emission Scanning Electron Microscopy (FESEM). Electrons are liberated from a field emission source and accelerated in a high electrical field gradient. Within the high vacuum column these so-called primary electrons are focused and deflected by electronic lenses to produce a narrow scan beam that bombards the object. As a result secondary electrons are emitted from each spot on the object. The angle and velocity of these secondary electrons relate to the surface structure of the object. A detector catches the secondary electrons and produces an electronic signal. This signal is amplified and transformed to a video scan-image that can be seen on a monitor or to a digital image that can be saved and processed further.

Figure 8: FESEM equipment unit

3.3   EXPERIMENTAL AND ANALYTICAL PROCEDURE

The methodology for this experiment comprises of three parts. The first is to assess the compatibility of solvent-membrane, the second one is to determine the solvent flux and rejection for pure solvent and catalyst solution and the last part is to determine the effect of pressure and catalyst concentration for rhodium-catalysed homogenous system. Membrane-solvent combination is prepared as shown in Table 4 below.

Table 4: Membrane-solvent combinations

Combination	Membrane	Solvent
1	STARMEM^{TM} 122	Ethyl acetate
2	STARMEM^{TM} 122	Toluene
3	STARMEM^{TM} 240	Ethyl Acetate
4	STARMEM^{TM} 240	Toluene

Membrane sheet with 1 cm wide and 2 cm length each is soaked in the respective solution for 24 hours. The physical changes are observed after 24 hours. The sheets are dried using the oven at about 35° C in order to ensure that the membranes are free from solvent and coating agent. The membrane sheets are observed through Field

Emission Scanning Electron Microscopy (FESEM) unit for microscopic view of the changes on the membrane structure.

By using 82 mm diameter of membrane disc, the membrane-solvent combination is tested in METcell to observe non-zero flux condition at pressure of 20 bar. The stable combinations are used for the subsequent experiments. Membrane pre-treatment is crucial before starting an experiment. First, the membrane is immersed inside pure solvent for about 2 hours. Then, permeation of the solvent in METcell at 20 bar is conducted in order to reduce the influence of flux decline (Scarpello *et al.*, 2002). The pre-treatment is stopped after 250ml of solvent is permeated and the permeability test is conducted. The cell is charged with 250 ml pure solvent again. The time taken for 30 ml of solvent to be permeated is recorded.

The procedure to study the effect of pressure and catalyst concentration is the same as above. The only different is that each parameter is manipulated for three readings which are 20 bar, 15 bar and 10 bar for pressure variation while the catalyst concentration is analysed at 0.05 mM, 0.08 mM and 1.00 mM.

As for pressure variation part, 30 ml of rhodium catalyst solution is prepared for each run. The solution is pressurized at the above mentioned pressure with stirred condition at 150 rpm. The permeation is stopped after half of the solution is permeated.

Same procedure is conducted for concentration variation but the pressure is fixed at 20 bar with stirred condition at 150 rpm.

Permeate and retentate are taken for UV-Visible spectrophotometric analysis. Prior to the retentate and permeate concentration measurement, standard solution analysis is required in order to produce absorbance vs. concentration calibration curve as shown in Appendix 2. Accurate concentration measurements by UV-Visible spectrophotometric are possible in this study. The $HRh(CO)(PPh)_3$ catalyst is detectable at concentration above 0.2 mM at a wavelength of 361 nm.

All the required research works are completed on time according to the Gantt chart in Figure 9 below.

Month	Jul 2010	Aug 2010				Sept 2010					Oct 2010			
Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
FYP briefing and meeting with lab technologist	■	■	■											
Membrane stability test				■	■									
Progress report 1					■									
Membrane pre-treatment						■								
Rejection test for STARMEM^{TM} 122						■								
Rejection test for STARMEM^{TM} 240						■								
Mid Sem break							■	■						
Pressure manipulation STARMEM^{TM} 122									■					
Concentration manipulation STARMEM^{TM} 122									■					
Pressure manipulation STARMEM^{TM} 240										■				
Concentration manipulation STARMEM^{TM} 240									■					
FESEM (original membrane)													■	
UV-Vis analysis													■	

POSTER PRESENTATION (week 12)

Figure 9: Gantt chart for final year project 2

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CHAPTER 4

RESULT AND DISCUSSION

4.0 MEMBRANE STABILITY TEST

Not all the membranes were found to be physically stable in solvents investigated. Thus, with this physical observation, the membranes are found suitable to be used for the next level of analysis, the flux permeability study and rejection test. From the physical observation both STARMEM^{TM} series produced physical changes after immersing the membrane inside the pure ethyl acetate solvent. STARMEM^{TM} 122 curled at the end of the membrane edge while STARMEM^{TM} 240 folded significantly. However the active layers of both membranes did not disintegrate during the immersion. No physical changes observed for the membrane series in pure toluene solvent. Figure 10 below shows the changes of the membrane before and after the soaking. In contrast, Scarpello *et al.* (2002) reported that both membranes are physically stable in ethyl acetate. However, the characteristic of 'physically stable' was not described clearly.
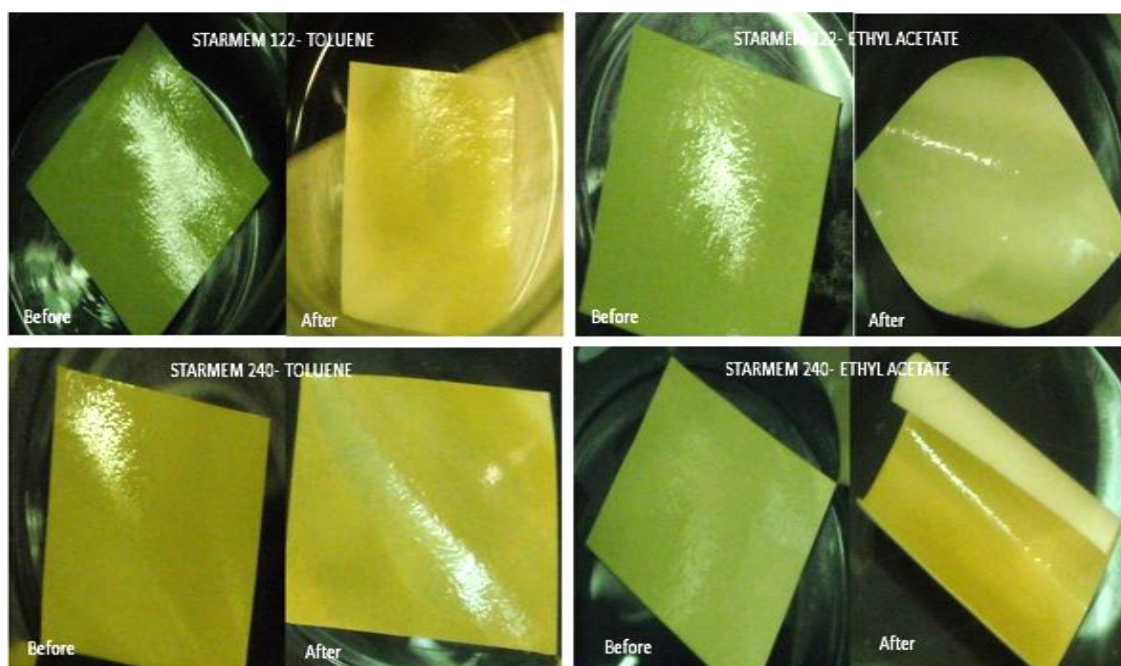

Figure 10: Physical changes of STARMEM^{TM} series after soaking test

The results of FESEM image in Figure 11 until Figure 14 below show the changes in cross-section morphology of the membranes before and after soaking for both membranes in ethyl acetate and toluene respectively. From the image, it is quite difficult to relate the curling effect that happened physically with the result of the images scanned. Inefficient cutting method can be the caused of the image to be difficult to interpret. The difference is observed for all samples comparing to the original image. There might be possibility that these membrane changes its structure after being soaked in to the system due to swelling, membrane dissolution or membrane disintegration as reported by Darvishmanesh *et al.* (2010). It is observed that there are changes in the thickness of the membrane which are not uniform for all membrane. The support layer of the membrane can also be affected by the solvent used.

(a)   (b)

Figure 11: The image of the STARMEM^{TM} 122: a) before soaking in ethyl acetate, b) after soaking in ethyl acetate

(a)   (b)

Figure 12: The image of the STARMEM^{TM} 122: a) before soaking in toluene, b) after soaking in toluene

