

CHAPTER III EXPERIMENTAL

3.1 Materials

The following materials were kindly donated by UOP LLC; cellulose acetate powder and zeolite: NaA; CaA; NaX; NaY; and silicalite. The solvents used was acetone from Labscan Co., Ltd. Polyethylene glycol (PEG) MW400 was obtained from Fluka Co., Ltd. Carbon dioxide (CO₂), methane (CH₄), propane (C₃H₈), propylene (C₃H₆) and nitrogen (N₂) were used in this study. CO₂ and N₂ were obtained from Prax Air Co., Ltd. whereas CH₄, C₃H₈ and C₃H₆ were acquired from TIG Plc.

3.2 Methodology

3.2.1 Preparation of Liquid-Solid Adsorbent

NaX-zeolite was dried at 350°C for two hours in order to remove the amount of water trace in its pores of zeolites. 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. The method was shown in Figure 3.1.

3.2.2 Preparation of Mixed Matrix Membrane

The mixed matrix membranes were prepared by solution casting method as schematically shown in Figure 3.2. First, zeolites were dried in order to remove the amount of water trace in its pores of zeolites. For NaX, NaY, and Silicalite, they were dried at 350°C for two hours. For NaA and CaA, they were dried at 300°C for two hours. Second, a casting solution was prepared by admixing an adsorbent in acetone and stirred for three hours. This was done to remove the air in the pores of adsorbent materials. Next, cellulose acetate powder (acetyl content =

39.8%) was added to the solution and stirred for an overnight to obtain the homogeneous solution. The casting solution was then degassed using a vacuum pump. The bubble free solution was poured and cast on the top of horizontal surface of a clean glass plate. The thickness of casting layer was adjusted by a cast knife as a 22-mil thick film. After that, a portion of the acetone was allowed to slowly evaporate for 20 seconds brought about forming a film on the upper surface of the solution. The resultant membrane was gelled in an iced water bath at 3-4°C for one and half hours followed by being annealed in a hot water bath at 65-75°C for an hour to remove residual acetone. Finally, the membrane was dried by using air at room temperature between paper towels and two glass plates to prevent curling of the membrane.

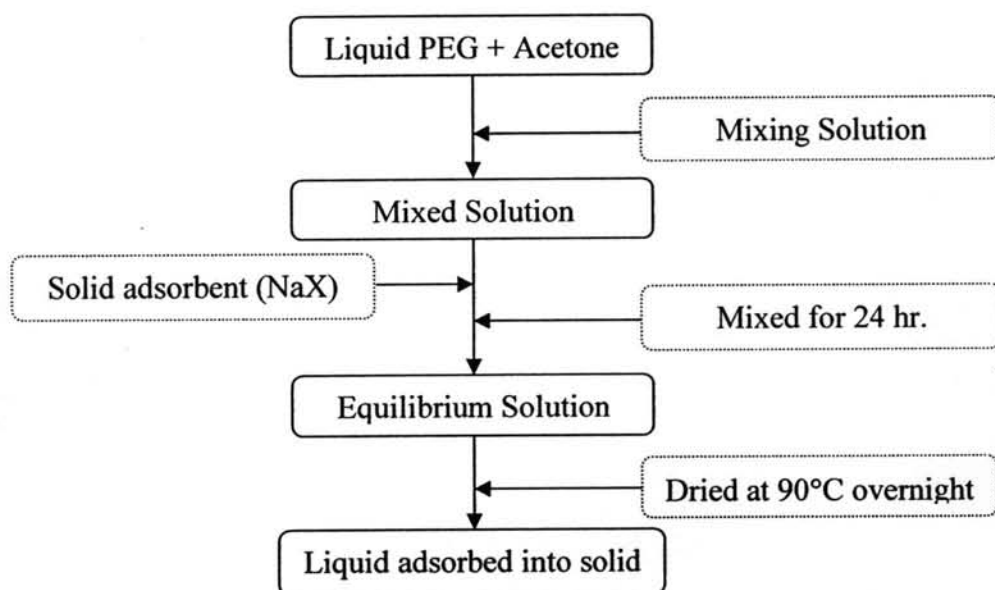


Figure 3.1 Liquid adsorbed into solid preparation procedure.

