

CHAPTER III

EXPERIMENTAL

3.1 Materials

The following materials were kindly donated by UOP LLC; Ultem polymer obtained from General Electric Co., Ltd.; NaX, AgX and Silicalite. N-methylpyrrolidone (NMP) used as a solvent and the silane agent was 3-aminopropyltrimethoxysilane (APTS) were purchased from Fluka Co., Ltd. Gases used in this study were Nitrogen (purity > 99.999%) from Prax air Co., Ltd. and Propane (purity > 99.5%), Propylene (purity > 99.5%) from National Petrochemical Public Company Limited.

3.2 Membrane Fabrication

3.2.1 Mixed Matrix Membranes without Modification of Zeolite Surface

Before preparation MMM, all zeolite used were dried at 300 °C in order to remove the amount of water trace in zeolite pore. All mixed matrix membranes were developed by solution-casting method as schematically shown in Figure 3.1 the concentration of the polymer solution was 25 wt% Ultem in NMP as a solvent. The preparation started first by mixing zeolite, both with and without modification surface of zeolite with NMP and well agitating to break up aggregation of zeolite particle for about 1 hour. The small amount, approximately 10% of total Ultem solution was added into the slurry and mixed for 6 hours. The technique of “priming” zeolite particle with a small amount of polymer solution is believed to make zeolite particles more compatible with polymer matrix. (Koros *et al.* 2002) It is also believed to promote greater affinity/adhesion between zeolite particles and polymer chains. Remaining Ultem solution was introduced into the slurry and further mixed for 12 hours until the homogeneous of zeolite-Ultem solution was formed. After mixing step the casting solution was degassed using a vacuum pump. The bubble free solution was casted onto a clean glass plate. The thickness of MMM film was adjusted by a caster knife. The cast MMM film was allowed to slowly

evaporate at 60°C for overnight then further annealed and dried at 150°C for 12 hours in order to initiate the crosslinking reaction and to remove the residue solvent.

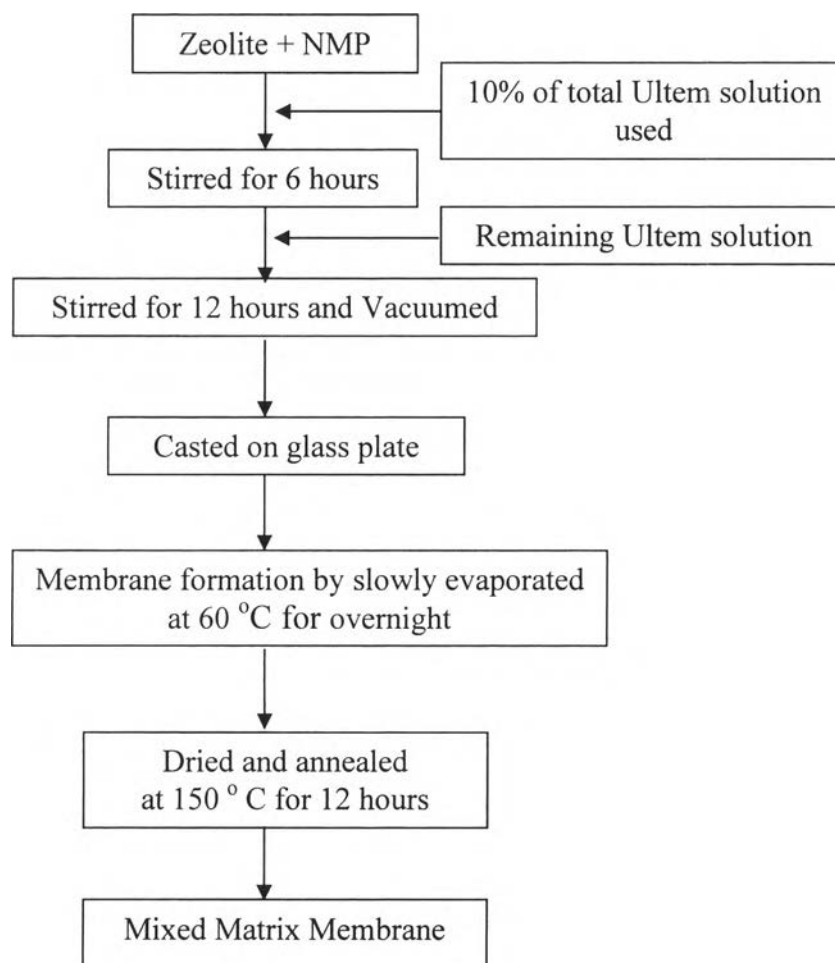


Figure 3.1 Mixed matrix membrane preparation procedure.