(a)   (b)
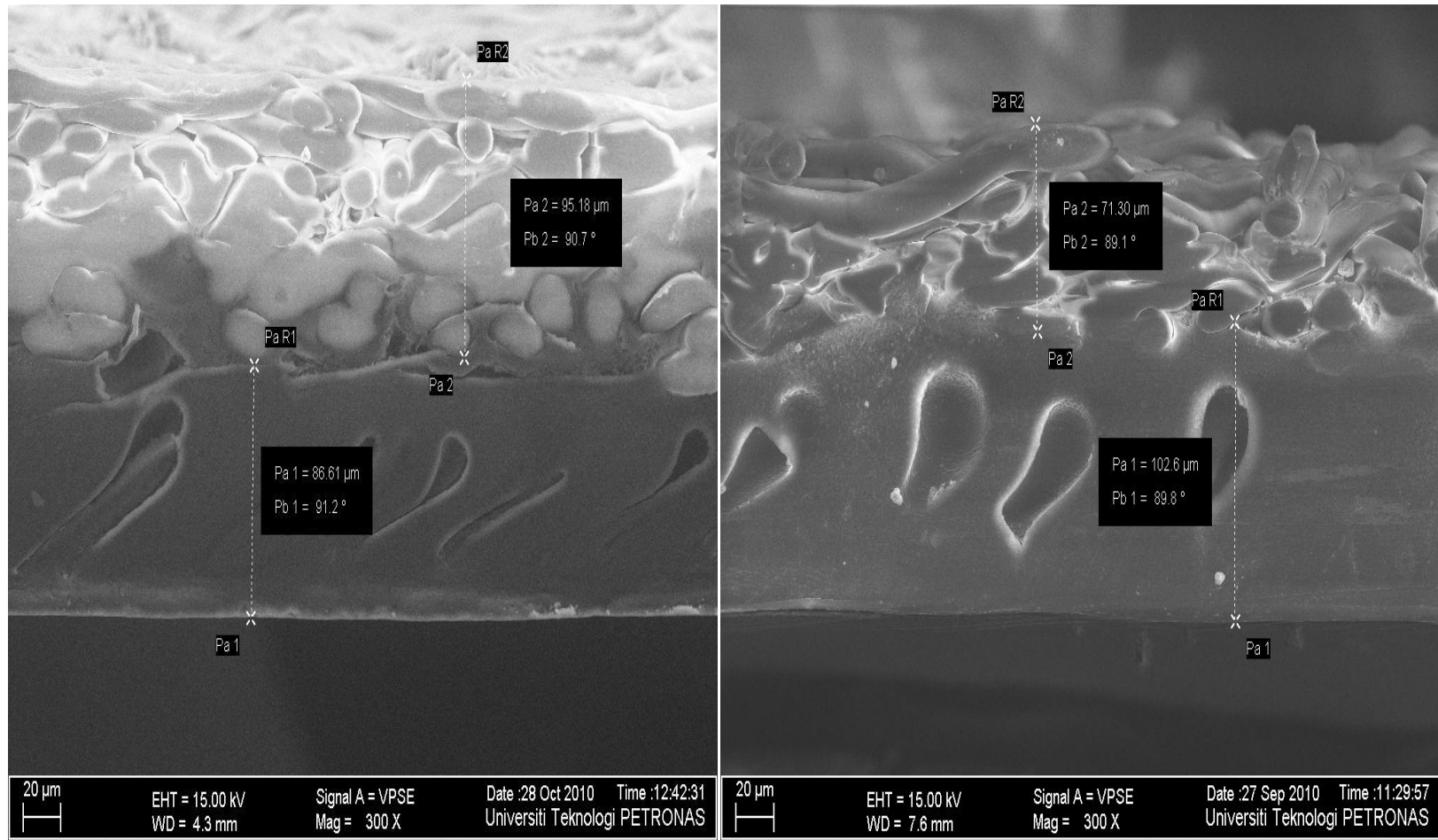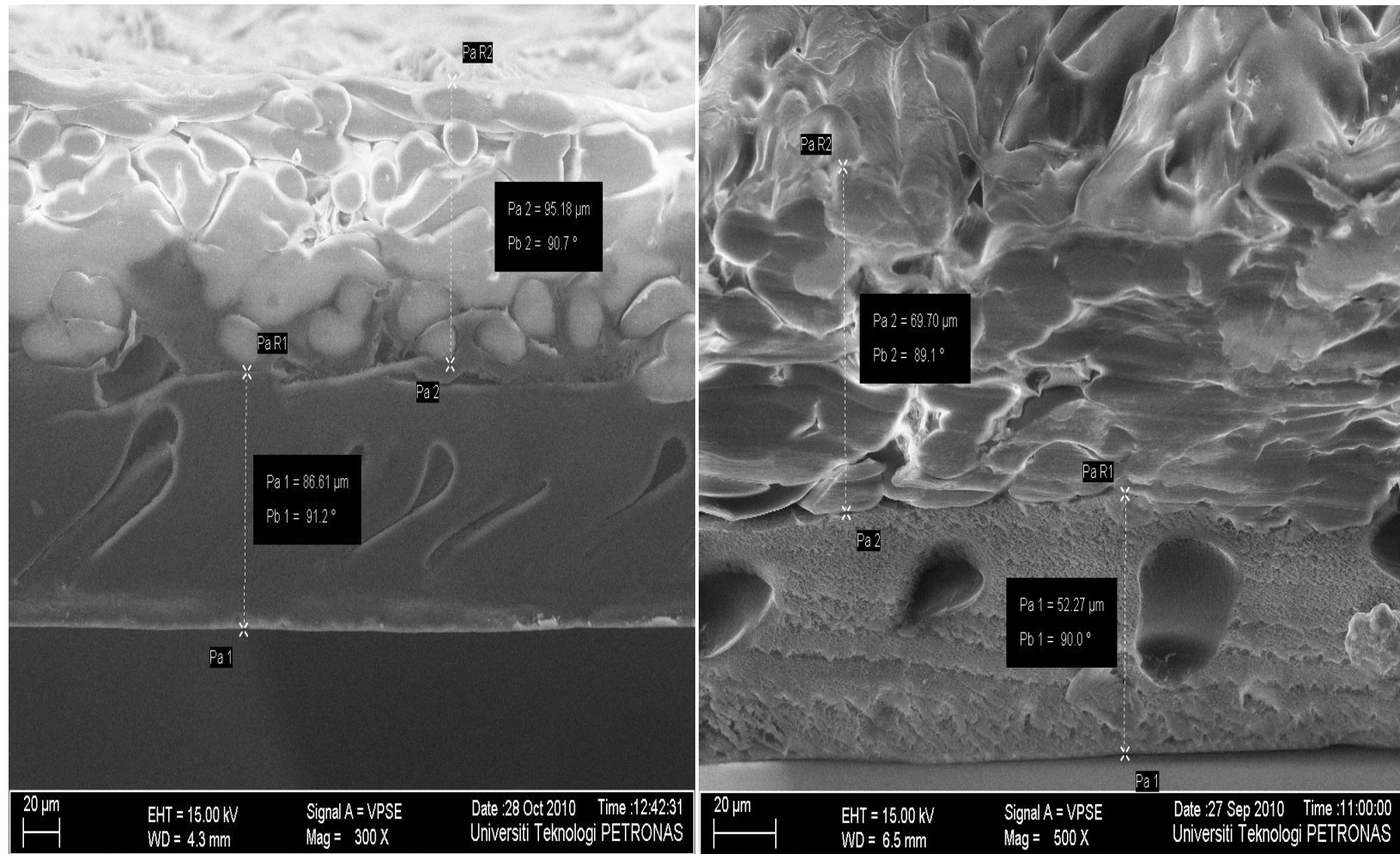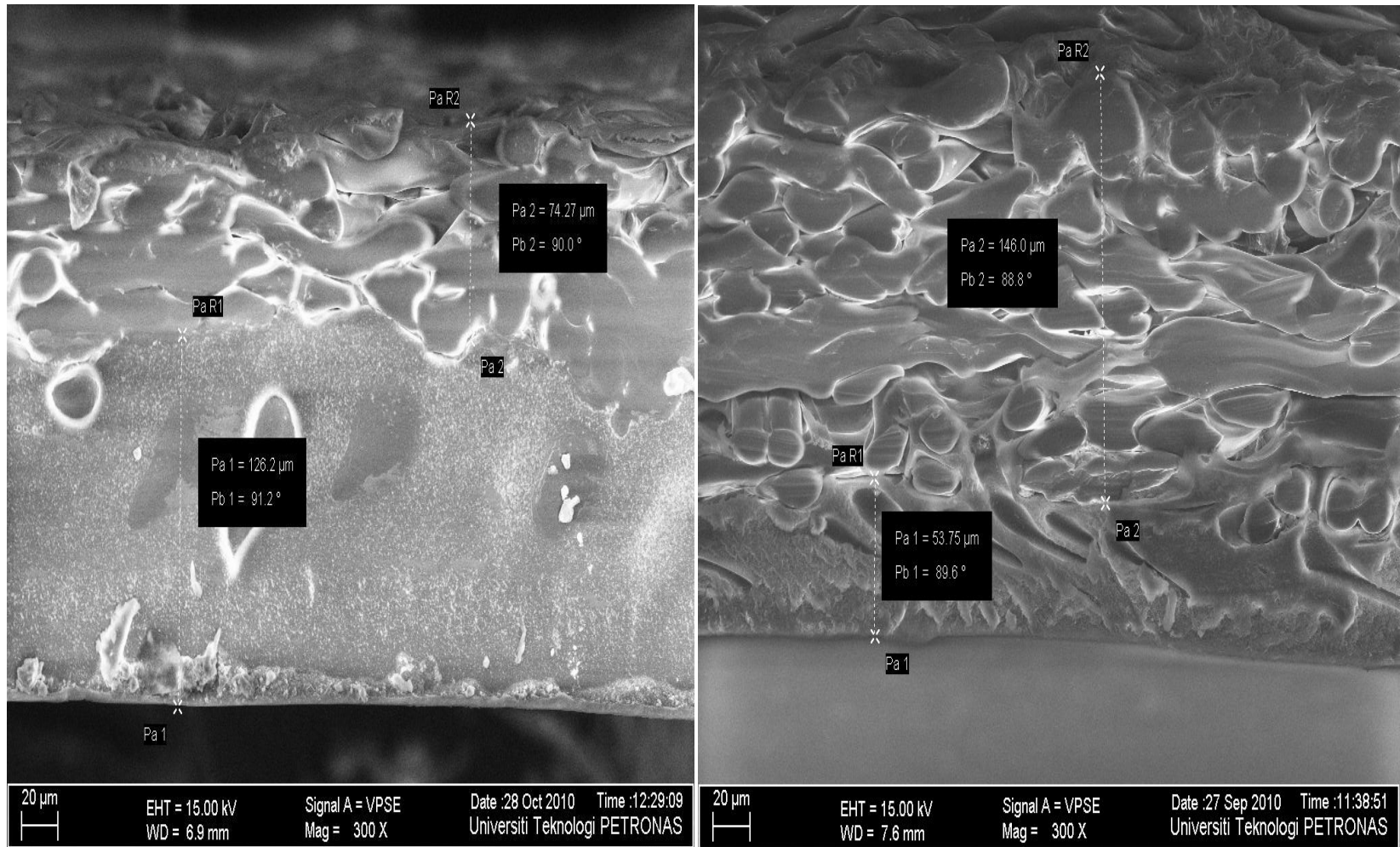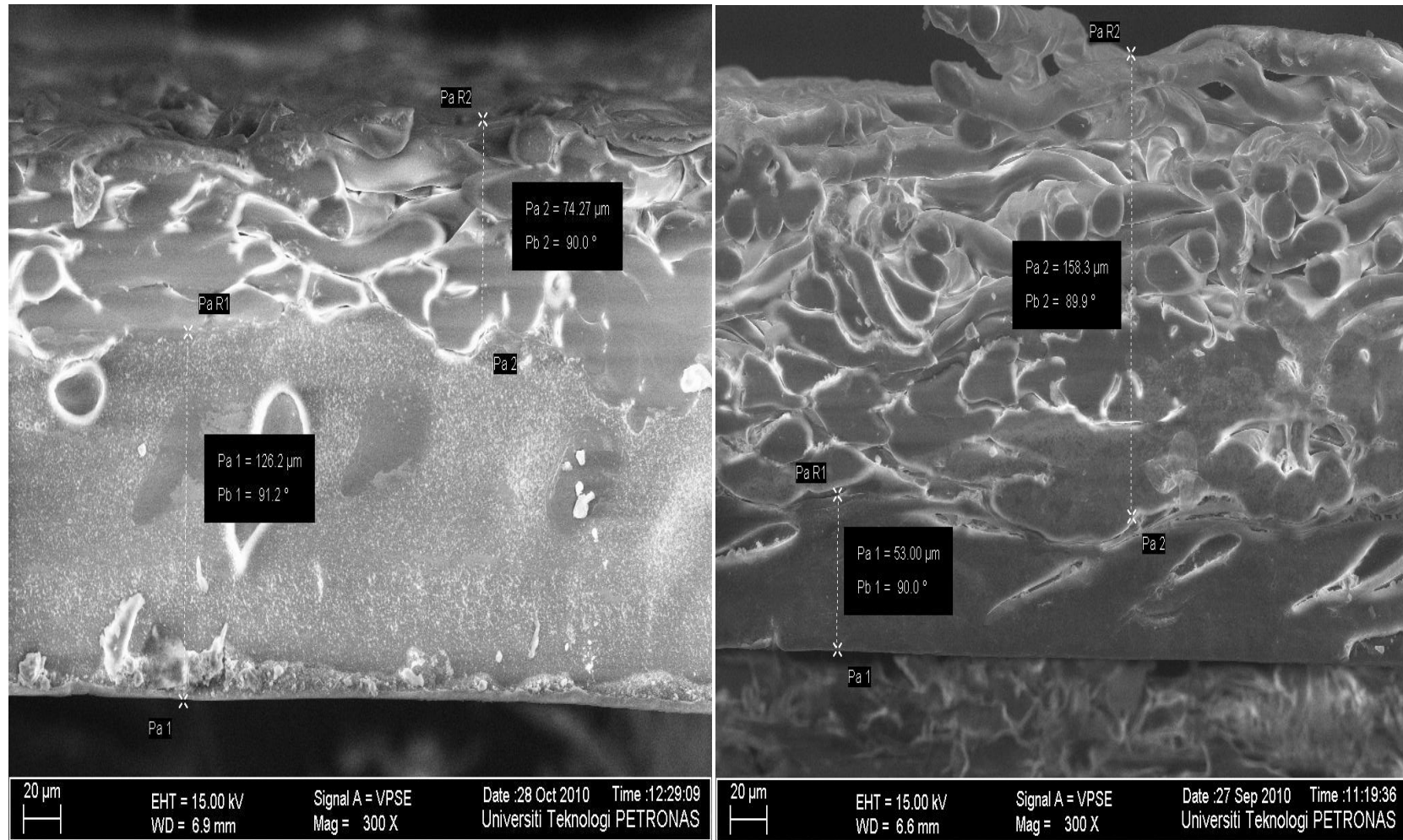
Figure 13: The image of the STARMEM[TM] **240: a) before soaking in ethyl acetate, b) after soaking in ethyl acetate**

