

**PURIFICATION OF SORBITOL USING REVERSE  
OSMOSIS MEMBRANE: EFFECT OF CROSS  
FLOW VELOCITY AND TRANSMEMBRANE  
PRESSURE**

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OSMOSIS MEMBRANE: EFFECT OF CROSS  
FLOW VELOCITY AND TRANSMEMBRANE  
PRESSURE**

**NUR ATHIRAH BINTI SABARUDIN**

Thesis submitted in partial fulfilment of the requirements  
for the award of the degree of  
Bachelor of Chemical Engineering (Biotechnology)

**Faculty of Chemical & Natural Resources Engineering  
UNIVERSITI MALAYSIA PAHANG**

JUNE 2014

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## **STUDENT'S DECLARATION**

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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## *Dedication*

*Special Dedication to:*

*My mom, Puan Wan Nor Aspalaila binti Wan Muda,*

*My Dad, Encik Sabarudin bin Mohd Arif,*

*My sisters and brothers,*

*My respected lecturers,*

*My friends and*

*My fellow colleagues*

*For all your care, support and believe in me*

## ACKNOWLEDGEMENT

In the name of Allah, the most gracious and the most merciful.  
May the peace and blessings be upon prophet Muhammad (SAW).

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## ABSTRACT

Sorbitol, a sugar alcohol that has formula of  $C_6H_{14}O_6$  also known as D-Glucitol. It can be obtained from the reduction of glucose, by changing the aldehyde group to hydroxyl group. It is very slow to metabolize in body, which lessen the chance of increasing the level of insulin, thus makes sorbitol is a good sweetener for diabetics. Since the use of sorbitol in the food industry more widely, sorbitol purity is a key factor before it is used as an additive in food. One of the method to purify sorbitol is application of membrane technology. However the membrane application is limited due to challenges such as decay in permeate flux and increases the complexity of membrane operations. Thus, this research was conducted to study the effect of cross flow velocity (CFV) and transmembrane pressure (TMP) on the flux in purification of sorbitol using the reverse osmosis membrane. The flux was determined from the calculated of permeate flow rate divide by the area of spiral wound membrane. In order to study the optimize condition for the process, the value of TMP and CFV was varied to obtained the flux value. While varying the CFV value, the TMP value was constant and when the TMP was varied, the CFV was constant. This study was conducted for 60 minutes, and every 10 minutes the permeate flow rate was collected to get the flux value. The solution was prepared with constant concentration of 10g/L in a 100 L solution in tank. The determination of sorbitol was analyze by refractometer. Based on the result obtained, increasing in CFV and TMP give the highest flux value.



## ABSTRAK

Sorbitol, sejenis gula alkohol yang mempunyai formula  $C_6H_{14}O_6$  juga dikenali sebagai D- Glucitol Ia boleh diperolehi daripada pengurangan glukosa, dengan menukar kumpulan aldehid kepada kumpulan hidroksil. Ia sangat lambat untuk dimetabolismekan dalam badan, dimana dapat mengurangkan peluang peningkatan tahap insulin maka menjadikan ianya sesuai sebagai pemanis bagi pesakit kencing manis. Oleh kerana penggunaan sorbitol dalam industri makanan sangat meluas, ketulenan sorbitol adalah faktor penting sebelum ia digunakan sebagai bahan tambahan dalam makanan. Salah satu kaedah untuk menuliskan sorbitol adalah penggunaan teknologi membran. Walau bagaimanapun penggunaan membran adalah terhad disebabkan cabaran seperti penurunan fluks dan meningkatkan kerumitan operasi membran. Oleh itu, kajian ini telah dijalankan untuk mengkaji kesan halaju aliran silang (CFV) dan tekanan transmembran (TMP) pada fluks dalam penulenan sorbitol menggunakan membran osmosis berbalik. Fluks ditentukan daripada kadar aliran isipadu dibahagi dengan luas membran. Dalam usaha untuk mengkaji keadaan yang paling optimum untuk proses, nilai halaju aliran silang dan tekanan transmembran telah diubah untuk memperolehi nilai fluks. Apabila nilai nilai halaju aliran silang diubah, maka nilai tekanan transmembran adalah tetap dan sebaliknya. Kajian ini dijalankan selama 60 minit, dan setiap 10 minit kadar aliran isipadu dikumpulkan untuk mendapatkan nilai fluks. Larutan sorbitol disediakan dengan kepekatan tetap pada 10g/L di dalam larutan tangki 100 L. Sorbitol ditentukan dengan menganalisis menggunakan refraktometer. Hasil yang diperolehi menunjukkan peningkatan halaju aliran silang (CFV) dan tekanan transmembrane (TMP) memberi memberikan nilai fluks yang maksimum.

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## LIST OF SYMBOLS

J	Flux
LMH	Flux through the membrane
t	Time
Q	Volume flow rate

## LIST OF ABBREVIATIONS

CCD	Central Composite Design
CFV	Cross flow velocity
FTIR	Fourier transform infrared spectroscopy
ICP-MS	Inductively coupled plasma – mass spectrophotometer
MW	Molecular weight
MWCO	Molecular weight cut off
OFAT	One factor at a time
RO	Reverse osmosis
RSM	Response surface methodology
SEM	Electric resistance tomography
TMP	Transmembrane pressure



# 1 INTRODUCTION

## *1.1 Background of research*

Polyols was commonly used for personal care industry, both the cosmetics and toiletries sectors and began to play an important role in a market characterized by growing interest in alternatives to animal-derived ingredients. Therefore, polyols were incorporated as excipients in the manufacture of the essential ranges of personal care products like toothpaste, creams and lotions, make-up, perfumes or deodorants.

Sugar alcohols were a class of polyols in which sugar's carbonyl, ketone or aldehyde is reduced to the corresponding primary or secondary hydroxyl group. Sorbitol is a sugar alcohol that has the molecular weight of 182.17 g/mole and melting point of 95°C while the boiling point is 296°C. The IUPAC name for sorbitol is (2S,3R,4R,5R)-hexane-1,2,3,4,5,6-hexol. Sorbitol is about 60% as sweet as sucrose with 1/3 fewer calories. It has been safely used in processed foods for almost half a century, also used in other products such as pharmaceuticals and cosmetics. (Baines *et al.*, 1975), U.S. Patent 3927201, states that the refractive index of sorbitol as a 70% solution in water is 1.45.

Developed in the 1950s, sorbitol was the most consumed sugar alcohol. In all applications, demand for sorbitol was largely a function of its unique combination of functional properties as a humectant, sweetener, bulking agent, stabilizer, softener, emulsifier, and its surface-active properties. Applications in personal care products (mainly toothpaste), food and confections and in the manufacture of vitamin C accounted for 78% of world consumption in 2007 and continue to account for over 75% of world demand in the near future. (Bizzari *et.al*)

Although sorbitol were less effective than xylitol in controlling the caries, but the lower cost of sorbitol makes it to be more demanding to be used as the sugar substitute in the food manufacturing. It is also be consumed by people in relatively large amount without side-effects. However, they can act as a laxative. It does not promote caries because it metabolized either slowly or not at all in dental plaque. For people with diabetics, there was some restriction for them to consume the glucose continuously. But, they was advised to take the artificial sweetener such as sorbitol, that has a fewer calories than glucose. It has been recommended used in dietetic food products which it lends

palatability and bulk. It was believed converted to glycogen to be liberated as glucose without producing hyperglycemia.

In this study, a reverse osmosis membrane was used in performing the purification process that increased the purity of sorbitol. However, during the process problems such as flux declination during the separation process, that can be reduced by applying a high transmembrane pressure and high cross flow velocity, thus will improve membrane flux and reduce the fouling of the membrane. Reverse Osmosis has many advantages over the conventional water and wastewater treatment operations and processes. It is able in removing many dissolved substances efficiently, yet produce good quality finished water. It does not require any addition of chemicals into the water for separation. The separation of the dissolved substances from the influent is achieved physically or physico-chemically. It is essentially a molecular squeezing process, using a semi-permeable membrane which causes water molecules to separate from the contaminants then the separated water molecules pass through to the inside of the membrane to a holding reservoir. Recently, reverse osmosis has been used in treating boiler feed water, in addition to industrial and process wastewaters. Boilers are found throughout the chemical processing industry and the primary method to treat boiler wastewater is ion-exchange. However, reverse osmosis has been demonstrated to be more cost effective than this demineralization process.

The International Union of Pure and Applied Chemistry (IUPAC) define reverse osmosis as a “pressure-driven process in which applied transmembrane pressure causes selective movement of solvent against its osmotic pressure difference” (IUPAC 1996). Reverse osmosis (also called hyperfiltration, is capable of the highest filtration level possible, including separating dissolved salts and removing bacteria, pathogens and organics from water. The applications of reverse osmosis include but are not limited to separation and concentration of solutes in many fields, such as chemical and biomedical industry, food and beverage processing, and water and wastewater treatment.

Wide application of reverse osmosis such as for seawater desalination, groundwater treatment, and for tertiary treatment to reclaim the secondary effluent for advance reuses purposes. In addition to removing total dissolved solid, reverse osmosis is very effective in reducing other minerals, ammonia, and total organic carbon (TOC). The membranes

remove 90 percent of total dissolved solid. It is useful for separating thermally and chemically unstable products.

## ***1.2 Motivation***

Nowadays, the number of patients with diabetics had achieved a very alarming number. One of the factors of diabetics is the diet that is taken by the people worlds wide besides obesity and sedentary lifestyles (Vischer et al, 2009). Unstable diet with high content of sugars can causes diabetics. Surprisingly, Malaysia has one of the world's greatest numbers of diabetic cases among its population with 2.6 million registered patients (Adie, 2012). Untreated diabetics may cause chronic renal failure (Mauro *et al*, 2001). Sugar substitute products regulate sugar intake by consumers, which helps promote healthy weight maintenance and improve blood glucose control. Hence, play an important role in the health and well-being of an individual. The benefits of consuming these substitutes sugar are highly valuable for all people that also have skin smoothing properties. (Chen, 1985)

Interest now arises because of their multiple potential health benefits. They are non-cariogenic (sugar-free tooth-friendly), low-glycaemic (potentially helpful in diabetes and cardiovascular disease), low-energy and low-insulinaemic (potentially helpful in obesity), low-digestible (potentially helpful in the colon), osmotic (colon-hydrating, laxative and purifying) carbohydrates. Glycaemic Index values on replacing sucrose were independent of both intake (up to 50 g) and the state of carbohydrate metabolism.

Although it is not an essential nutrient, they contribute to clinically recognized maintenance of a healthy colonic environment and function. A role for polyols to hydrate the colonic contents and aid laxation is now recognized by physicians and favors saccharolytic anaerobes and aciduric organisms in the colon, purifying the colon of endotoxic, putrefying and pathological organisms, which has clinical relevance. Polyols also contribute towards short-chain organic acid formation for a healthy colonic epithelium.

### ***1.3 Problem statement***

Lacking of previous research that in purification of sorbitol using the membrane separation techniques. The problem of this research is to find the most appropriate condition that can enhance the purification of sorbitol using the low pressure reverse osmosis membrane. The purification of sorbitol is important in order to remove the impurities to produce pure product. The impurities will then reduce the quality of sorbitol hence the economic value will be low.

Although application of membrane purification is the most effective process at present, the challenges for membrane purification of sorbitol because of problem faced is decay in permeate flux, which cause by fouling. In order to reduce the fouling by varying the transmembrane pressure and cross flow velocity to the optimum condition thus enhances the membrane flux.

Thus, this research proposed studying the purification process of sorbitol using membrane technology and determining the factors that affect the permeate flux. The suitable parameters that will be determined include cross flow velocity (CFV) and transmembrane pressure (TMP).

### ***1.4 Objectives***

The main objective of this research is to purify Sorbitol by using Low Pressure Reverse Osmosis Membrane System.

This research also has a few specific objectives that are:

- i. To determine the effect of cross flow velocity and transmembrane pressure on flux during sorbitol filtration.
- ii. To determine the optimum condition of sorbitol flux using RSM.

## ***1.5 Scope of this research***

The scopes of study are:

- 1) Purification using GE Membrane was focused in this study.
- 2) Transmembrane pressure and cross flow velocity was adjusted to determine the membrane flux.
- 3) The volume of permeate and retentate was collected every 10 minutes.
- 4) The sample was analyzed by Fourier Transform Infra-Red (FTIR), refractometer, and ICPMS.
- 5) The optimization condition of operating parameters, including cross flow velocity (CFV) and transmembrane pressure (TMP) has been done by the Response Surface Methodology (RSM)

## ***1.6 Thesis outline***

This report contains five chapters that consist of first chapter that includes the background of research, problem statement, objectives of the research, scopes of study, and thesis outline. Second chapter is the literature review on sorbitol, purification, membrane, CFV, TMP, membrane cleaning and analysis performed. Methodology was discussed in chapter 3 which comprises of materials and experiment procedures. The discussions of the findings are delivered in Chapter 4 that discusses about effect of cross flow velocity (CFV) and transmembrane pressure (TMP) towards permeate flux. While Chapter 5 presents the conclusion and recommendation of this study.

## 2 LITERATURE REVIEW

### 2.1 Overview

Table 2-1: Properties of sorbitol and xylitol

Sorbitol	Xylitol
Synonym: D-Glucitol, D-Sorbitol	Synonym: D-Xylitol, D-xylitol-Pentane-1,2,3,4,5-pentol
Chemical formula: C <sub>6</sub> H <sub>14</sub> O <sub>6</sub>	Chemical formula: C <sub>5</sub> H <sub>12</sub> O <sub>5</sub>
Molecular weight : 182.17 g/mole	Molecular weight : 152.15 g/mole
Boiling Point: 295 °C ( 563.00 fF)	Boiling Point: : 216°C (420.8°F)
Melting Point: 75 °C ( 167.00 fF)	Melting Point: 94°C (201.2°F)
Critical Temperature: Not available.	Critical Temperature: Not available
Specific Gravity: 1.489 (Water = 1)	Specific Gravity: 1.52(Water = 1)
Solubility in water: 55% @ 25°C	Solubility in water: Easily soluble in cold water. Soluble in methanol.
Color: colorless or white	Color: White
Odor: odorless	Odor: Odorless

Based on Table 2.1, it shows the comparison properties of sorbitol and xylitol, both is a sugar alcohol that they might shows or exhibit the same characteristics. However the chemical formula was different where sorbitol, has six number of carbon while xylitol has only five number of carbon. The properties of sorbitol (IUPAC) are fairly similar to those of its stereoisomer, mannitol. However the solubility of sorbitol in water is significantly higher than mannitol. At 25°C the solubility of sorbitol in water is only approximately 55% while for xylitol easily soluble in cold. Sorbitol is sparingly soluble in organic solvents like ethanol, and glycerol and practically insoluble in ether, ketone and hydrocarbons. The relatives sweetness to sucrose are varies among different sugar alcohols. The relative sweetness of xylitol is 100%, mannitol is 40-50% and sorbitol, 60%. (Schiweck *et al.* 1994)

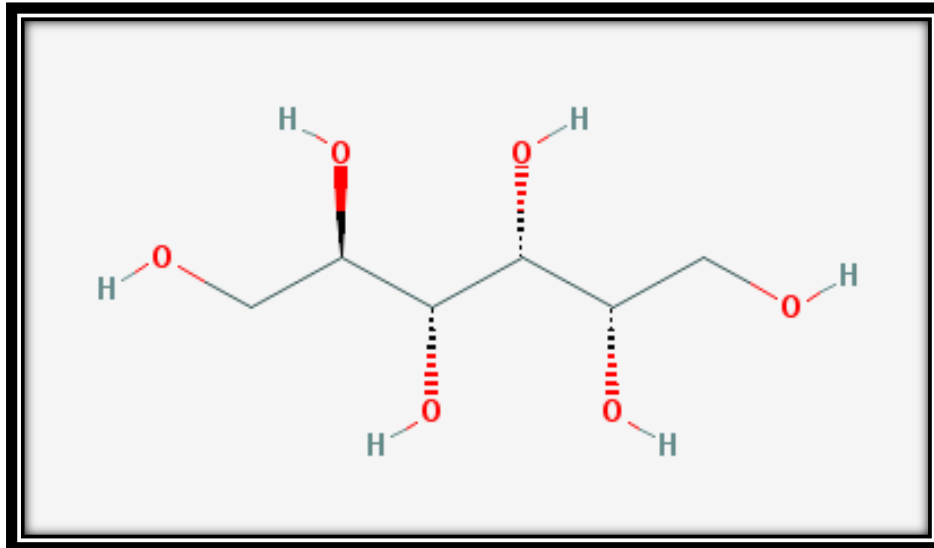


Figure 2-1: Sorbitol structure formula

Sorbitol sweetener is a sugar alcohol that's derived from glucose by modifying one of the chemical groups so that it's metabolized more slowly by the body. This reduces the insulin surge that normally occurs when sugar is ingested. Sorbitol can be found naturally in some fruits or can be obtained from glucose catalytic hydrogenation and at only 2.6 calories per serving it offers significant calorie advantages over table sugar (Cazetta *et al*, 2005). This is because fermentation process involves biological systems which are less controllable and more complex than straightforward chemical reactions, the variability in products derived by fermentation is often greater than in products derived by chemical synthesis. Thus, the impurity profile of a fermentation product may be more complex and less predictable than that of a synthetic product. (Canary, 2010) Usually the production of sorbitol is accomplished in a hydrogenation process however, reactions like hydrolysis and hydrogenation may be involved in the same reaction system due to the rapid development of research.

Besides of the application in food industry, in pharmaceutical and medicine it can be used to decrease cellular edema and medicine it can be used to determine increase of urinary output. The chemical structure of sugar alcohol allows them to be absorbed more slowly in the body than regular sugars. Therefore, they have smaller impact on blood insulin level. Individuals, who should not eat sugar such as diabetic patient, now can consume the artificial sweetness, due to the sweet taste of sugar alcohols combined with the independence of insulin when metabolized by body. (Weymarn, 2002)

The reduced caloric values are due to the facts that sugar alcohol is only partially absorbed in the upper intestine. Thus, a large part of the ingested sugar alcohols reached the large intestine, where bacteria degrade it. (Schiweck *et al.*, 1994) sugar alcohol is commonly used for production of reduced-calorie products, because of the reduced caloric value compared to most sugar. However, incomplete absorption can result in diarrhea, a gastrointestinal effect, hence the daily intake of polyols should not exceed 20g (Anon, 2001)

## **2.2 Purification**

Purification is a process that removes the remaining impurities which typically are similar to those of target product. (R. Ghosh, 2006). According to Shawn(1999), the first chromatographic techniques that is paper and thin-layer chromatography were used to separate sugars, but the separations were limited with respect to number of recognized analyses, presented poor resolution and not always quantitative, while the efficient methods for purify specific carbohydrate to high degree of purity using the semipermeable membrane reverse osmosis.

For the identification of all, polyols still did not have single method that is universally applicable. In previous preliminary analyses of plant material, paper chromatography is very frequently used where the application of this method requires little specialized equipment and because it has proved most useful for examining material. Purification in chemical industries is important in pharmaceutical industries such that positive results of quality control and cost of purification step or steps are that won't be available without optimized process conditions and choosing the best purification method.(Salehparhizkar, 2009).Table 2-2 summarize the different types of separation process by using different separation methods.



