

CHAPTER III

RESEARCH METHODOLOGY

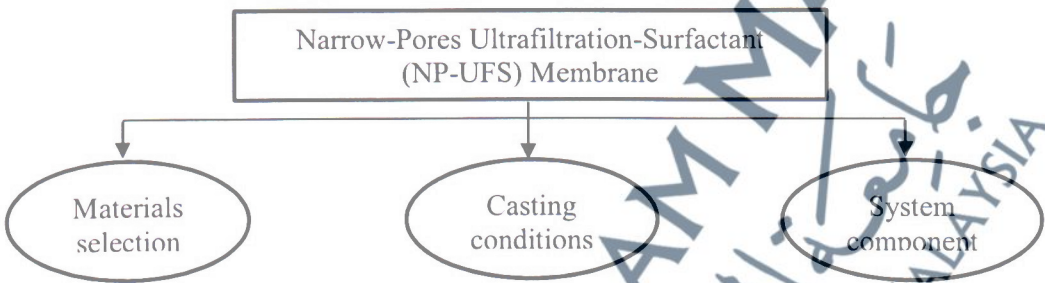
3.1 General Preview

This chapter describes on the development of newly ultrafiltration-surfactant (UFS) membranes for protein separation. The materials selection, dope preparation, membranes fabrication, performance evaluation and characterization are well discussed in this section. In addition, characterization of developed UF membranes based on Scanning electron microscope (SEM) allowed determination the effects of preparation factor on the performances and membranes morphological structures. Besides, Fourier-transform infrared spectrophotometer (FTIR) is used to determine the functional group of additives or surfactants in the membrane matrix of the polymer used. Thermogravimetry analysis (TGA) also will be used in order to determine the thermal stability of the polymer solution. In order to produce high performance UFS membranes, membrane is fabricated using a simple casting machine at different conditions based on an established dry/wet phase inversion technique.

3.2 Research Design

Fabrication of narrow-pores ultrafiltration-surfactant (NP-UFS) membranes can be influenced by many factors and some of them are given in Figure 27.