Figure 14: The image of the STARMEM^{TM} 240: a) before soaking in toluene, b) after soaking in toluene

4.1 PERMEABILITY OF STARMEM^{TM} MEMBRANES IN TOLUENE

The flux study is conducted for the stable membrane in order to observe for the membrane's ability to permeate the solvent. Solvent used was only toluene since ethyl acetate caused physical changes to the membrane. The results of the experiment are as follows (Figure 15).
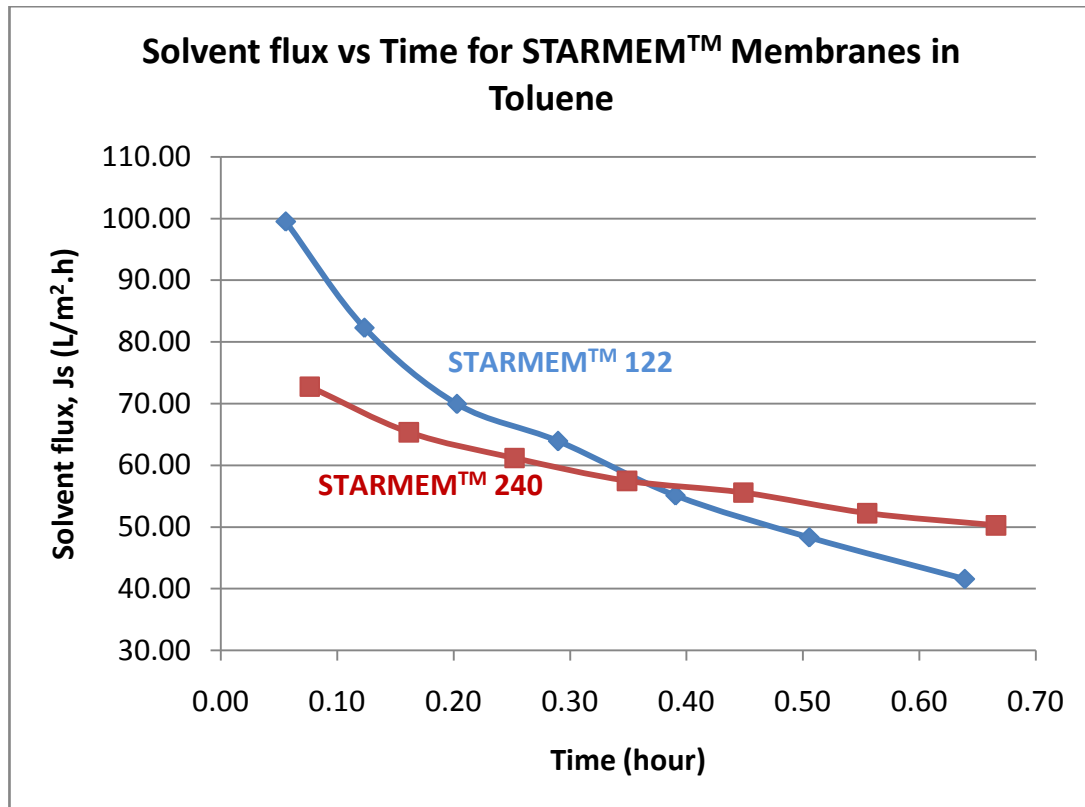
Figure 15: Solvent flux vs. time for STARMEM^{TM} series

After a few runs of experiments, the solvent flux of STARMEM^{TM} 240 was found to be higher than the flux for STARMEM^{TM} 122 which is mentioned by the manufacturer that the molecular weight cut off (MWCO) for STARMEM^{TM} 122 is lower compared to STARMEM^{TM} 240. From the graph, STARMEM^{TM} 240 achieved steady state faster than STARMEM^{TM} 122 in accordance with the slope of the line. The trend of flux for STARMEM^{TM} 122 is quite similar with the one reported by Luthra *et al.* (2002). The flux for STARMEM^{TM} 122 was 39 L/m^2.h which comparable to the value from the

above trend which is 42 L/m^2.h. For STARMEM^{TM} 240, the permeability was 38 L/m^2.h which has significant different with the above value, 50 L/m^2.h.

The pressure used for the filtration can cause the difference in flux value since when operating with high pressure, the time taken to collect the certain value of permeate will be faster. Thus, according to the flux formula, as the time taken is lower, the flux will be higher. In this study, the operating pressure was 20 bar while the operating pressure reported by Luthra was 30 bar. From the manufacturer specification sheet, the permeability of pure toluene at 55 bar for STARMEM^{TM} 122 and STARMEM^{TM} 240 were 30 L/m^2.h and 20 L/m^2.h, respectively which is logical to the assumption made. Zhao *et al.*(2006) did not conduct the permeability test for pure solvent but however even for the aqueous system (ethanol as solvent) the flux of STARMEM^{TM} 122 (320 L/m^2.h) was higher than STARMEM^{TM} 240 (164 L/m^2.h) under the pressure of 30 bar. Darvishmanesh *et al.* (2010) reported that for pure toluene solvent, STARMEM^{TM} 122 gives lower flux (35 L/m^2.h) than STARMEM^{TM} 240 (60 L/m^2.h) which is the same with the result for this experiment in terms of the flux trending.

The MWCO is not useful in predicting the permeability of the membrane in the organic solvent system as it does in aqueous system. As for the significant different of flux value for this experiment compared to the other literature, one of the possible reason is the membrane pre-conditioning. Different method of pre-conditioning the membrane prior to testing can contribute to the different value of flux (Yang *et al.*, 2001). It is assumed that no pre-conditioning or pre-treatment is conducted by the manufacturer before obtaining the flux of pure toluene since it was not specified in the sheet. While for Luthra *et al.* (2002), pure toluene was passed through the membrane once to allow the benchmarking of toluene flux for the membrane. As for this experiment, the membrane was immersed in the solvent for 2 hours before washing it with pure toluene (250 ml) as the procedure of membrane pre-conditioning. The flux did not able to reach better steady state condition as the supply of solvent was in the batch mode. The experiment need to be stopped after the cell has permeated the maximum volume. Depressurizing

and opening the unit to add the solvent to continue the process caused the flux to be unstable.

Flux decrease was found to be common for permeability of pure solvent study in all literature. The main explanation for this phenomenon could be the membrane compaction under high pressure. It is found that for all NF membrane, there will be a significant or slight decrease of flux at the early stage followed by gradual decrease of flux (Luthra *et al.*, 2002). Organophilic membrane tends to swell depending on the solvent and process condition. As the pressure is applied to membranes, the swollen structure is compressed which results in the membrane to be compacted. The reversibility and irreversibility of the membrane compaction depend on the mechanical properties of the membrane (Priske *et al.*, 2010). Compaction of membrane could alter the pore structure to be tighter which consequently reduced the solvent flux. According to Silva *et al.* (2005), the membrane compaction is a slow process, so the data taken from dead-end cell with limited period of time ranging from 1 to 3 hours may not be accurate to predict the stable solvent flux. Therefore, limited time of permeability test of pure solvent is also a plausible explanation for the flux to be at higher value for this experiment.