Table 2-2: Different types of separation process

Type of system	Characteristics	Process	Result	References
Ultrafiltration (in organic membrane)	Pore size: 20 100nm 0.8 m long Filtration area - 0.2m <sup>2</sup>	Purification of different liquid materials from food industry	-Amaranth starch solution was concentrated 5 times while eg blend approximately 2- times in one step ultrafiltration process.	Hinkova <i>et al.</i> (2005).
Ion exchange chromatography (HPLC)	Column matrix	Purification of monosaccharide	-Bulk of contaminants was removed. - 8 µg of glucose contamination per milligram of ultrafiltered enzyme remained.	Hodgins. G. W.L. <i>et al</i>
Nanofiltration	Polysufone membrane	Purification of xylose from hemicellulose hydrolyzate feeds	Purified xylose	Sjoman <i>et al.</i> (2006)
Tangential flow microfiltration and ultrafiltration	Polyvinylidene fluoride (PVDF) membrane	Separation of hyaluronic acid from the fermentation broth of <i>Streptococcus</i> <i>Zooepidemicus</i>	Hyaluronic acid was separate from fermentation broth	Zhou <i>et al.</i> (2006)

### **2.3 Reverse osmosis membrane**

The use of reverse osmosis (RO) membrane to remove salts and impurities from water has been a recognized technology to improve water quality. RO was being used in producing variety of high purity needs including industrial boiler feed, pharmaceutical waters, electronic industry supply and other process industries. It is proved to be used in water consolidation with microfiltration (MF), ultrafiltration (UF) evaporation or other water processes. The use of RO in wastewater is valuable application because permeate water can be reused and waste water stream becomes a resources in the stream. The benefits include reduced discharge, reduced purchases and conservation of water resources. (Zibrida *et al.*, 2000).

Composition of the feed water largely controlled the performance of RO system where the quality of the feed water will determine the amount and type of pre-treatment necessary to make an RO an economical process. (Zibrida *et al.*, 2000)

### **2.4 Membrane applications**

Membrane is a physical barrier that allows certain compounds to pass through and can be classified according different pore size or molecular weight cut-off into four different types that is reverse osmosis (RO), nanofiltration (NF), ultrafiltration (UF) and microfiltration (MF).

Reverse osmosis is a separation technique which operates at about normal ambient temperatures, or slightly above or below and can be used for concentration and purification of liquids without a phase change. It separates salts and small molecules at relatively high pressures and has advantage over traditional evaporation techniques of removing water content, that it does not cause loss of juice flavour and colour dedgradation due to effect of high temperature. In this study, reverse osmosis was used for sorbitol purification.

Ultrafiltration can separate extremely small particles and dissolved molecules from fluids. It cannot separates molecules of similar size but only molecules which differ by at least one order of magnitude in size. The molecular, chemical or electrostatic of sample can affect the permeability of the filter medium. Particulates matter ranging in size from 1000 to 1000000 molecular weight are retained by certain ultrafiltration membranes, while water will pass through. UF membrane can be used for both purify

materials passing through the filter and also to collect materials retained by the filter. (Munir, 2006).

Microfiltration membrane can remove particles or biological entities ranging between 0.025  $\mu\text{m}$  to 10  $\mu\text{m}$  from fluids by passage through a microporous medium such as membrane filter. (Munir, 2006) Reverse Osmosis is even more attractive in land-constrained areas because of reduced land requirement. It is anticipated that reverse osmosis processes will be used more widely in water industry to replace the large conventional water treatment systems. Reverse osmosis was first developed in the 1950's by the US government to provide fresh drinking water for the Navy, and since then, advances have made it much more feasible for obtaining purified water from wastewater. (Tansel *et al.*, 2000)

The uses of membrane for separations are becoming increasingly important in the process industries, where the membrane acts as a semipermeable barrier and separation occurs by the membrane controlling the rate of movement of various molecules between two liquid phases, two gas phase or a liquid and a gas phase. The two fluid phases are usually miscible and the membrane barrier prevents actual, ordinary hydrodynamic flow. (Geankoplis., 2003)

The membrane filtration techniques becoming a mainstream technology since early 1990's. With a number of advantages such as huge efficiency, simple equipment, convenient operation and low energy consumption, the technology has become one of the most important industrial separation techniques and has been applied extensively to various fields such as food industry and pharmaceutical. (Jing Howard, 2010). Hinkova, (2000) reported that progressively increasing transmembrane pressure and cross-flow velocity resulted in 13-26% improvement of permeate flux.

Din *et al.*, (2012) states that membrane technology is considered as one of the most effective process for water and wastewater treatment. It is a compact system, economically feasible and has high pollutant removal efficiency. In the past, pressure-driven membrane processes such as RO had gained special attention due to its effective removal of pollutants, especially those with low concentrations. The use of RO is limited due to high operational cost especially when high pressure is applied. Therefore,

low pressure reverse osmosis membrane (LPROM) has been introduced to water and wastewater industries in the past few years. (Din *et al.*, 2012)

Most of LPROM are multi-layer thin film composed of complex polymers. The active membrane surface layer normally consists of negatively charged sulphone or carboxyl group. This helps the membranes in improving of fouling resistance against hydrophobic colloids, proteins, oils and other organics. In order to increase water flux, a charged hydrophilic layer is attached to a hydrophobic UF support membrane. This makes the membrane favorable for the orientation of water dipoles. Flux is inversely proportional to the membrane thickness. Generally, LPROM contains corrugated skin surface that can improve flux significantly. It produces specific flux more than 60 L/m<sup>2</sup>.h MPa (flux per membrane area and per net driving pressure) at low operating pressure. This flux rate is about double the flux of the previous generations of composite RO membrane. (Din *et al.*, 2012)

Some advantages the production of food by using membrane processes, which it has better technological and nutritional functionalities, efficient use of material and resource, and give reduced negative impact to the environment. The main benefits of membrane is improved the production process by consistence high quality of permeate/retentate, reduced operating costs, low maintenance and pressure drop, chemical and temperature resistance and long membrane operating life and the recovery of valuable products that previously would have been lost to waste. (Scott, K. 1998) Reverse Osmosis have some advantages, over evaporation when concentrating sugar solution that can prevent the camelization and save energy. (Yurong *et al.*, 1987).

However, the use of membranes cannot be applied widely due to membrane fouling. The fouling was dependent on the pore size and cause by the large particles (250nm) or coagulants. It is influenced the rejection of particles in MF and NOM in UF and NF. It is also stated that the low molecular weight acids that passed through the NF membranes and the rejection are dependent on the deposit of membrane. The mechanism of fouling can be such as pore plugging, cake formation (internal pore adsorption that reduces the internal pore diameter and charge of the deposit. In order to achieve unfouled membrane, the extent of rejection is influenced largely by the pore size or molecular weight cut-off (MWCO).The charge interactions, bridging, and hydrophobic interactions may play important role in fouling effect. For membrane

separation process of reverse osmosis the size of particle are about 0.001 $\mu$ m and the molecular weight are in the range about 100 to 1000 Dalton. (Schafer *et al.*, 2000)

In order to reduce the membrane fouling, the optimum condition of TMP and CFV should be applied as shown in Table 2.2. Defrance and Jaffrin,(1998) reported that increase in TMP will increase the membrane flux and Matsuura,(1971) shown that the concentration of sugar increase after the membrane purification process by reverse osmosis membrane. Due to fouling will increase in operational cost and lower the process efficiency. (Vrouwenvelder *et al.*, 2002)

Reverse osmosis (RO) is the most economical method of removing 95% to 99% of all contaminants. The pore structure of RO membranes is much tighter than UF membranes. RO membranes are capable of rejecting practically all particles, bacteria and organics >300 daltons molecular weight (including pyrogens). Natural osmosis occurs when solutions with two different concentrations are separated by a semi-permeable membrane. Osmotic pressure drives water through the membrane; the water dilutes the more concentrated solution; and the end result is equilibrium.

Because reverse osmosis membranes are very restrictive, they yield very slow flow rates. Storage tanks are required to produce an adequate volume in a reasonable amount of time. Reverse osmosis also involves an ionic exclusion process. Only solvent is allowed to pass through the semi-permeable reverse osmosis membrane, while virtually all ions and dissolved molecules are retained (including salts and sugars). The semi-permeable membrane rejects salts (ions) by a charge phenomenon action: the greater the charge, the greater the rejection. Therefore, the membrane rejects nearly all (>99%) strongly ionized polyvalent ions but only 95% of the weakly ionized monovalent ions like sodium.

Membrane separation processes find their application in almost all branches of food and biotechnological industry. Apart from the biotechnology, the most wide-spread applications are in dairy and beverage industries, e.g. for whey protein concentration and purification (Sschkoda and Kessler, 1997), whey desalination and demineralisation, milk standardisation by ultrafiltration, etc. In the beverage industry, membranes are applied for beer and wine stabilisation to prevent the microbial decomposition, for the yeast and colloid removal, or for non-alcoholic beer production by pervaporation

(Karlsson & Tragardh,1996). Membranes are also very useful in fruit and vegetable juices production for juice purification by ultrafiltration or concentration by reverse osmosis or nanofiltration (Koseoglu *et al.* 1991).

Reverse osmosis is the most economical and efficient methods for purifying tap water if the system is properly designed for the feed water conditions and the intended use of the product water. Reverse osmosis is also the optimum pretreatment for reagent-grade water polishing systems. Reverse Osmosis is the reverse process of spontaneous osmosis. The osmosis process can be reverted by adding external pressure on the salty side so that some of the fresh water molecules on the salty side will end up on the fresh water side. The problem is that the osmotic pressure tends to force water to the more saline side, which is opposite of the desired outcome. To overcome this tendency, the osmotic pressure can be overcome by the applied pressure, forcing water from the saline side to the less saline side. Reverse osmosis is schematically presented in Figure 2.2.

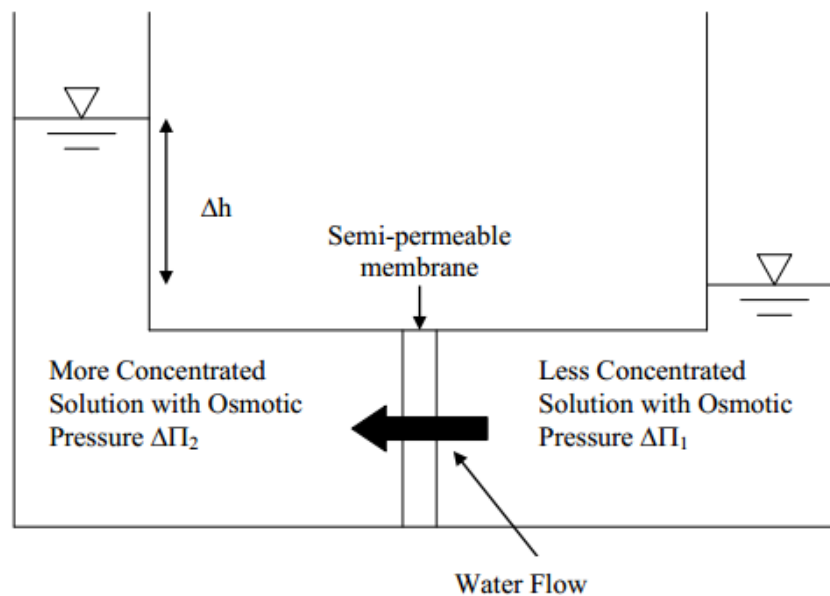


Figure 2-2: Simple osmosis

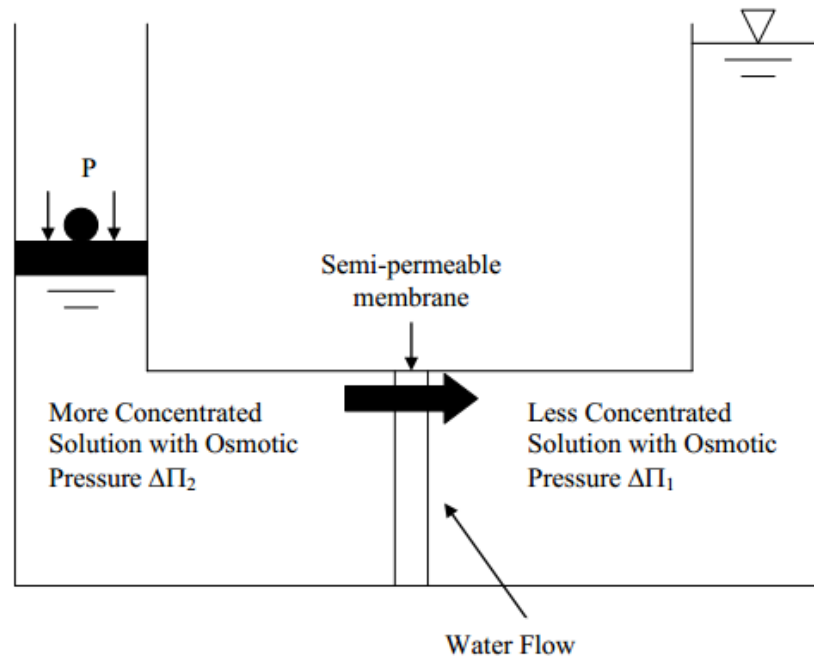


Figure 2-3: Simple Reverse Osmosis

As the pressure increases, the migration of fresh water to salty water slows down. If enough external pressure is applied, an osmotic equilibrium will be reached and the water stops going from the fresh water side to the salty side.

Table 2-3 shows the molecular weight cut off of different types of membrane such as ultrafiltration, nanofiltration, microfiltration and reverse osmosis. While Table 2-4 shows the effect of difference types of membranes towards separation process.

Table 2-3: The MWCO for different type of membranes

Process	Pore Diameter (nm)	Pressure	MWCO	References
UF (PLHK)	18.20	1	100	A.I. Schafer et al. (2000)
(PLTK)	9.62	1	30	
(PLGC)	5.18	3	10	
(PLCC)	3.72	3	5	
NF (CA-UF)	<3.72	5	5	
(TFC-SR)	<0.64	5	<0.18	
(TFC-S)	<0.64	5	<0.18	

(TFC-ULP)	<0.64	5	<0.18	
Microfiltration	>0.1 $\mu\text{m}$	<2 bar	>5000 kDa	Osada et al. (1992)
Ultrafiltration	100-2 nm	1-10bar	5-5000 kDa	
Nanofiltration	2-1 nm	3-20 bar	0.1-5 kDa	
Reverse osmosis	<1nm	10-80 bar	<100Da	

Table 2-4: The effect of difference types of membrane

Membrane	Parameter	Result	References
0.25 m <sup>2</sup> ceramic membrane	CFV :1-5 m/s	<ul style="list-style-type: none"> <li>Flux increase by 10L/h.m<sup>2</sup></li> <li>TMP rise moderately and then stabilize in 15 min until critical flux</li> </ul>	Defrance and Jaffrin (1998)
Reverse Osmosis membrane	Glucose-water  Maltose-water  Lactose-water	<ul style="list-style-type: none"> <li>Concentrated from 0.1 to 1.5M</li> <li>Concentrated from 0.03 to 0.11M</li> <li>Concentrated from 0.04 to 0.22M</li> </ul>	Matsuura(1971)

Increasing of juice purity and retention of almost 50 % of colour impurities by microfiltration is one of the most important results of this study. For nanofiltration tests, a special cross-flow testing cell with adjustable tangential speed of 0|3 m s<sup>-1</sup> has been designed.

Reverse-osmosis membrane is a membrane, which impedes the passage of a low-molecular weight solute, is placed between a solute-solvent solution and a pure solvent.



The solvent diffuses into the solution by osmosis. A reverse pressure difference is imposed which cause the flow of solvent to reverse, as in the desalination of seawater. This process also used to separate other low-molecular- solutes, such as salts, sugar and simple acids from solvent.

## ***2.5 Spiral wound membrane***

There are four major types of membrane modules that are currently employed in membrane applications that is tubular, hollow fibre , plate and frame and spiral wound) which all have various benefits and limitations. (Somasundaran, 2006)

Spiral wound membrane becomes the most popular and economical form for RO membrane because it have been invented by polymer chemists that can be cast with a significant range of flow and feed pressure requirements along with a significant range of inorganic salt and organic rejection capabilities. Thus, it offers the highest membrane packing area capability which results in the smallest water plant foot print size while maintaining reasonable anti-fouling characteristics, when compared to other membrane configurations like hollow fibre, tubular or plate-and-frame. A wide range of materials was used in constructions of the membrane offer an optimal blend of low material cost, high quality, chemical stability and physical ruggedness. It also offers the highest degree of automatic manufacturing capability, reasonable labor cost, high quality, and high yields.

Figure 2-4 shows the overview of spiral wound membrane of reverse osmosis membrane process. The membrane used in this study was spiral wound membrane. From the first picture shows that the feed solution enters the membrane, and out stream flow for permeate and retentate (concentrate on the product). Solution passes through the membrane and was collected in the permeate collection material, while rejected solution stay in the feed channel spacer. Picture 2-5 shows the membrane layer that is feed channel starter, membrane, permeate collection material. Feed solution that was rejected by the membrane passes straight through the membrane element, and the purified water passes through the membrane layer and moves via spiral layers to outlet at central tube. (Picture 6-8)

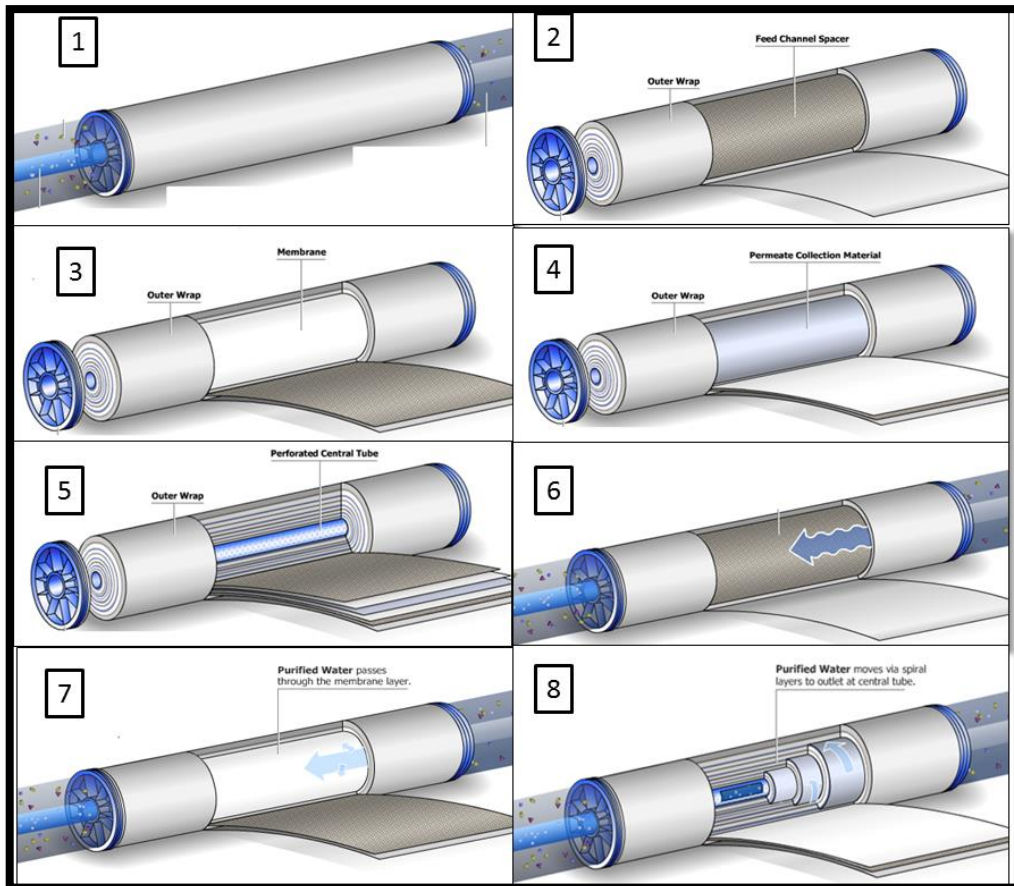


Figure 2-4: Process flow through spiral wound membrane

Usually, there are two types of operation mode on mode in membrane separation, which are dead-end filtration and cross flow filtration (Figure 2-5 and 2-6) (Kim et al., 1993). Across flow or tangential mode operation system is often preferred for many industrial application due to lower fouling tendency compared to the dead-end filtration. In order to increase flux rate, (product passing across the membrane) as well as reduce the gel or cake layer which deposits on the membrane surface, an appropriate use of cross flow velocity and transmembrane pressure is widely applied. (Scott, 1995)

Table 2-5: Comparison between dead end filtration and cross flow filtration

Dead End Filtration	Cross Flow Filtration
Complete flow feed was forced through the membrane and filtered matter is accumulated on the membrane surface.	Constant turbulence feed flow rate flow along the membrane surface.
Useful technique for concentrating compounds.	Excellent way to filter fluids with high concentration of filterable matter.
One flow of feed perpendicular to the membrane surface. Water pushed to the membrane by pressure.	Feed flow parallel to the membrane surface.
No rejected stream, all passed through the membrane as permeate.	Have rejected stream, retentate and permeate.