FIGURE 27: Factors Affecting the Performance of UF Membrane

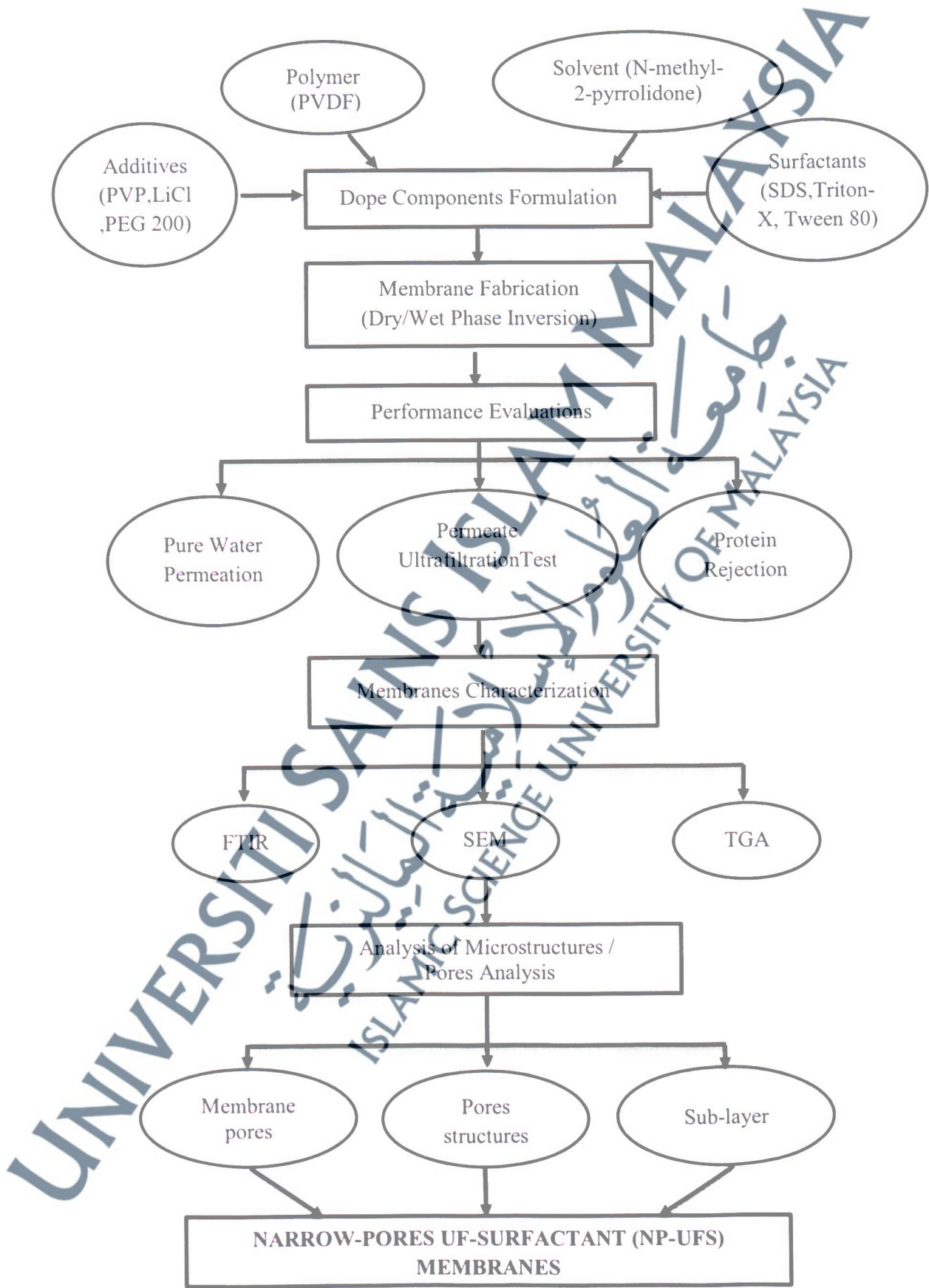


The parameters that determine the performance of NP-UFS membrane are including of polymer selection, solvent and non-solvent selection, dope solution preparation method, casting parameters, flux, and protein rejection test. The experimental framework of this study is illustrated in Figure 28.

3.3 Materials Selection

Materials selection is one of the most important parameter in membrane preparation especially in dope formulation. System components can be either a binary, tertiary or quaternary which can influences the membrane final structures and properties. Based on Loeb-Sourirajan technique, the composition of solution consists of polymer, solvent and sometimes non-solvent(s) or additive(s).

FIGURE 28: Research Framework



3.3.1 Polymer

The Polyvinylidene fluoride (PVDF) membranes have been widely used in ultrafiltration (UF) due to its semi-crystalline polymer with good chemical resistance, mechanical strength and properties, excellent thermal and chemical stability. PVDF is a polymer which one of the most popular hydrophobic membrane materials, having the molecular structure as shown in Figure 29 below. In this study, Kynar® PVDF resins were used for membrane preparation. Kynar® PVDF resins have exceptional chemical resistance compared to other membrane materials such as cellulose acetate, polyamide, polypropylene, polysulfone, and polyethersulfone resins. In addition Kynar® PVDF resins have excellent chemical resistance to harsh chemicals such as chlorine, bromine, hydrogen peroxide, alcohols, sodium hypochlorite, chlorine dioxide, inorganic acids and organic acids.

FIGURE 29: Repeating Unit of PVDF Monomer

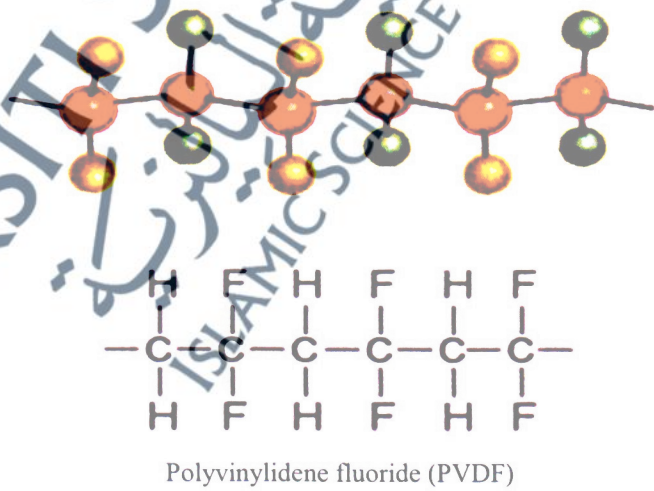


Table 4 shows physical, mechanical and thermal properties of PVDF membranes. Due to the hydrophobic nature of PVDF materials, however it might

cause difficulty in the preparation of highly porous membrane. It has been reported that the morphology and properties of PVDF asymmetric membranes could be adjusted by dissolving a third component as additives in the casting solutions consisting of a polymer and a solvent. In general, the additives were considered as pore forming agents. Then, these additives would enhance the membrane fluxes and reduce rejection. In order to develop narrow pores ultrafiltration-surfactant (UFS) membrane, dope solutions for this study are prepared in the range 13-19%. The less concentration of polymer solutions tends to produce a more hydrophilic and porous membrane. Therefore, this aspect will help on the determination of better membranes performance with a highly permeation rate.