3.2.2 Mixed Matrix Membrane with Modification of Zeolite Surface

In order to alleviate or eliminate the interfacial void between zeolite and polymer phase an aminofunctional silane agent was introduced as a compatibilizer modifying the zeolite surface for enhancing interaction with the polymer. In this work 3-aminopropyltrimethoxy silane was investigated for which its chemical structure is given in Figure 3.2.

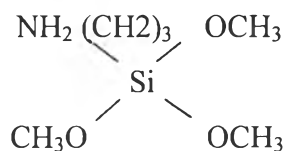


Figure 3.2 Chemical structure of 3-aminopropyltrimethoxy silane (APTS).

The procedures for fabricating MMM with modification of zeolite surface were similar to those for MMM without modification of zeolite surface, except that before fabricating zeolite studied were modified in order to promote greater adhesion between polymer chains and zeolite surfaces by utilizing aminofunctional silane agent. Zeolite particles were firstly dispersed to a solution of APTS in 95/5 w/w methanol/water at 70 °C for 3 hours. The amount of APTS is approximately a half of zeolite used based on weight. When the silanted zeolite preticipated, it was washed three times in methanol to remove the excess of APTS and then dried overnight at 110 °C. This modified materials were used further to fabricate MMMs.

3.3 Characterizations

The cross-sectional morphology of mixed matrix membranes was observed with a scanning electron microscope (SEM:JEOL 5200) and the interactions between silane coupling agent and zeolite surface were investigated using a FTIR spectroscope (BRUKER EQUINOX, model 55/s).

3.4 Gas Permeability Measurements

The experimental setup used for the determination of gas permeability is schematically shown in Figure 3.4. The gas permeability measurement was performed by utilizing a 7.5 centimeters in diameter membrane sample, which was placed inside the membrane-testing unit with an O-ring forming a seal around the edge as shown in Figure 3.5. The membrane was supported by a metal plate. The various test gases; N₂ , C₃H₆ and C₃H₈ were individually introduced into the

membrane testing unit at 50 psia. The permeate side was maintained at atmospheric pressure, resulting in differential pressure driving force of 50 psia across the membrane testing unit. The experiment was performed at room temperature. After a time interval estimated to be sufficient for attaining steady state, flux of gas was measured using a bubble flow meter. After changing to the new gas, membrane testing unit has to be purged and vacuumed at permeate side by a vacuum pump in order to remove the old gas from membrane.

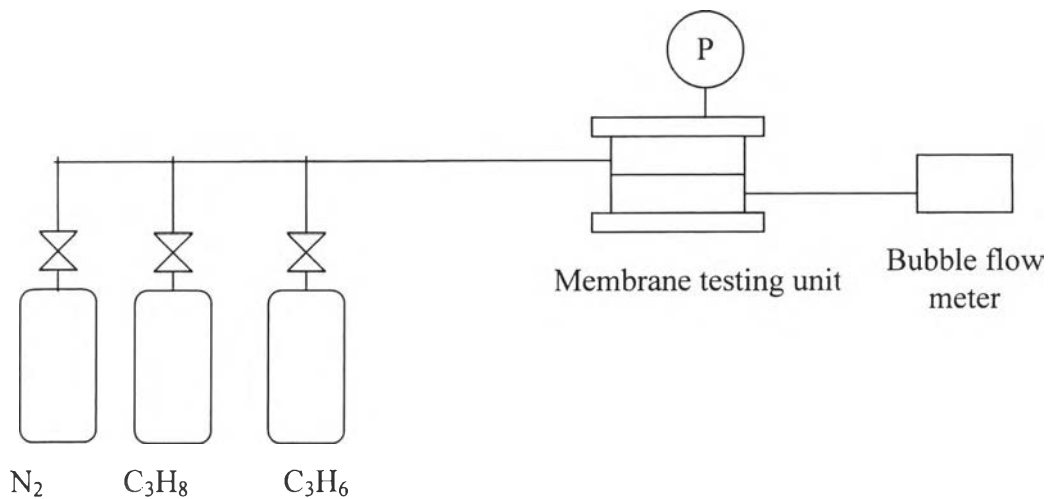


Figure 3.3 Schematic of the experimental setup for measuring gas permeability.

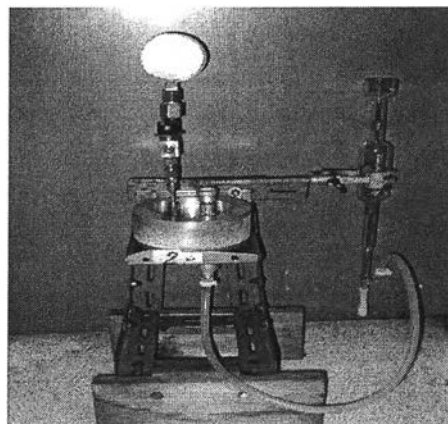


Figure 3.4 Photograph of the membrane testing unit.