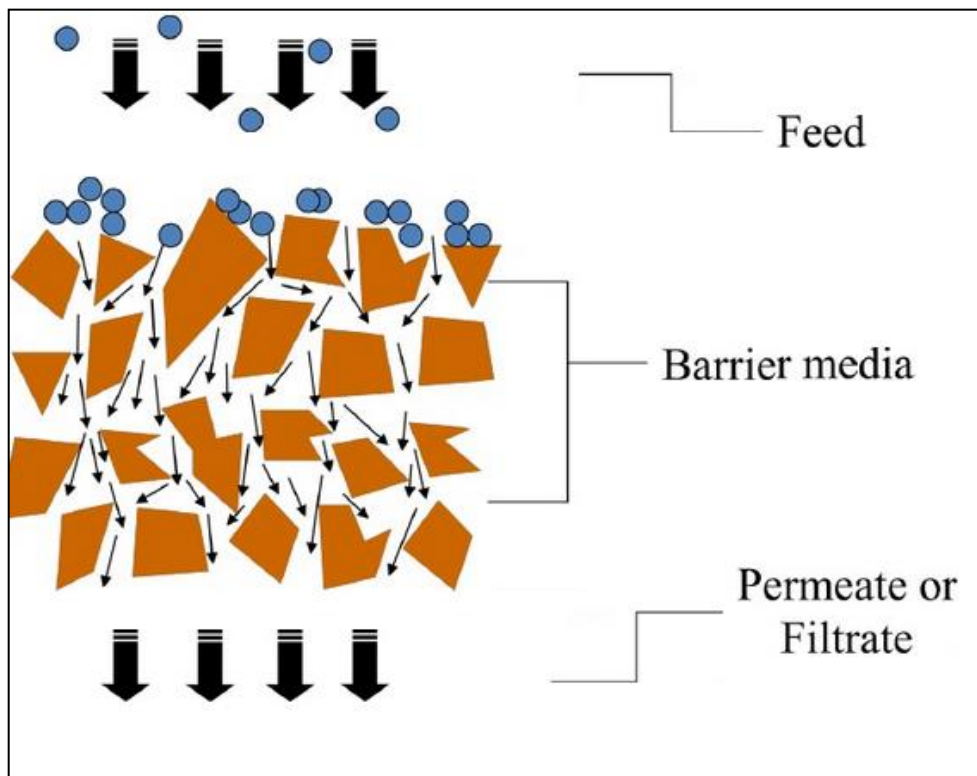


Figure 2-5: Dead End Filtration mechanism

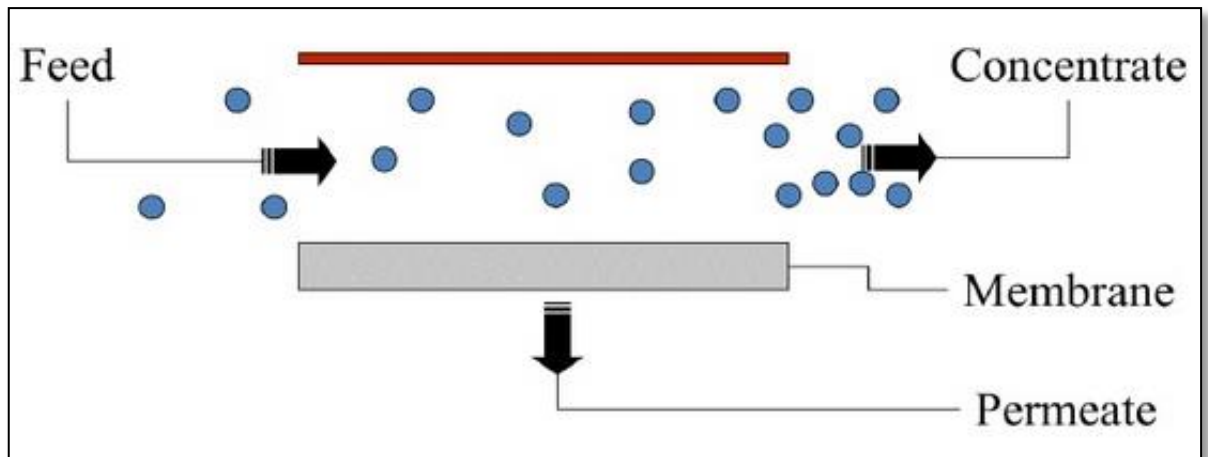


Figure 2-6: Cross Flow Filtration Mechanism

## 2.6 Flux

From the investigation of fouling on Reverse Osmosis membranes, the membrane flux performance depended on both hydrodynamic conditions (flux and cross flow velocity) and solution component was largely independent of virgin membrane properties. When the cross flow velocity low, it affected the flux performance moderately, severe flux reduction occurred at high initial flux, at low pH and high calcium concentration. Fouling can cause significant loss of productivity and added the operational cost. (Chuyang *et al.* 2006)

Definition of flux, J which is the flow rate of permeate, amount of fluid passing through the membrane and usually given in terms of volume per unit time per unit membrane area. (Affleck, 2000). From equation 2.1, the parameters applied to identify flux declination and the efficiency of membrane processes are as follows:

$$J = \frac{Q}{A} \quad \dots\dots\dots (2.1)$$

Where J is the flux through the membrane (LMH), Q is the permeate flow rate (LPM) and A is the membrane area (m<sup>2</sup>). (Sakinah *et al.*, 2007)

Membrane purification of sorbitol was evaluated by using by using the permeate flux. From equation 2.2, the permeate flux was calculated by measuring the quantity of permeate collected during a certain time and dividing it by effective membrane area. (Li *et al.*, 2006)

$$\text{Permeate Flux, } J = \frac{\text{Permeate volume}}{\text{Membrane} \times \text{time}} \text{ (Lm}^{-2}\text{h}^{-1}\text{)} \dots\dots\dots (2.2)$$

## **2.7 Membrane cleaning**

Various techniques used to reduce membrane fouling such as backwashing that can improve membrane permeability and reduce fouling, thus leading to stable and optimal operating condition. Backwashing or chemical cleaning method has been widely applied in many industries. (Sakinah and Zularisam, 2013)

It was found that backwashing during a membrane filtration process was successfully remove most of the reversible component of the foulant layer leading to reduced TMP increases and permeate flux decrease. Backwashing enables the system to operate longer before it stopped for intensive physical or chemical cleaning. It's frequency is a vital parameter in successful long term operation of the system. (Smith et al, 2005)

Backwashing systems can use chlorinated or unchlorinated water with or without air scour depending on the membrane system. Backwashes are typically frequent ( every 5 minutes to hours) and of short duration (3 to 10 seconds) depending on the membrane system and specific feed water quality conditions. Backwashing a system filter means reversing and increasing the water's flow to flush out accumulated debris and particles from the membrane. (Satterfield, 2005)

For maintaining or re-establishing the permeate flux after the membranes are fouled, there are two methods in microfiltration that is membrane backwash and membrane pre-treatment. For membrane backwash, the membrane was backwashed that only takes a few minutes in order to prevent the continuous accumulation of solids on the membrane surface. If the backwash is incapable of restoring these flux, the membrane are chemically cleaned.

Reverse-pumping of water was referred to the backwashing. It is a form of preventive maintenance to prevent further clogging of the filter medium. Backwashing in water treatment plants solutions that deliver the very strong force with well-executed flush times and speeds required for effective backwashing. (www.grundfos.com)

## **2.8 Cross flow velocity (CFV)**

Studies have been conducted by several researches that cross flow velocity is used to reduce the effect of membrane resistance due to polarization and fouling or gel layer on the membrane surface. (Mohammad, 2008)

The obtained results showed that permeate flux increased linearly with increasing cross-flow velocity and a high cross-flow velocity was more effective to reduce fouling of MF membrane than that of UF membrane. The formation of a reversible fouling layer was actually membrane. (Choi. H *et al.*, 2005)

Hwang and Sz,(2010) studied on operation condition on the filtration flux for solute rejection and membrane fouling in BSA/ dextran binary suspension cross flow microfiltration shows that filtration flux was increased 30-50% when increased in cross flow velocity or transmembrane pressure. Besides that, cross flow velocity also influence on the formation of the fouling layer during biological suspension. (Choi *et al.*)

## **2.9 Transmembrane pressure (TMP)**

Transmembrane pressure has also been studied as operating parameter in measure and control membrane fouling in research on the separation of proteins from an aqueous solution by dead-end filtration. (Lin *et al*, 2008). Transmembrane pressure is defined as the difference in pressure between two sides of membrane. It is a valuable measurement because it describes how much force is needed to push fluid referred as the feed through the membrane. A low transmembrane pressure indicates a clean, well function membrane. On the other hand, a high transmembrane pressure indicates a dirty or fouled membrane wit reduced filtering abilities. The ideal transmembrane pressure varies for different membranes and is usually available from the company that produced or distributed particular membrane. (www.ehow.com)

Trans Membrane Pressure (TMP): The driving force for flux. In our cross-low microfiltration system, it is measured as the difference between retentate and permeate pressures at the inlet and outlet. (Mayank, 2007)

As result of the low resistance, the filters achieve exceptionally high fluxes under low operational pressure. Consequently, the transmembrane pressure is substantially lower

with microsieves (0,1-0,01 bar) than it would be with, for example, ceramic membranes (1-10 bar), making these filters less invasive and suitable for delicate pressure sensitive products like algae. (Kuiper *et al*, 1998)

### ***2.10 Refractometer***

The refractive index of a refractometer, also known as the Brix value (BV), is constant for a substance under standard conditions of temperature and pressure. The BV, a measure of total soluble solids in solution, correlates closely with the molar fractions of the components. BVs have been widely used to determine the concentration of substances such as drugs, food, fruit juices, dietary formula, and parenteral nutrition solution. (Chang *et al*, 2005)

The refractometer is a precision optical instrument that has been widely used to measure the concentration of sugar, protein, fruits, drugs, parenteral nutrition, and dietary formulas. Refractometers of excellent quality are available through food service equipment catalogues for about USD 150. The hand-held refractometer, is about 20 cm in length and 2 cm in diameter, which permits bedside measurement. The BV of an solution is a physical property of that solution which is remarkably constant and reproducible under varying conditions of concentration, pH, and temperature. The reliability of these physical characteristics can be used to confirm the identity of substances, to analyze mixtures, and to measure the dietary substance. (Chang *et al*, 2005)

Dissolved sugar changes the refractive index of water substantially. Degrees Brix (symbol °Bx) is the sugar content of an aqueous solution. One degree Brix is 1 gram of sucrose in 100 grams (%w/w) of the solution. If the solution contains dissolved solids other than pure sucrose, then the °Bx only approximates the dissolved solid content.

### ***2.11 Fourier transform infra-red (FTIR)***

One of the great advantages of infrared spectroscopy is that virtually any sample in virtually any state may be studied. Liquids, solutions, pastes, powders, films, fibres, gases and surfaces can all be examined with a judicious choice of sampling technique. (Barbart, 2004)

In infra-red spectroscopy, IR radiation was passed through a sample, and some of the infra-red will passed through (transmitted) and some of it will be absorbed by the sample. The resulting spectrum represents the molecular absorption and transmission, creating a molecular structures produce the same infra-red spectrum, making it useful for several types of analysis. (Mariey *et al.*,2001)

FTIR can be used to identify unknown materials, determine quality or consistency of sample and amount of components in mixture. It has been a workhorse technique for materials analysis in the laboratory for over seventy years. The spectrum represents the fingerprint of a sample with absorption peaks which corresponds to the frequencies of vibrations between the bonds of the atoms making up the material. Therefore infrared spectroscopy can result in a positive identification (qualitative analysis) of every different kind of material. In addition, size of peaks in spectrum is a direct indication of the amount of material presents (Salimi *et al.*, 2003)

### ***2.12 Response surface methodology (RSM)***

Response Surface Methodology (RSM) method being useful for the optimization the conditions of test variables in maximizing the response of an experiment. The response is maximized, using RSM based on many reports revealed. RSM can be utilized to assess the relationship between response and independent variables as well to optimize the relevant processes. Beside the period of research also can be decreased.

RSM employs a low-order polynomial equation in a pre-determined region of independent variables, in order to locate the optimum values of independent variables to obtain the best response which was analyzed later. Using RSM, process optimization was faster for gathering experimental research results than conventional, time consuming one-factor-at-a-time approaches. (Alim *et al.*, 2000)



Thus, the advantages of using this software that all the experimental units are used in evaluating effects, resulting in the most efficient use of resource. Then, wider ranges of conditiond with the minimum of resources of the effects are evaluated and for estimating main effects and interaction, a factorial set of treatment is optimized. (Lin *et al*, 2008).

### ***2.13 Inductively Coupled Plasma – Mass Spectrophotometer***

Inductively coupled plasma-mass spectrometry (ICP-MS) combines the ionization efficiency of the argon plasma with the sensitivity and selectivity of mass spectrometric separation. Elements have primary ionization potentials has about 80% that are attainable using an argon plasma as the ionization source; therefore, absolute detection limits are consistently in the 0.1 µg/L range. Analyte ions produced in the plasma are sampled at the instrument-plasma interface through concentric orifices in a pair of nickel sampling cones. (Gabarino and Taylor, 1996)

The principles of ICP-MS were electrostatic lenses accelerate and focus the ion beam into the quadrupole mass analyzer. By applying a pair of radio frequencies and direct-current potentials to the quadrupole rods, only ions with a specific mass-to-charge ratio ( $m/z$ ) are transmitted to the detector; ions with other  $m/z$  ratios collide with the rods and are lost. The ions impinge on a Channeltron-type detector where a current pulse is generated and counted. The detector is mounted 90 degrees off-axis to minimize background signal contributions from photons emitted by the plasma. Inherent to the technique is the capability of selectively measuring all isotopes of a given element, thereby providing isotopic ratio information and allowing isotope dilution analysis to be performed.

Spectral interferences result in the analysis of an element primarily from spectral overlap of isotopes from other elements or molecular ion species that cannot be resolved from the analyte ion. Concurrent elemental isotopic interferences may be eliminated or reduced by a chemical separation to remove the interfering element, by mathematical correction, or by selection of an alternative isotope, whenever possible. In this case of direct spectral overlap from a concomitant ion, use of a different analytical isotope is the best alternative for eliminating interferences. When the analyte is monoisotopic, chemical separation or mathematical correction are the only alternatives.

Inductively coupled plasma mass spectrophotometry, (ICP-MS) is an analytical technique which has undergone rapid development and seen application in diverse fields. As the argon ICP is an ion atmospheric pressure ion source, laser ablation sampling must also be carried out in atmospheric pressure ambient gas in order to ensure no contamination transport of the ablation products to ICP. The expansion of laser induced plasma above the ablation site was constrained by the ambient gas to a volume of only a few cubic millimetres under these condition. The redeposition of ablated material onto the sample substrate following by laser light pulse was promoted by the dissipation of this plasma. This, together with other possible transport losses and condensation onto the sidewalls as the plasma plume exits the ablation hole, reduces the ablation yield and may influence measured element concentrations. (Eggins *et al.*, 1998)

### ***2.14 Heavy metals***

Heavy metals was commonly defined as those having a specific density of more than 5 g/cm<sup>3</sup>. The main threats to human health of heavy metals are exposure to lead, cadmium, mercury and arsenic. (arsenic a mettaloid but usually classified as heavy metal). It have been used in many areas for thousands of years. Early applications including pigments of glazing ceramics, building materials, and pipes for transporting water. (Jarup, 2003)

Any metallic chemical element that has relatively high density and toxic or poisonous at low concentrations was referred as heavy metals. It was including mercury (HG), cadmium (Cd), arsenic (As), chromium (Cr), thallium (TI) and lead (Pb). Heavy metals are natural components of the Earth's crust that cannot be degraded or destroyed. To a small extent they enter our bodies via food, drinking water and air. Some heavy metals are essential as trace elements to maintain the metabolism of human body. Heavy metal poisoning could occur at higher concentrations that for instance resulting from drinking contaminated water, breathe a high ambient air concentrations near emission sources or intake via the food chain. Heavy metals tend to bioaccumulate, which are increases in the concentration of a chemical in a biological organism over time make it dangerous compared to the chemical concentration in the environment. (www.lenntech.com)

Arsenic is a grey-appearing chemical element with atomic number 33, symbol As in the periodic table also termed a metalloid and is considered to be a heavy metal, and arsenic toxicity shares some features with poisonings by other heavy metals. It can exist in a metallic state in three forms (yellow, black, and gray; with gray predominating) and in ionic forms. Historically, it has been used as a medicinal agent, a pigment, a pesticide, and an agent with intent to harm. Up until 2003, arsenic in the form of chromated copper arsenate was used in the US as a treatment to prevent insect infestation of wood used in building. In 2003 the use of this compound was banned by the US Environmental Protection Agency.

Arsenic was mixed with copper or lead to strengthen alloys containing these materials. Some of its compounds react with proteins, mainly the thiol portions, and uncouples the process of oxidative phosphorylation, thus inactivating most cellular functions. Consequently, arsenic and some of the substances it combines with are deadly poisons to most biologic systems, except for a few bacterial species. Arsenic was used in making insecticides and weed killers and thought to be carcinogenic that has potential to cause cancer.

Arsenic can be found as a contaminant in food and water sources such as shellfish and other seafood, as well as fruits, vegetables, and rice; are the foods most commonly contaminated. Resulting from industrial exposure, contaminated wine or illegally distilled spirits can typically arsenic poisoning can occur.