TABLE 4: Physical, mechanical and thermal properties of PVDF membranes

Property	Value
<i>Physical properties</i>	
Material form	Pellets
Category	Homopolymer
Molecular weight of repeat unit (g/mol)	45.0
Specific gravity	1.77-1.79
Water absorption, immersion for 24 hours (%)	0.01-0.03
<i>Mechanical properties</i>	
Flexural strength @5% strain (psi / MPa)	8500-11000 / 58-76
Tensile Yield Elongation (%)	5-10
Tensile Yield Strength (psi / MPa)	6500-8000 / 45-55
Tensile Break Elongation (%)	50-200
Tensile Break Strength (psi / MPa)	5000-8000 / 34-55
Tensile Modulus (psi / MPa)	200000-335000 / 1379-2310
<i>Thermal properties</i>	
Melting temperature (°F / °C)	323-342 / 162-172
Melting point (°C)	162-172
Melt viscosity (kp)	47-53

3.3.2 Solvent

In this study, the organic chemical used in dope solution is N-methyl-2-pyrrolidone (NMP, >99%) supplied by Merck, Darmstadt, Germany. Formula structure of NMP is shown in Figure 30. Physical and chemical properties for NMP are tabulated in Table 5. Three steps of solvent exchange fluids included water, ethanol and n-hexane are used in order to produce the membrane with the best permeation properties. Tap water used as the coagulation medium and was temperature controlled at 25°C.

FIGURE 30: Formula Structure of N-methyl-2-pyrrolidone (NMP)

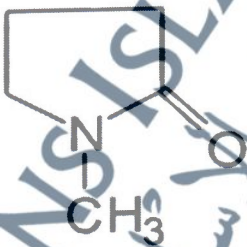


TABLE 5: Properties of N-methyl-2-pyrrolidone (NMP)

Physical and Chemical Properties	
Property	Unit value
Formula molecule	C ₅ H ₉ NO
Molecular weight (g/mol)	99.1
Solubility in water	Very good
Boiling point (°C)	202
Freezing point (°C)	-23.6
Relative vapour density (air=1)	3.4
Relative density of the vapour/air-mixture at 20°C (air=1)	1.00
Vapour pressure, Pa at 25°C	66
Liquid Viscosity (Pa.s)	1.66x10 ⁻³

3.3.3 Additives

In order to improve membrane performance, hydrophilic polymers or polymeric additives such as polyvinyl-pyrrolidone (PVP) and poly(ethylene glycol) (PEG) have been investigated as the suitable additives to membrane solution (Bowen et al., 2001). In this research, PVP K29-32 and PEG were added to the PVDF casting solutions separately. The influences of these additives on structure and properties on the membranes were investigated.

Besides, it has been shown that the main role of PVP in membrane formation is as pore forming agents. A small molecular weight polymeric additive such as PVP added tends to increase membrane separation performance especially for liquid separation. PVP also act as an agent for suppressing macropores formation in the phase inversion membranes, much less a demixing enhancer or a permeate flux enhancer. PVP addition can induce the enlargement of macrovoids in the membrane as well as the significant increase in membrane permeability (Han et al., 2002). The molecular structure and physical properties of this PVP additive are showed as in Figure 31 and Table 6 respectively.

FIGURE 31: Molecular Structure for Polyvinylpyrrolidone (PVP)

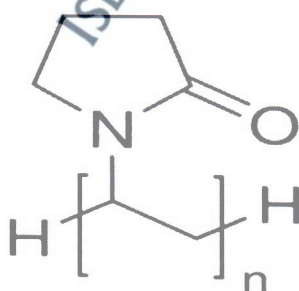


TABLE 6: Physical properties of PVP K29-32

Properties	State
Appearance	White solid (powder)
Odor	Off-white powder; odorless
Solubility	Soluble in water
Density	1.66 gcm ⁻³
Melting point	100°C (212 F)
Specific gravity	1.25
% volatiles by volume @ 21°C (70 F)	0

In general, PEG has been used to increase porosity and reduce the skin layer thickness of the membrane. The addition of PEG has an influence on the casting solution properties, permeation properties and resulting membrane structures. By adding PEG as a pore reducing agent in the solution containing NMP, PVDF membranes with improved fluxes and solute rejections will be obtained. In other words, PEG could induce or suppress the macrovoids expansion for PVDF membrane-forming systems depending on its molecular weight. The molecular structure and physical properties of this PEG additive are showed as in Figure 32 and Table 7, respectively.

FIGURE 32: Molecular Structure for Poly(ethylene glycol) (PEG)



TABLE 7: Physical properties of PEG

Properties	State
Physical form	Liquid
Odor	Off-white ; odorless
Solubility	Soluble in water
Stability	Stable under ordinary condition (hygroscopic)
Density	1.12 gcm ⁻³
Viscosity at 100°C	4.3 cSt

3.3.4 Surfactants

In this study, there are three types of surfactants were added to the polymer casting solution which are Sodium dodecyl sulfate (SDS), Triton X-100, and Tween 80 added to the polymer casting solution.

SDS as hydrophilic surfactant (strong anionic surfactant) can affect the membrane performance, thus membrane morphology and porosity of sub-layer of the membrane. It is an organic compound with the formula $\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$ as the molecular structure shown in Figure 33. The physical properties for SDS are tabulated in Table 8.

