# 3 MATERIALS AND METHODS

## *3.1 Overview*

This paper presents a production of flat sheet mixed matrix membrane for the treatment of copper ions. Various concentration of flat sheet mixed matrix membrane were produced from PES/NMP/PVP and Amberlite IR 120H cation resin using dry-wet spinning method. The 4 samples of different concentration of mixed matrix membrane were undergo characterization. This involved scanning of pores size and morphological of mixed matrix membrane, water permeability, static adsorption and lastly regeneration. The main testing equipment used including Atomic Absorption Spectroscopy (AAS). From the results that obtained, we determined which concentration of PES has the maximum adsorption of copper ions from batch experiment and cross flow filtration.

## *3.2 Introduction*

This paper presents the overall methodology of the formation of mixed matrix membrane with various concentration to determine which concentration in effectiveness in ion exchange uptake of copper ions. All the chemicals and methods used were briefly explained on how to conduct the experiment. Furthermore, pictures have been presented to ease the understanding of methodology.

## *3.3 Chemicals*

Radel Solvay Advanced Polymer polyethersulfone (PES) was used as a base membrane polymer. Amberlite IR120H, polyvinylpyrolidone (PVP), Hydrochloric acid was obtained from Sigma-Aldrich. N-methylpyrolidone, NMP and copper (II) sulphate pentahydrate from Mercks Malaysia and hydrochloric acid from AR&MJ. The dope polymer solution is prepared from PES/NMP/PVP with various weight concentration and fixed amount of Amberlite IR120H cation resin. The Amberlite IR120H cation resin from *Fluka* used to uptake of copper ions. Copper ion solution used in binding experiment was prepared by diluting copper (II) sulpate pentahydrate. Hydrochloric acid (37%) was used for elution in batch binding and regeneration of flat sheet mixed matrix membrane in cross flow filtration.

## *3.4 Production of Flat Sheet Membrane*

In the production of flat sheet membrane, the procedures including preparation of Amberlite IR120H, mixed matrix membrane dope solution preparation and membrane casting process.

### *3.4-1 Preparation of Amberlite IR120H*

Amberlite IR120H was washed with de-ionised water in order to remove the impurities and dried at 80$^{o}$C for 24 hours in drying sample oven as shown in Figure 3-1(Dizge *et al.*, 2009). Dried resin was ground using *Retsch* branded ultracentrifuges grinder and sieved the resin in the range of 38-45 μm using stainless steel siever as shown in Figure 3-2.

Figure 3-1: Drying of Amberlite IR120 resin.

Figure 3-2: Grinded and sieved resin.

### *3.4-2 Mixed Matrix Membrane Dope Solution Preparation*

PES and NMP were used as membrane forming material and solvent respectively while PVP was used as additive. Since the research is studied on the effect concentration of PES toward the adsorption capacity, the composition of PES were varied in the range of 23wt% to 30wt% in Table 3-1(Zhen-Liang Xu, 2004). The use of molecular weight of PVP at 10,000wt with suitable content at 2-5wt% tends to produce high permeation flux and good solute rejection (Wang *et al.*, 1999). Therefore, PVP used was fixed at 5wt% for every dope polymer concentration. Each solution was made in 500ml of SCHOTT reagent glass bottle. The formula (3.4-1) used on how to obtain the quantity of dope polymer content using 500mL of dope polymer solution is equivalent to 500g of PES.

$$PES\ content = \ 500g\ PES\ X\ 23wt\% = 115g\ PES$$

$$Since\ PVP\ is\ fixed\ at\ 5wt\%, PVP\ content = 500g\ PES\ X\ 5wt\% = 25g\ PVP$$

$$Therefore, NMP\ content = 500g\ PES\ X\ 72wt\% = 360mL\ of\ NMP \qquad (3.4\text{-}1)$$

The formula were repeated for the rest of dope polymer concentration from 25wt% to 30wt%.