

CHAPTER III EXPERIMENTAL

3.1 Materials

3.1.1 Chemicals

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

Table 3.1 Chemicals used in the experiments

Name	Source	Purity	CAS number
<i>p</i> -xylene	Merck	99.95 %	106-42-3
<i>o</i> -xylene	Merck	99.95%	95-47-6
<i>m</i> -xylene	Merck	99.95%	108-38-3
ethylbenzene	Fluka	99.95%	100-41-4
toluene	J.K. Baker	99.95%	108-88-3
n-nonane	Lab-Scan	99.95%	111-84-3

3.1.2 Adsorbents

Commercially available 2.0X, 2.5X and Y zeolites (5% LOI) exchanged with di-valence series cations, *Ba*, *Sr*, *Ca* and *Mg*, were used as adsorbents.

3.2 Experiment

3.2.1 Multi-component Pulse Test: Low Concentration Study

Figure 3.1 shows the experimental set up for the multi-component pulse test at low concentration. Water content, %LOI, of each adsorbent was adjusted to the desired value. Zeolite was packed in a 70 ml column with 0.75 and 158 cm as the inside diameter and length. Glass wool was placed at both ends of the

column. The column was positioned in a controlled temperature hot box and connected to the pulse test unit. Then, a desorbent, toluene, was fed to the column at a constant flow rate of 1.2 ml/min. After the column was saturated by the desorbent, the heater was turned on. The fan inside the column was used to circulate the air in order to unitize the temperature. Feed was the mixture of *p*-xylene, *m*-xylene, *o*-xylene, ethylbenzene and *n*-C₉ with 20 wt% each. After reaching the desired temperature, 2 ml of the feed was injected into the column through the two-way valve; at the same time, the fraction collector was started to collect system effluent every 2 min. Each fraction would contain approximately 2.4 ml of the sample. The sample was further analyzed by a Hewlett Packard gas chromatograph equipped with an FID detector and AT™ WAX capillary column. The concentrations of all components were plotted versus the time or volume and then the *p*-xylene selectivities were calculated.

3.2.2 Multi-Component Breakthrough Test: High Concentration Study

The experiment was similar to the Multi-component Pulse test as shown in Figure 3.2. But after the column was saturated by the desorbent and the desired temperature was reached, the two-way valve was switched to let the feed flow through the column. The fraction collector was started at the same time to collect the system effluent every 2 min. After 100 ml of the sample was collected, the two-way valve was switched to let the desorbent flow instead and the collector was replaced by another collector. The desorbent was allowed to pass through the column at the same volume, 100 ml, and the samples were collected continuously. The sample collected by the first fraction collector was called adsorption part sample and the sample collected by the second one was called desorption part sample. Both kinds of sample were analyzed for their concentration by means of the same gas chromatograph Hewlett Packard with FID detector. By using the same method as the multi-component pulse test, *p*-xylene selectivity was calculated.

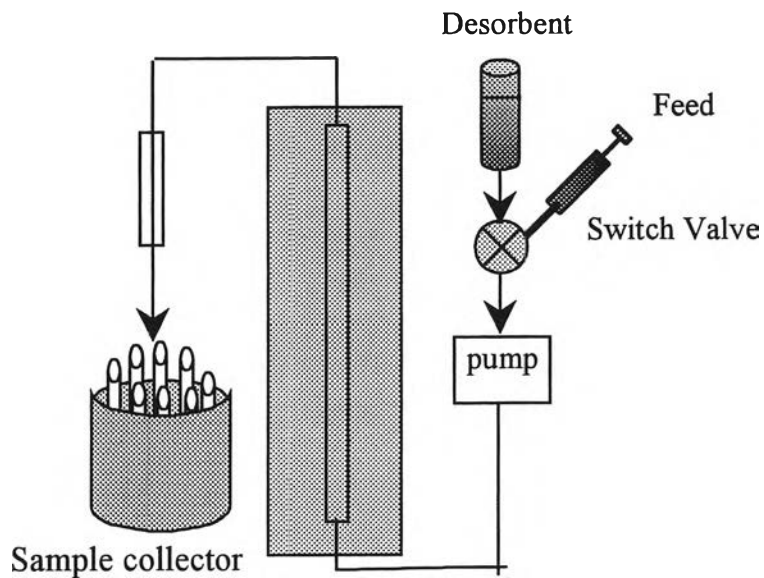


Figure 3.1 Experimental setup of Pulse Test Technique (Low Concentration Study).

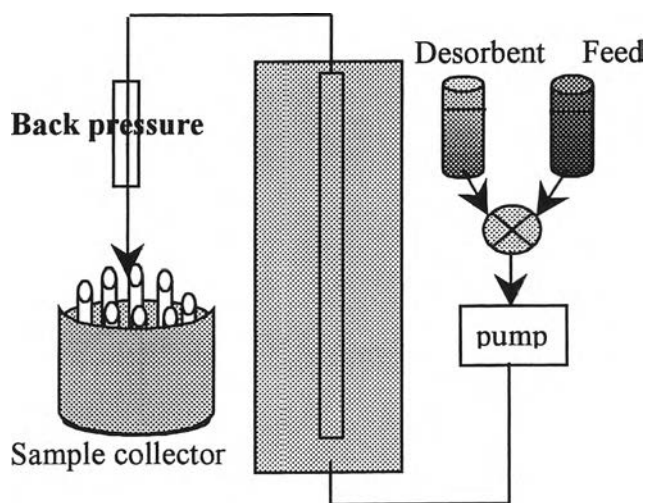


Figure 3.2 Experimental setup of Breakthrough Technique (High Concentration Study).