FIGURE 33: Molecular Structure of Sodium Dodecyl Sulfate (SDS)



TABLE 8: Physical properties of Sodium dodecyl sulfate (SDS)

Properties	State
Physical form	Dry powder (White or cream-color)
Odor	Odorless
Solubility	Soluble in water
Stability	Stable under ordinary condition
Density	1.01 gcm ⁻³
Melting point	206 °C (403 °F; 479 K)
Surface tension	39.5 dyn/cm at 25 °C

On the other hand, Triton X-100, $C_{14}H_{22}O(C_2H_4O)_n$ ($n = 9-10$) was used as non-ionic surfactant in the polymer casting solution. The "Triton X" series of detergents are produced from octylphenol polymerized with ethylene oxide. The number ("-100") relates only indirectly to the number of ethylene oxide units in the structure. X-100 has an "average of 9.5" ethylene oxide units per molecule, with an average molecular weight of 625. Triton X-100 is a nonionic detergent, 100% active ingredient, which is often used in chemical applications to solubilize proteins. Triton X-100 has no antimicrobial properties. It is considered a comparatively mild non-denaturing detergent (Sigma Aldrich). The molecular structure and physical properties of this Triton X-100 surfactant are showed as in Figure 34 and Table 9, respectively.

FIGURE 34: Molecular Structure of Triton X-100

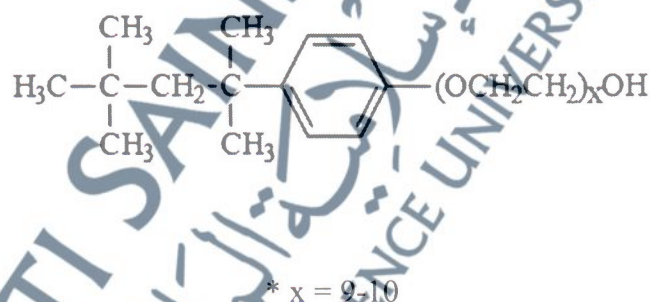


TABLE 9: Physical properties of Triton X-100

Properties	State
Appearance	Liquid, clear to slightly hazy, colorless to light yellow
Solubility	Soluble in water
Density	1.07 gm ⁻³
Melting point	6 °C (43 °F; 279 K)
Boiling point	270 °C (518 °F; 543 K)
Specific gravity	1.065 at 25°C (approx. 1.07 g/mL)

The third surfactant used in this study is Tween 80 ($C_{64}H_{124}O_{26}$). This synthetic compound is a viscous, water-soluble yellow liquid. This surfactant, Tween 80 is derived from polyethoxylated sorbitan and oleic acid. The hydrophilic groups in this compound are polyethers also known as polyoxyethylene groups, which are polymers of ethylene oxide. In the nomenclature of polysorbates, the numeric designation following polysorbate refers to the lipophilic group, in this case the oleic acid. The molecular structure and physical properties of Tween 80 surfactant are showed as in Figure 35 and Table 10, respectively. According to Liu et al. (2011), it cannot be thoroughly washed out from the structure of membrane due to its high molecular weight. The addition of Tween 80 enhances the membrane hydrophilicity because of the hydrophilic groups of Tween 80 exposure on the membrane surface. Therefore, it can lead to the macrovoids formation and also finger-like pores in the sub-layer (Amirilargani et. al., 2009).

FIGURE 35: Molecular Structure of Tween 80

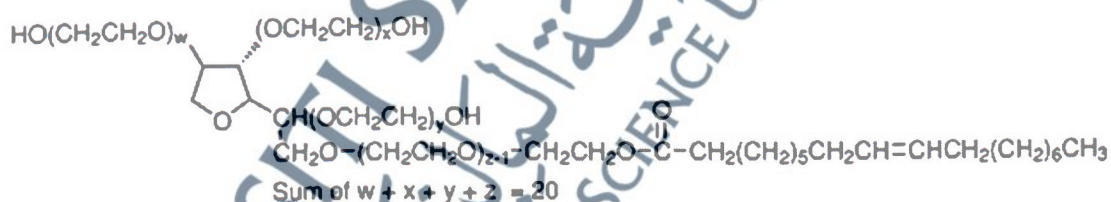


TABLE 10: Physical properties of Tween 80

Properties	State
Physical form	Liquid, clear to slightly hazy faint yellow solution.
Odor	Odorless
Solubility	Easily soluble in cold water, hot water. Soluble in methanol.
Stability	Stable under ordinary condition
Density	1.06–1.09 g/mL
Melting point	-20.556°C (-5°F)
Boiling point	>100°C (212°F)
Specific Gravity	1.06 - 1.10 (Water = 1)

3.4 Formulation of Polymer Solution

In general, a polymer solution formulation for asymmetric fabrication consists of polymer, solvent, and non-solvent. This can be called as multicomponent casting formulation. Sometimes, this formulation consists of four components where polymeric additives were added. However, in this experiment there is no non-solvent used since the characteristic of PVDF polymer has become such jelly-like. According to Wang (a) (2000), the multicomponent polymer dope incorporates certain amounts of non-solvent so that the dope condition is near the bimodal boundary thus facilitating the necessary rapid coagulation in the dry gap for effective phase separation.

