CHAPTER III
EXPERIMENTAL

3.1 Materials

The following materials were kindly donated by UOP LLC; the silicone rubber used in this work was RTV615A and a curing agent RTV615B from General Electric Co., Ltd.; zeolite: NaX; two types of cellulose acetate supporting membrane coated on non-woven cloth, which are porous and dense supporting membranes. Cellulose acetate powder (acetyl content of 39.8%) was purchased from Aldrich Chemical Co., Ltd. The solvents used were cyclohexane and acetone from Labscan Co., Ltd. Polyethylene glycol (PEG) MW400 was obtained from Fluka Co., Ltd. Gases used in this study were carbon dioxide (CO_2), methane (CH_4), and nitrogen (N_2). CO_2 and N_2 were obtained from Prax Air Co., Ltd. whereas CH_4 was acquired from TIG Plc.

3.2 Membrane Preparation

There were two types of MMMs fabricated according to the following methods.

3.2.1 Mixed Matrix Membranes from Cellulose Acetate Supporting Membrane

The solid-liquid-polymer mixed matrix membranes studied were prepared using PEG as liquid, NaX-zeolite as solid and silicone rubber as polymer phase. The liquid adsorbed solid consisting of PEG adsorbed into NaX was dispersed into silicone rubber and then coated on a cellulose acetate supporting membrane. The detailed of preparation is presented in the following sections.

3.2.1.1 Preparation of Liquid Adsorbed Solid

NaX-zeolite was dried at 400°C in order to remove the amount of water trace in its pores. The preparation started by dissolving the liquid PEG with

acetone. After being well mixed, the dried solid (NaX) was admixed into the solution and stir until the homogeneous solution was formed. The obtained solution was allowed for 1 day to achieve equilibrium before being dried at 90°C for overnight to remove the residual acetone.

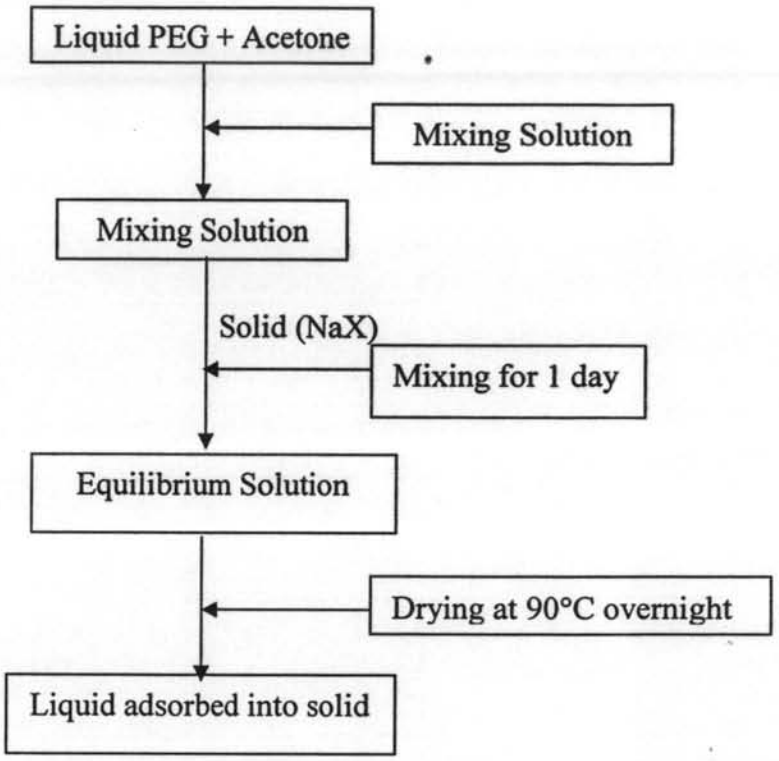

Figure 3.1 Liquid adsorbed into solid preparation procedure.

3.2.2.2 Membrane Fabrication

Mixed matrix membranes were prepared by solution-casting and solvent evaporation methods as schematically shown in Figure 3.2. The solution was formed by mixing 2.7 grams of RTV615A silicone rubber with 0.3 grams of RTV615B curing agent in 7 ml. of cyclohexane. After mixing together, the prepared liquid adsorbed solid about 30% of total solution was introduced into the solution. The casting solution was then degassed using a vacuum pump. The bubble free solution was casted on a cellulose acetate sheet. The thickness of casting layer was adjusted by a caster knife as a 22-mil thick film. The cast film was allowed to cure at

a temperature of 90°C for one hour in order to initiate the crosslinking reaction and to remove the residue solvent. Finally, mixed matrix membranes were placed in oven at a temperature of 170°C and 190°C for 30 minutes in order to treat the membrane.