4.1.1   Solvent flux study for different pressure

For the solvent study of pressure variation in catalyst solution, the flux of the catalyst solution was found to be lower compared to the pure solvent as the pore of the membrane can be blocked by the catalyst which indirectly causes the flux value to be lower. From Figure 16, it is obvious that as the pressure of the system is increased, the flux value also increases. This trend is supported by the Scarpello *et al.* (2002) finding which also had a proportional relationship for solvent flux with pressure. Shi *et al.* (2006) stated that nanofiltration is a pressure driven physical process. So, pressure boost is effective in increasing the flux.
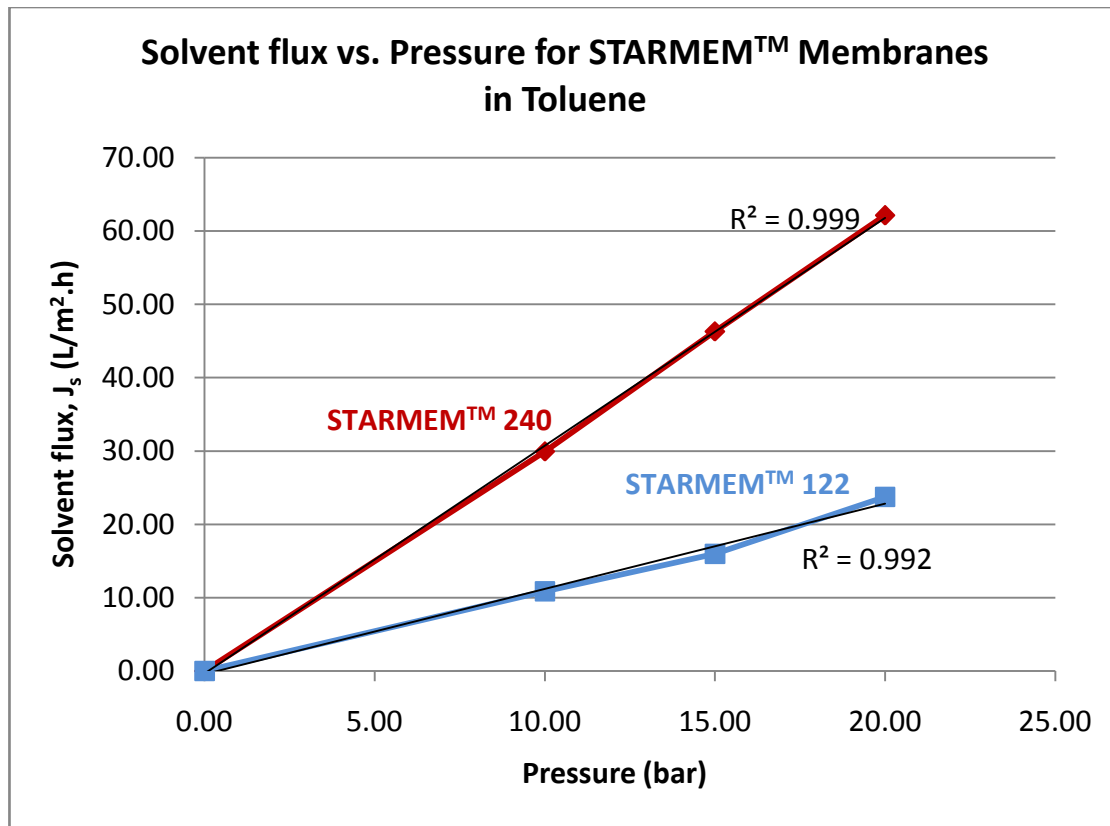
Figure 16: Solvent flux vs. pressure for STARMEM^{TM} series

4.1.2 Solvent flux study for different concentration

For concentration variation, Figure 17 shows that increasing the initial concentration of the solution will reduce the solvent flux. Scarpello *et al.* (2002) and Shi *et al.* (2006) confirmed that the increase in initial concentration caused a reduction in flux value. The increase in surface or pore fouling contributes to the decreasing of flux. In addition, concentration polarization can also be the cause of the flux reduction over concentration. The concentration polarization could create a layer at the membrane-surface interface. The concentration of solute within that layer is higher compared to the bulk of the solution. The layer holds up the movement of component through the membrane since the increase of osmotic pressure across the membrane reduces the driving force of mass transfer. Consequently, the permeability is reduced.
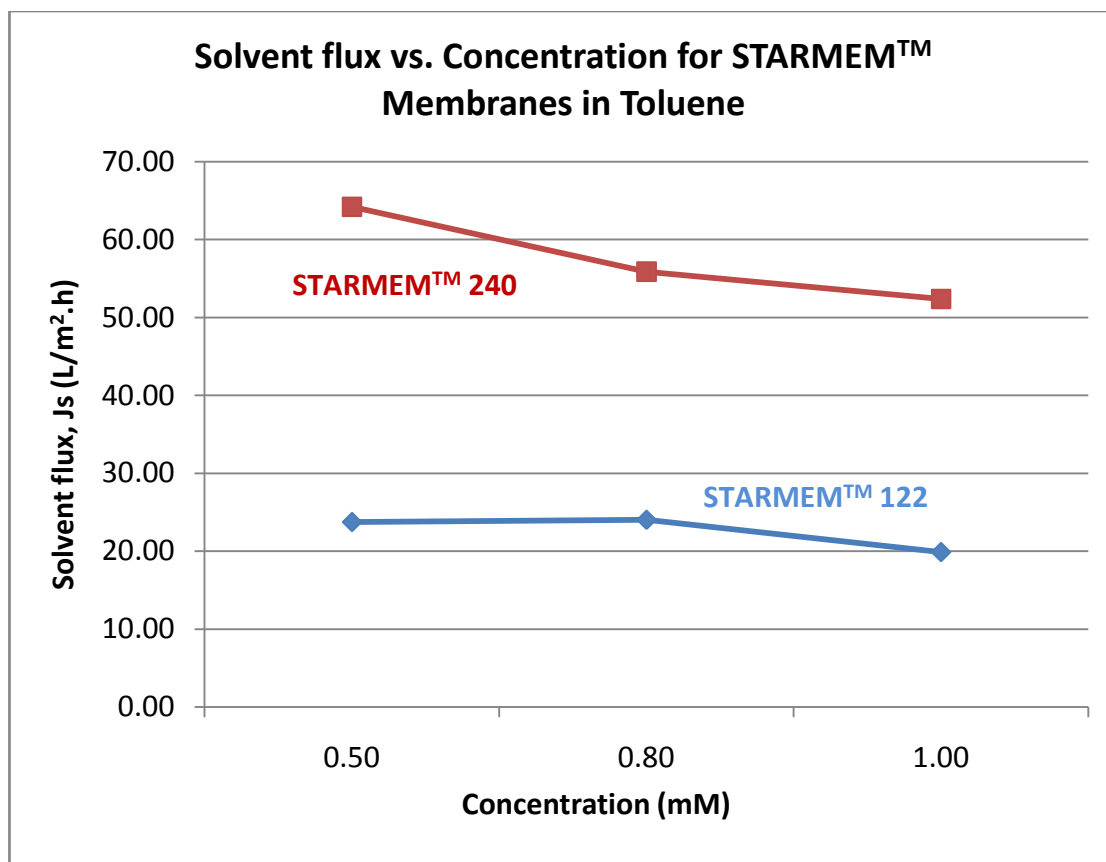
Figure 17: Solvent flux vs. concentration for STARMEM^{TM} series

4.2   REJECTION OF STARMEM^{TM} MEMBRANES IN TOLUENE

The rejection value for STARMEM^{TM} 240 was higher compared to STARMEM^{TM} 122 for both, pressure variation and also concentration variation study. STARMEM^{TM} 240 is reported to have less rejection of Wilkinson catalyst (which is also one of the Rh-based catalysts) compared to STARMEM^{TM} 122 by Scarpello *et al.* (2002). However for this experiment, as shown in Figure 18 and Figure 19, the result is the opposite. From STARMEM^{TM} 240, when the pressure increases, the rejection also slightly increases. Scarpello et al reported that one of the plausible reasons is due to the membrane compaction that is partially reversible which occurs on the active layer of the membrane. The pore of the membrane is tightened due to the increase in pressure. Therefore it prevents the catalyst particles to pass through. For the concentration part, as the concentration is increases, there will be an increase pore fouling phenomena which cause the rejection to be higher.

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For STARMEM^{TM} 122, the result was unstable. The instability of the result may caused by the membrane structure problem. There might be possibilities that the membrane may break during the experiment which causes the separation to be lower. Membrane break is usually due to the handling of the unit such as applying pressure abruptly to the system. It can be concluded that the pore of STARMEM^{TM} 122 may need extra care compared to STARMEM^{TM} 240. Since the pore is smaller, according to their molecular weight cut off, pressure applied can result in the membrane pore to be damaged.
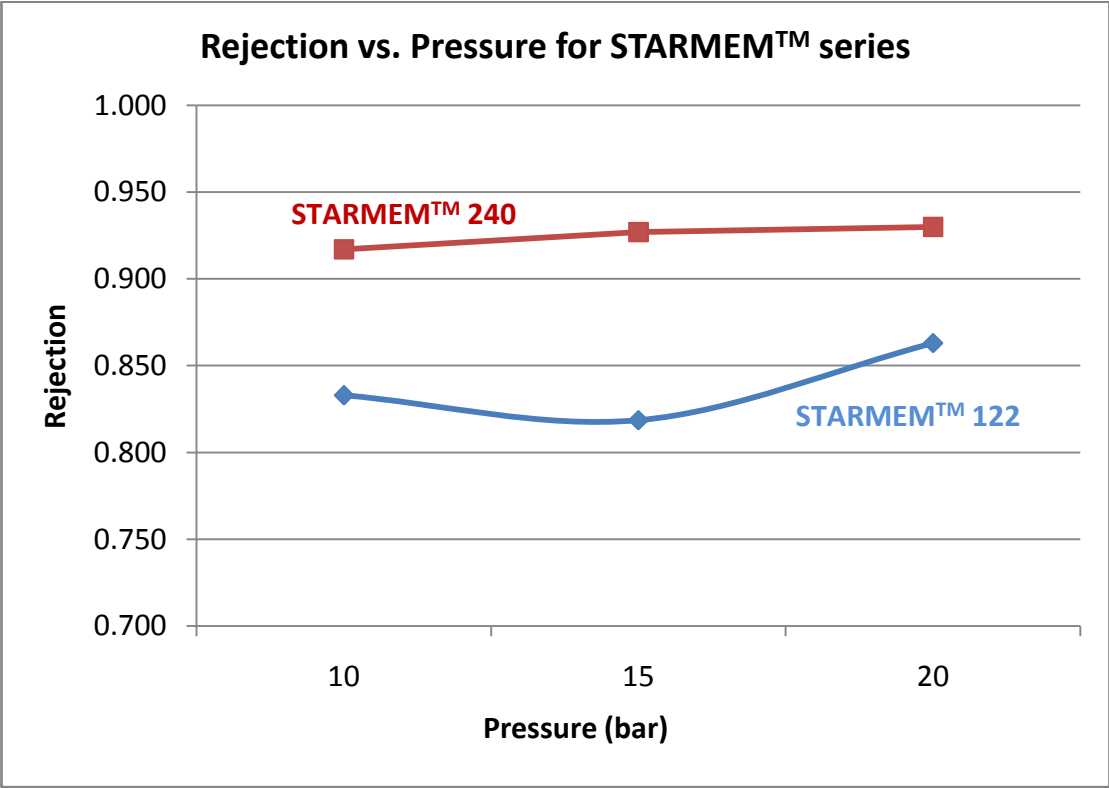