3.4.1 Multi Component Dope Preparation

Polymer was first dried for at least 24 hours in a vacuum oven at a temperature of about $100 \pm 2^\circ\text{C}$ in order to remove all absorbed water vapor. The existence of water in the polymer solution will influence the quality of a polymer solution. Round bottom flask was used to prepare the dope solution. Thermometer was used to measure the temperature during the mixing process. Processing temperature was controlled at the desired range. The function of stirrer is to ensure that the polymer, solvent and non-solvent are well-mixed to form a homogeneous solution.

Firstly, the solvent was poured into the round bottom flask until the temperature increase to $50\text{--}60^\circ\text{C}$. Thermometer measured the temperature while heater controlled the required temperature range. When temperature has reached 50°C ,

polymer is added gradually to the solution. The stirrer continuously stirred the solution while the PVDF polymer has been dissolved then the additive was added after the entire polymer completely dissolved. The solution is being stirred for 1-2 hours before surfactant is added into the polymer solution. The best time for surfactant added is 3 hours before the polymer solution is completely done. The mixing temperature must be controlled and maintained not exceed over than 70°C to prevent the solvent loss because the dope solution tend to vaporize. Besides, it also to prevent the dope solution to become scorched and the color is becoming darker. However the low temperature will not encourage the mixing process. Throughout the mixing process, the stirring rate was maintained between 300 to 400 rpm. Figure 36 and Figure 37 shows the apparatus for preparing the dope solution and the process of making multi-components dope solution respectively.

After all the component had been mixed up in the flask, the dope solution was left over 6 to 8 hours to stir until the solution is homogeneous. After the solution was fully dissolved, the dope was cooled in room temperature before it was poured into a clean bottle and degassed using Branson Ultrasonics bath to remove trapped micro-bubbles of gases and particles prior to a casting process. In addition, Table 11 below show dopes formulation for the preparation of membranes.

FIGURE 36: Apparatus for Dope Solution Preparation

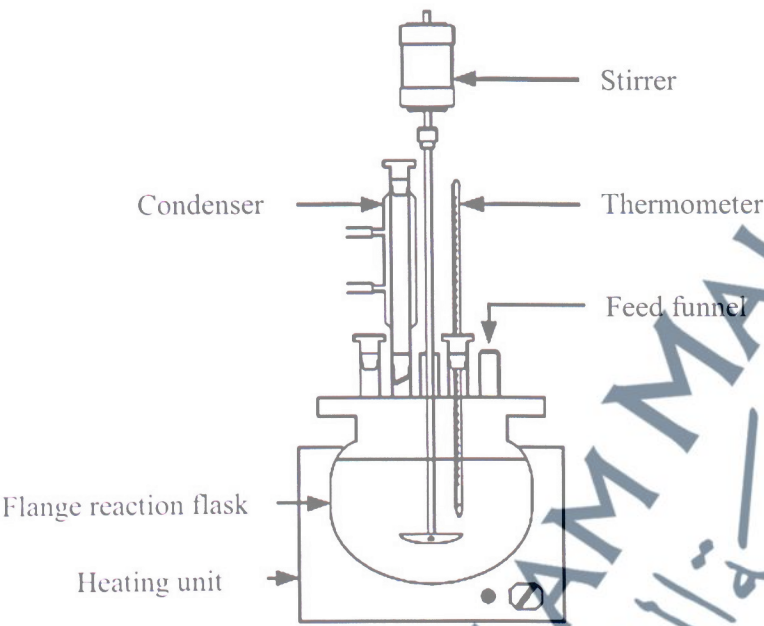


FIGURE 37: The Process of Making Multi-Components Dope Solution



TABLE 11: Dopes formulation for the preparation of UFS membranes

Membranes	PVDF (wt%)	NMP (wt%)	Additives			Single & Dual Surfactant		
			PVP (wt%)	LiCl (wt%)	PEG 200 (wt%)	SDS (wt%)	Triton X (wt%)	Tween 80 (wt%)
PN-1	13	87	-	-	-	-	-	-
PN-2	15	85	-	-	-	-	-	-
PN-3	17	83	-	-	-	-	-	-
PN-4	19	81	-	-	-	-	-	-
PN-3P	17	80	3	-	-	-	-	-
PN-3L	17	80	-	3	-	-	-	-
PN-3G	17	80	-	-	3	-	-	-
PN-3PS	17	78	3	-	-	2	-	-
PN-3PT	17	78	3	-	-	-	2	-
PN-3PW	17	78	3	-	-	-	-	2
PN-3GS	17	78	-	-	3	2	-	-
PN-3GT	17	78	-	-	3	-	2	-
PN-3GW	17	78	-	-	3	-	-	2
PN-3PSW	17	78	3	-	-	1	-	1
PN-3PTW	17	78	3	-	-	-	1	1
PN-3GSW	17	78	-	-	3	1	-	1
PN-3GTW	17	78	-	-	3	-	1	1

3.5 Membrane Fabrication

3.5.1 Membrane Casting

Asymmetric ultrafiltration (UF) membranes were fabricated according to the dry/wet phase separation process using manual casting blade which is a casting knife purchased from BYK Instrument as shown in Figure 38. The casting process was conducted at room temperature ($30 \pm 2^\circ\text{C}$) and approximately 84% relative humidity. A small amount of casting solution was poured onto a glass plate with a casting knife gap setting of $150\mu\text{m}$ ($1.5 \times 10^{-4}\text{ m}$) and at an appropriate casting shear.