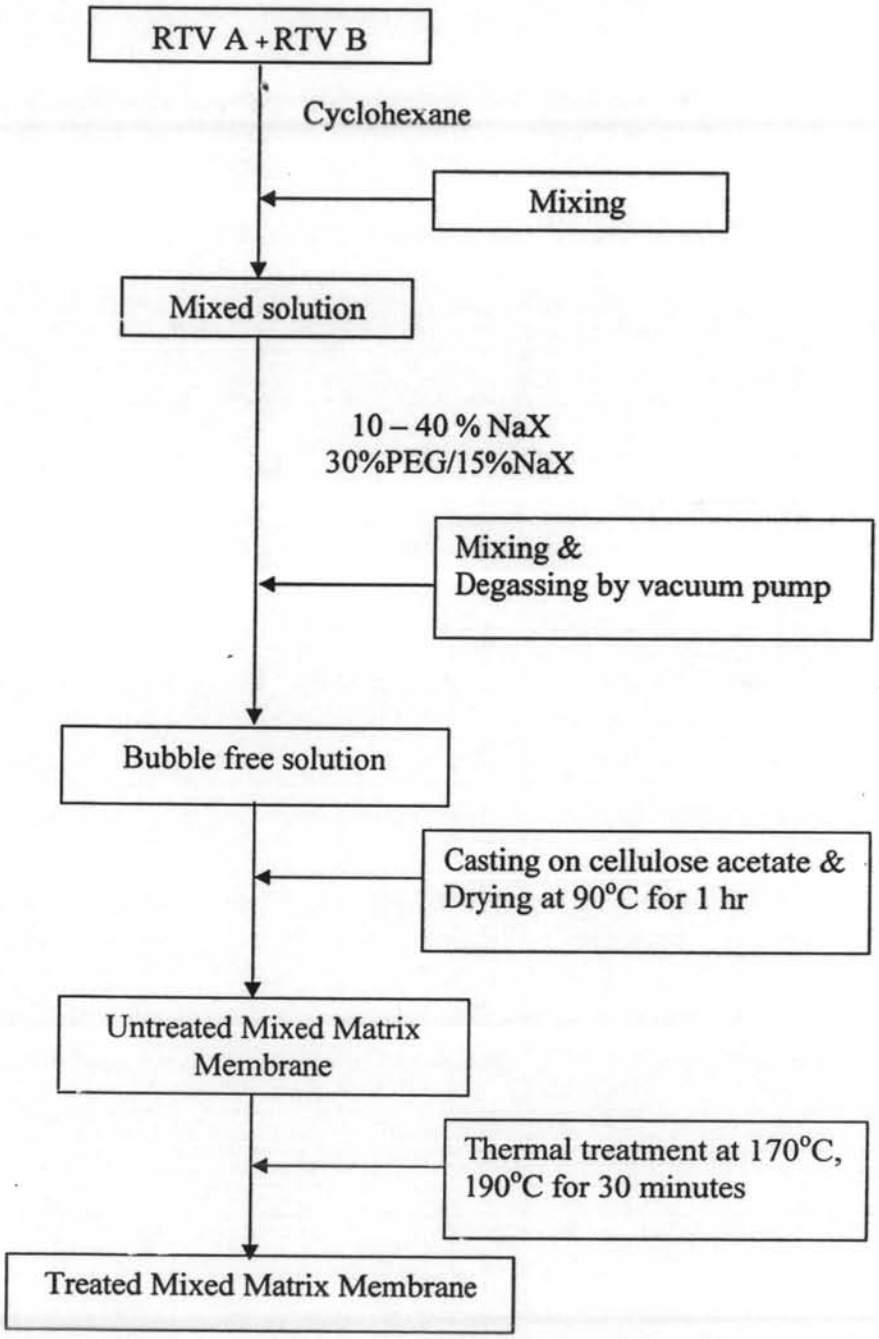

Figure 3.2 Fabrication procedure for cellulose acetate supporting MMM.

3.2.2 Mixed Matrix Membranes Fabricated from Cellulose Acetate Powder

These membranes were also prepared by solution casting method as schematically shown in Figure 3.3. A casting solution was prepared by admixing NaX-zeolite in acetone and stirred about 3 hours. This was done to ensure removal of air in the pores of adsorbent materials. Then, cellulose acetate powder (acetyl content = 39.8 %) was added to the solution and stirred until a suspended homogeneous solution was obtained. The casting solution was then degassed using a vacuum pump. The bubble free solution was poured on the top of horizontal surface of a clean glass plate and a portion of the acetone is allowed to slowly evaporate until a film forms on the upper surface of the solution. The thickness of casting layer was adjusted by a caster knife as a 22-mil thick film. After an additional set time of 20 seconds, the resultant membrane was gelled in an ice water bath (4-5 °C) followed by being annealed in a hot water bath at 65-75 °C for one hour to remove residual acetone. The membrane was dried by using air at room temperature between paper towels and two glass plates to prevent curling of the membrane. Finally, mixed matrix membranes were placed in oven at a temperature of 170 °C and 190 °C for 30 minutes in order to treat the membrane.