It can enter through erosion and weathering of soils, minerals, and ores into the groundwater. Arsenic can occur in four forms in the environment, i.e.  $As^{5+}$ ,  $As^{3+}$ ,  $As^0$ ,  $As_{-3}$ , which depend on the redox conditions. Thus, the natural As content can range between decimal values and tens of mg/L. The anthropogenic sources of As are: coal combustion, mining of mineral resources, battery production, soil fertilization and metallurgical industry. ([www.medicinenet.com](http://www.medicinenet.com))

### 3 METHODOLOGY

#### 3.1 Introduction

In Chapter 3, the methodology was discussed with a specific focus on the sample of sorbitol preparation, purification of sorbitol using reverse osmosis (RO) membrane, membrane cleaning and analysis the purity of sorbitol. The method of purification of sorbitol by using reverse osmosis (RO) membrane simply because saving the cost and does not using high energy. The method of purification of sorbitol used the membrane filter which is focused on the effect of transmembrane pressure (TMP) and cross-flow velocity (CFV).

#### 3.2 General methodology

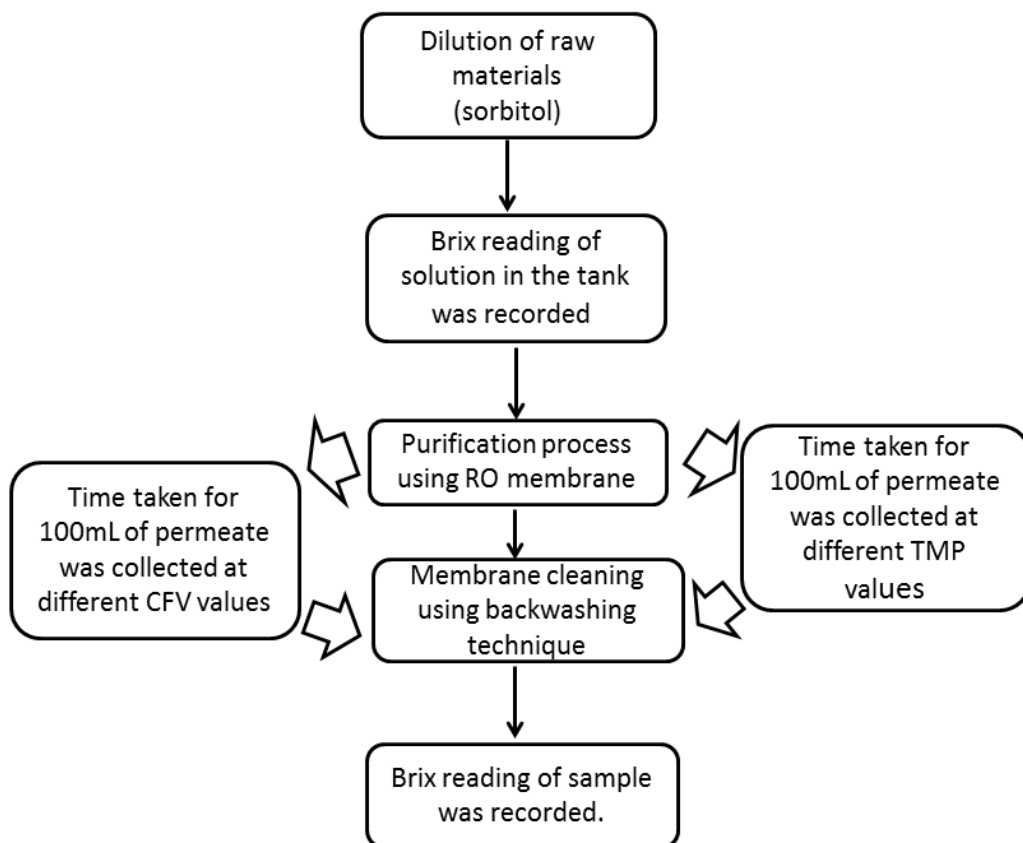


Figure 3-1: Process flow for sorbitol purification

### ***3.3 Fourier Transform Infrared Spectroscopy (FTIR)***

FTIR was used to separate individual frequencies of energy emitted from an infrared source that was accomplished by using a prism or grating. An infrared prism works exactly the same as a visible prism which separates visible light into its color (frequencies). For a liquid sample, a GE plate was used for placing the liquid sample.

After the GE plate was set up, the computer was switched ON. Then OMNIC software was opened and waits up for equipment validation. After the equipment was ready, sample background was collected. After that, sample was collected and rinsed the GE plate with toluene and dried with Kim Wipes tissue. Lastly saved the peak produced.

### ***3.4 Heavy metal determination using ICP-MS***



Figure 3-2: ICP-MS Agilent 7500 series

For the sample having greater than 0.5 percent solids appropriate dilution was required. Water samples must be filtered through a 0.4 micrometers ( $\mu\text{m}$ ) membrane filter (particle-size limit for dissolved constituent definition in most water quality studies is 0.45  $\mu\text{m}$ , Hem, 1985) and was acidified with ultrapure nitric acid to pH less than 2. Detection limits and lower limits of quantitation for analytes included in this method, has been specified in Figure 3-3. The calibration limit for all elements is 500  $\mu\text{g/L}$  for

sample having analyte concentrations exceeding the calibration range must be diluted and re-analyzed. The concentration range of the analyte that normally present in natural water was used as guidance for selecting the calibration range for each analyte. Alternate isotope having a lower natural abundance or by increasing the concentration of the upper calibration standard was being used in order to adjust the concentration range. If an alternative isotope is used evaluation of isobaric or polyatomic ion interferences must be determined. (Gabarino and Taylor, 1996)

Sample solutions are introduced into the ICP as an aerosol that is carried into the center of the plasma (superheated inert gas). The plasma desolvates the aerosol into a solid, vaporizes the solid into a gas, and then dissociates the individual molecules into atoms. This high temperature source (plasma) excites the atoms and ions to emit light at particular wavelengths, which correspond to different elements in the sample solution. The intensity of the emission corresponds to the concentration of the element detected. (Velez, 2009)

[The calibration limit<sup>1</sup> for all elements is 500 micrograms per liter]

Element <sup>2</sup>	Detection limit <sup>3</sup>	Quantitation limit <sup>4</sup>
<sup>7</sup> Li	0.03	0.06
<sup>9</sup> Be	0.02	0.08
<sup>11</sup> B	0.4	2.4
<sup>27</sup> Al	0.2	0.42
<sup>52</sup> Cr	0.2	0.54
<sup>51</sup> V	0.08	0.28
<sup>55</sup> Mn	0.06	0.23
<sup>59</sup> Co	0.008	0.03
<sup>60</sup> Ni	0.03	0.16
<sup>63</sup> Cu	0.02	0.1
<sup>66</sup> Zn	0.08	0.39
<sup>75</sup> As	0.6	2.4
<sup>88</sup> Sr	0.01	0.08
<sup>95</sup> Mo	0.09	0.22
<sup>111</sup> Cd	0.1	0.46
<sup>137</sup> Ba	0.1	0.34
<sup>205</sup> Tl	0.05	0.14
<sup>208</sup> Pb	0.06	0.29
<sup>238</sup> U	0.06	0.26

<sup>1</sup> Limit using injection method calibration scheme; linear range extends to approximately 1 mg/L.

<sup>2</sup> Elements listed show specific isotope used for measurement.

<sup>3</sup> Detection limit at the 99 percent confidence level calculated following the method of Skogerboe and Grant (1970).

<sup>4</sup> Quantitation limit, the experimentally determined analyte concentration that can be reported with 99 percent confidence. Calculated as  $QL = t \times s$ , where  $t$  is the student  $t$  statistic at 99 percent confidence level and  $s$  is the standard deviation of the analyses of a synthetic standard at a concentration of 5 times the detection limit for the respective analyte.

Figure 3-3: Detection and quantitation limits in micrograms per litre for ICP-MS using internal standard manifold injection

### ***3.5 Preparation of sample***

Sorbitol powder obtained from Merck was diluted with 100L of distilled water in a 100L tank was prepared. (Figure 3-4)



Figure 3-4: Reverse osmosis membrane system

### ***3.6 Purification process***

A little modification will be made in conducting this experiment based on the Sakinah et al., (2008) in order to make this method is suitable for this research.

Reverse osmosis membrane is used for the separation of sorbitol solution. The pressure pump will be adjusted to varieties of transmembrane pressure (TMP) and the pressure use in this process ranging from 1.5 to 3.0 bars. The valve will be adjusted to varieties the cross flow velocity (CFV) that range from 0.042 to 1.57 m/s.

Then 100mL of permeate sample was collected at every 10 minutes for 60 minutes and the time taken was recorded. (Azimah, 2010, p. 35-36)



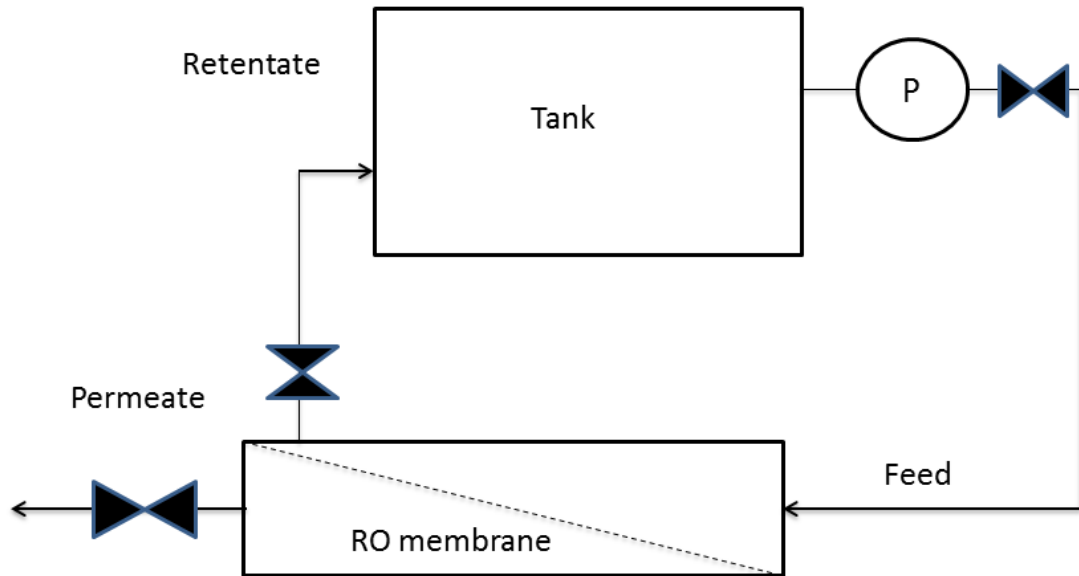


Figure 3-5: Purification operation diagram

Figure 3-5 shows how the membrane operation during the purification process. Brix reading of sorbitol solution in the tank was measured and recorded. Then the feed valve was adjusted to certain angle to obtain the CFV 0.402 m/s and valve of retentate was adjusted to obtain TMP 2 bars. The retentate lines was directed to the feed tank while the permeate line to another tank. The permeate valve was fully opened in allowing the permeate to flow completely during the process. Switch ON the panel to switch on the pump. Then, the solution was circulate for 10 minutes and 100mL of permeate was collected in a measuring cylinder with time taken was recorded. The purification process was continuously run until 60 minutes. Next, the process was repeated with another value of parameter.



Figure 3-6: Permeate sample was collected



Figure 3-7: Collected sample

### ***3.7 Membrane cleaning***

The membrane was cleaned by using the backwashing method using water to remove the fouling form in the membrane where the membrane is flushed with water from the permeate line for about 15 to 20 minutes. (Azimah, 2010, p.37) The membrane was rinsed with water for several times before continue with the next experiment.



Figure 3-8: Membrane was cleaning using backwashing method

### ***3.8 Brix reading***

All the retentate sample collected was taken for brix reading using pocket refractometer. Lastly the sample reading was compared with the reading of sample before the purification. The refractometer well was cleaned using distilled water and Kim Wipes Tissue.



Figure 3-9: Refractometer

### ***3.9 Optimization of sorbitol purification using Response Surface Methodology (RSM)***

The optimization has been done by using the Response Surface Methodology (RSM). It is a collection of mathematical and statistical technique that can be used for studying several effects of several factors at different level and their influence on each other. The low and high values from each parameter have been selected from the screening process. Using this method, the period of research has been decreased instead the response was maximizes.

The objective of RSM was to optimize the response based on the factors investigated. Its study begins with a definition of a problem concerning which response was measured and how it is measured and which variables to be explored. Then the experiment plan was designed.

Design Expert 7 software was used in developing the experimental plan for RSM, also used in analyzing the data collected by performing analysis of variance (ANOVA). Three-dimensional graphs and contour plots was plotted if the model looks good. In brief, a good model must be significant and the lack-of-fit must be insignificant. The value of various coefficient of determination,  $R^2$  must be close to 1.

Three dimensional response surface and contour plots were generated in investigating the interactive effects of any two variables on the response by evaluating two variables

at a time while holding the other at constant level. A three dimensional plot gives a clearer geometrical representation of the nature and extent of the interaction between the variables and response within the experimental range studied (Hui et al., 2010)

The response surface design was developed based on central composite design (CCD). In order to describe the nature of the response surface in the optimum region, a central composite design (CCD) with two levels was performed. The processing parameter involved was shown in Table 3.1. the designs involves 14 runs and variables measured was cross flow velocity (CFV) and transmembrane pressure (TMP).

Table 3-1: Parameter and levels used in RSM

Variables	Unit	Low	high
CFV	m/s	0.5	1
TMP	bar	1.5	2.5

Table 3-2: Design Layout of RSM

Standard	Run	Factor 1	Factor 2
		A: TMP bar	B: CFV m/s
11	1	2.00	0.75
2	2	2.50	0.50
6	3	3.00	0.75
14	4	2.00	0.75
10	5	2.00	0.75
5	6	1.00	0.75
1	7	1.50	0.50
7	8	2.00	0.25
13	9	2.00	0.75
3	10	1.50	1.00
8	11	2.00	1.25
12	12	2.00	0.75
4	13	2.50	1.00
9	14	2.00	0.75

## 4 RESULT AND DISCUSSION

### 4.1 Introduction

Results of FTIR analysis was discussed in subtopic 4.2 and in sub topic 4.3 the results of ICP-MS analysis was discussed. The effect of CFV and TMP towards the flux for purification of sorbitol was obtained by varying the parameter. For CFV effect on the flux (1.5, 2.0, 2.5, and 3 bar) was discussed in the subtopic 4.4 and TMP effect on flux was discussed in subtopic 4.5.

The calculation in obtaining the flow rate using the equation below:

$$Q, \text{Volume flow rate} = \frac{\text{Volume of permeate (mL)}}{\text{Time taken (s)}} \quad \text{----- Eq. 1}$$

For determination of flux, equation below was used:

$$J, \text{Flux} = \frac{Q \left(\frac{\text{mL}}{\text{s}}\right)}{\text{Membrane area (m}^2\text{)}} \quad \text{----- Eq. 2}$$

### 4.2 Fourier Transform Infrared spectroscopy (FTIR)

FTIR spectrum analysis was performed in order to identify functional groups of sorbitol and study the chemical changes of chemical structure in the sample. In this study FTIR spectroscopy was used as a technique to characterize the sample after the purification process. Figure 4-1 and 4-2 shows the result of FTIR analysis of sample at condition 1.042m/s CFV and 2 bars.

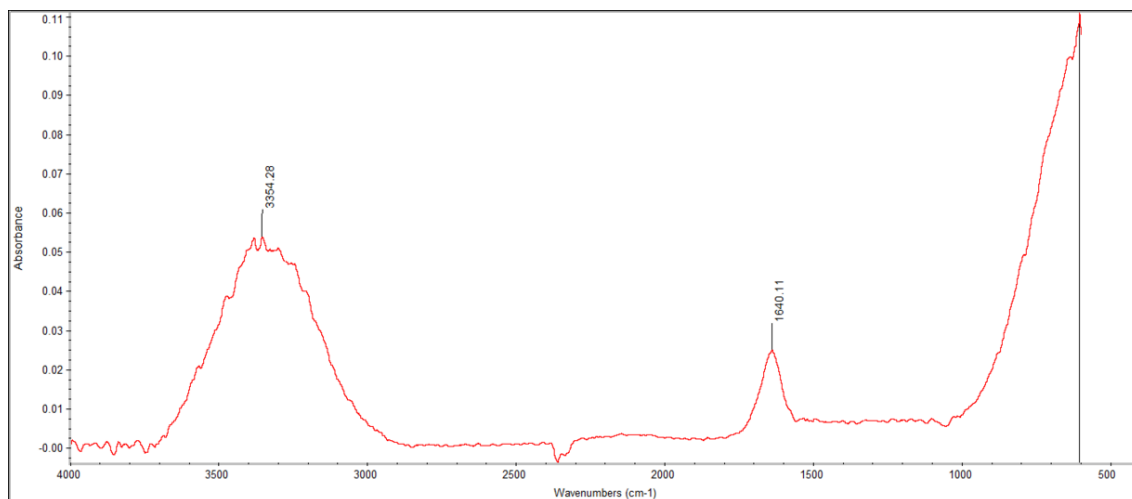


Figure 4-1: Sample before purification

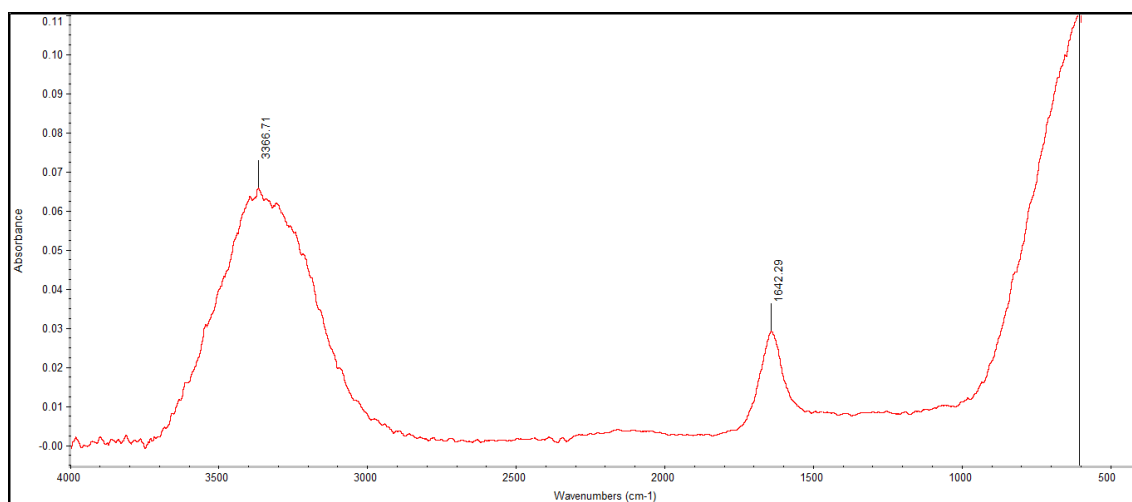


Figure 4-2: Sample after the purification

Based on Figure 4-1 and 4-2, it shows the comparison of peak value between the sample before and after the purification process. Before the process, FTIR spectrum clearly shows the absorption bands in region 3354.28 and 1640.11 due to C=C stretching vibration and –OH stretching vibration. These absorption bands were due to hydroxyl group and alkene group in sample. It shows that the value increases for both peaks, that is from 3354.28 to 3366.71 and 1640.11 to 1642.29. Increased value of –OH peak was assumed that the success of purification.



