

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, LiX; cellulose acetate supporting membrane coated on non-woven cloth. Activated carbon (AC) was obtained from Aldrich Chemical, Inc. The solvent used was cyclohexane from Carlo Erba Reagenti Co., Ltd. Polyethylene glycol (PEG) MW400 and diethanolamine (DEA) were obtained from Fluka Co., Ltd. Gases used were carbon dioxide (CO₂), methane (CH₄), and nitrogen (N₂). CO₂ and N₂ were obtained from Prax Air Co., Ltd. whereas CH₄ was acquired from TIG Plc.

3.2 Membrane Preparation

The solid-liquid-polymer mixed matrix membranes studied were prepared by using different types of liquids such as PEG or DEA. Such of liquids were individually adsorbed into solids used i.e. activated carbon, NaX, LiX. The adsorbed solid was then dispersed in silicone rubber and coated on cellulose acetate. The detail of preparation was separated into two parts as follows:

3.2.1 Preparation of Liquid Adsorbed Solid

There were a series of liquid adsorbed solids in this study which are following: 10-30%PEG/AC, 10-30%DEA/AC, 10-30%PEG/NaX, 10-30%DEA/NaX, 10-30%PEG/LiX and 10-30%DEA/LiX.

First of all, AC, NaX and LiX used were dried at 400 °C in order to remove the amount of water trace in their pores. The preparation started by dissolving the liquid (PEG or DEA) with acetone. After well mixing, the dried solids (AC, NaX or LiX) were admixed into the solution and stir until the homogeneous

solution was formed. The obtained solution was left for 1 day to achieve equilibrium before dried at 90 °C for overnight in order to remove the acetone.

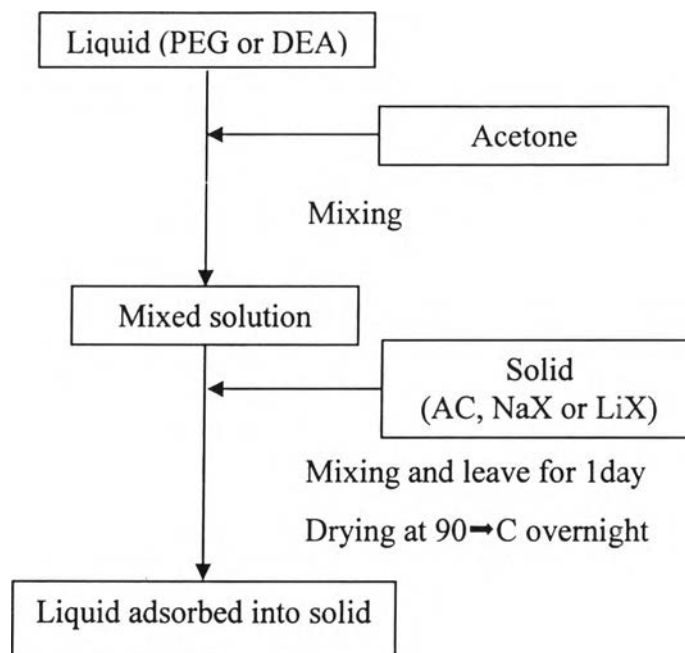


Figure 3.1 Liquid adsorbed solid preparation procedure.

3.2.2 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 5-30% 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.

