## CHAPTER III EXPERIMENTAL

## 3.1 Materials

Most of materials were kindly donated by UOP LLC. Ag-LZ-210, Ag-X and Ag-A zeolites were used as a carrier. Polysulfone support membrane casted on non-woven cloth was used as a backing. Polyethylene glycol (PEG) MW400 was used as plasticizers. The silicone rubber used in this study was RTV615A and RTV615B by General Electric Co. Ltd. The cyclohexane solvent was obtained from Carlo Erba Reagenti. Gases used in this study were ethylene polymerization grade obtained from National Petrochemical Co. Ltd., ethane gas 99.99% purity obtained from Thai Industrial Gas Co.Ltd. and nitrogen gas 99.999% purity obtained from Prax Air.

## 3.2 Membrane Preparation

A casting solution was prepared by admixing 0.6 g Ag-zeolite and 7 ml cyclohexane, then, admixing 1.512 g RTV615A and 0.168 g RTB615B. The casting solution was then coated on a polysulfone membrane with a desired thickness by a caster. The polysulfone membrane of 10 x 10 cm in size, which was placed on a glass plate, was heated in an oven at 82°C for 30-60 min. The membranes prepared for the experiments were categorized in 6 different types as follows:

Type I (SR/Ag-zeolite/PS) consisted of three subtypes of which they were polysulfone membranes coated by a casting solution of silicone rubber added with each of Ag-zeolites. The Ag-zeolites used were Ag-LZ-210, Ag-A and Ag-X.

Type II (SR/PS) was a polysulfone membrane coated by a silicone rubber casting solution.

Type III (SR/PEG/PS) was a polysulfone membrane coated by a casting solution of silicone rubber and polyethylene glycol (PEG). The membrane preparation procedure of Type I to Type III is shown in Figure 3.1.

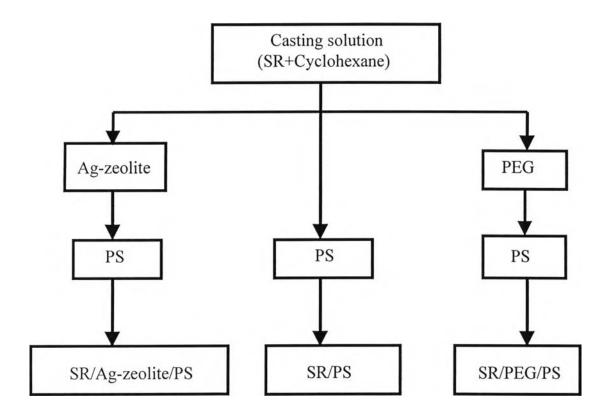


Figure 3.1 Membrane preparation procedure (I).

Type IV (SR/PEG-PS) was a treated polysulfone membrane coated by a silicone rubber casting solution. The treated polysulfone membrane (PEG-PS) was prepared by soaking a polysulfone membrane in a 20% PEG in aqueous solution for an hour and being dried at 105°C for 3 hours.

Type V (SR/Ag-zeolite/PEG-PS) consisted of three subtypes of which they were treated polysulfone membranes coated by the same casting solution as used for Type II

Type VI (SR/Ag-zeolite( $H_2O$ )/PEG-PS) consisted of three subtypes which were different from Type V in that each zeolites was added by water and then mixed with silicone rubber. Figure 3.2 shows the membrane preparation procedure of Type IV to Type VI.

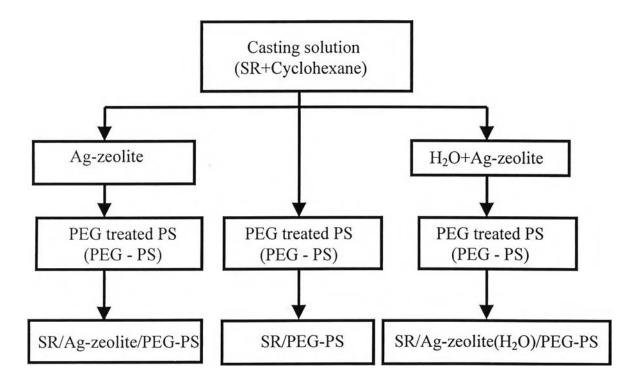


Figure 3.2 Membrane preparation procedure (II).

## 3.3 Design and Experimental Setup

The prepared mixed matrix membrane having a diameter of 7.5 cm was placed for test inside the membrane-testing unit with an O-ring forming a seal around the edge. The membrane was supported by a metal plate. There

were 7 steps to test the membrane. First, N<sub>2</sub> gas was passed through the membrane-testing unit and maintained at a pressure of 50 psig. The permeate side of the testing gas was exposed to atmospheric pressure via a bubble flow meter for flux measurements. Second, shut off the N<sub>2</sub> and turn on C<sub>2</sub>H<sub>6</sub> and purge for 5 minutes by opening the outlet valve at the retentate side. Third, turn off the outlet valve at the retentate side and allow C<sub>2</sub>H<sub>6</sub> to permeate through membrane for 2-3 hours to wash out gas N<sub>2</sub> in the membrane phase. Fourth, read flux of C<sub>2</sub>H<sub>6</sub> for 30-60 min or to get steady state flux. Fifth, shut off the tested C<sub>2</sub>H<sub>6</sub> and turn on gas N<sub>2</sub> and purge for 5 min. Sixth, turn off outlet valve and read flux N<sub>2</sub> (same as step 4). Seventh, repeat step 2 for C<sub>2</sub>H<sub>4</sub> and N<sub>2</sub>, respectively. For each membrane, it took 2 days to finish test. The schematic diagram of the experimental setup is shown in Figure 3.3 and the cross section of the membrane testing unit is shown in Figure 3.4.

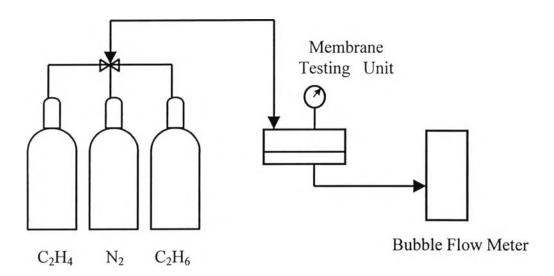


Figure 3.3 Schematic diagram of the experimental setup.

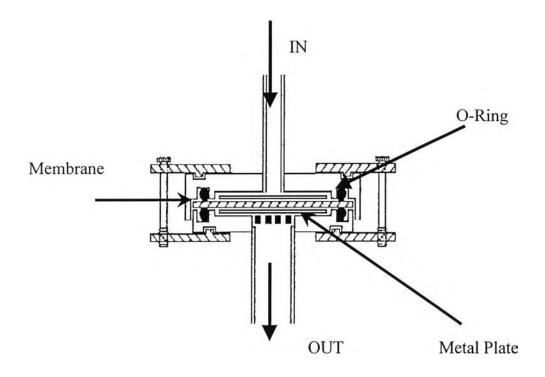


Figure 3.4 The cross section of the membrane testing unit.