Figure 18: Rejection vs. pressure for STARMEM^{TM} series
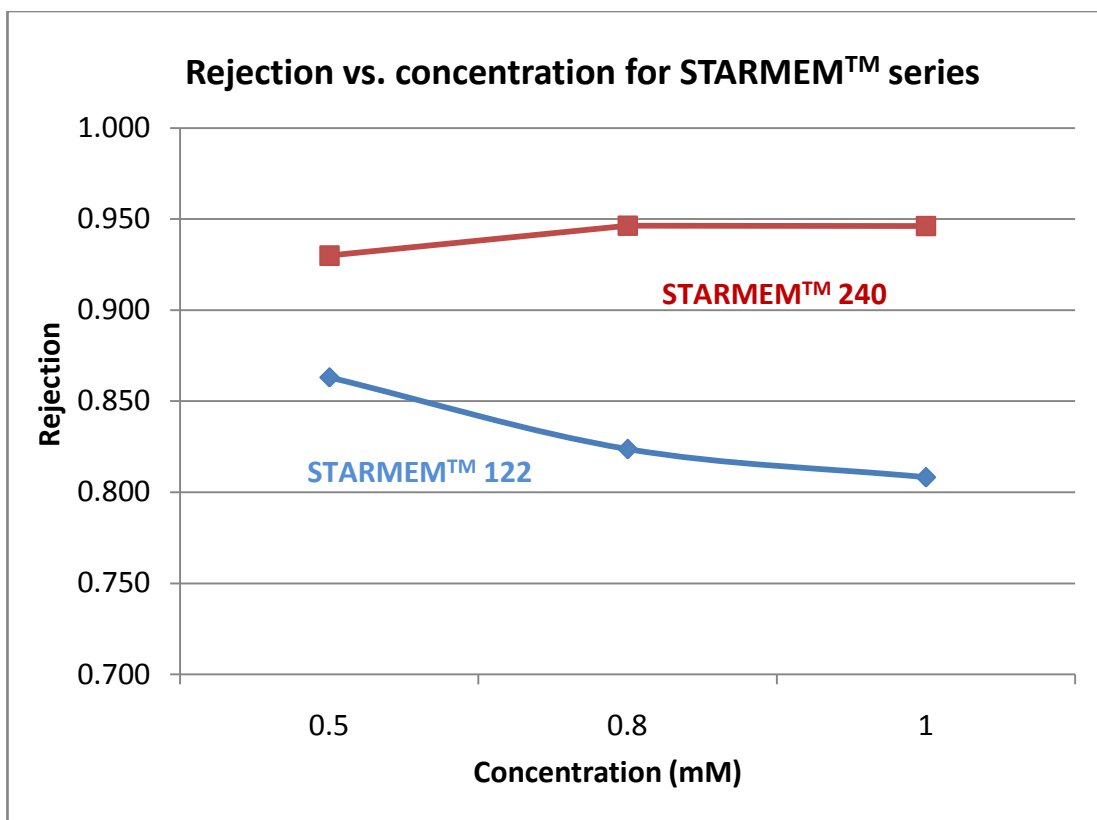
Figure 19: Rejection vs. concentration for STARMEM^{TM} series

UV-visible spectrophotometer analysis shows the absorbance at low wavelength for the solvent is unstable as absorbance peaks are observed at many points as shown in Appendix 2. The concentration determination can be affected by the instability of absorbance which leads to various trending obtained for rejection study.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

From the research, nanofiltration is proven to be able to separate the $HRh(CO)(PPh)_3$ catalyst from a homogeneous system without having to cause catalyst degradation which usually occurs at high temperature. The compatibility of membrane with the catalyst and solvent is the utmost important step before filtration process as the membrane can reacts with the catalyst and solvent used which may cause it to be less effective for separation. Ethyl acetate was found to be not suitable for STARMEM^{TM} 122 and STARMEM^{TM} 240 as it shows physical changes even before the experiment started.

Changes are observed from FESEM image for the cross section of membrane before and after soaking test. Thus, it is proven that immersing the membrane into solvent does give an impact to the membrane structure. Since the morphology is observed before the membranes undergo the filtration process, further analysis can be conducted by observing the membrane structure changes using FESEM unit after completing the filtration process.

The flux of the pure solvent needs a longer time to be at steady state condition. When a membrane is not permeating at its steady flux condition, the value of flux will not have a good trend. The filtration unit for this research is in batch mode. The solvent permeates at high rate which makes the volume of solution inside the cell unit runs out fast which is lesser than one hour. Opening the seal to refill the cell with the solvent might affect the membrane as it is being pressurize and depressurize frequently. Thus, a continuous mode of supplying solvent to the cell can be helpful to reach steady flux condition. Increase in pressure results in increasing flux proportionally. Concentration variation does not produce a similar pattern for flux value. However, it can be seen that the flux decreases with increasing concentration.

The rejection of HRh(CO)(PPh)$_3$ catalyst for pressure and concentration variation is found to be high which is from 0.8 to 0.95. However, the literature reported the value to be almost reaching 1 involving Wilkinson catalyst, which is also one of rhodium based catalyst. The rejection at different pressure and concentration does not produce a comparable trend for this research. Since the result might be affected by absorbance instability during the UV-visible spectrophotometer analysis, alternative method for concentration analysis can be conducted by using Atomic Absorption Spectroscopy (AAS) which analyzes the concentration of specific metal element in sample.

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APPENDICES

Appendix 1: Experimental Raw Data

Permeability test result for STARMEM^{TM} membranes

Membrane-solvent combination	Volume (L)		Time (h)		Solvent flux, J_s (L/m^2.h)
STARMEM^{TM} 122	0.00	0.00	0.00	0.00000	0.00
	0.03	0.03	0.06	0.05583	99.50
	0.06	0.03	0.12	0.06750	82.30
	0.09	0.03	0.20	0.07944	69.93
	0.12	0.03	0.29	0.08694	63.90
	0.15	0.03	0.39	0.10083	55.10
	0.18	0.03	0.51	0.11500	48.31
	0.21	0.03	0.64	0.13361	41.58
STARMEM 240	0.00	0.00	0.00	0.00000	0.00
	0.03	0.03	0.08	0.07639	72.73
	0.06	0.03	0.16	0.08500	65.36
	0.09	0.03	0.25	0.09083	61.16
	0.12	0.03	0.35	0.09667	57.47
	0.15	0.03	0.45	0.10000	55.56
	0.18	0.03	0.56	0.10639	52.22
	0.21	0.03	0.67	0.11056	50.25

Solvent flux result for pressure variation experiment

Membrane-solvent	Pressure (bar)	Volume (L)	Time (h)	Solvent flux, J_s (L/m^2.h)
STARMEM^{TM} 122-Toluene	0.00	0.00	0.00	0.00
	10.00	0.02	0.26	10.88
	15.00	0.02	0.17	15.97
	20.00	0.02	0.12	23.75
STARMEM^{TM} 240-Toluene	0.00	0.00	0.00	0.00
	10.00	0.02	0.09	29.94
	15.00	0.02	0.06	46.30
	20.00	0.02	0.04	62.11

Solvent flux result for concentration variation experiment

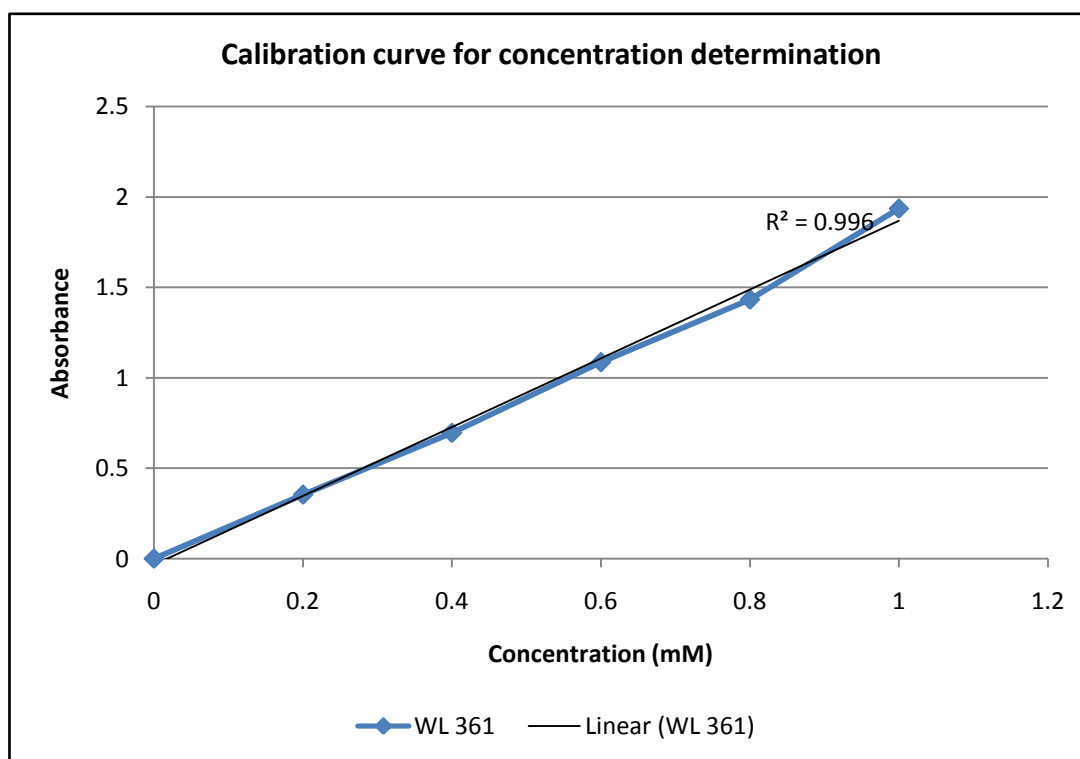| Membrane-solvent	Concentration (mM)	Volume (L)	Time (h)	Solvent flux, J_s (L/m^2.h)
STARMEM^{TM} 122-Toluene	0.50	0.02	0.12	23.75
	0.80	0.02	0.12	24.04
	1.00	0.02	0.14	19.88
STARMEM^{TM} 240-Toluene	0.50	0.02	0.04	64.18
	0.80	0.02	0.05	55.87
	1.00	0.02	0.05	52.36

