CHAPTER III
EXPERIMENTAL

3.1 Materials

All chemicals used in the experiment are shown in Table 3.1. *KY* and *KBaX* zeolites were supplied by UOP LLC. Their properties are shown in Table 3.2.

Table 3.1 Chemicals used in the experiment

Name	Source	Purity	CAS number
p-xylene	Merck	99.95%	106-42-3
o-xylene	Merck	99.95%	95-47-6
m-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
helium	TIG	99.995%	7440-59-7

Table 3.2 Properties of the *KBaX* and *KY* zeolites from the BET analysis*

Details	*KBaX* zeolite	*KY* zeolite
Composition	Ba 20 wt%, K 4.96 wt%	K 12 wt%
Langmiur surface area (m^2/g)	684.14	813.69
Micropore volume (ml/g)	0.23	0.27
Average pore diameter (^{o}A)	16.32	17.29

3.2 Experiment

3.2.1 Single Component Adsorption Experiment

This adsorption study of the C_8 aromatics and toluene on *KY* and *KBaX* zeolites was performed using a stirred-well reactor at atmospheric pressure and at 40, 65, and 90 °C. The reactor, as used by Ngamkitidachakul, 2000, was made of stainless steel 304 and its thickness is 0.4 cm. The volume is about 100 ml with 6.5 and 4.5 cm as the height and inside diameter. The top plate of the reactor was held in place with six bolts. Heat was provided by heating coils and controlled by an PID controller with ±1 °C in the temperature range of 30 – 700 °C. The reactor was connected with a pressure gauge and a K-type thermocouple through the top plate. An o-ring was placed between the top plate and the reactor to prevent any leak. Solution inside the reactor was constantly stirred using a 0.25 inch magnetic bar. To avoid any vortex formation during the stirring, two baffles were placed inside the reactor. A sample from the reactor was drawn by a syringe through the sampling port that was on the top plate (See Figure 3.1). The sample was then injected into a Fison gas chromatograph equipped with a mass spectrograph 800 series and a RSL-150 capillary column for the analysis. The GC/MS used He with 99.95% purity as a carrier gas. The gas was supplied by the Thai Industrial Gas Co., Ltd. Each cycle of the analysis required about 4 minutes to complete before the next injection. The conditions used for the analysis in this research were summarized in Table 3.3.

Table 3.3 GC conditions for the analysis

Setting	Condition
Injection temperature	220 °C
Oven temperature	50 °C for 1 minute; goes up to 150 °C with the rate of 50 °C; stay at 150 °C for 1 minute
Detector temperature	180 °C
Inter face temperature	200 °C
Carrier gas	Helium 99.995% purity

In order to determine water content in a zeolite, a small amount of the zeolite was weighed and then calcined at 900 °C for 3 hours, where water in the zeolite was completely eliminated. The adsorbent was weighed again. Its water content on the weight percent basis, which is indicated by %LOI, could then be calculated. To adjust the water content, the adsorbent was dried at 350 °C for 3 hours. Then, the zeolite weight was calculated from the desired water content. After that, the water content was varied from 1.2%, 2.4%, and 4.5% by letting the adsorbent adsorb moisture from air.

The experiment was started by adding all the components according to Tables 3.4 to 3.7 into the reactor. The weight ratio of zeolite and solution was set at 1 to 4 in each batch. The solution was then constantly stirred and the temperature was set at 40 °C. The system was left until it reached equilibrium. The equilibrium of the system was observed by measuring the liquid solution with the GC/MS until a constant concentration of each species was observed. Normally, the system reached its equilibrium within two hours.

3.2.2 Multi-Component Pulse Test

The *KY* adsorbent water content was adjusted to the desired value. Then the zeolite was packed in a 70 ml column with 0.75 and 158 cm

as the inside diameter and length. Quartz wool was used to block the adsorbent at both ends of the column. After the column was packed with the adsorbent and connected to the pulse test unit, the desorbent pump was turned on. Toluene was passed through the packed column at a constant flow rate of 1.2 ml/min. The column was placed in a temperature controlled hot box. The operating temperature was varied from 40 – 90 ^{o}C. A fan was used to circulate the air in order to unitize the temperature. The system was left until it reached steady state, which usually takes approximately 2 hours. The feed consisted of *n*-C_9, *p*-xylene, *o*-xylene, *m*-xylene, and ethylbenzene with 20% each. A tracer, *n*-C_9, was selected so that it would not be adsorbed by the adsorbent. The feed was then injected through the feed injector; at the same time, the fraction collector was started to collect the system effluent every 2 minutes. Each fraction would contain approximately 2.4 ml of the sample. Each fraction was analyzed for its concentration by means of a gas chromatograph. The results from the GC were entered into the Sorbex database, provided by the UOP LLC. The program calculates the selectivity of *p*-xylene over other components.