### ***4.3 Inductively Coupled Plasma –Mass Spectrophotometer (ICP-MS)***

Arsenic occurs in many minerals, usually in conjunction with sulphur and metals, and also as a pure elemental crystal. It is a naturally occurring chemical element found throughout our environment and living systems. Many cases of chronic As poisoning result from drinking water whose as concentration is high.(Czapla *et al*,2013) Arsenic compounds are used in manufacturing variety of products and may enter our environment directly or indirectly from industrial effluent and atmospheric deposition.

As guideline from Canada in drinking water, the maximum allowable level of arsenic from 50 to 25 µg/L and the U.S. from 50 to 10 µg/L. (Kapaj *et al*, 2006) Based on Table 4-1, arsenic concentration in the sample was less than 0.06363 ppb equal to 0.06363µg/L it is below the allowable limit content in the sample. With this concentration it does not give negative health effect to human body such as risk of skin, internal organ, and lung cancers.

Long term exposure (over many years) to high levels of inorganic arsenic was known to contribute risk of cancer and affect the gastrointestinal tract, liver, kidneys, lungs and epidermis. While short term exposure to high level of inorganic arsenic can also cause various health effects such as nausea, diarrhea, and vomiting.

Cadmium is a soft silver-white metal that was found naturally in the earth's crust. However it was common found in combinations with other elements in the environments such as cadmium oxide, cadmium chloride and cadmium sulphide. It was used in metal plating and making of pigments, batteries and plastics.

Exposure to cadmium over a long period time can cause severely irritate and bothering to stomach if eating food with high levels of cadmium. While breathing close to it can caused irritation and damage to the lungs. But if the low dosesof cadmium was expose to human for a long period time it can cause kidney damage. So it is important to make sure the low concentration of cadmium in the sample.

Based on Table 4-1 cadmium content in the sample was 0.016 ppm and (Jarup, 2003) state that the minimum allowable content of cadmium in order kidney damage would not occur is 200 ppm. At this concentration it is in the range if allowable limit of cadmium content, thus it will not cause any harm to be consumed.

Table 4-1: Result of concentration of heavy metal contain in the sample

NO.	PARAMETER	RESULTS	UNIT
1	Arsenic (As)	< 0.06363	ppb
2	Cadmium (Cd)	0.016	ppm

#### ***4.4 Effect of cross flow velocity on permeate flux***

One of the critical operating conditions in influencing the efficiency of membrane filtration was cross flow velocity. It is a highly important parameter for creating an optimal condition for sorbitol purification. The experiment was performed at various cross flow velocity (CFV) as one factor at time (OFAT) values that was 0.204, 1.042, 1.47 and 1.57 m/s, while the other parameter which is transmembrane pressure was kept constant at 2 bars in order to get actual nature of dependence. The process duration was 60 minutes at ambient temperature.

##### ***4.4.1 Effect on flux at CFV 0.204 m/s***

Table 4-2 shows that the result of time taken for 100 ml permeates solution at the CFV 0.204 m/s. From the collected data, flux can be calculated from equation 1 and 2.

Table 4-2: Result of permeate flux at CFV 0.204 m/s

Time(min)	Flow Rate	
	(mL/s)	Flux(mL/m2s)
10	5.4288	0.1454
20	3.7864	0.1014
30	3.9920	0.1069
40	3.9154	0.1049
50	3.9761	0.1065
60	3.7425	0.1002

Figure 4-3 shows the pattern of permeate flux over time under CFV of 0.204 m/s. The plotted pattern shows that the permeate flux declined after the first 10 minutes about

30.26%, from 0.1454 mL/m<sup>2</sup>.s to 0.1014 mL/m<sup>2</sup> and then increased back after 20 minutes about 5.14%. The permeate flux pattern shows that the flux was unstable when it decreased again at 40 minutes time and lastly decreased back about 5.91% at the last 60 minutes of operating time. It was assumed that flux was decreased due to accumulation of sorbitol on the surface of membrane.

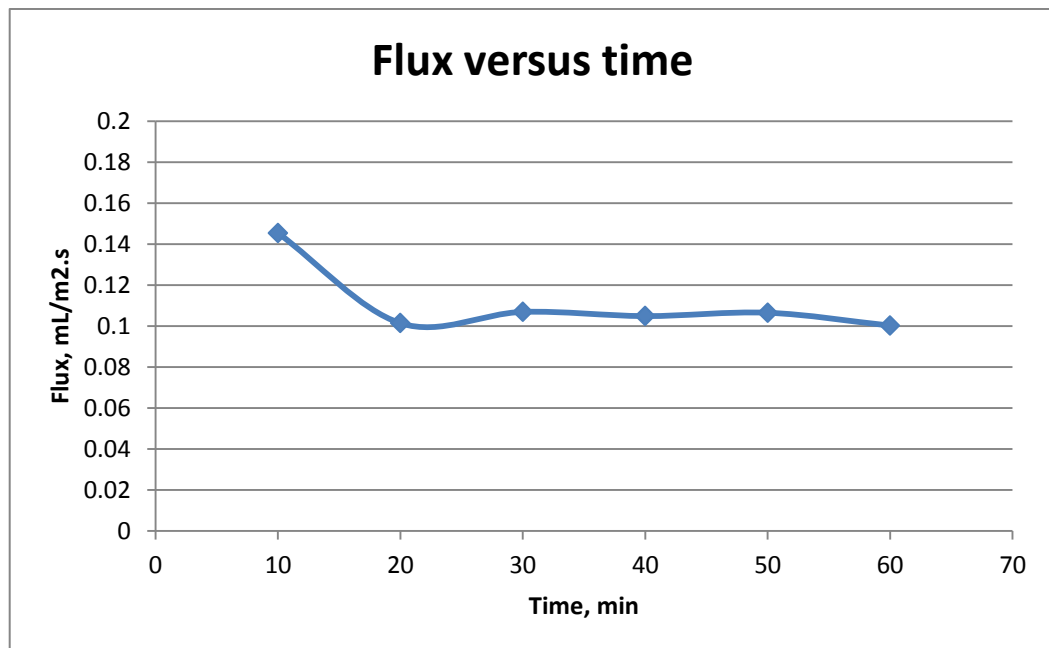


Figure 4-3: Flux pattern at CFV of 0.204 m/s

#### 4.4.2 Effect on flux at CFV 1.042 m/s

Table 4-3 shows that the result of time taken for 100 ml permeates solution at the CFV 1.042 m/s. From the collected data, flux can be calculated from equation 1 and 2.

Table 4-3: Result of permeate flux at CFV 1.042 m/s

Time(min)	Flow Rate	
	(mL/s)	Flux(mL/m2s)
10	3.3411	0.0894
20	3.4435	0.0922
30	3.2938	0.0882
40	3.0572	0.0818
50	2.3685	0.0634
60	2.6157	0.0700

Figure 4-4 shows the pattern of permeate flux over time under CFV of 1.042m/s. The plotted pattern shows that the permeate flux was increased at 10 minutes to 20 minutes, from 0.0894mL/m<sup>2</sup>.s to 0.0922mL/m<sup>2</sup>.s, but starting declined after 20 minutes until at 50 minutes, from 0.0922 mL/m<sup>2</sup>.s to 0.0634 mL/m<sup>2</sup>.s and then increased back at the last 60 minutes to 0.0700 mL/m<sup>2</sup>.s. The declined percentage from 0.0922 mL/m<sup>2</sup>.s to 0.0634 mL/m<sup>2</sup>.s, was about 31.24%. Compared to flux pattern at CFV 0.402 m/s from Figure 4.1, the percentage flux decreased at CFV 1.042 m/s was slight higher when increased in value of CFV due to the turbulence flow that created by high cross flow velocity that scrubbed the foulant on membrane surface, hence decreased foulant accumulation on membrane surface hence decreased foulant accumulation on membrane surface that lead to high permeate flux.

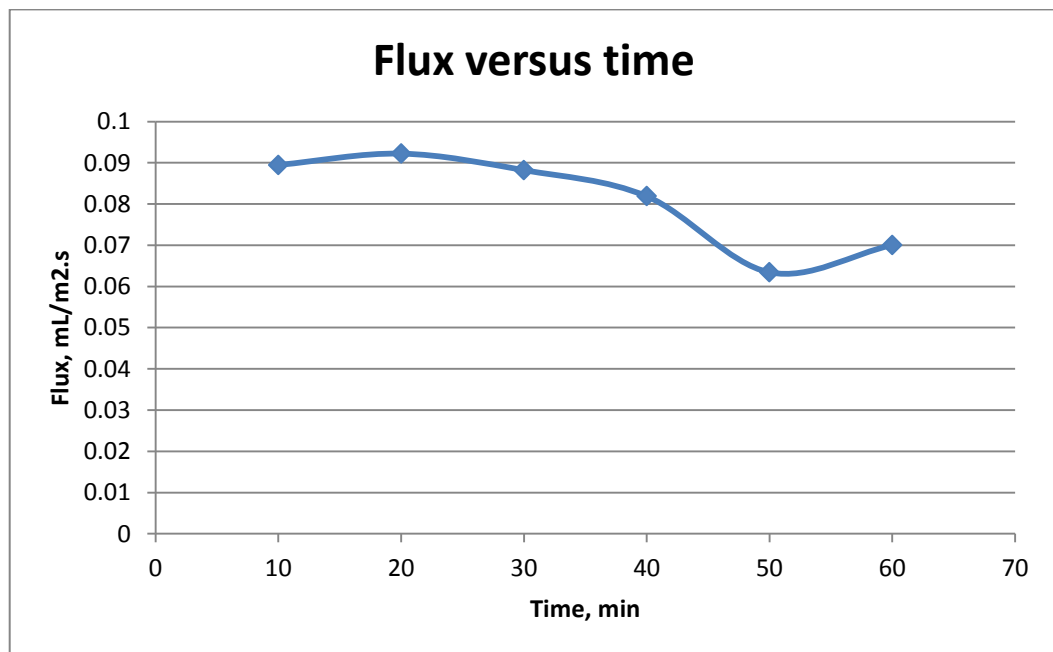


Figure 4-4: Flux pattern at CFV of 1.042 m/s

#### 4.4.3 Effect on flux at CFV 1.47 m/s

Table 4-4 shows that the result of time taken for 100 ml permeates solution at the CFV 1.47 m/s. From the collected data, flux can be calculated from equation 1 and 2.

Table 4-4: Result of permeate flux at CFV 1.47 m/s

Time(min)	Flow Rate	
	(mL/s)	Flux(mL/m <sup>2</sup> s)
10	1.9428	0.0520
20	1.9417	0.0520
30	1.9409	0.0519
40	1.9227	0.0515
50	1.8762	0.0502
60	1.8653	0.0499

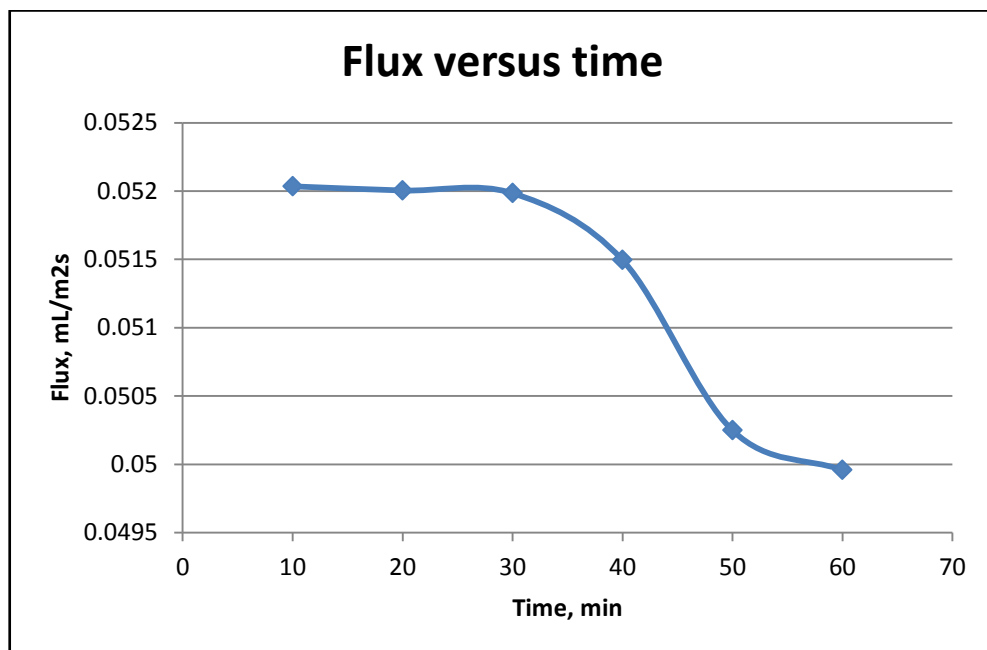


Figure 4-5: Flux pattern at CFV of 1.47 m/s

Pattern of permeate flux over time under CFV of 1.47m/s was shown in figure 4-5. The plotted pattern shows decline gradually until the last 60 minutes. It shows that the permeate flux was steady for the first 30 minutes but started to decreased after 30 minutes of process until the last 60 minutes, from 0.0519 mL/m<sup>2</sup>.s to 0.0499 mL/m<sup>2</sup>.s about 3.8%. Flux value at 1.47 m/s was lower compared to flux value at CFV of 0.1042 m/s in Figure 4-4.

#### 4.4.4 Effect on flux at CFV 1.57 m/s

Table 4-5 shows that the result of time taken for 100 ml permeates solution at the CFV 1.57 m/s. From the collected data, flux can be calculated from equation 1 and 2.

Table 4-5: Result of permeate flux at CFV 1.57 m/s

Time(min)	Flow Rate	
	(mL/s)	Flux(mL/m <sup>2</sup> s)
10	1.4543	0.0389
20	1.4537	0.0389
30	1.4522	0.0389
40	1.4432	0.0387
50	1.4194	0.0380
60	1.4150	0.0379

Figure 4-6 shows the pattern of permeate flux over time under CFV of 1.57m/s. The plotted pattern shows that the permeate flux was steady for the first 30 minutes but started to decline after 30 minutes of process until the last 60 minutes, from 0.0519 mL/m<sup>2</sup>.s to 0.0499 mL/m<sup>2</sup>.s about 3.8% . From the figure 4.4 shows that at time 40 minutes the cake deposition on the membrane was started.

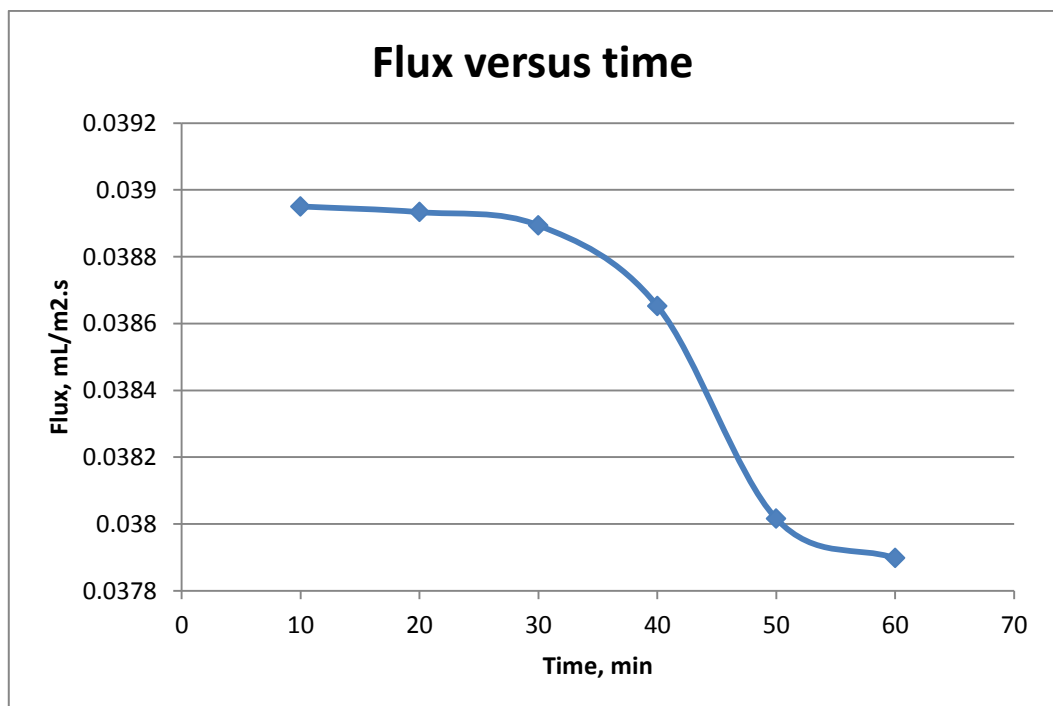


Figure 4-6: Flux pattern at CFV of 1.57 m/s

#### ***4.4.5 Comparison of effect on flux at different CFV value***

Cross flow velocity (CFV) is one of the operating conditions which is a critical parameter that influences the efficiency of membrane separation. This is to create an optimal environment for sorbitol purification from the solution as to minimize the fouling effect. This study investigates the effect of CFV on the permeate flux as well as the determination of sorbitol and the results are shown in Figure 4-7. Based on this figure, 0.204 m/s and 1.042 m/s of CFV exhibited 30% and 23% flux declination compared to 1.47 m/s and 1.57 m/s of CFV that only 2.56%. In other words, the detrimental effect of lower CFV (0.204 m/s and 1.042 m/s) to permeate flux was more pronounced as it showed greater flux decline and higher fouling rate than 1.47 m/s and 1.57 m/s of CFV. This could be related to the compression of fouling layer that covered the membrane surface at low CFV as a function of filtration time and afterward this fouling layer was considerably became more concentrated of thick layer. This saturated layer hindered permeates flux through the membrane pore, which later generates low filtrate. This scenario is apparently attributed to the physical scouring effect of CFV at the membrane surface. Increase in operating CFV resulted in increase in the in the shearing stress at the membrane surface, which redistributes the solute particles at the membrane surface. CFV of 1.47 m/s and 1.57 m/s produced relatively lower fouling layer than 0.204 m/s and 1.042 m/s of CFV, which subsequently easy to remove out from the membrane surface by using hydraulic cleaning. Thus, it is worth noting that high CFV, 1.57 m/s presumably implied greater membrane flux recovery, which consequently contributed to the numerous usages of membrane for longer operation time without replacement. Higher CFV implies a higher steady state flux than lower CFV, which suggests that less fouling occurred on the membrane surface and the pores.