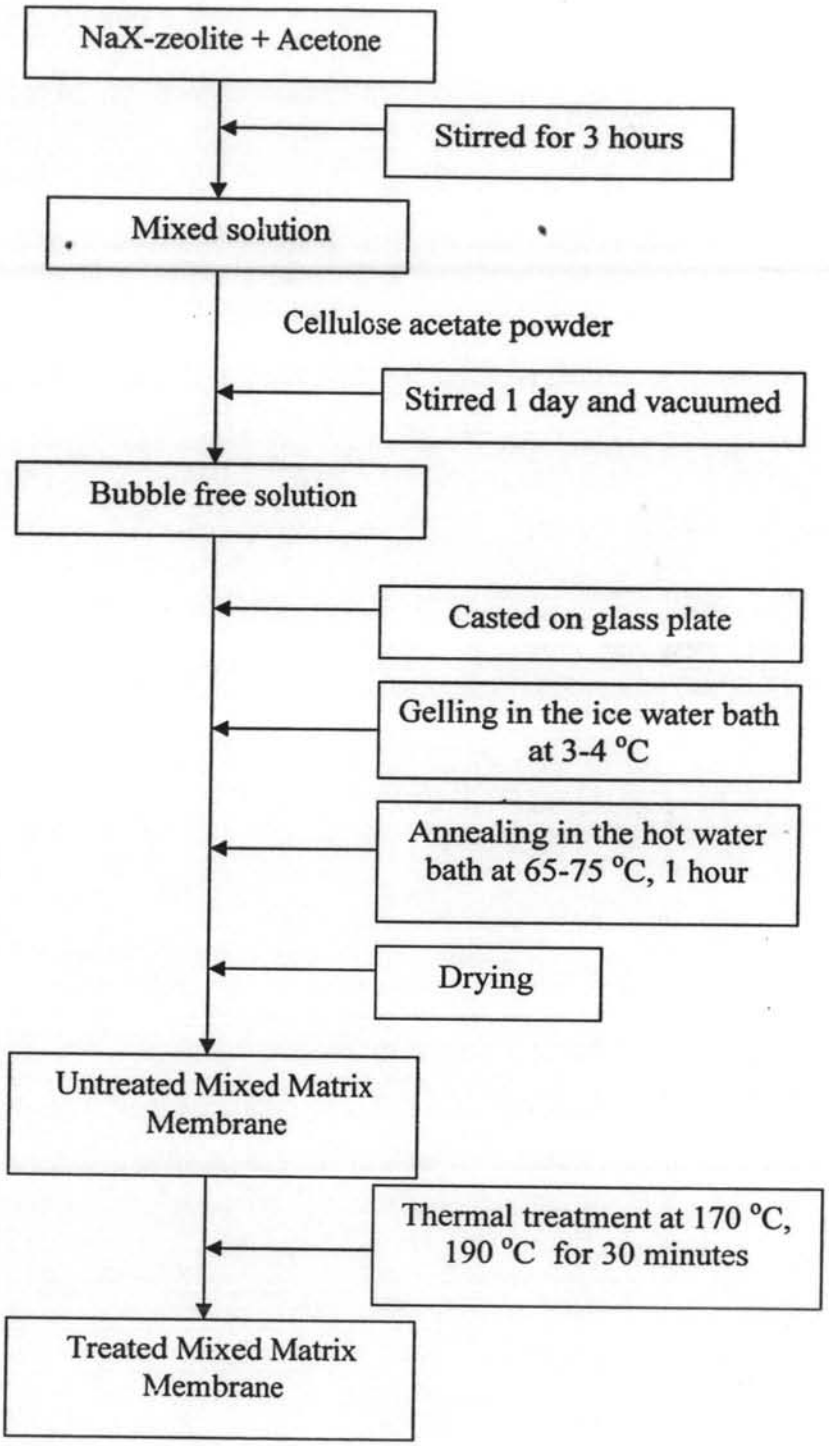

Figure 3.3 Fabrication procedure for cellulose acetate powder MMM.

3.3 Gas Permeability Measurements

The experimental setup used for determination of gas permeability is schematically shown in Figure 3.4. The mixed matrix membrane was tested by placing a 7.5 cm in diameter sample inside a membrane testing unit with an O-ring sealing the edge as shown in Figure 3.5 with a porous metal plate supported the membrane. Then, the various test gases; N_2, CH_4, and CO_2 were individually introduced into the membrane testing unit at room temperature and a constant pressure between 25 and 250 psi. The permeate side was maintained at atmospheric pressure. After a steady state is reached, flux of gas was measured using a bubble flow meter.

