

CHAPTER III EXPERIMENTAL

3.1 Materials

3.1.1 Chemicals

All chemicals used in the experiments are shown in Table 3.1. They were treated by silica to remove contaminants before use.

Table 3.1 Chemicals used in the experiments

Name	Source	Purity
<i>p</i> -xylene	Sigma-Aldrich	99.95%
<i>o</i> -xylene	Sigma-Aldrich	99.95%
n-heptane	Sigma-Aldrich	99.95%
1-hexene	Sigma-Aldrich	99.95%
<i>p</i> -diethylbenzene	Sigma-Aldrich	99.95%
N-methyl-2-pyrrolidone	Sigma-Aldrich	99.95%

3.1.2 Other Materials

Silicalite-1 was supported by UOP LLC and polyimide (Ultem) was obtained from GE Plastics.

3.2 Experiments

3.2.1 Mixed Matrix Membrane Preparation

Mixed matrix membranes (MMM's) were prepared by solution casting and solvent evaporating methods. Polyimide solution was first prepared by dissolving 25 wt% polyimide in 75 wt% N-methyl-2-pyrrolidinone (NMP). Silicalite-1 of 10, 20, and 30 wt% was mixed with NMP to remove air bubbles in the

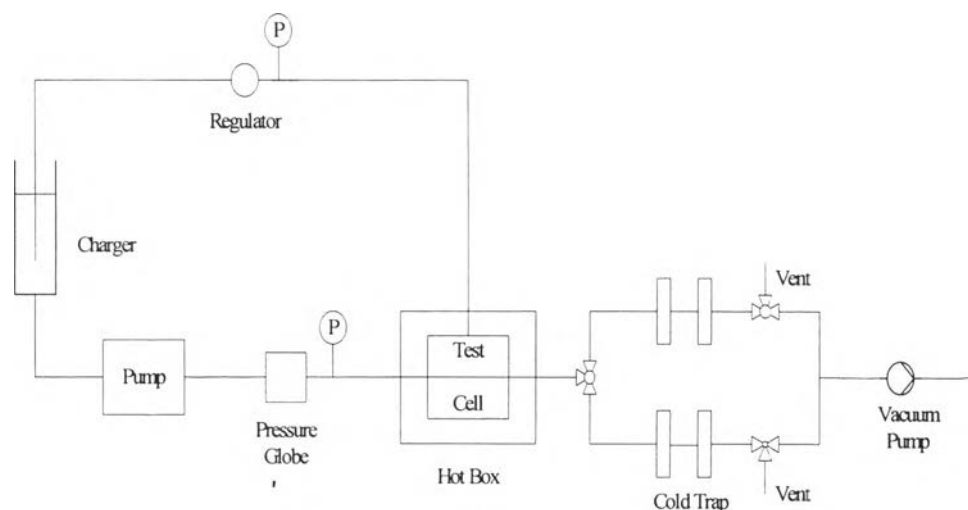
pores of zeolite. It was then added, and then shaken until a well-mixed solution was obtained. The solution was degassed with a vacuum pump, cast on a clean glass plate with a casting knife of 15 mil clearness, and then left in an oven at 60°C overnight. After the membranes were detached from the glass plate, residual solvent was removed by drying subsequently at 100, 130 and 160°C for 1 h each.

The cross-sectional morphology of membranes was observed with scanning electron microscope (SEM, JEOL JSM-5200).

3.2.2 Pervaporation Testing

Figure 3.1 shows schematics of the experimental setup and membrane pervaporator. The membranes were tested in pervaporation apparatus that included a water pump, a test cell which held the membrane and was placed in a hot box, cold trap and vacuum pump. Exposed membrane was 34.2 cm² and feed circulation was about 9 cm³/min. A feed mixture contained a 1:1:1:1 ratio of *p*-xylene, *o*-xylene, paraffin, and olefin. Temperature at 120°C was used for all experiments. The feed was maintained at 120 psig to ensure that it was in the liquid phase. For all experiments, the permeate pressure was maintained under vacuum. The feed, permeate and retentate were analyzed by a Hewlett Packard 5890 series II gas chromatograph with an AT™-WAX capillary column and equipped with a FID.

(a)



(b)

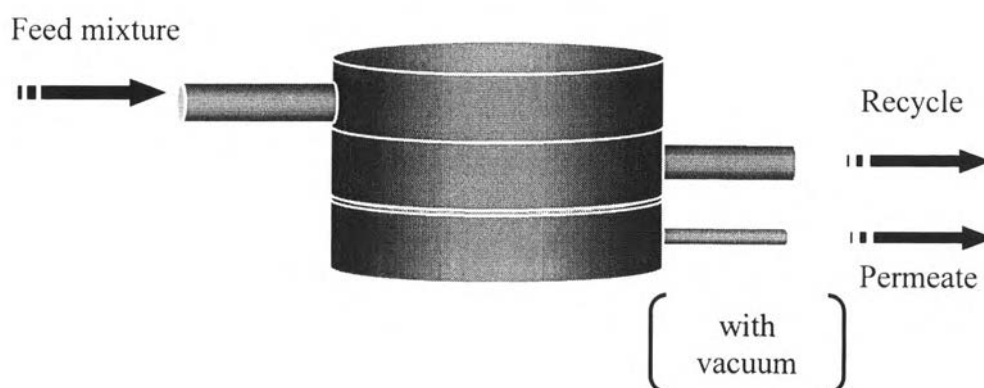


Figure 3.1 Schematics of pervaporation process: (a) Flow chart of pervaporation process; (b) the membrane pervaporator.

3.2.3 Single Component Adsorption Experiments

This adsorption studied C_8 aromatics and paraffin on silicalite-1. The experiment was started with adding all components according to Tables 3.2 to 3.4 and silicalite-1 into vials. The weight ratio of silicalite-1 and solution was set at 1 to 4 in each batch. The vials were agitated constantly at room temperature. The system

was left until it reached equilibrium. The equilibrium of the system was observed by measuring the liquid solution with the gas chromatograph.

Table 3.2 Sample preparation for *p*-xylene solution

Concentration (wt%)	<i>p</i> -xylene (g)	n-nonane (g)	Silicalite-1 (g)
1.25	0.05	3.95	1.00
2.5	0.10	3.90	1.00
5	0.20	3.80	1.00
10	0.40	3.60	1.00
20	0.80	3.20	1.00
40	1.60	2.40	1.00
60	2.40	1.60	1.00

Table 3.3 Sample preparation for *o*-xylene solution

Concentration (wt%)	<i>o</i> -xylene (g)	n-nonane (g)	Silicalite-1 (g)
1.25	0.05	3.95	1.00
2.5	0.10	3.90	1.00
5	0.20	3.80	1.00
10	0.40	3.60	1.00
20	0.80	3.20	1.00
40	1.60	2.40	1.00
60	2.40	1.60	1.00

Table 3.4 Sample preparation for n-hexane solution

Concentration (wt%)	n-hexane (g)	n-nonane (g)	Silicalite-1 (g)
1.25	0.05	3.95	1.00
2.5	0.10	3.90	1.00
5	0.20	3.80	1.00
10	0.40	3.60	1.00
20	0.80	3.20	1.00