



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 *X* and *Y* zeolites (3.5% LOI and 3% LOI, respectively) exchanged with monovalence series cations, *Cs*, *Rb*, *K*, *Na* and *Li*, were used as adsorbents in this study.

3.2 Experiment

3.2.1 Dynamic Adsorption: Multi-component Pulse Test

Figure 3.1 shows the experimental set-up for the multi-component pulse test. A 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 to block the adsorbent. The column was positioned in the controlled temperature hot box and connected to the pulse injection unit. The experiments started with feeding

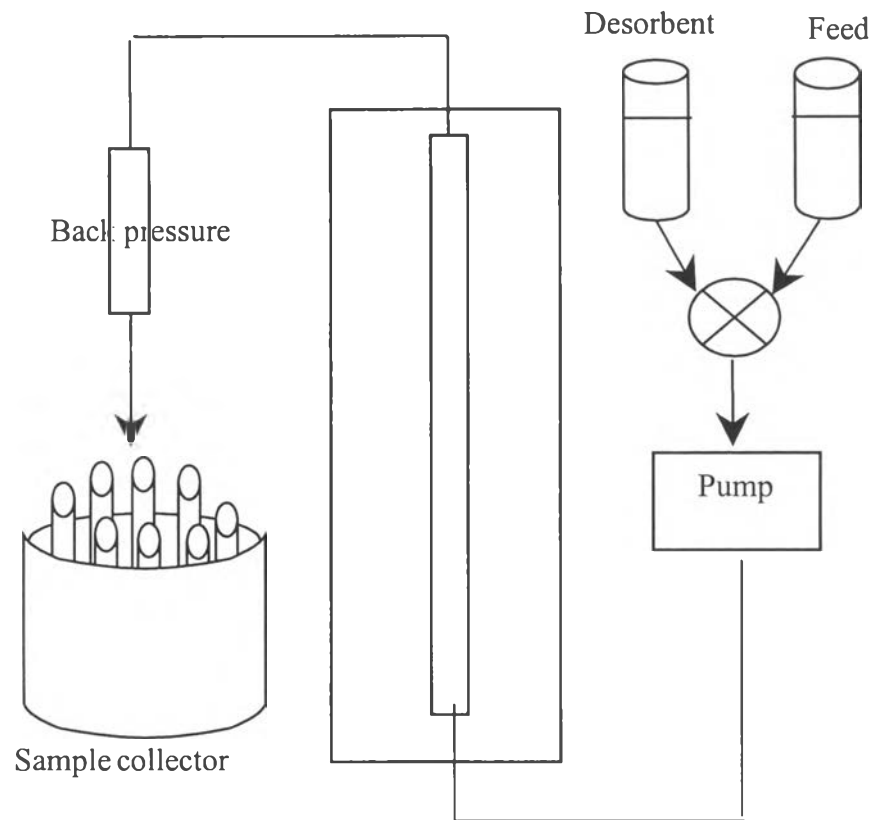


Figure 3.1 The experimental set-up for the dynamic adsorption experiments

the desorbent, toluene, to the column at a constant flow rate of 1.2 ml/min. After the column was saturated with the desorbent, a heater was turned on. A feed solution mixture consisted of *p*-xylene, *m*-xylene, *o*-xylene, ethylbenzene and *n*-C₉ with 20% by wt each. After reaching the desired temperature, 150 °C, 2 ml of the feed was injected into the desorbent stream, which continuously flow through column, through the two-way valve. At the same time, the fraction collector was started to collect the system effluent every 2 min. Each fraction, approximately 2.4 ml of the sample, was analyzed by a Hewlett Packard 5890 series II gas chromatograph equipped with a FID. The concentrations of all components were plotted versus time or volume and then the *p*-xylene selectivity was calculated.

3.2.2 Dynamic Adsorption: Breakthrough Test on KY Zeolite

In this experimental, only *KY* zeolite was used as the adsorbent and there were five different compositions of the feed, with the experimental set-up was similar to the multi-component pulse test as shown in Figure 3.1. After the column was saturated with the desorbent and the desired temperature, 150 °C, 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 the desorbent and another fraction collector was replaced the old one. 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 the adsorption part sample and the sample collected by the second one was called the desorption part sample. All samples were analyzed for their concentration by means of a Hewlett Packard 5890 series II gas chromatograph. The *p*-xylene selectivity was then calculated.