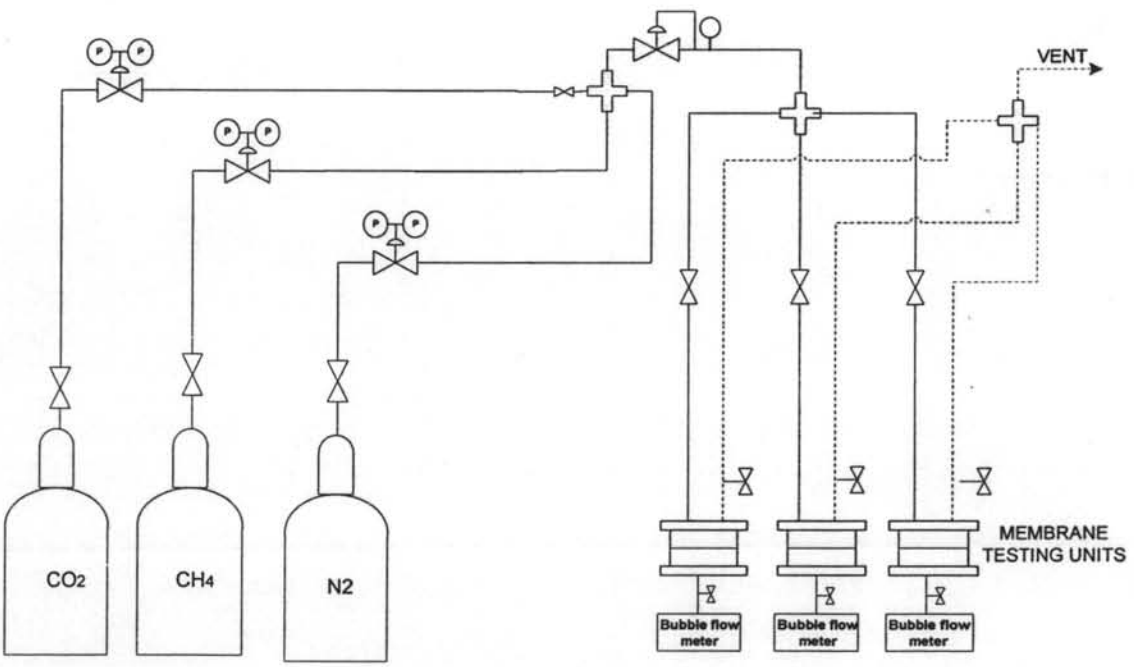

Figure 3.4 Schematic of the experimental setup for measuring gas permeability.

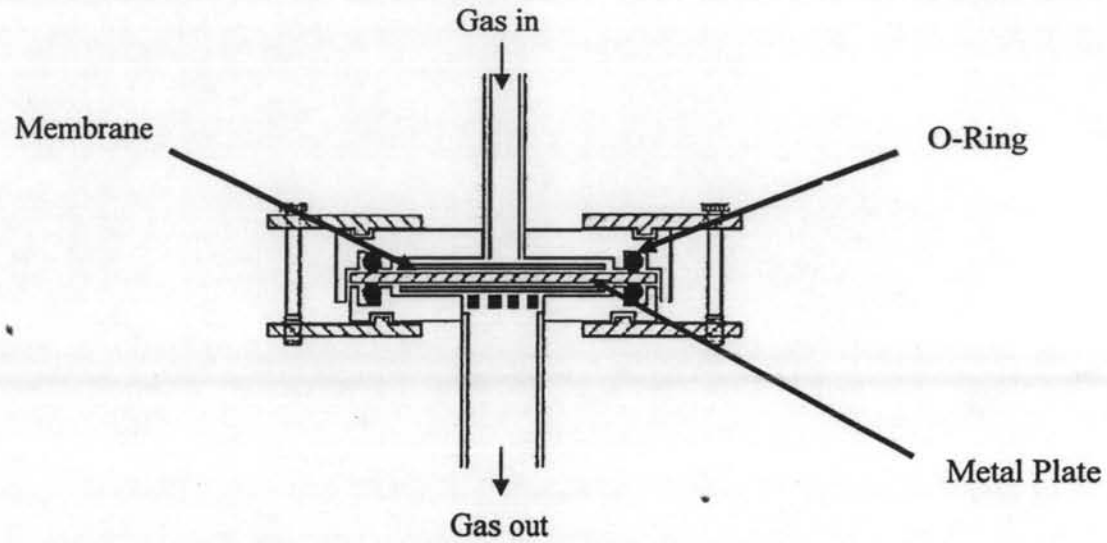

Figure 3.5 Schematic of the membrane testing unit.