

## CHAPTER III EXPERIMENTAL

### 3.1 Materials

o-, m-, and p-xylenes (99.95% purity, Merck Co.)  
Ethylbenzene (99.95% purity, Fluka Co.)  
Iso-octane (99.95% purity, Merck Co.)  
Nonane (99.95% purity, Lab-Scan Co.)  
Hexane (99.95% purity, Merck Co.)  
Hexene (99.95% purity, Merck Co.)  
Octane (99.95% purity, Merck Co.)  
Octene (99.95% purity, Merck Co.)  
Nitrogen gas  
Adsorbent : NaY, silicalite, and activated carbon  
Silicone rubber  
Polysulfone  
Polyimide (Ultem 1000)  
N-methylpyrrolidinone (NMP)

### 3.2 Equipment

Extractor  
Pervaporation unit  
Gas chromatograph (AT Wax Column, length = 60 m, ID: 0.32mm, film thickness = 0.5 $\mu$ m, carrier gas = H<sub>2</sub>)

### 3.3 Methodology

#### 3.3.1 Membrane Selection

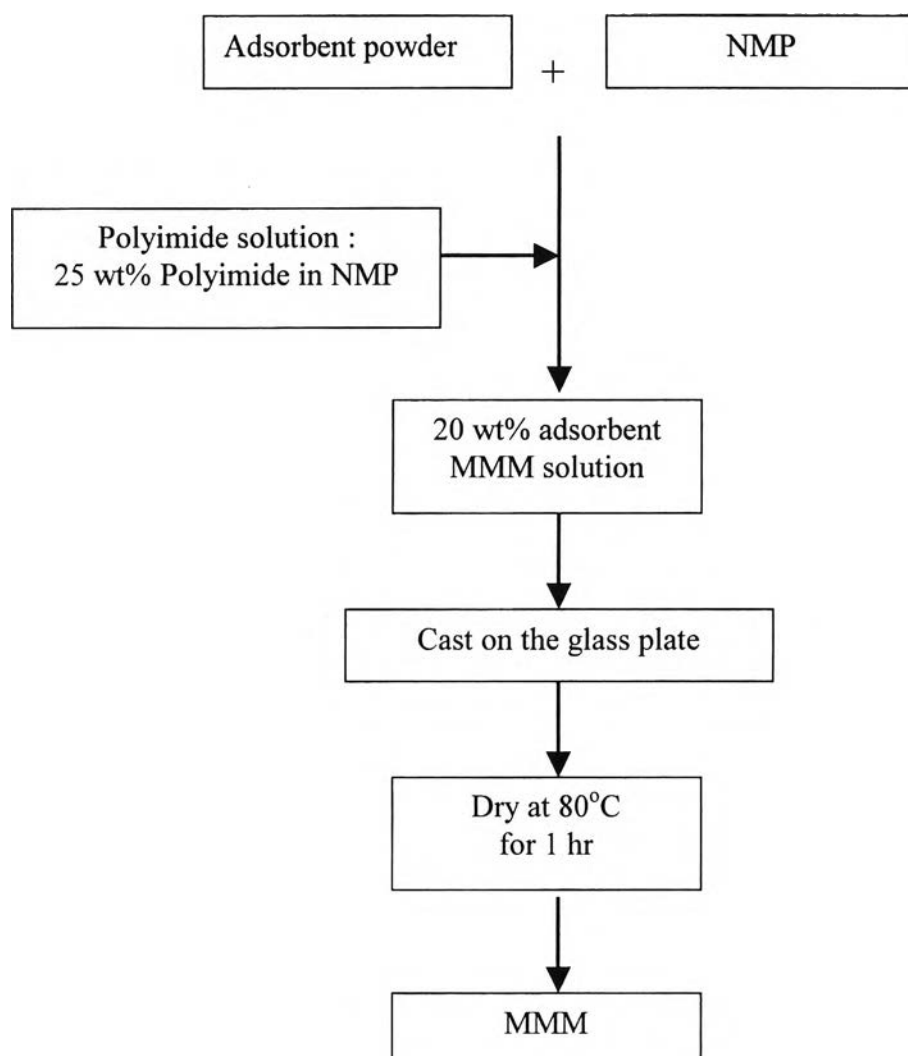
In this research, silicone rubber, polysulfone and polyimide (Ultem 1000) membrane were prepared. To select a membrane with stability, the membrane was soaked in the C<sub>8</sub>-aromatics feed solution (p-xylene, m-xylene, o-xylene, ethylbenzene and n-nonane). Any physical changes of the membrane were observed. The membrane was also tested in the extraction unit with the feed solution. Only membranes without any damage were chosen for experiments.

### 3.3.2 Mixed Matrix Membrane Preparation

From the membrane selection method, it was found that polyimide membrane was selected for further study. MMM's were prepared by solution-casting and solvent evaporation methods. The polyimide solution was formed by mixing 25 wt% polyimide with 75 wt% N-methylpyrrolidinone (NMP). Then, adsorbent powder was mixed with NMP to remove the air bubble in the adsorbent pores. The polyimide solution and the adsorbent were mixed together and shaken to obtain a well-mixed solution. In this research, the total amount of an adsorbent in MMM's was 20 wt%. The mixture was degassed using a vacuum pump. The bubble-free mixture was cast on a clean glass plate. The membrane thickness was set at 15 mil by adjusting a caster knife. The cast film is left in an oven 80°C for 1 hr to remove the solvent. The schematic of membrane preparation is shown in Figure 3.1.