Appendix 2: Raw Data for UV-Visible Spectrophotometric Analysis
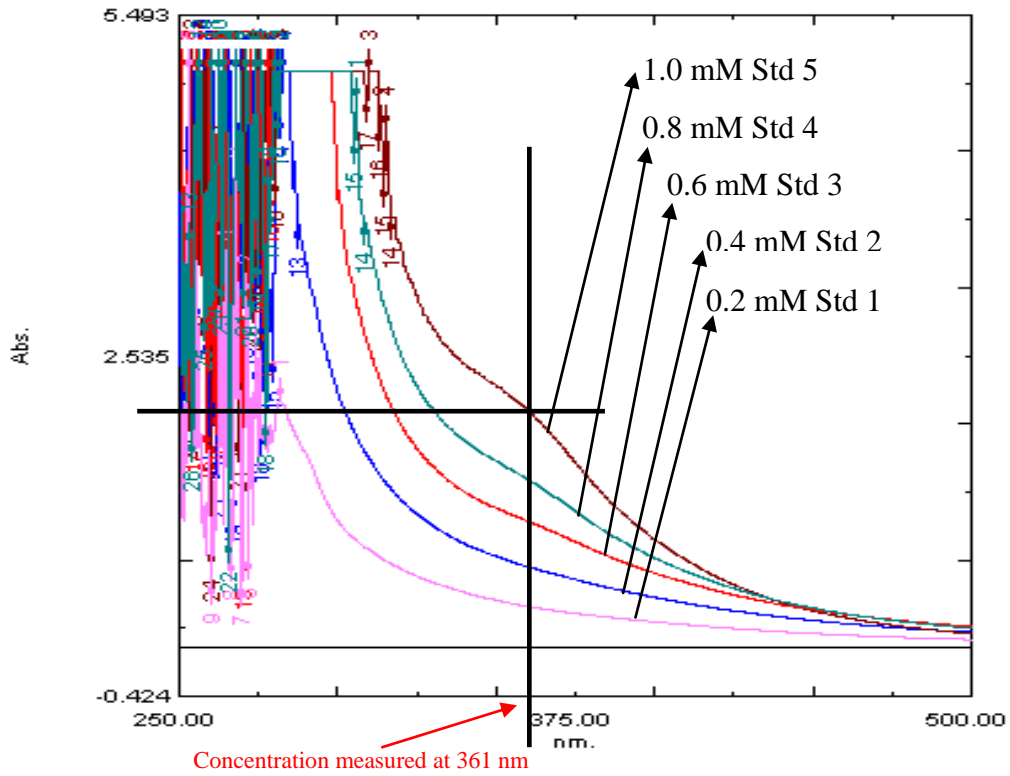
Standard Solution for UV-Visible analysis

Standard Solution	Concentration (mM)	Absorbance at 361 nm
Std 0	0	0
Std 1	0.2	0.354
Std 2	0.4	0.695
Std 3	0.6	1.086
Std 4	0.8	1.432
Std 5	1	1.935

* Standard solution is rhodium catalyst solution in toluene

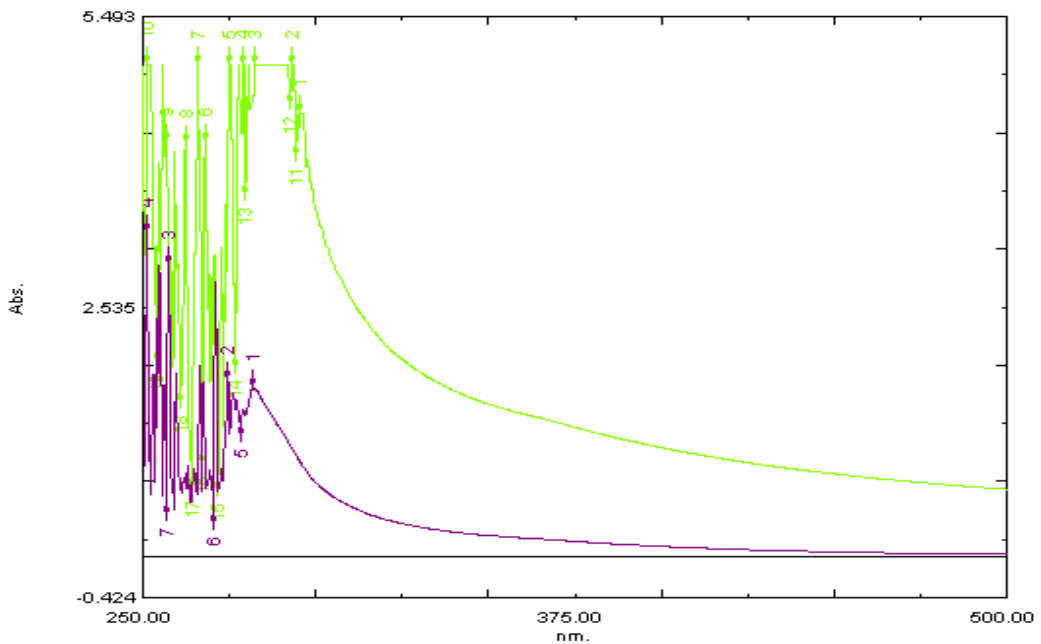## Calibration Curve for Concentration Determination using UV-Vis Spectrophotometer
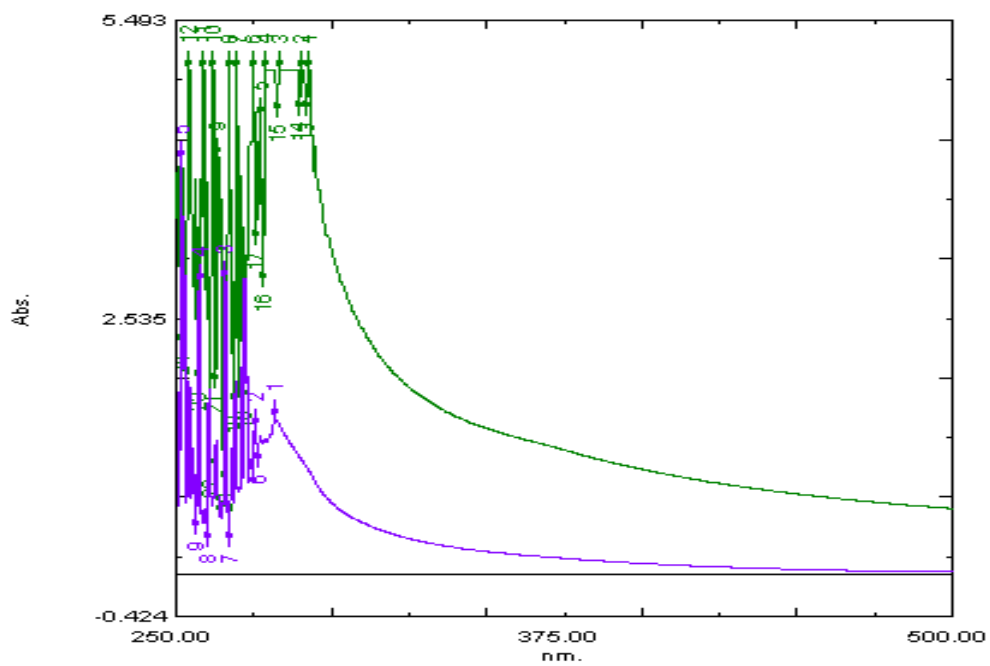
Absorbance vs. Wavelength for Standard Solution

1.0 mM Std 5

0.8 mM Std 4

0.6 mM Std 3

0.4 mM Std 2

0.2 mM Std 1

Concentration measured at 361 nm

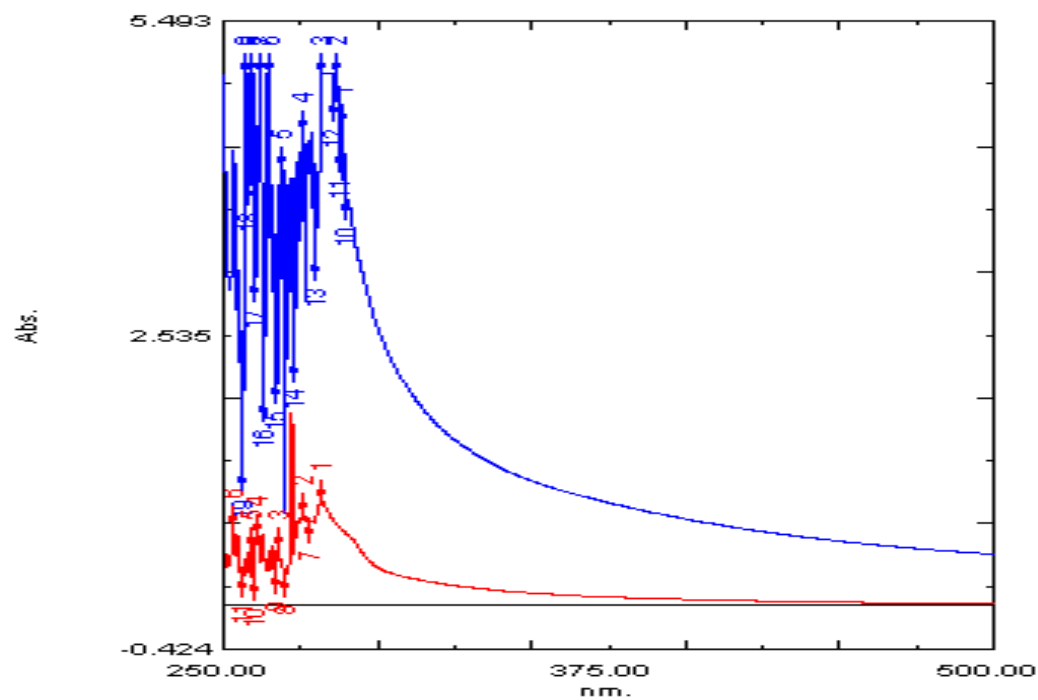## Absorbance graph for STARMEM^{TM} 122
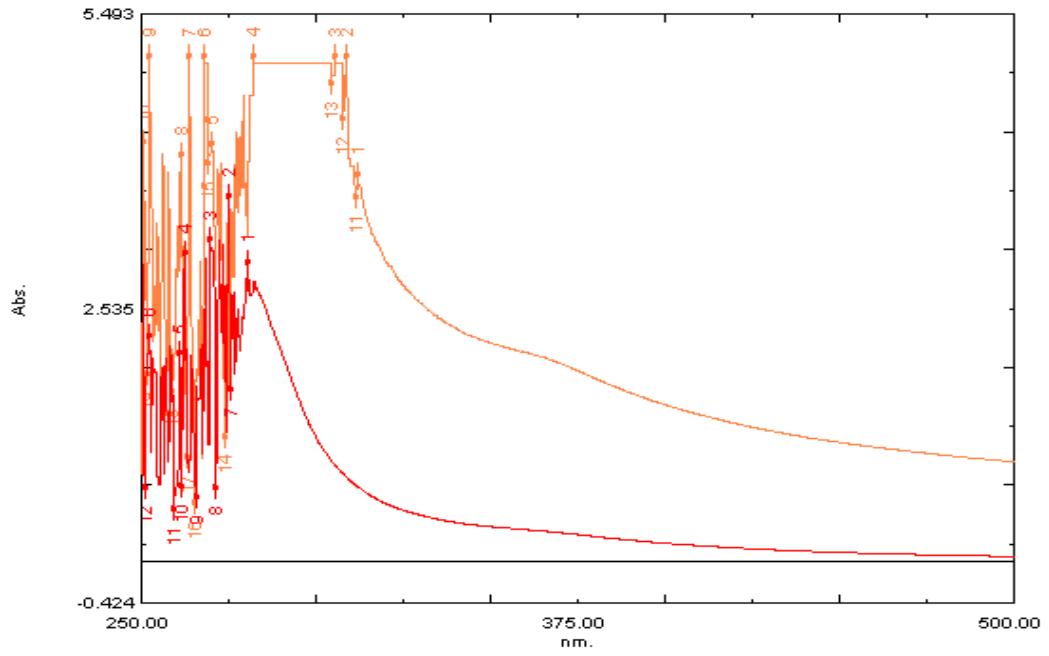
Pressure = 10 bar at 0.5 mM
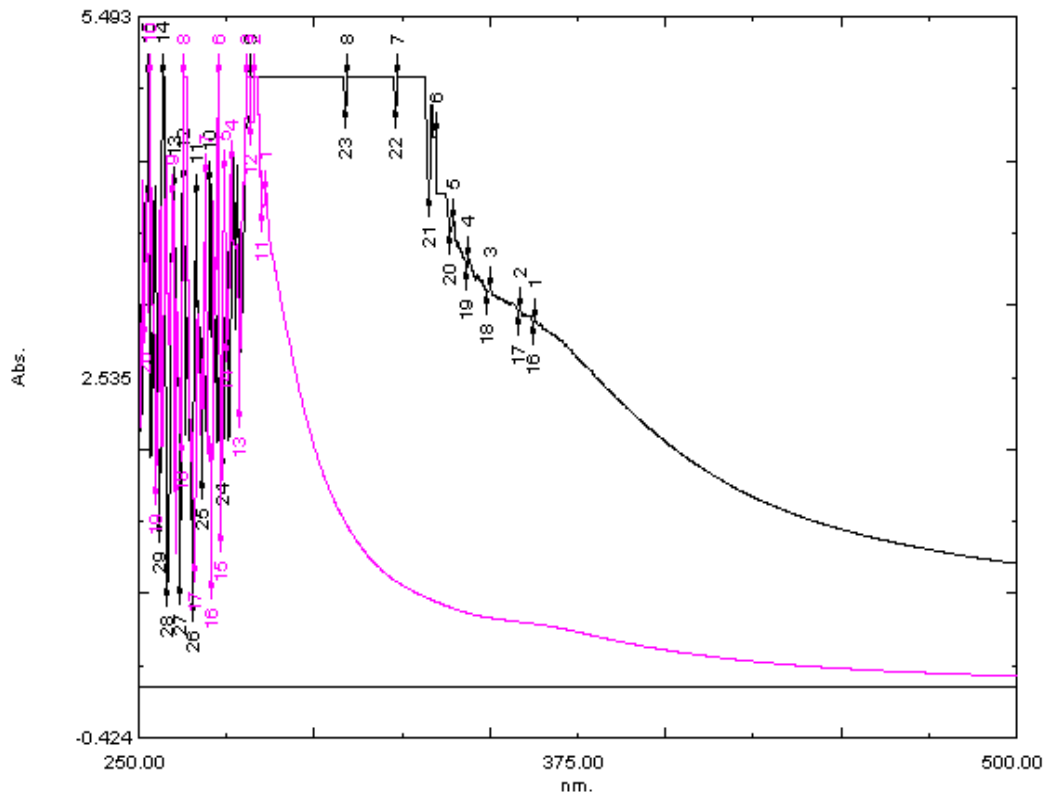
Pressure = 15 bar at 0.5 mM