FIGURE 38: Membrane Casting Knife



3.5.2 Coagulation Bath

After the membrane had been cast, the glass plate support together with the membrane was then immersed into the coagulation bath (composed of water as the coagulation medium). There were three phases of coagulation bath need to be completed in this study. The first bath contained tap water and the glass plate with the membrane was

immediately immersed in the water after casting had been done. While immersing the glass plate into the first coagulation medium, it is important to be careful in order to have a good thin film membrane and to prevent it from tore apart. The solvent exchange was immediately observed as the membrane was turned into a nice white thin film membrane. After the membrane was separated from the glass plate, the membrane was transferred to new water bath and then it left for 1 day.

The thin film membrane was then transferred to the second coagulation bath compose of ethanol for 1 day. Then, the membrane was again transferred to the third coagulation bath which containing n-hexane and soak for only 1-2 hours to cramp the membrane structure for it to be strong. After the process coagulation complete, the membrane was dried for at least 48 hours at room temperature to remove any residual organic compounds. The use of the liquid exchange treatment is to prevent changes in the structures and properties of membranes caused by large capillary forces during drying. This is important when non-volatile solvents are used with water as the non-solvents. When membranes are dried afterwards, water will be removed first and the solvent left behind may damage the membrane structure.

3.6 Performances Evaluation

3.6.1 Ultrafiltration Permeation Rig

The schematic diagram of ultrafiltration permeation rig is shown as in Figure 39. Using the well-known Solvent Resistance Stirred Cells (Figure 40), a circular disk membrane was cut and mounted in a stainless steel, cylindrical membrane test cell by a porous support and tightened by a rubber *O*-ring. Effective area of the membrane

being mounted under the cell is $1.3856 \times 10^{-3} \text{ m}^2$. The operating pressure for filtration process was supplied by pressurized nitrogen gas. The nitrogen gas outlet pressure is regulated using a pressure regulator where the equilibrium pressure is shown in the pressure gauge. A pressure relief valve was installed between the nitrogen gas and cell.

FIGURE 39: Schematic Diagram of UF Filtration Set-Up

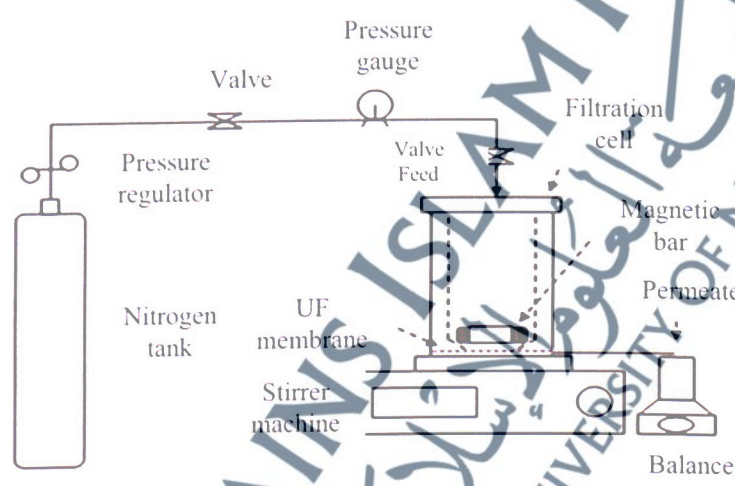
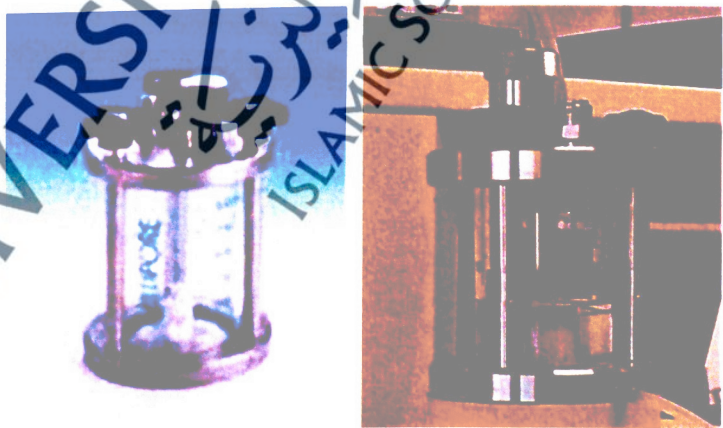