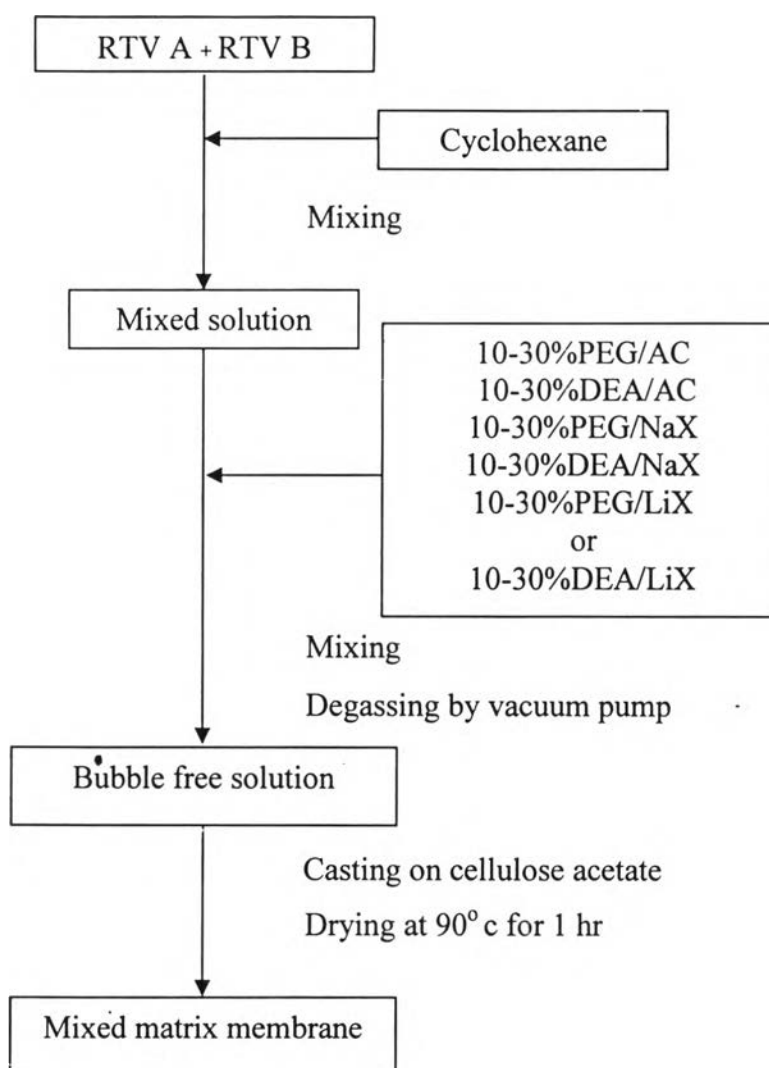


Figure 3.2 Membrane fabrication procedure.

3.3 Gas Permeability Measurements

The experimental setup used for the determination of gas permeability is schematically shown in Figure 3.3. The gas permeability measurement was performed by utilizing a 7.5 centimeters in diameter membrane sample, which was supported by a metal plate and placed inside the membrane-testing unit with an O-ring forming a seal around the edge as shown in Fig. 3.4. The various test gases; N₂, CH₄ and CO₂ were individually introduced into the membrane testing unit at 10-100 psia at the feed side of the membrane while the permeate side of the membrane was at atmospheric pressure. 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, the membrane testing unit has to be purged and vacuumed at permeate side by a vacuum pump in order to get out the old gas from membrane.

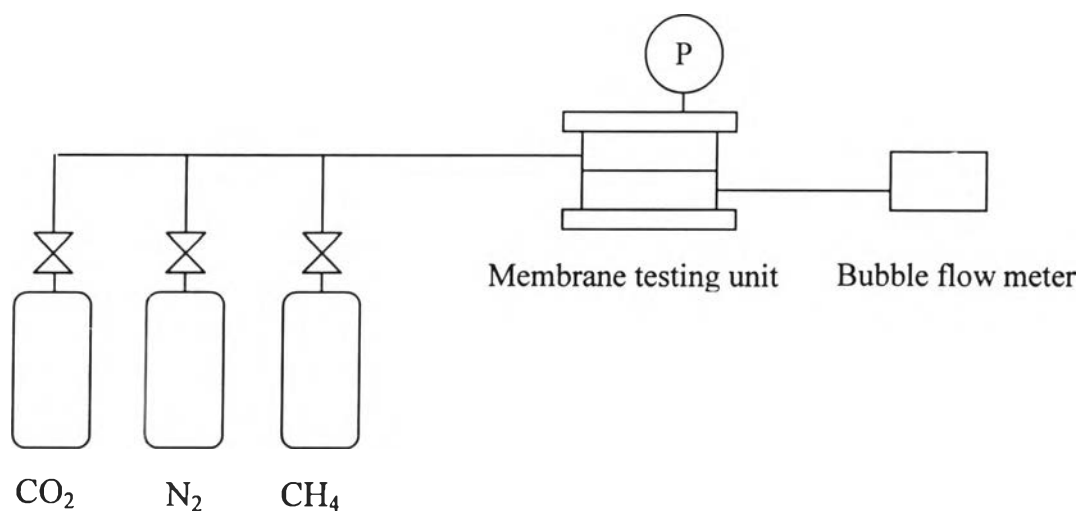


Figure 3.3 Experimental setup for measuring gas permeability.

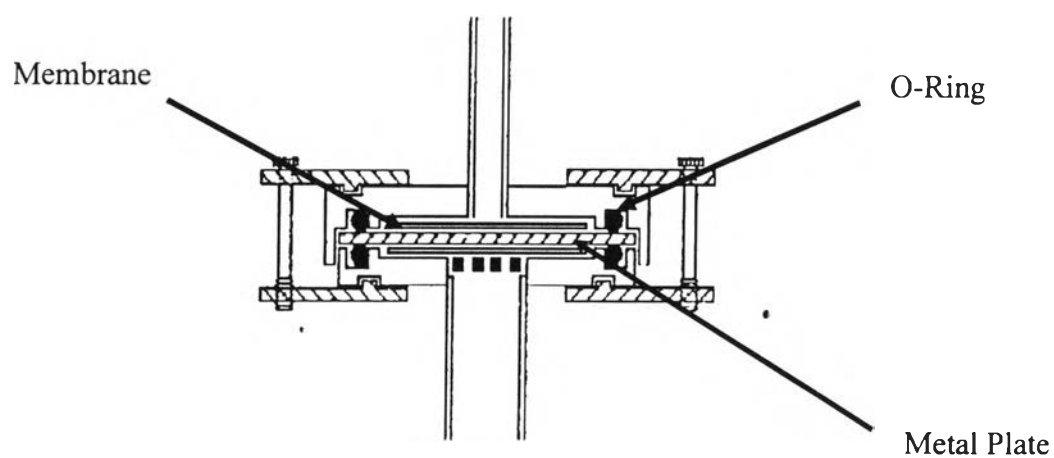


Figure 3.4 Schematic of membrane testing unit.