Pressure = 20 bar at 0.5 mM

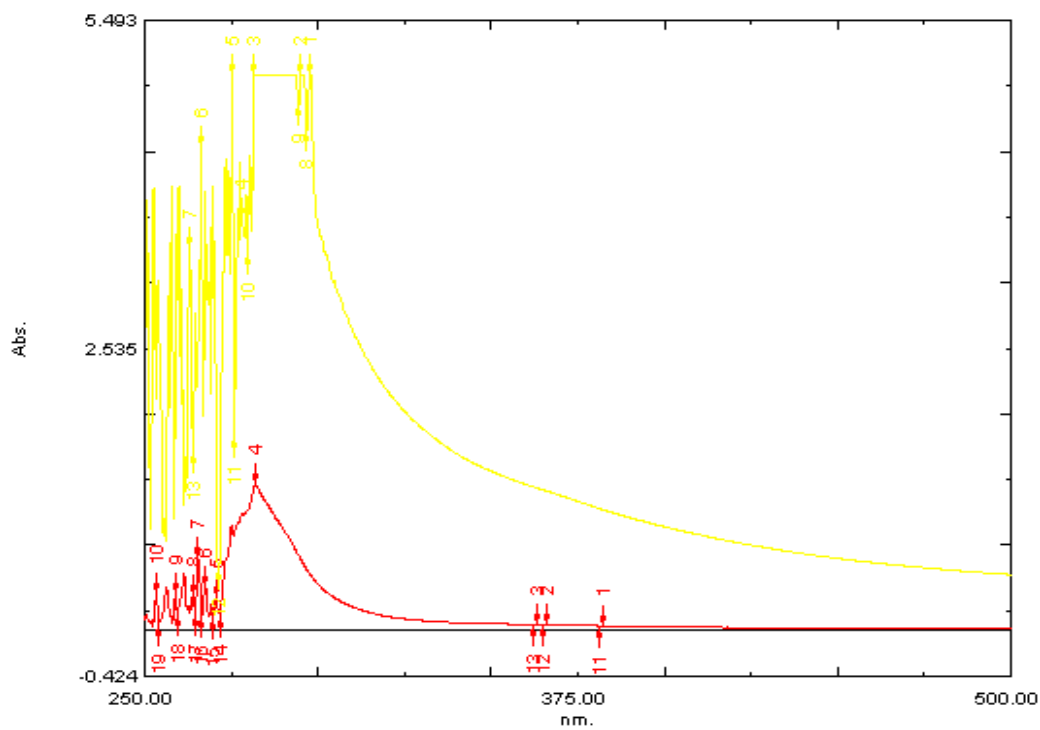**Concentration = 0.8 mM at 20 bar**

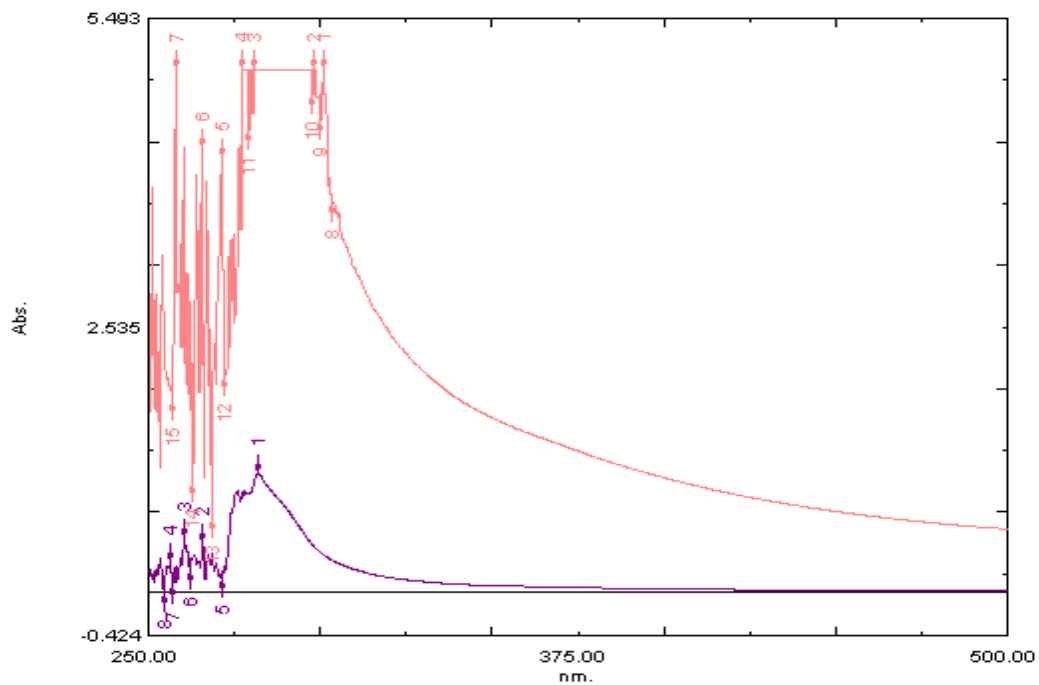**Concentration = 1.0 mM at 20 bar**

Absorbance graph for STARMEM^{TM} 240
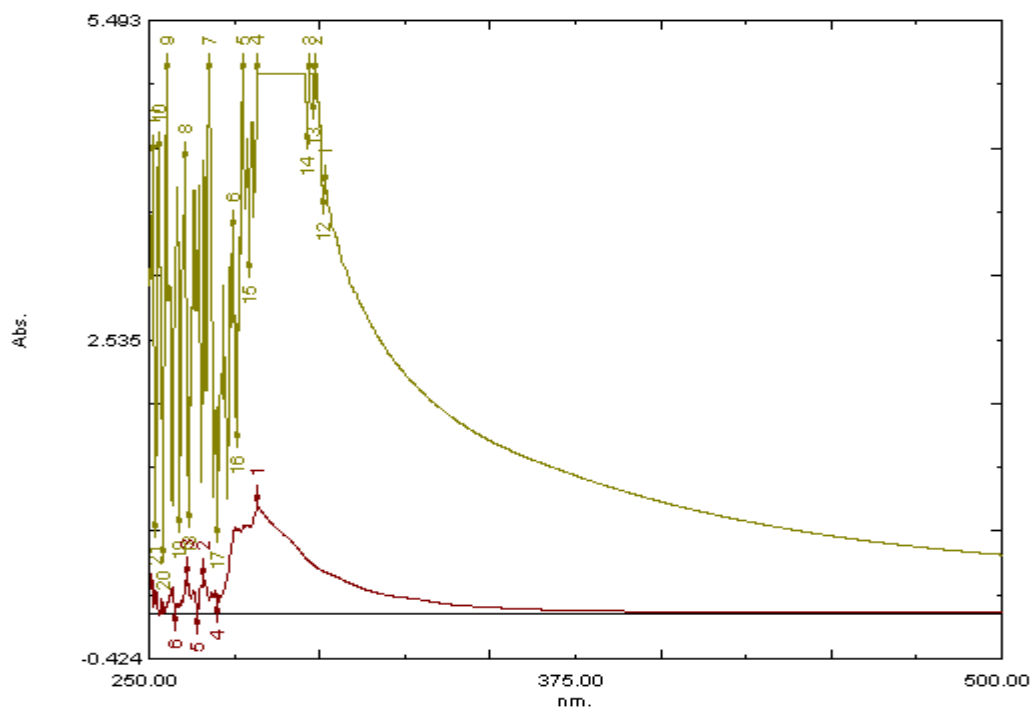
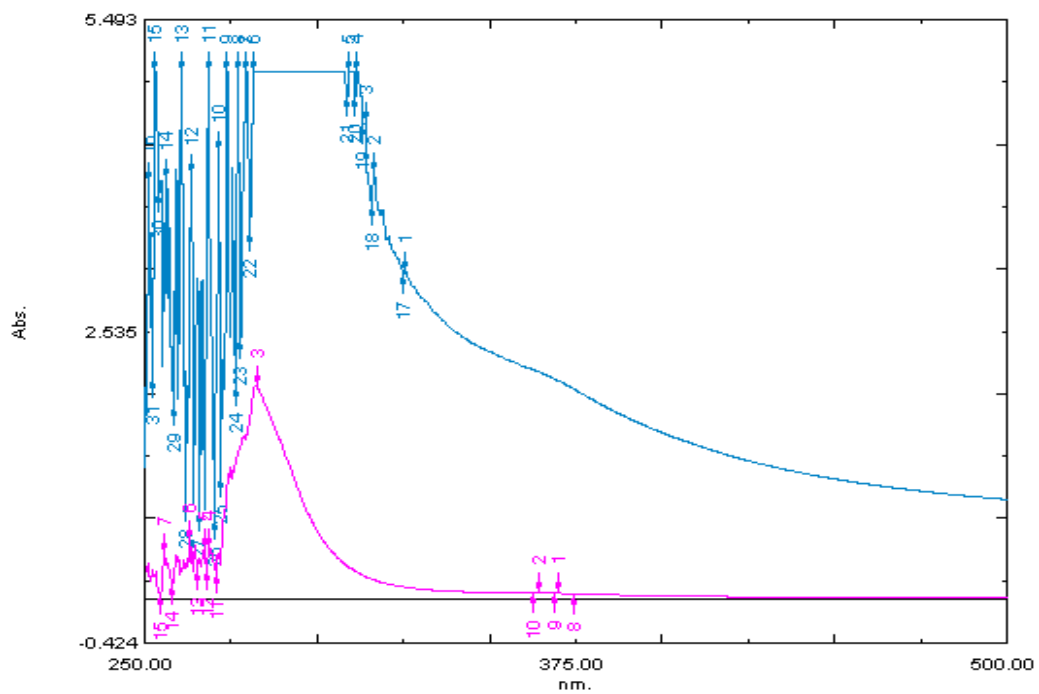## Pressure = 10 bar at 0.5 mM