FIGURE 40: Dead-End Stirred Cells for UF Test



3.6.2 Ultrafiltration Permeation Test

Ultrafiltration (UF) permeation test was carried out using dead end filtration unit at different pressure. For each operating pressure, distilled water was used to determine the pure water permeability (PWP) parameter/constant. PWP parameter was obtained from the slope of volume flux versus pressure. Prior to the each UF test, membrane was subjected to be pressurized till about 300 kPa for 1 hour. The flux was equilibrated for the passage of the first 30 ml permeate whilst the following 10 ml permeate was collected for concentration analysis. All the results presented are averaged data obtained through three membranes samples with a variation of about $\pm 10\%$. The permeate was collected and weight was measured by an electronic balance for every minute. The volume flux was calculated based on the weight and time as stated in Equation 1.

$$\text{Volume flux, } J_v = \frac{\text{Volume permeation rate, } V}{\text{Membrane area (A)} \times \text{time (t)}} \quad (1)$$

where,

J_v = the permeate flux of salt aqueous solution or pure water flux ($\text{L}/\text{m}^2\text{h}$),

A = the effective area of membrane (m^2),

t = the time (h),

V = Volume of permeate solution collected, (L).

For pure water flux, the permeability of each membrane was determined by the measurement of water flux as a function of applied pressure (Equation 2).

$$P_m = \frac{J_v}{\Delta P} \quad (2)$$

where,

P_m = Permeability (L/m²hkPa),

J_v = Flux (L/m²h),

ΔP = Pressure (kPa).

3.6.3 Pure Water Permeation (PWP) Test

As PWP parameter and flux are obtained, the membrane permeation test is carried out according to the procedure stated in Section 3.6.2 at six different operating pressures that are 50, 100, 150, 200, 250 and 300 kPa, the permeation test is carried out using distilled water. The flux and observed rejection are determined for each operating condition. In order to reduce the polarization effects, the stirring speed is fixed at 400rpm or 41.881 rad/sec. After each permeation test, UF membranes surface is rinsed with distilled water. The pure water permeability is measured to ensure the original flux (pure water) is recovered before next permeation test.

3.6.4 Protein Rejection

By using dead end filtration, the circular membrane was cut to test the performance with protein rejection. Molecular weight cut-off (MWCO) is a pore characteristic of membranes and is related to solute rejection for a given molecular weight of a solute. The molecular weight has a linear relationship with the pore radius or pore size of a membrane. Therefore, two types of proteins were chosen for the solute rejection and estimation of MWCO which are Bovine serum albumin (BSA, 69kDa), Egg albumin (EA, 45kDa). The protein solutions were prepared at a concentration of 0.1 wt% in phosphate buffer (0.5M, pH 7.2) using distilled water and filtered through each of membranes. During UF process, the permeate protein concentration were collected over measured time intervals in graduated tubes, and tube contents were analyzed for protein content by using UV-Visible spectrophotometer at a wavelength of 280 nm (Nurul et al., 2013). Solute rejection is calculated based on the concentration of feed and permeate solutions as Equation 3.

$$R(\%) = \left(1 - \frac{C_p}{C_f}\right) \times 100 \quad (3)$$

where,

R = Percentage rejection of protein (%),

C_p = Concentration of protein in permeate (g/mL),

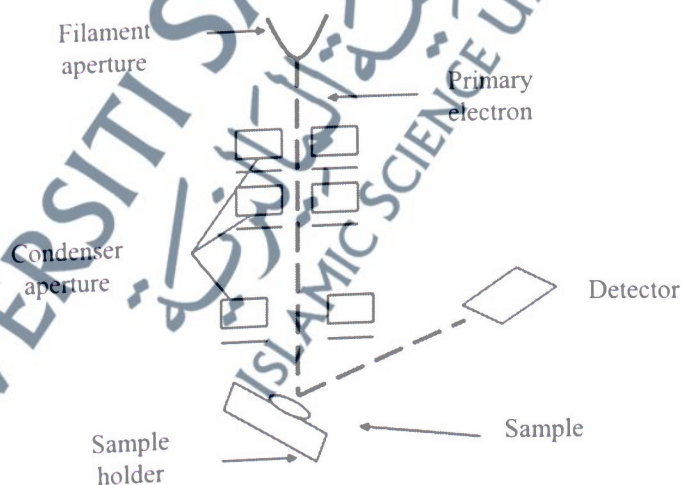
C_f = Concentration of protein in feed (g/mL).

3.7 Structural Characteristics of Asymmetric UF Membranes

3.7.1 Scanning Electron Microscopy (SEM)

Scanning Electron Microscope (SEM) is a very useful technique for membrane morphological determination. This method is used for the determination of membrane structure, membrane thickness and membrane surface. The principle of the Scanning Electron Microscope is illustrated in Figure 41. A narrow beam of electrons with kinetic energies in the order 1-25 kV hits the membrane sample. The incident electrons are called primary (high-energy) electrons, and those reflected are called secondary electrons. Secondary electron (low energy) are not reflected but liberated from atoms in the surface, determine the imaging (what is seen on the screen or on the micrograph).