Table 3.4 Samples preparation for *p*-xylene

Concentration (wt%)	toluene (g)	*p*-xylene (g)	*n*-nonane (g)	Zeolite (g)
1.25	28.4400	0.3600	1.4400	7.2000
2.5	28.0800	0.7200	1.4400	7.2000
5	27.3600	1.4400	1.4400	7.2000
10	25.9200	2.8800	1.4400	7.2000
20	23.0400	5.7600	1.4400	7.2000

Table 3.5 Samples preparation for *m*-xylene

Concentration (wt%)	toluene (g)	*m*-xylene (g)	*n*-nonane (g)	Zeolite (g)
1.25	28.4400	0.3600	1.4400	7.2000
2.5	28.0800	0.7200	1.4400	7.2000
5	27.3600	1.4400	1.4400	7.2000
10	25.9200	2.8800	1.4400	7.2000
20	23.0400	5.7600	1.4400	7.2000

Table 3.6 Samples preparation for *o*-xylene

Concentration (wt%)	toluene (g)	*o*-xylene (g)	*n*-nonane (g)	Zeolite (g)
1.25	28.4400	0.3600	1.4400	7.2000
2.5	28.0800	0.7200	1.4400	7.2000
5	27.3600	1.4400	1.4400	7.2000
10	25.9200	2.8800	1.4400	7.2000
20	23.0400	5.7600	1.4400	7.2000

Table 3.7 Samples preparation for ethylbenzene

Concentration (wt%)	toluene (g)	ethylbenzene (g)	*n*-nonane (g)	Zeolite (g)
1.25	28.4400	0.3600	1.4400	7.2000
2.5	28.0800	0.7200	1.4400	7.2000
5	27.3600	1.4400	1.4400	7.2000
10	25.9200	2.8800	1.4400	7.2000
20	23.0400	5.7600	1.4400	7.2000