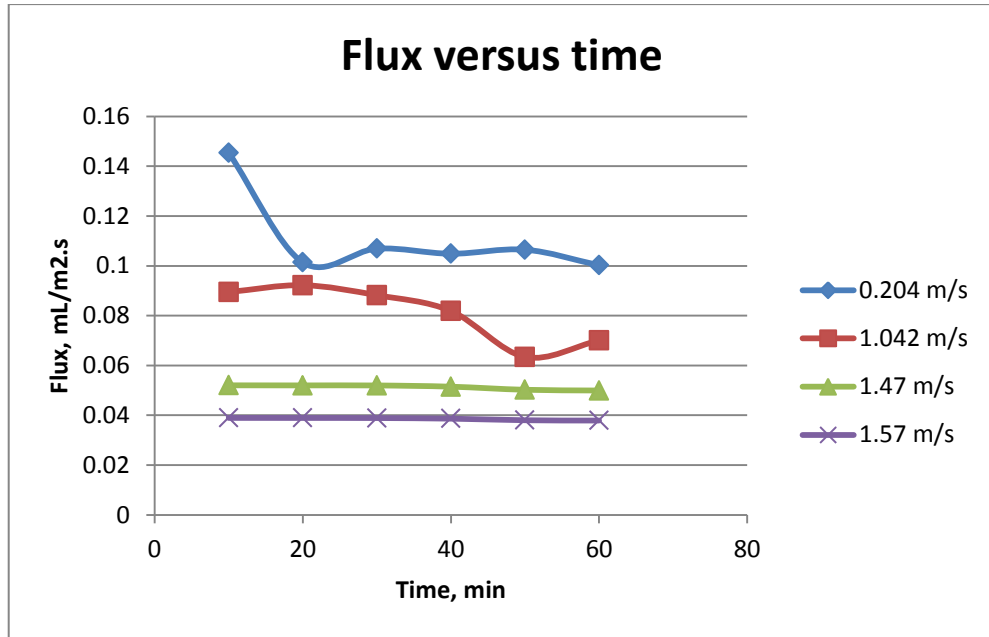


Figure 4-7: Comparison of Flux pattern at different CFV values

#### 4.5 Effects of transmembrane pressure on permeate flux.

The experiment was performed at various transmembrane pressure (TMP) values that was 1.5, 2.0, 2.5 and 3.0 bar, while the other parameter which is cross flow velocity (CFV) was kept constant at 1.042 m/s in order to get actual nature of dependence. The process duration was 60 minutes at ambient temperature. At higher transmembrane pressure, more solute was convected towards the membrane surface, causing severe pore plugging. (rai *et al.*, 2006)

##### 4.5.1 Effect on flux at TMP 1.5 bars

Table 4.-6 shows that the result of time taken for 100 ml permeates solution at the TMP 1.5 bars. From the collected data, flux can be calculated from equation 1 and 2.

Table 4-6: Result of permeate flux at TMP 1.5 bars

Time(min)	Flow Rate (mL/s)	Flux(mL/m <sup>2</sup> s)
10	1.8839	0.0505
20	1.5788	0.0423
30	1.5775	0.0423
40	1.5765	0.0422
50	1.5047	0.0403
60	1.4826	0.0397



Figure 4-8 shows the change in permeate flux pattern over time under TMP of 1.5 bars. The plotted pattern shows that the permeate flux was decreased from 0.0505 mL/m<sup>2</sup>.s to 0.0423 mL/m<sup>2</sup>.s, about 16.24% . Then it was stable for 20 minutes and decreased again to 0.0403 mL/m<sup>2</sup>.s and lastly to 0.0397 mL/m<sup>2</sup>.s. The flux reaches stabilization, but decreased back due to the accumulation of flocculent on the membrane surface.

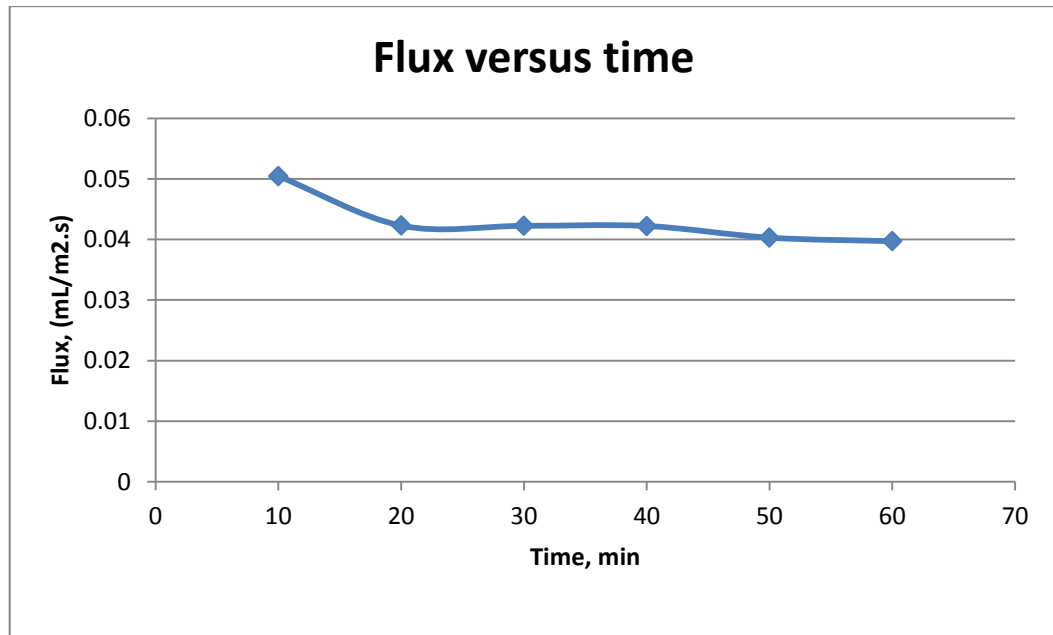


Figure 4-8: Flux pattern at TMP of 1.5 bars

#### 4.5.2 Effect on flux at TMP 2 bars

Table 4-7 shows that the result of time taken for 100 ml permeates solution at the TMP 2 bars. From the collected data, flux can be calculated from equation 1 and 2.

Table 4-7: Result of permeate flux at TMP 2 bars

Time(min)	Flow Rate (mL/s)	Flux(mL/m <sup>2</sup> s)
10	3.3523	0.0898
20	3.4435	0.0922
30	3.2959	0.0883
40	2.8810	0.0772
50	2.6157	0.0701
60	2.6144	0.0700

Figure 4-9 shows the flux pattern of sorbitol purification at TMP 2 bars. Based on the graph, the permeate flux increased from 0.0898 mL/m<sup>2</sup>.s to 0.0922 mL/m<sup>2</sup>.s after 10 minutes operation time. Then declination of permeate flux can be seen after 20 minutes from 0.0922 mL/m<sup>2</sup>.s to 0.077 mL/m<sup>2</sup>, about 16.49 % of declination percentage before it stabilize at 50 minutes of operation time. It was assumed that the cake adsorption to the membrane surface was started at 20 minutes operation times. Thus, when the flocculation of sorbitol increased the permeate decline also increased.

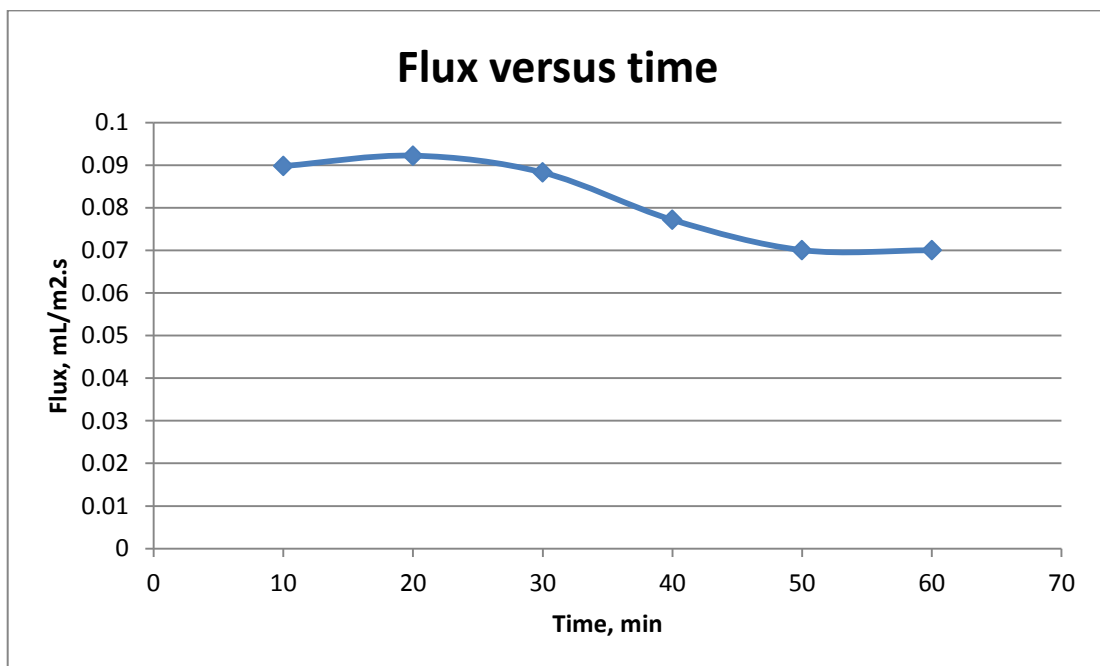


Figure 4-9: Flux pattern at TMP of 2 bars

#### 4.5.3 Effect on flux at TMP 2.5 bars

Table 4-8 shows that the result of time taken for 100 ml permeates solution at the TMP 2.5 bars. From the collected data, flux can be calculated from equation 1 and 2.

Table 4-8: Result of permeate flux at TMP 2.5 bars

Time(min)	Flow Rate (mL/s)	Flux(mL/m <sup>2</sup> s)
10	8.8731	0.2376
20	7.2727	0.1948
30	6.8259	0.1828
40	6.4851	0.1737
50	6.3775	0.1708
60	6.3532	0.1702

Figure 4-10 shows the decline of permeate flux at TMP 2.5 bars. Based on the graph, permeate flux was continuously decreased with time until the flux reached steady state at 50 minutes of purification process. After the first 10 minutes of operation, the cake adsorption was started, and flocculation starting blocking the membrane. Hence the permeate flux decline as increasing with time. The declination percentage was about 28.36%, from 0.2376 mL/m<sup>2</sup>.s to 0.1702 mL/m<sup>2</sup>.s.

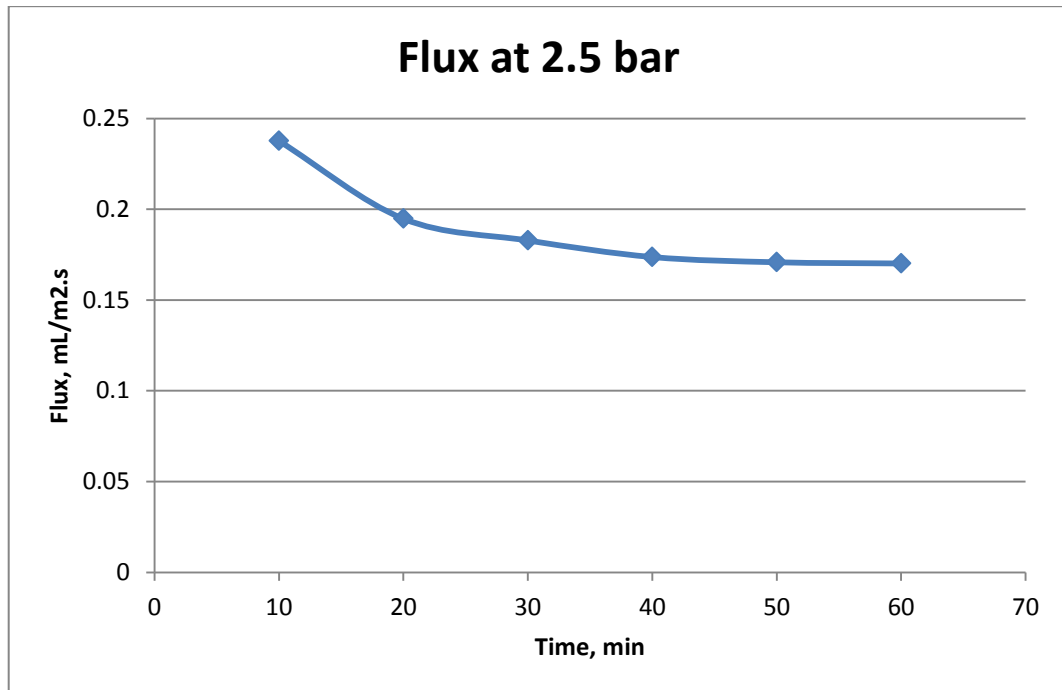


Figure 4-10: Flux pattern at TMP of 2.5 bars

#### 4.5.4 Effect on flux at TMP 3 bars

Table 4-9 shows that the result of time taken for 100 ml permeates solution at the TMP 3 bars. From the collected data, flux can be calculated from equation 1 and 2.

Table 4-9: Result of permeate flux at TMP 3 bars

Time(min)	Flow Rate (mL/s)	Flux(mL/m <sup>2</sup> s)
10	11.4025	0.3054
20	9.8135	0.2628
30	9.8522	0.2639
40	6.7935	0.1819
50	5.4318	0.1455
60	4.9213	0.1318

Figure 4-11 shows the flux pattern of sorbitol purification process at TMP 3 bars. Initially the flux was decreased from 0.3054 mL/m<sup>2</sup>.s to 0.2628 mL/m<sup>2</sup>.s, after the first 10 minutes. Suddenly, it increased to 0.2639 mL/m<sup>2</sup>.s, about 0.41 % after 20 minutes of operation time. This might be cause by the system still unstable. However after 30 minutes, it can be seen that the flux was gradually decreased with time until the last minutes of operation, due to the flocculation of sorbitol at the membrane surface.

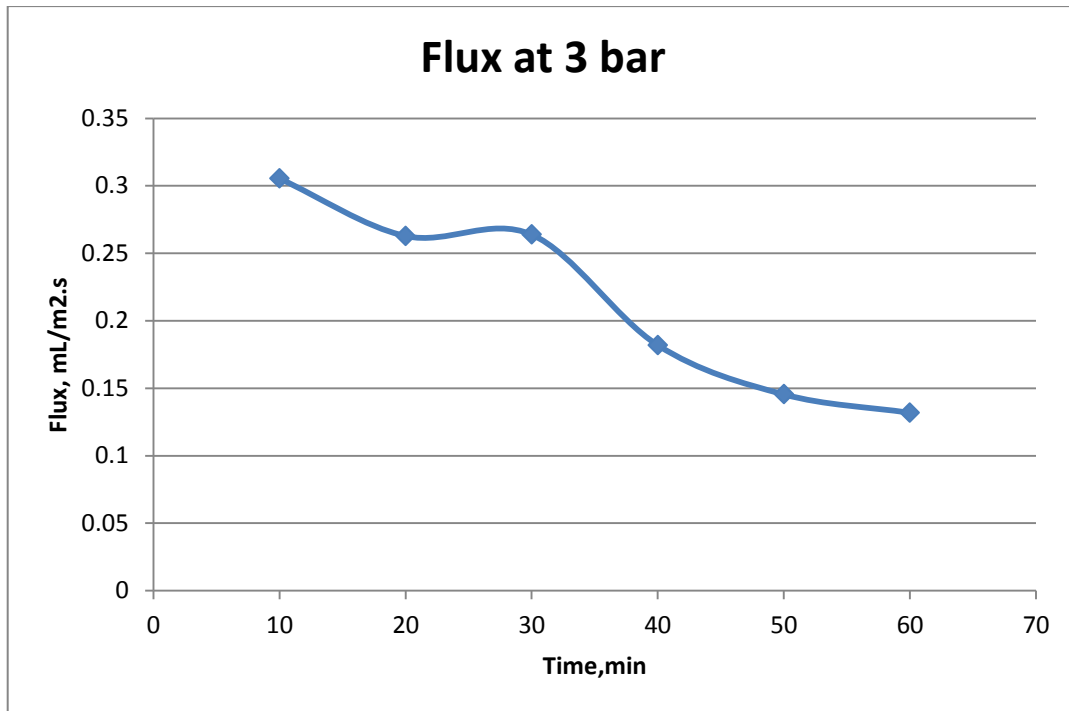


Figure 4-11: Flux pattern at TMP of 3 bars

#### 4.5.5 Effect on flux at different TMP values

Figure 4-12 shows the flux declines as a function of time regardless of various transmembrane pressure (TMP) applied during the purification of sorbitol. Based on this figure, at TMP (3bar) the flux declination was the highest about 50% compared with the TMP of 2.5 bar, 2 bar and 1.5 bar that is only 28%, 23% and 22%. Increment in the operational TMP enhances the flux declination and thud accelerated the operation to achieve a steady state flux condition. This is due to the effect of compaction of cake layer. As the flux increases, it was assumed that more solute mass (sorbitol) was brought to the membrane surface, which increases pore plugging and resistance to flow. Therefore, it is worth to note that the cake layer deposited over the membrane plays an important role in the Sorbitol purification. In fact, severe flux decline was observed at a higher operational TMP. Besides, this supposition could be evidently correlated with Darcy's Law equation (Equation 1) that expresses the relationship between the permeate flux(J), applied TMP( $\Delta p$ ) and total resistance (Rt). Based on this equation, when increase in TMP the total resistance increase and consequently lower the permeate flux. High TMP clearly indicates maximal permeate flux but unfortunately it would also increase the rate of membrane fouling, meanwhile low TMP reduces as it produces low brix reading times.

$$J = \frac{\Delta p}{\mu R} \quad \dots\dots\dots \text{Eq. 3}$$

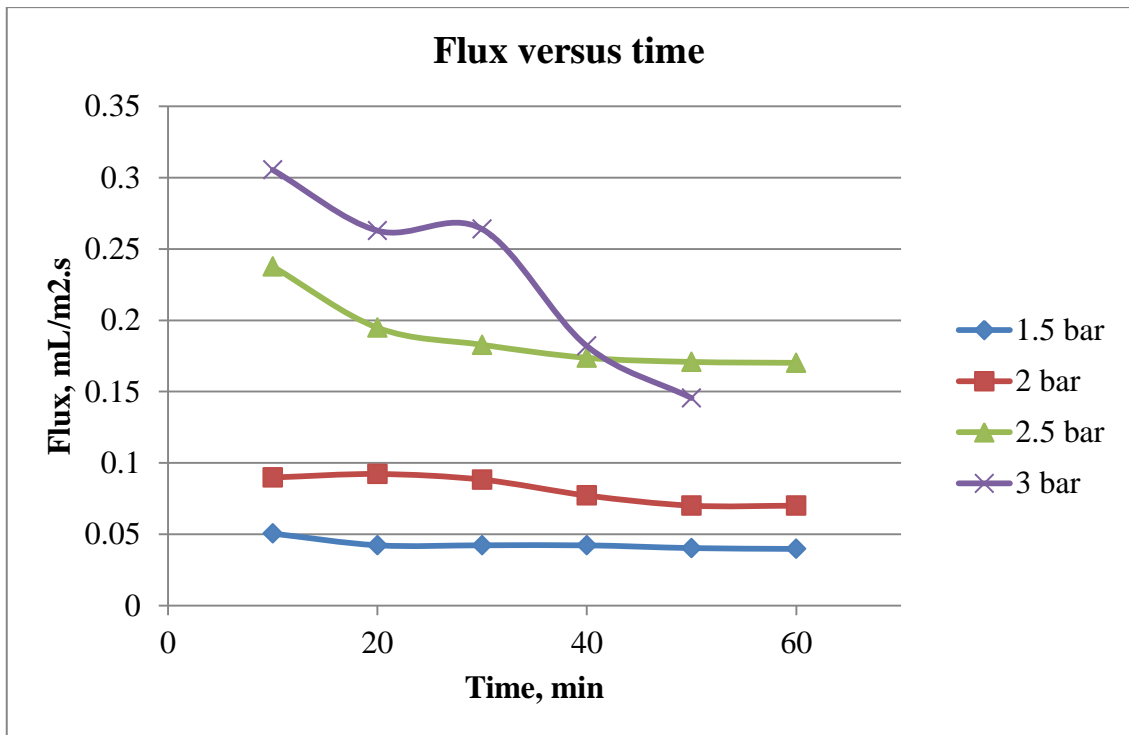


Figure 4-12: Comparison of permeate flux at different TMP values

#### 4.6 Effect of flux declination during sorbitol purification

After 60 minutes of purification process, the solution was subjected to the membrane permeation test. The relative flux, the permeate flux,  $J$  is shown in Figure 4-13 as a function of time for the purification of sorbitol at CFV 0.204m/s which shows the best pattern of permeate flux. The process shows flux decline from the moment of filtration was started. The fouling that happened in these cases could most probably be attributes to the clogging of sorbitol in the membrane. With respect to the permeate flux,  $J$  dropped to 0.101mL/m<sup>2</sup>.s from 0.145mL/m<sup>2</sup>.s within 10 minutes and then increases to 0.107mL/m<sup>2</sup>.s within 10 minutes and then stabilized until it decreased back to 0.100mL/m<sup>2</sup>.s at the last 10 minutes. At the steady state stage, the cake layer was presumably achieved an equilibrium thickness. In order to recover the initial flux, hydraulic and chemical cleaning has to be applied onto the fouled membrane. The fouling is due to high content of sorbitol in the feed. Carrol et al., 1999 and Lee et al., 2004 was supporting well this finding.