3.2.3 Equilibrium Adsorption Isotherm: Autoclave Work on KY Zeolite

This adsorption study of the C₈ aromatics in the presence of toluene on KY zeolite was performed using a designed reactor at 30, 60, 90, 120 and 150 °C. The same feeds as in the breakthrough test were used. The experimental set-up is shown in Figure 3.2. The reactor used was in the cylindrical shape. The top plate of the reactor was held in place with six bolts. There are two different level valves connected to the reactor. The hollow cylindrical basket containing an adsorbent was placed inside the reactor. Heat was provided by a heater, which was controlled by a PID controller. A Teflon[®] o-ring was placed between the top plate and the reactor to prevent any leak. The mixer attached to the top plate constantly stirred the solution inside the reactor. The speed of the mixer can be adjusted by the control box. A sample from the reactor was withdrawn periodically. The sample was then analyzed by the gas chromatograph.

The experiment was started with packing KY zeolite in the basket. The basket was then placed in the reactor followed by adding the feed into the reactor. The temperature was first set at 30 °C. Once the system reached equilibrium, which, normally, took about three hours, samples were collected and analyzed by the GC. The temperature was then raised to other temperatures.

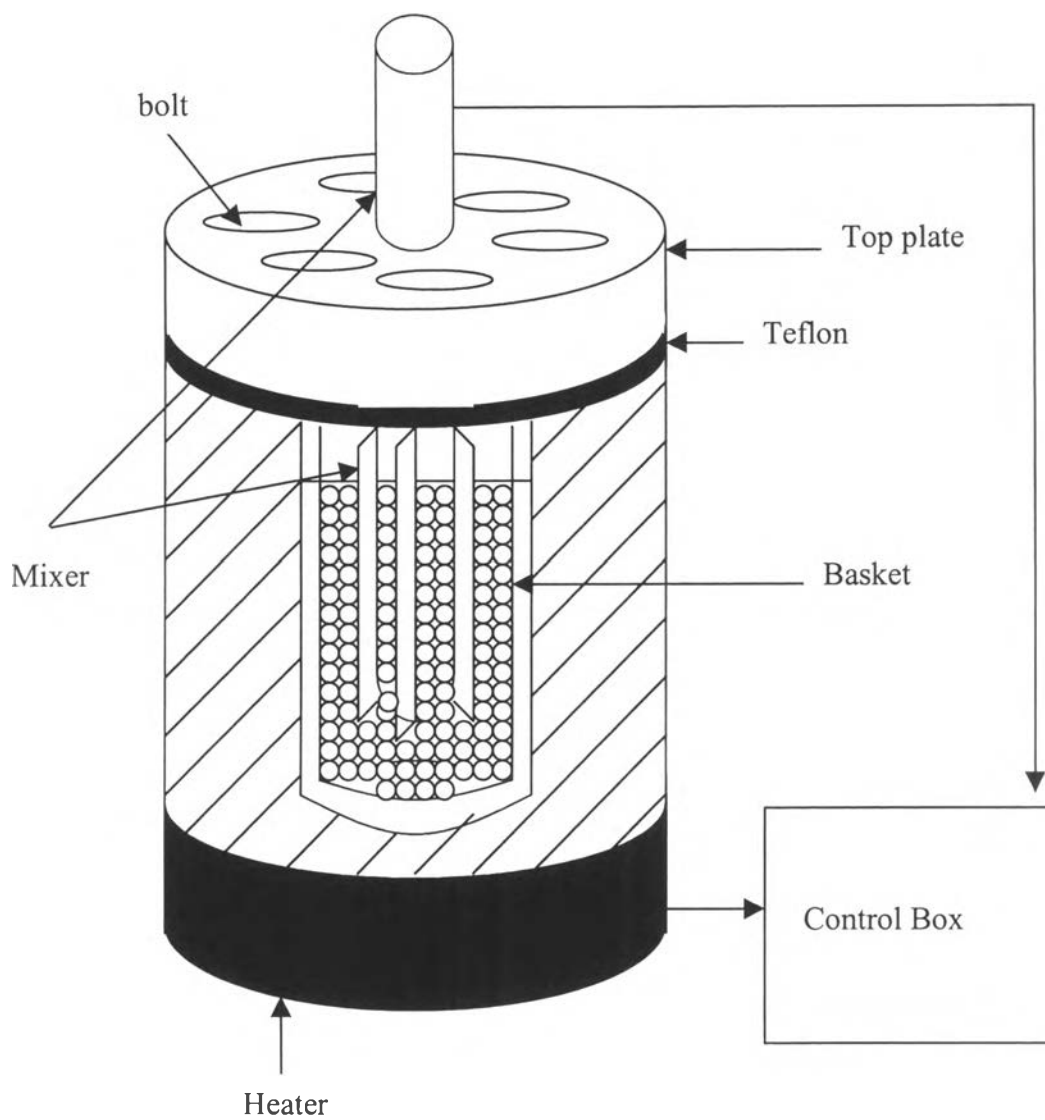


Figure 3.2 The experimental set-up for the equilibrium adsorption experiments