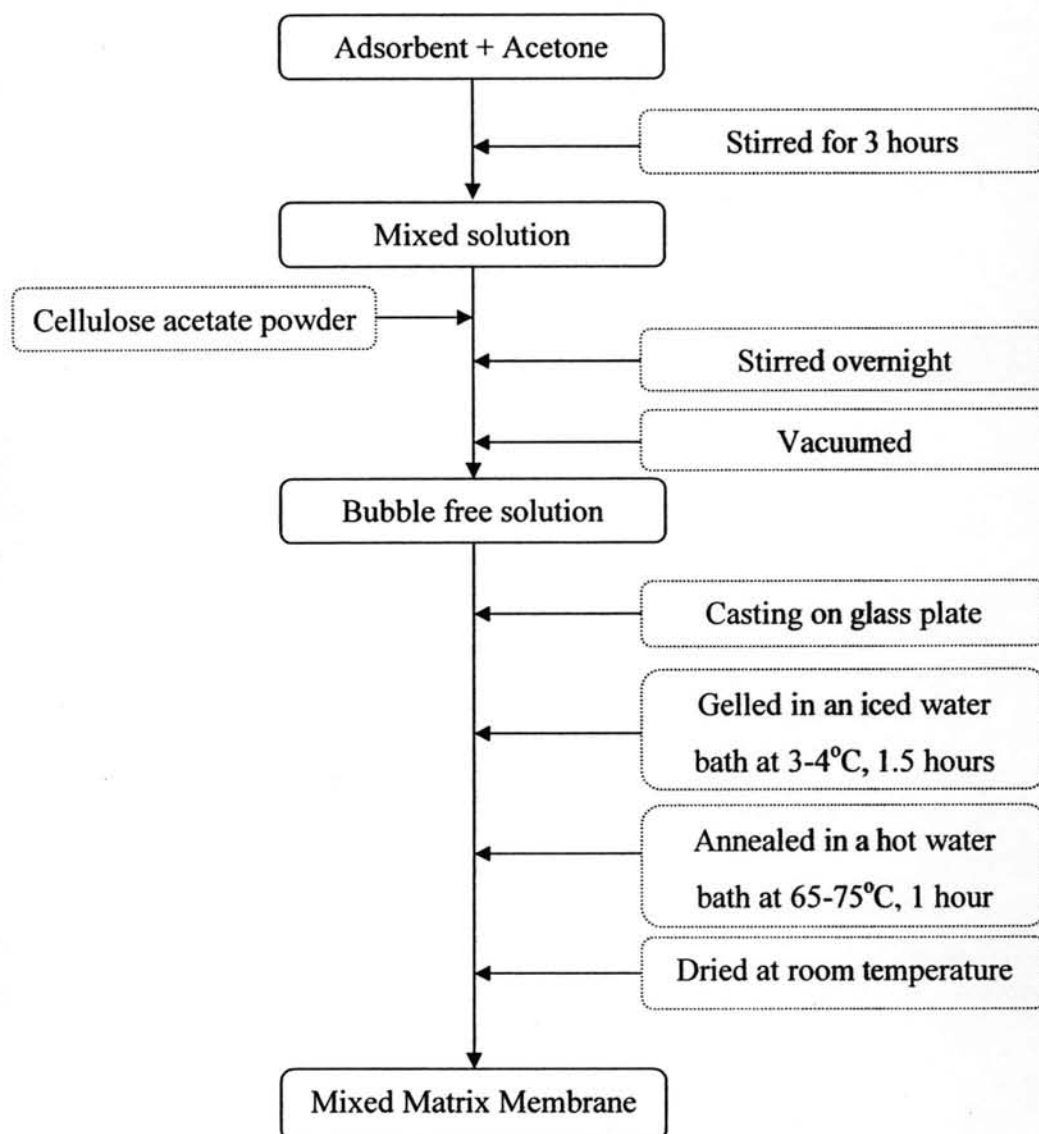


Figure 3.2 Fabrication procedure for MMM.