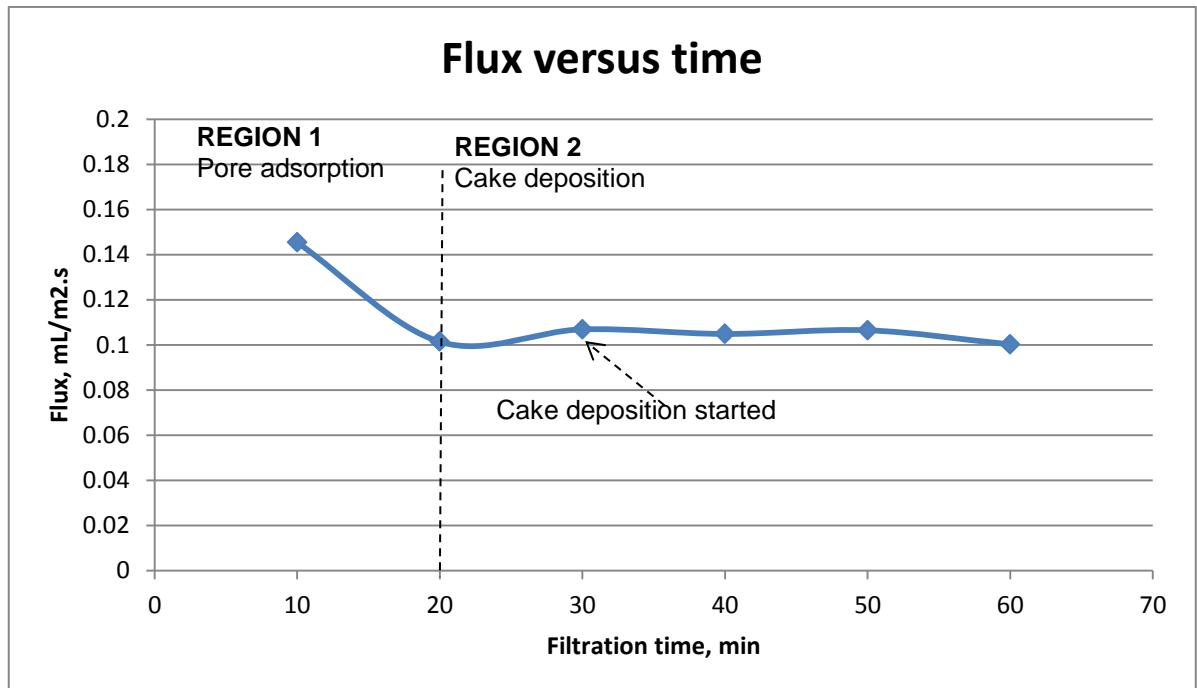


Figure 4-13: Flux pattern at CFV 0.204 m/s

#### 4.7 Brix reading of sorbitol solution

Figure 4-14 shows reading brix of sorbitol solution in retentate line as a function of time. It shows the increment in brix reading of sorbitol solution in retentate stream. Based on figure, before the purification process the brix reading was 0.9% and after 10 minutes of process the value increases to 1.1%. It constant for 20 minutes and the increases to 1.2% after 30 minutes of operation. At the last of 60 minutes the brix value increased to 1.3%. From this result, it was assumed that sorbitol purification was accomplished with the increased of brix percent.

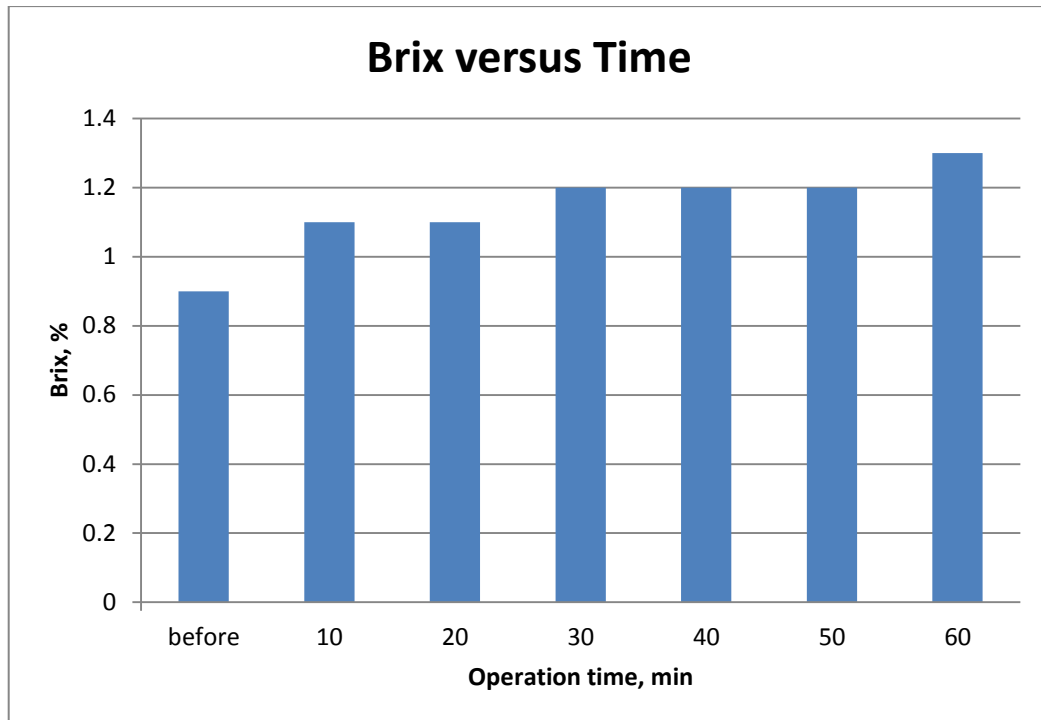


Figure 4-14: Brix reading of retentate solution with operation time

#### ***4.8 Optimization using Response Surface Methodology***

The aim of response surface methodology (RSM) was to determine the optimum operating conditions for the system and to optimize the response based on factors investigated. In this study, parameter of cross flow velocity (CFV) and transmembrane pressure (TMP) were selected for RSM and central composite design (CCD) was applied to identify the optimum CFV and TMP value in order to maximize the flux.

The response surface design was developed based on central composite design (CCD) whereby the factorial portion was a full factorial design with all the combinations of the factors at two levels (high, +1 and low, -1 levels), while the centre points (coded level 0), which is the midpoint between the high and low levels, the axial or star points for which all but one factor was set at 1 and the one factor was set at the outer value corresponding to an  $\alpha$  equal to 2. The experimental plan was generated by using Design Expert version & software as shown in Table 4-10. The design involves 14 runs and the response variables measured was the flux.



Table 4-10: Design layout and experimental result

Standard	Run	Factor 1 A: TMP bar	Factor 2 B: CFV m/s	Response 1 Flux mL/m <sup>2</sup> .s
11	1	2.00	0.75	0.115
2	2	2.50	0.50	0.177
6	3	3.00	0.75	0.268
14	4	2.00	0.75	0.113
10	5	2.00	0.75	0.114
5	6	1.00	0.75	0.015
1	7	1.50	0.50	0.043
7	8	2.00	0.25	0.116
13	9	2.00	0.75	0.116
3	10	1.50	1.00	0.026
8	11	2.00	1.25	0.31
12	12	2.00	0.75	0.113
4	13	2.50	1.00	0.199
9	14	2.00	0.75	0.101

The levels of flux at each experimental point are given in Table 4-10. This table shows that standard error no 8 which was run no. 11 gave the highest permeate flux with 0.31 mL/m<sup>2</sup>.s . The operating parameter to of Standard 8 was 2.00 bars and 1.25 m/s. The lowest permeate flux was 0.015 mL/m<sup>2</sup>.s which was detected as Standard order no. 5 with the operating parameter were 1.00 bar and 0.75 m/s.

Then the results were input into Design Expert software for further analysis. Examination of the Fit Summary output was revealed that quadratic model statistically significant for the flux rate. Therefore this model was used to represent the response for further analysis.

#### 4.8.1 Optimization of sorbitol purification process flux

In order to produce high purity of sorbitol, two factors of process were carried out in this study. The factors are cross flow velocity (CFV) and transmembrane pressure (TMP). The experimental plan exhibiting the different combinations of cross flow velocity (CFV) and transmembrane pressure (TMP). In this study, a full factorial central composite design (CCD) involving 14 runs was carried out. Apparently, the multiple regression equation for Flux after the process cross flow velocity (CFV) and transmembrane pressure (TMP) as the main variables was as follows:

(Coded)

$$\text{Flux} = +0.019 + 0.023*A + 0.0016*B + 0.0031*AB + 0.0068A^2 + 0.000944B^2 \quad \dots\dots\dots \text{Eq. 4}$$

(Actual)

$$\text{Flux} = +0.075957 - 0.081098*TMP - 0.065453*CFV + 0.024585*TMP*CFV + 0.027170*TMP^2 + 0.015108*CFV^2 \quad \dots\dots\dots \text{Eq. 5}$$

According to Equation 5, the largest value of estimated regression coefficient for sorbitol purification (TMP=0.081098). Thus, it is worth to note that the transmembrane pressure was the prime factor which had the greatest effect on sorbitol purification. Consequently, this condition enhances the purification process.

The coefficient determination,  $R^2$  was found to be 0.9973 which corresponding to the purity of sorbitol. The value of  $R^2$  is a measure of the total variation of the observed values of sorbitol purification about the mean explained by fitted model, which often described in percentage. This apparently indicates that 99.73% of the total variation in sorbitol purification is explained by the fitted model (Equation 4). Based on table 4.10, the differences between predicted  $R^2$  and adjusted  $R^2$  are less than 0.01, which implies that the predicted  $R^2$  is in reasonable agreement with the adjusted  $R^2$ .

In addition the ratio of the adequate precision was greater than 4 that are 79.98 which indicate adequate model discrimination. Moreover in this study, the statistical analysis of the model was carried out using Fisher's statistical test for the analysis of variance (ANOVA) and the result was tabulated in Table 4-11. The F-value is the ratio of mean

square due to the regression to the mean square error. The F-value of the sorbitol purification is 526.31 which is greater than the tabulated F the null hypothesis is rejected at the  $\alpha$  level of significance and infers that the unexplained variation. In this case the nu; hypothesis is rejected at <0.0001 level of significance for sorbitol purification. This  $\text{prob}>F$  (<0.05) indicated that the model terms were significant. Furthermore, according to Table 4-11, A, A<sup>2</sup>, B<sup>2</sup>, A<sup>3</sup> and B<sup>3</sup> were a significant model. The lack of fit F-value of 1.21 implied that the lack of fit was not significant.

Table 4-11: ANOVA table (partial sum of square) for quadratic model (response flux)

Source	Sum Of Squares	dF	Mean Square	F Value	p-value Prob> F	
Model	0.0075	5	0.0015	526.31	<0.0001	significant
A-TMP	0.0064	1	0.0064	2214.42	<0.0001	
B – CFV	0.00012	1	0.000012	4.12	0.0819	
AB	0.000038	1	0.000038	13.17	0.0084	
A <sup>2</sup>	0.0011	1	0.0011	388.19	<0.0001	
B <sup>2</sup>	0.000008	1	0.00000802	2.79	0.1385	
Residual	0.00002	1	0.0000029			
Lack of Fit	0.0000066	2	0.0000033	1.21	0.3719	Not significant
Pure error	0.000014	5	0.000005			
Cor Total	0.0076	12				
Std. Dev	0.00169			R <sup>2</sup>		0.9973
Mean	0.025			Adj R <sup>2</sup>		0.9955
Adeq Pre	79.983			Pred R <sup>2</sup>		0.9858

This model can be used to predict the reject flux rate within the limits of the experiment. The normal probability plot of residuals and the plot of the residuals versus the predicted response for both the rejection and flux rate are shown in Figure 4-15, 4-16

and 4-17. Figure 4-15 revealed that it has no obvious pattern and unusual structure. It also shows equal scatter above and below the line. This implies that the model proposed is adequate and there is no reason to suspect any violation of the independence or constant variance assumption.

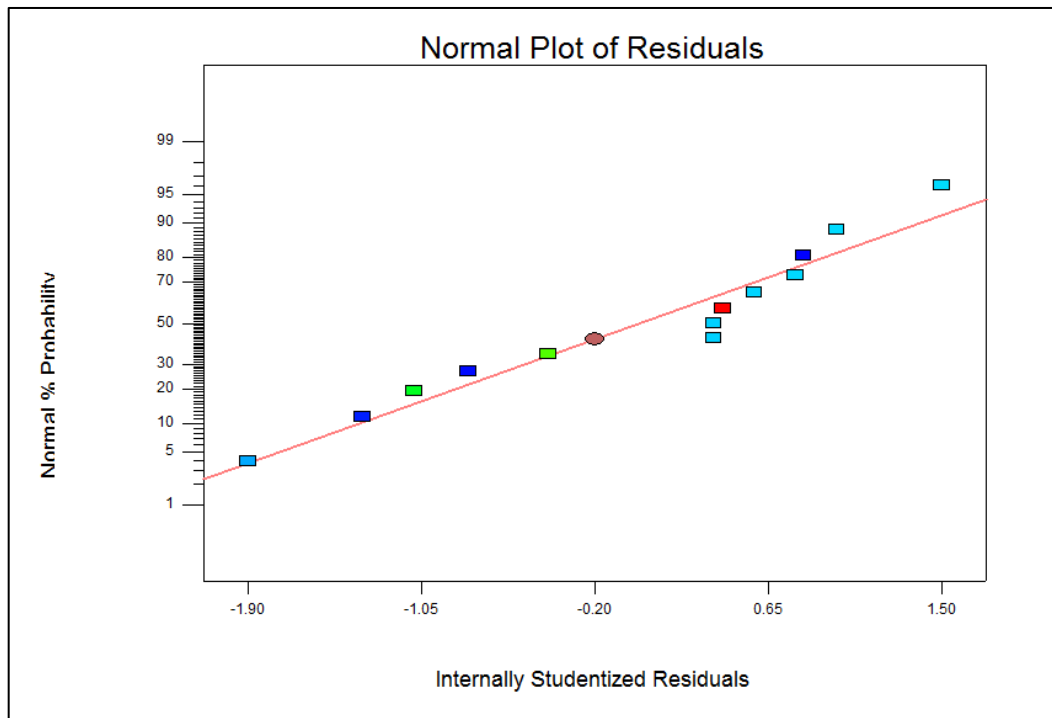


Figure 4-15: Normal probability plot of residual for rejection rate

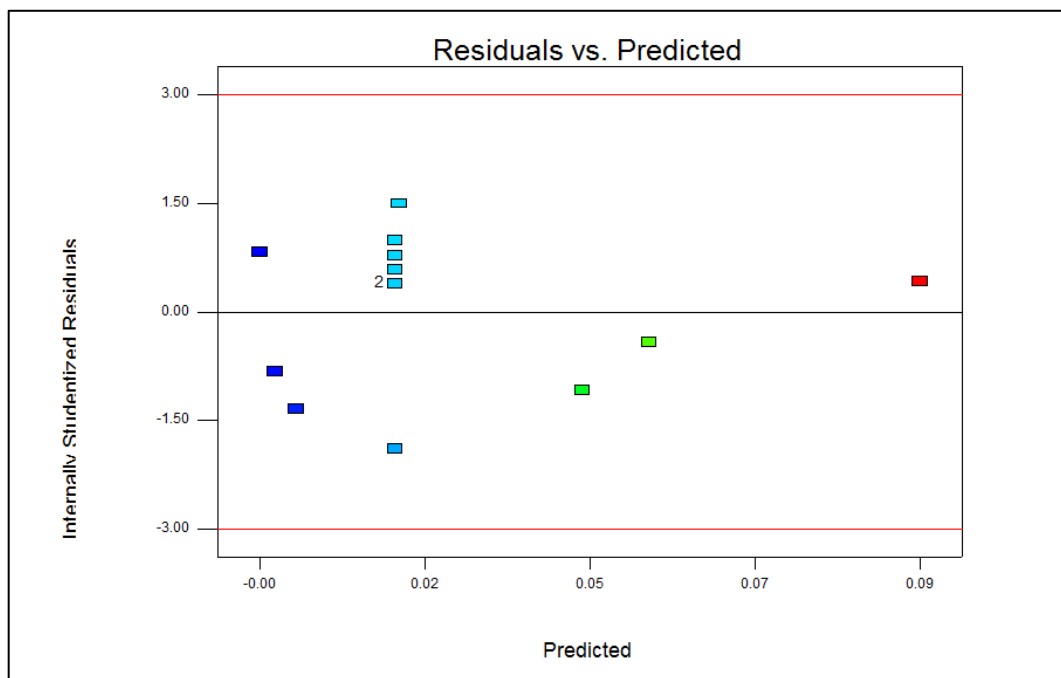


Figure 4-16: Plot of residuals versus predicted response for rejection rate

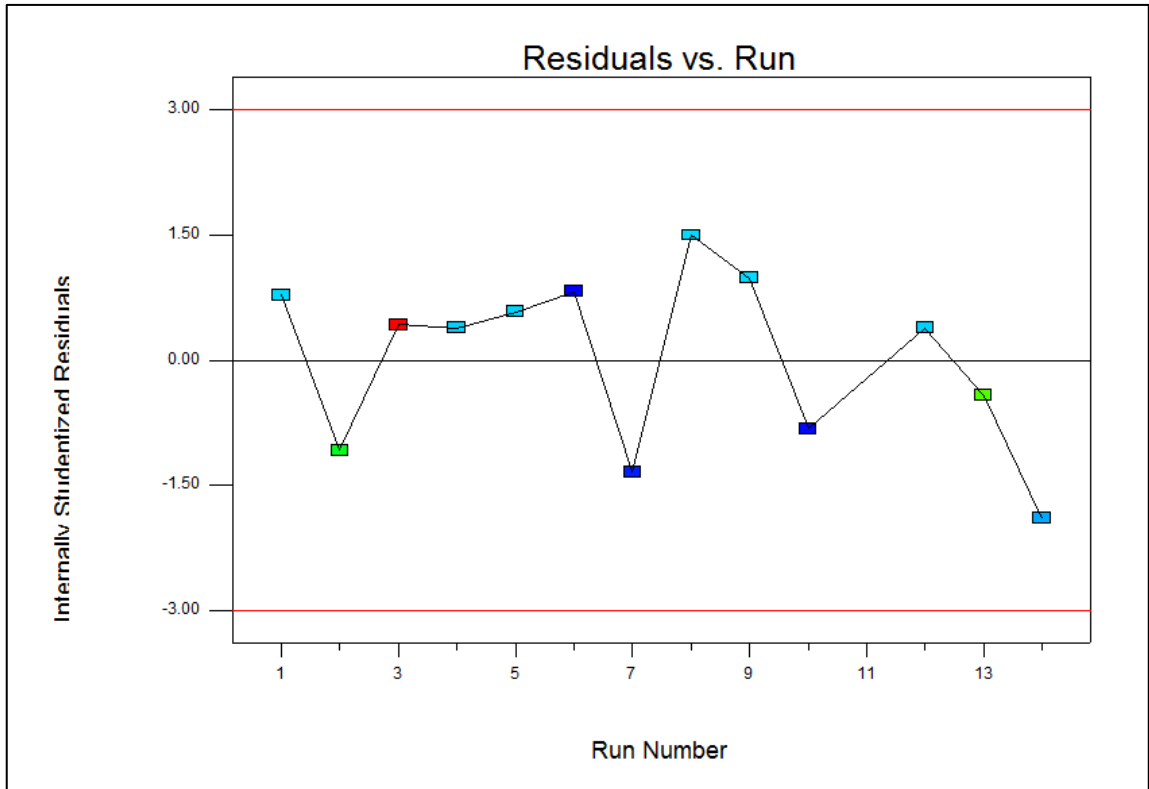


Figure 4-17: Normal Plot of Residuals Plot

Figure 4-18 shows the contour plot and response surface curves illustrate the effects of applied cross flow velocity (CFV) and transmembrane pressure (TMP) on flux. From the figure it reveals that equipment's limitations make the increase in cross flow velocity and transmembrane pressure results in increase in flux. It does not have further values due to those limitations. In brief, higher cross flow velocity and transmembrane pressure would consequence provide better high flux. This scenario is again presumably related to the size of sorbitol molecular structure effects by CFV and TMP towards the permeate flux. In addition, the high CFV and TMP help to increase the physical scouring effect at the membrane surface and improve back transport into the bulk solution. Also, shear stress generated from this this CFV helps to reduce the rate of cake deposition and the resultant cake thickness.