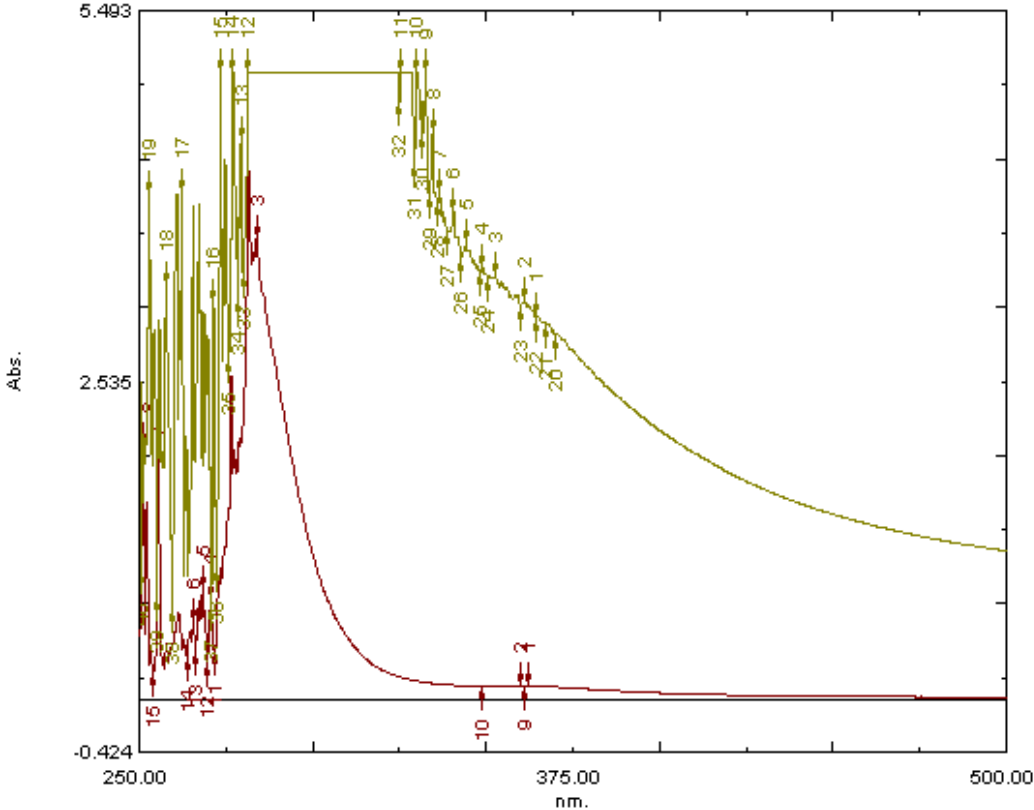## Pressure = 15 bar at 0.5 mM

Pressure = 20 bar at 0.5 mM

Concentration = 0.8 mM at 20 bar

Concentration = 1.0 mM at 20 bar

* **The higher line is for retentate and the lower line is for permeate concentration for all graphs.**

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Result of concentration of permeate and retentate solution using UV-Vis spectrophotometer

Membrane	Sample ID	Concentration	WL 361.0
STARMEM™ 122	S122_20bar_Permeate	0.079	0.086
	S122_20bar_Retentate	0.577	1.056
	S122_15bar_P	0.131	0.186
	S122_15bar_R	0.722	1.338
	S122_10bar_P	0.128	0.181
	S122_10bar_R	0.766	0.766
	S122_0.5mM_P	0.079	0.086
	S122_0.5mM_R	0.577	1.056
	S122_0.8mM_P	0.194	0.309
	S122_0.8mM_R	1.1	2.074
	S122_1.0mM_P	0.3	0.516
	S122_1.0mM_R	1.564	2.979

Membrane	Sample ID	Concentration	WL 361.0
STARMEM™ 240	S240_20bar_Permeate	0.054	0.036
	S240_20bar_Retentate	0.771	1.434
	S240_15bar_P	0.059	0.046
	S240_15bar_R	0.808	1.505
	S240_10bar_P	0.058	0.044
	S240_10bar_R	0.699	1.293
	S240_0.5mM_P	0.054	0.036
	S240_0.5mM_R	0.771	1.434
	S240_0.8mM_P	0.062	0.052
	S240_0.8mM_R	1.156	2.184
	S240_1.0mM_P	0.087	0.101
	S240_1.0mM_R	1.618	3.084

Rejection result for STARMEM^{TM} membranes

Membrane	Parameter	Rejection
STARMEM^{TM}122 – toluene	Pressure (bar)	
	10	0.833
	15	0.819
	20	0.863
	Concentration (mM)	
	0.5	0.863
	0.8	0.824
	1	0.808
STARMEM^{TM}240 - toluene	Pressure (bar)	
	10	0.917
	15	0.927
	20	0.930
	Concentration (mM)	
	0.5	0.930
	0.8	0.946
	1	0.946

Appendix 3: Sample of Calculations

Calculation of solvent flux

For combination of STARMEM$^{\text{TM}}$ 122 – toluene,

Volume of solution, V= 0.03 L

Time taken, t = 0.06 hr

Active membrane surface area, A_s = 0.0054 m^2

Solvent flux is calculated by the formula:

$$J_s = \frac{V}{A_s t}$$

$$J_s = \frac{0.03}{0.0054 \, (0.06\,)}$$

$$J_s = 99.50 \, m^2$$

Calculation of mass of catalyst for solution preparation

To prepare 100 ml of 1.0mM solution:

$$n = \frac{MV}{1000}$$

Where   n = mole of catalyst, g/gmol

M= concentration of solution, mM

V = volume of solution, ml

$$n = \frac{1.0(100)}{1000}$$

$$n = 1 \, x \, 10^{-4} \, mole$$

Mass of catalyst = n x MW

Where   MW = molecular of catalyst = 918.79

Mass of catalyst $= (1 \times 10^{-4}) \times 918.79$

                        $= 0.0919$ grams

Calculation for preparation of standard solution

Standard solution is made by preparing 100 ml of 1mM of rhodium catalyst solution and is diluted to get the concentration of solution at desired value. Solution is prepared 5ml for each concentration.

For 0.2 mM,

$$M_1V_1 = M_2V_2$$

$$(1 \times 10^{-4}) \; V_1 = (0.2 \times 10^{-3})(5)$$

$$V_1 = 1 \; ml$$

1ml of the 1.0mM solution needs to be diluted in 4 ml of pure toluene to get the solution at 0.2mM concentration.

The calculation for remaining concentration uses the same formula. The volume of 1.0mM solution need is as follows:

Concentration (mM)	V_1 (ml)	Volume of pure toluene (ml)
0.4	2	3
0.6	3	2
0.8	5	1
1.0	5	0

Calculation for rejection of catalyst

The rejection, r is calculated by the formula:

$$r = 1 - \frac{C_p}{C_r}$$

Where C_p = concentration of permeate from uv-visible analysis, mM

C_r = concentration of retentate from uv-visible analysis, mM

Taking for rejection of catalyst through STARMEM^{TM} 122 at 20 bar at concentration of 0.5mM,

C_p = 0.079 mM

C_r = 0.577 mM

$$r = 1 - \frac{C_p}{C_r}$$

$$r = 1 - \frac{0.079}{0.577}$$

$$r = 0.863$$

Appendix 4: Specifications Sheet for STARMEM[TM] Series

EVONIK INDUSTRIES

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uop
A Honeywell Company

UOP's Organic Solvent Nanofiltration Membranes - STARMEM™ Series

STARMEM™ 122; STARMEM™ 240

Specifications Sheet

Membrane Material	Polyimide
Flat Sheets	210 x 297 mm
Modules	Spiral Wound (2.5" x 12"; 2.5" x 40"; 4" x 40"; 8" x 40")
Maximum Pressure	60 bar
Maximum Temperature	50 °C
Stable in Solvents[1]	Alcohols (e.g. Butanol, Ethanol, Iso-propanol)
	Alkanes (e.g. Hexane)
	Aromatics (e.g. Toluene, Xylene)
	Ethers (e.g. Methyl-*tert*-Butyl-Ether)
	Ketones (e.g. Methyl-Ethyl-Ketone, Methyl-iso-Butyl-Ketone)
	Others (e.g. Acetonitrile, Butyraldehyde, Ethyl Acetate)

Membrane name: STARMEM™	122	240
Permeability[2] / $L\ m^{-2}\ h^{-1}$ Toluene at 55 bar	30	20
Molecular Weight Cut-Off / Dalton[3]	220	400

[1] Data referring to pure solvents. If you intend to use a different solvent please contact us for further advice.

[2] Performance Data are approximate.

[3] Based on rejection of normal alkanes dissolved in toluene, MWCO = molecular weight cut-off, defined as MW for 90% rejection.

For further information please contact us at emet@evonik.com.