3.2.3 Gas Permeability Measurements

The experimental setup used for determination of gas permeability is schematically shown in Figure 3.3. The mixed matrix membranes were 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 3.4 with a porous metal plate supported the membrane. Then, the various test gases; N_2 , CH_4 , C_3H_8 , C_3H_6 and CO_2 were individually introduced into the membrane testing unit at room temperature and a

constant feed pressure at 50 psi for N_2 ; 100 psi for CH_4 , C_3H_8 , and C_3H_6 ; and in range of 25-200 psi for CO_2 . Nitrogen was used to check leak of the membrane. The permeate side was maintained at vacuum condition. After a steady state was reached, the gas flow rate was measured using a bubble flow meter. The attained data were used to calculate the gas permeance in order to determine the selectivity.

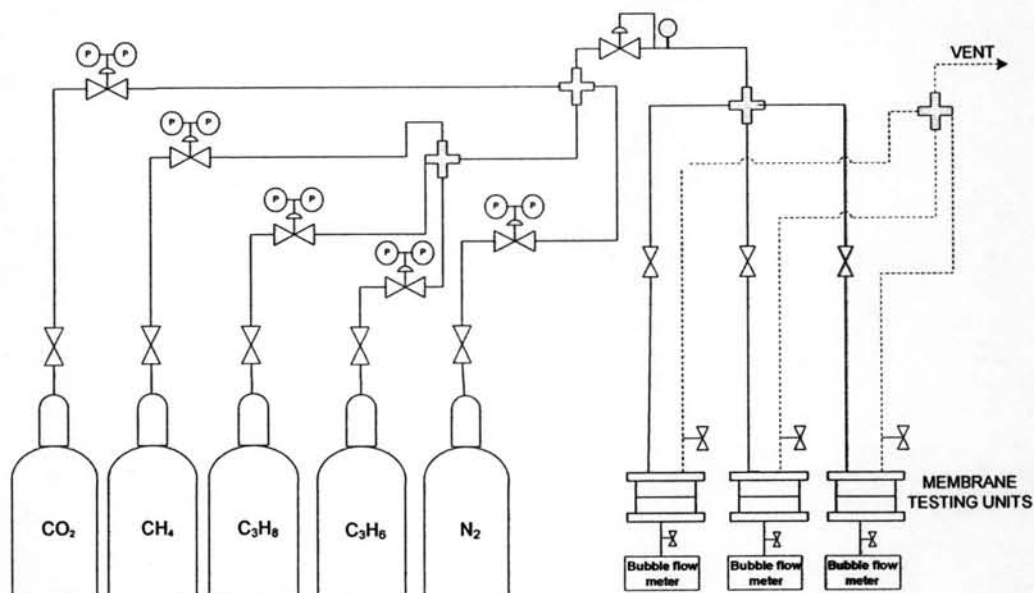


Figure 3.3 Schematic of the experimental setup for measuring gas permeability.

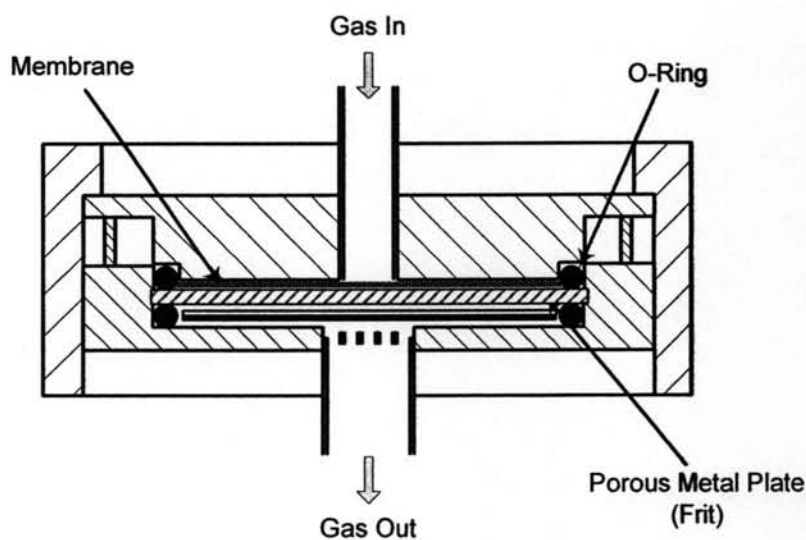


Figure 3.4 Schematic of the membrane testing unit