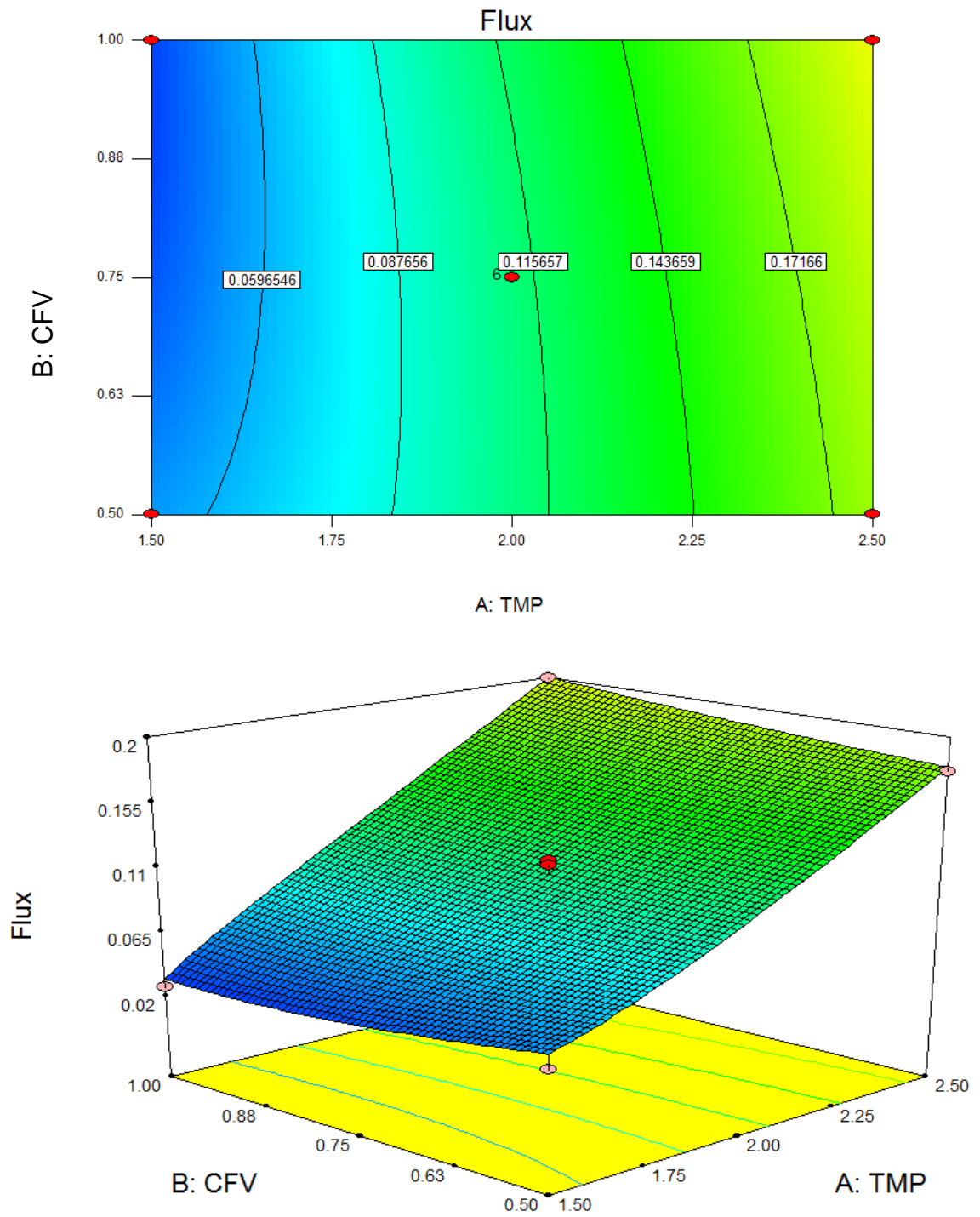


Figure 4-18: Contour plot and response surface curves of flux for interaction of CFV and TMP.

#### 4.8.2 Validation of Empirical Model Adequacy

Adequacy of the developed empirical models needs to be verified or validated in order to confirm the prediction accuracy. Thus, a series of additional experiments were conducted in order to validate the reliability of the model, using the conditions previously used and combined with additional experiments. Table 4-12 shows the results of operating conditions with experimental design in confirmation run. Actual values obtained and its associated predicted values from the selected experiments were compared for further residual and percentage error of analysis.

Table 4-12: Results of operating conditions with experimental design in confirmation run

No	CFV (m/s)	TMP (bar)	Predicted Flux (mL/m <sup>2</sup> .s)	Actual Flux (mL/m <sup>2</sup> .s)	Residual	% Error
1	1.00	2.50	0.199661	0.215	0.015339	7.13
2	0.75	2.00	0.113	0.116	0.003	2.58
3	0.50	2.50	0.177	0.163	-0.086	8.58

Equation used to calculate the error;

$$\text{Residual} = (\text{Actual value} - \text{Predicted value}) \dots\dots\dots (1)$$

$$\% \text{ Error} = \frac{\text{Residual}}{\text{Actual Value}} \times 100\% \dots\dots\dots (2)$$

Equation 1 and 2 was used in calculating the percentage error between actual and predicted value of response over selected range of operating levels. Results of Table 4-12 shows that the percentage error was in the range from 2.58% to 8.58% for permeate flux. Thus it implied that the empirical model developed were considerably accurate for responding term which is permeate flux as the percentage error between the actual and predicted values were well within 10% values, suggesting that the model adequacy was reasonably within the 90% of prediction interval.

## **5 CONCLUSION AND RECOMMENDATION**

### ***5.1 Conclusion***

In this research the purification of sorbitol was successfully carried out using reverse osmosis (RO) membrane. Value of sorbitol was increased after the purification process and any other impurities can be removed using reverse osmosis membrane. The substance contained in the sample can cause fouling on membrane surface. The optimized membrane operating parameters can control amount of fouling so that membrane can be used for a longer time period. The analysis of cross flow velocity (CFV) and transmembrane (TMP) for this research can be used for further studied and experiments for optimizing the process of sorbitol purification using reverse osmosis (RO) membrane.

The maximum permeate flux obtain was at 0.116 mL/m<sup>2</sup>.s when the operating conditions was at optimum was TMP=2.00 bars and CFV = 0.75 m/s. The combination between TMP and CFV enhance the permeate flux. Increasing the TMP causes a decline in permeate. Besides, objective which is to optimize the effect of TMP and CFV on permeate flux has been achieved by using Response Surface Methodology.

### ***5.2 Recommendation***

In order to enhance he purification process, additional time was needed for further study to give a longer time for the flux to reached steady state. Besides that, further study could be done on mechanical machine such as pump performance and impeller design that can optimize the process and increase the pump performance by changing the pump that can overcome the limitation in pump performance.

Moreover, other analysis method should be done to determine sorbitol content in sample such as High Performance Liquid Chromatography (HPLC) test. Field Emission Surface Electron Microscope (FE SEM) analysis should be done on sample for observation at high magnification and identifying the elements that compose a sample by detecting the energies of characteristic X-rays.



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# APPENDIX

Spiral Wound membrane area = 37.34 m<sup>2</sup>

Membrane length = 1.07 m

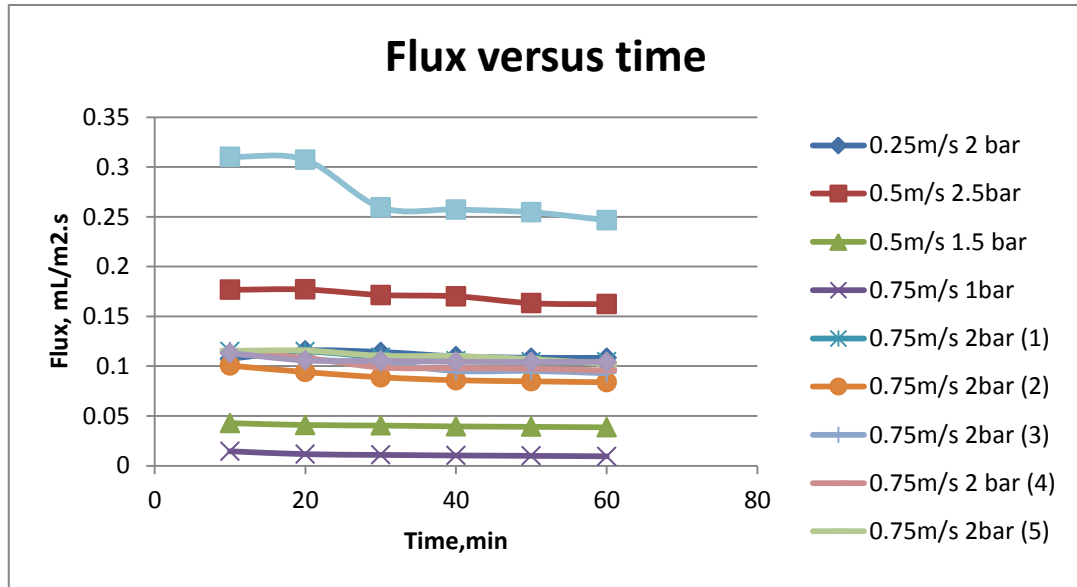


Figure A-1: Comparison of flux pattern for RSM

Response 1 Flux Transform: Power Lambda: 1.81

\*\*\* WARNING: The Cubic Model is Aliased! \*\*\*

Sequential Model Sum of Squares [ Type 1 ]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob>F	
Mean vs Total	8.343E-003	1	8.343E-003			
Linear vs mean	6.398E-003	2	3.199E-003	27.27	<0.0001	
2FI vs Linear	3.778E-005	1	3.778E-005	0.30	0.5975	
<u>Quadratic vs 2FI</u>	<u>1.115E-003</u>	<u>2</u>	<u>5.575E-004</u>	<u>194.31</u>	<u>&lt;0.0001</u>	<u>Suggested</u>
Qubic vs Quadra	6.563E-003	2	3.282E-006	1.21	0.3719	Aliased
Residual	1.352E-005	5	2.704e-006			
Total	0.016	13	1.224E-003			

“Sequential Model Sum of Squares [Type1]”. Select the higher order polynomial where the additional terms are significant and the model is not aliased.

### Lack of Fit Tests

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob>F	
Linear	1.159E-003	5	2.319E-004	85.74	<0.0001	
2FI	1.122E-003	4	2.804E-004	103.69	<0.0001	
<u>Quadratic</u>	<u>6.563E-006</u>	<u>2</u>	<u>3.282E-006</u>	<u>1.21</u>	<u>0.3719</u>	<u>Suggested</u>
Cubic	0.000	0				Aliased
Pure Error	1.352E-005	5	2.704E-006			

“Lack of Fit Tests”: Want the selected model to have insignificant lack-of fit.

### Model Summary Statistics

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	0.011	0.8451	0.8141	0.6178	2.893E-003	
2FI	0.011	0.8501	0.8001	0.6277	2.818E-003	
<u>Quadratic</u>	<u>1.694E-003</u>	<u>0.9973</u>	<u>0.9955</u>	<u>0.9858</u>	<u>1.076E-004</u>	<u>Suggested</u>
Cubic	1.644E-003	0.9982	0.9957			Aliased

+Case(s) with leverage of 1.0000: PRESS statistic not defined

“Model Summary Statistics” Focus on the model maximizing the “Adjusted R-Squared” and the “Predicted R-Squared”

Table A-1: Diagnostics Case Statistics

Standard Order	Actual Value	Predicted Value	Residual	Leverage	Internally Studentized Residual	Externally Studentized residual	Influence on Fitted Value DFFITS	Cook's Distance	Run Order
1	3.362E-003	4.932E-003	-1.570E-003	0.519	-1.337	-1.435	-1.491	0.322	7
2	0.044	0.045	-1.271E-003	0.519	-1.083	-1.099	-1.142	0.211	2
3	1.352E-003	1.975E-003	-6.230E-004	0.798	-0.819	-0.798	1.588	0.443	10
4	0.054	0.054	-3.242E-004	0.798	-0.426	-0.400	-0.796	0.120	13
5	4.997E-004	-1.233E-004	6.230E-004	0.798	0.819	0.798	1.588	0.443	6
6	0.092	0.092	3.242E-004	0.0798	0.426	0.400	0.796	0.120	3
7	0.020	0.019	9.472E-004	0.860	1.497	1.681	*4.17	*2.30	8
9	0.-16	0.019	-2.957E-003	0.151	-1.895	-2.514	-1.061	0.107	14
10	0.020	0.019	9.065E-004	0.151	0.581	0.551	0.233	0.010	5
11	0.020	0.019	1.219E-003	0.151	0.781	0.757	0.319	0.018	1
12	0.019	0.019	5.958E-004	0.151	0.0382	0.357	0.151	0.004	12
13	0.020	0.019	1.534E-003	0.151	0.983	0.980	0.414	0.029	9
14	0.019	0.019	5.958E-004	0.151	0.382	0.357	0.151	0.004	4

\*Exceeds limits

Proceed to Diagnostic Plots (the next icon in progression). Be sure to look at the:

- 1) Normal probability plot of the studentized residuals to check for normality of residuals.
- 2) Studentized residuals versus predicted values to check for constant error.
- 3) Externally Studentised Residuals to look for outliers, i.e., influential values.
- 4) Box-Cox plot for power transformations.

If all the model statistics and diagnostic plots are OK, finish up with Model Graphs icon.

Table A-2: IR Absorptions for Representative Functional Groups

Functional Group	Molecular Motion	Wavenumber (cm <sup>-1</sup> )
Alkenes	C=C stretch (isolated)	3100-3010
	C=C stretch (conjugated)	1690-1630
Aromatics	C=C stretch	1600-1475
Alcohols	O-H stretch	3400-3300
	C-O stretch	1260-1000



Design-Expert Software  
(Flux)<sup>1.81</sup>

Lambda

Current = 1.81

Best = 1.81

Low C.I. = 1.4

High C.I. = 2.12

Recommend transform:

Power

(Lambda = 1.81)

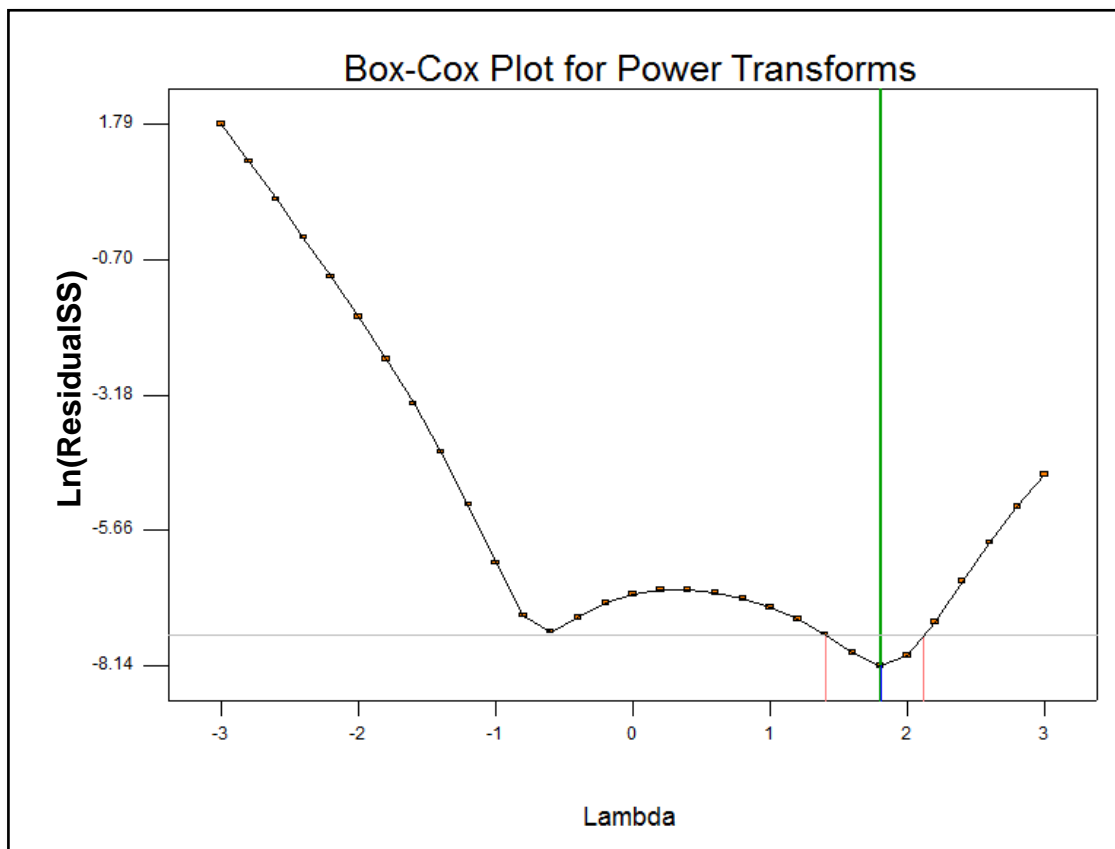


Figure A-2: Box-Cox Plot