### 3.3.3 Extraction Testing

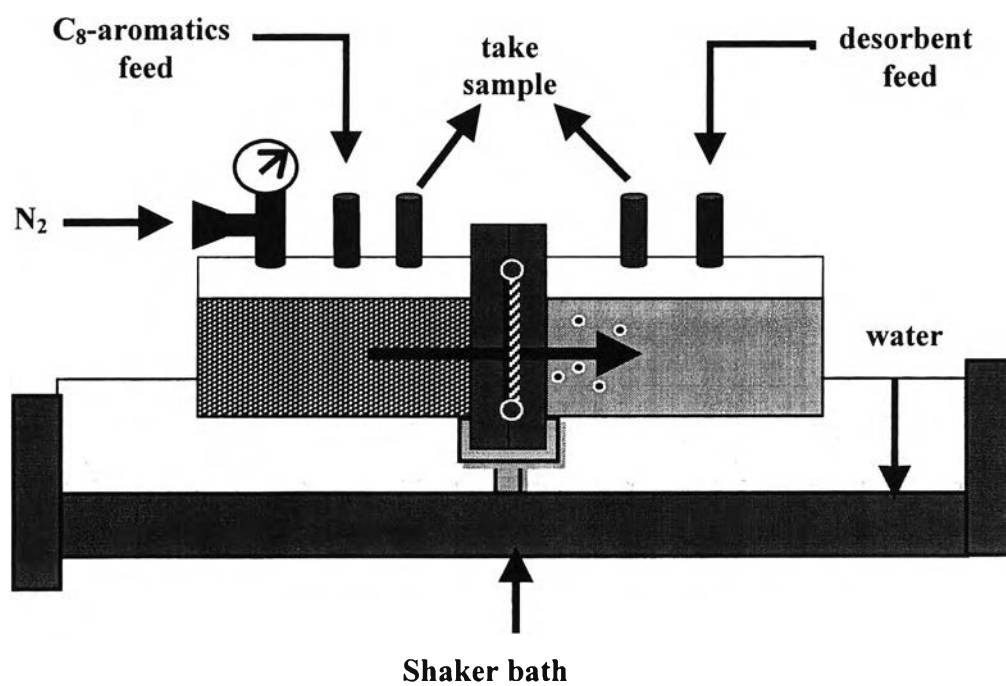
Figure 3.2 shows the experimental set up. A MMM was placed in contact with a stainless-steel porous support in the middle of the extractor. The C<sub>8</sub>-aromatics and a tracer (nonane) were mixed together. Then, 95 ml of this solution was fed in one side of the extractor. The other side was filled with 95 ml of the desorbent (i-octane). Nitrogen gas was applied to the C<sub>8</sub>-aromatics and tracer mixture through the valve at 15 psi. The extractor was placed on the shaker bath. Samples from both sides were withdrawn periodically. A chromatograph equipped with AT Wax column was used for quantitative analysis. The study was carried out at room temperature.



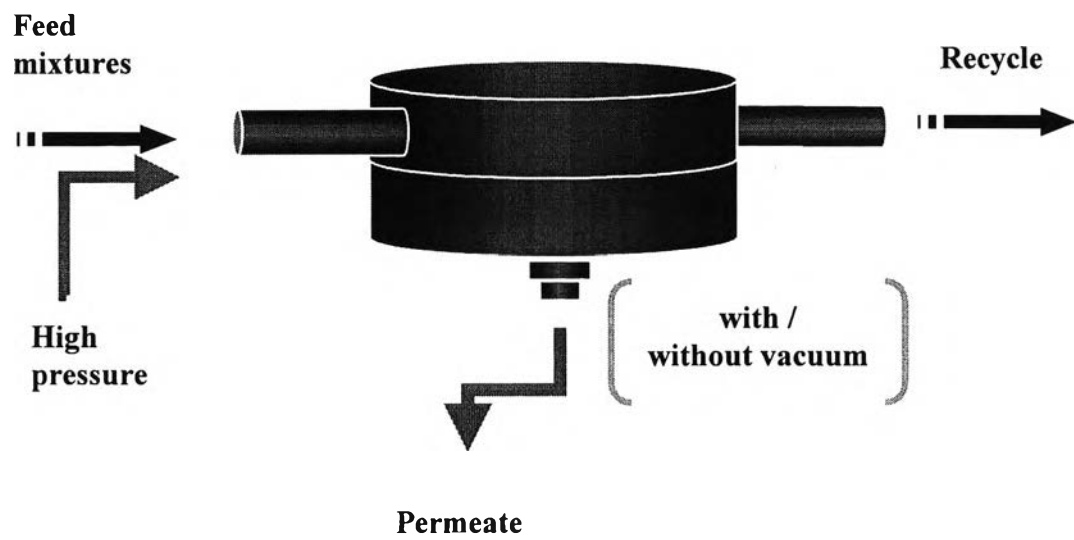
**Figure 3.1** MMM preparation method.

#### 3.3.4 Pervaporation Testing

Figure 3.3 shows the pervaporation unit set up. The unit was placed in the hot box, where the temperature can be controlled. A MMM was placed in the middle of the unit. Feed mixture containing paraffin, olefin and aromatics was fed to the upper side at the flow rate of 9 ml/min and the pressure inlet of 1200 psi. For the permeate side, the pressure was controlled as the vacuum. The vapor that passes through the membrane was condensed in the condenser and detected by the same gas chromatograph.



**Figure 3.2** Schematic diagram of the extraction unit.



**Figure 3.3** Schematic diagram of the pervaporation unit.