FIGURE 41: The Principle of Scanning Electron Microscopy



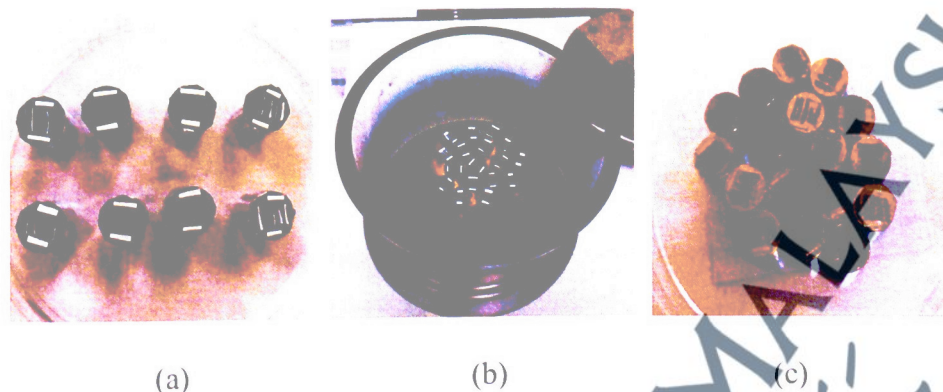
For this purpose, the pieces were immersed in liquid nitrogen for 60-90 s and were frozen. The membranes were snapped under liquid nitrogen to give a

generally consistent and clean break. The membranes were mounted on sample stubs using a double-sided adhesion tape in lateral position (Figure 43a). The membranes were then sputter coated with a thin film of gold for producing electric conductivity (Figure 43b). After the samples were sputtered with gold (Figure 43c) by an automatic gold coater (JFC 1600), they were scanned by employing a JEOL JSM 6360LA Scanning Electron Microscope (Tokyo, Japan) as shown in Figure 42 under magnification of 500x to 5000x with potentials of 20kV.

FIGURE 42: JEOL JSM 6360LA Analytical Scanning Electron Microscopy (SEM)



When a membrane is placed in the electron beam, the sample can be burned or damaged, depending on the type of polymer and accelerating voltage employed. This can be avoided by coating the membranes with a conducting layer (gold palladium) to prevent charging up the surface. Scanning electron microscopy allows a clear review of the overall structure of the membranes; the top surface, the cross section and the bottom surface can all be observed nicely. In addition, the porosity and the pore size distribution can be estimated from the micrographs obtained.

FIGURE 43: Membrane Samples for SEM Analysis

3.7.2 Fourier-Transform Infrared (FTIR)

Fourier-Transform Infrared (FTIR) spectroscopy is another efficient tool for the study of molecular structure. Because an organic molecule can absorb and convert the infrared radiation of frequencies into the energy of molecular rotation, an IR spectrum can offer the information of the entire molecule. Although it is true that certain groups of atoms give rise to band at or near the same frequency, it still provides useful information to analyze the structure of molecules. Therefore, the persistence of the characteristic bands that permits to obtain useful structural information by simple inspection and reference to generalized charts of characteristics group frequencies (Silverstein, 2005).

Molecular orientation and chemical structures of PVDF blend polymer membranes were examined using transmission Variance 3100 FTIR Excalibur Series as shown in Figure 44. Knowledge of vibrational frequencies of functional groups allow for chemical identification of at least a class of compounds. Each of the

membrane samples were mounted in position with skin surface facing the infrared beam. The samples were placed on the sample holder and all spectra were recorded in the wave number range of $4000\text{--}1000\text{ cm}^{-1}$ by cumulating 32 scans at a resolution of 2 cm^{-1} .

FIGURE 44: Varian 3100 FTIR Excalibur Series



3.7.3 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) is an analytical technique used to determine a material's thermal stability and its fraction of volatile components by monitoring the weight change that occurs as a specimen is heated. TGA instruments can be divided into two general types which are vertical and horizontal balance. Vertical balance instruments have a specimen pan hanging from the balance or located above the balance on a sample stem. On the other hand, the horizontal balance instruments normally have two pans (sample and reference) and can perform the measurements.

The thermogravimetric behavior was characterized by a thermogravimetric analysis instrument (Mettler Toledo Gas Controller GC 200 shown in Figure 45). The sample is heated under nitrogen or synthetic air with constant heat rate while the difference of the mass during this process is measured. A mass loss indicates that a degradation of the measured substance takes place. The reaction with oxygen from the synthetic air for example could lead to an increase of mass. All measurements were performed with polymer samples (about 5 mg) in aluminium pans under dynamic nitrogen atmosphere, in the temperature range from 30°C to 900°C with a heating rate of 10°C/min and gas flow of 100 mL/min. The test was carried out under nitrogen atmosphere (20 ml/min) in order to prevent thermoxidative degradations.

FIGURE 45: Thermogravimetric Analysis (TGA)