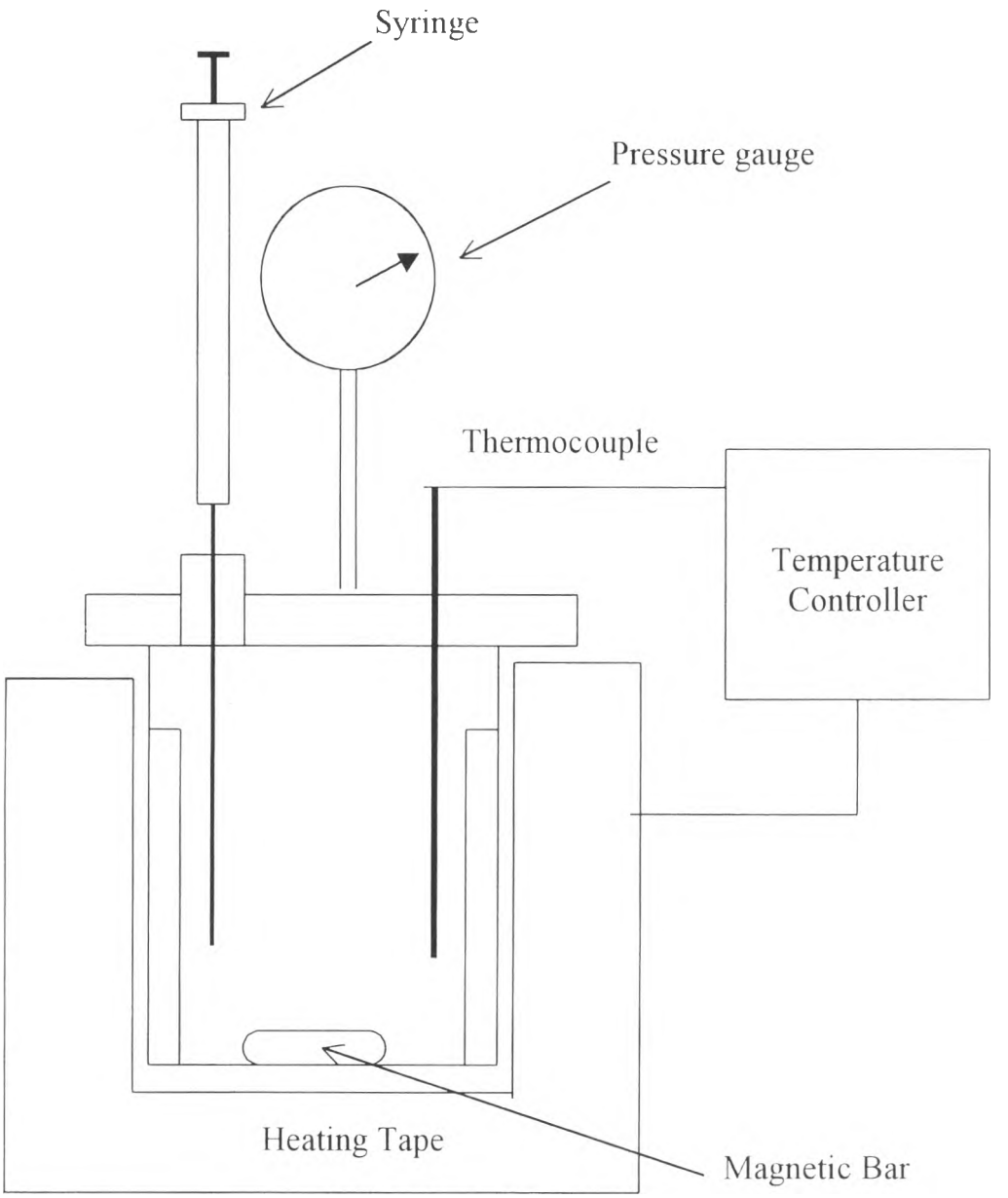

Figure 3.1 Experimental setup for single component adsorption experiment

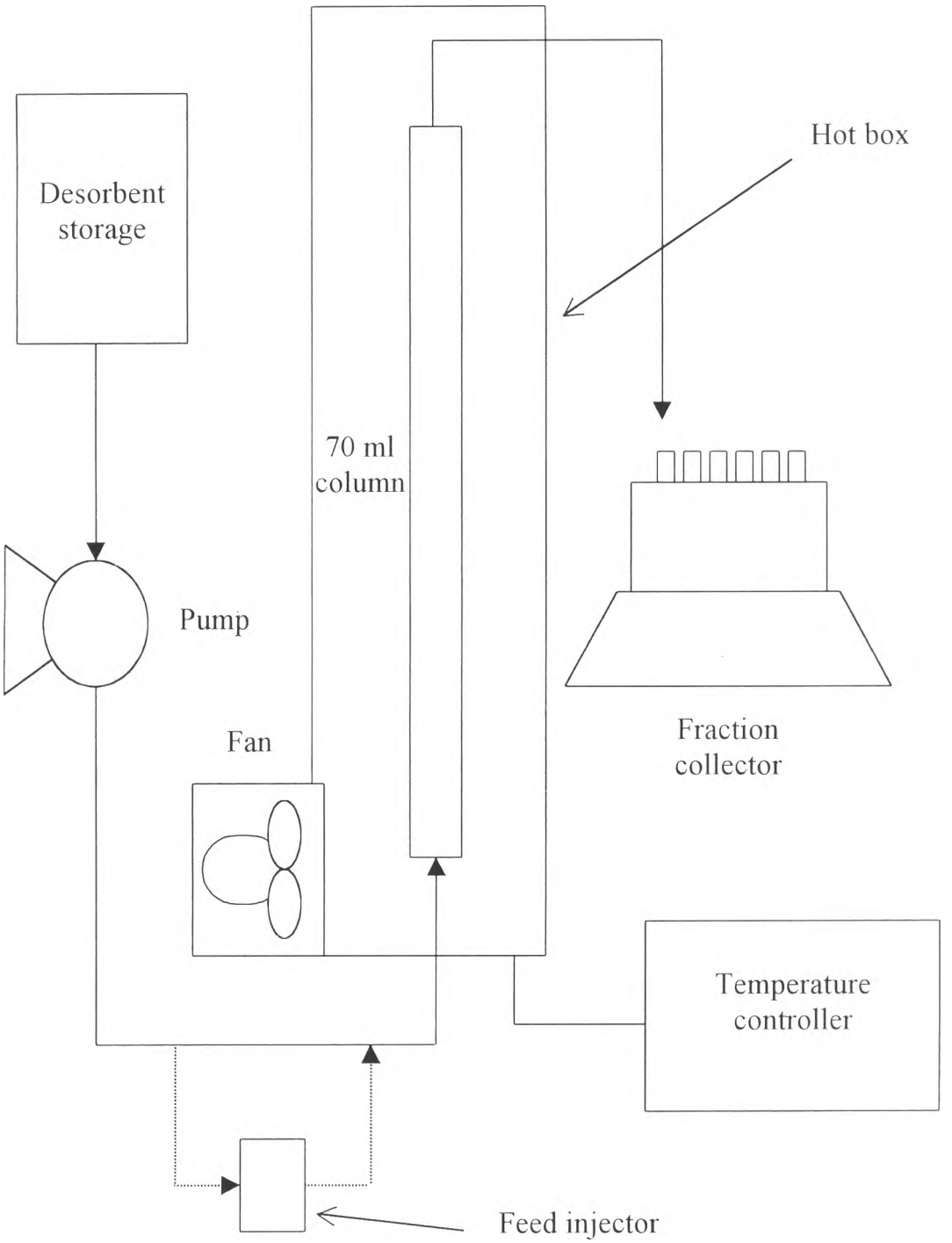

Figure 3.2 Experimental setup of a pulse test unit