

CHAPTER III

EXPERIMENTAL

This adsorption study of the C_8 aromatics and toluene on *KBaX* and *KY* zeolites was performed using a stirred-well reactor at atmospheric pressure and at 40, 65 and 90°C. Illustration of the complete reactor system is shown in Figure 3.1.

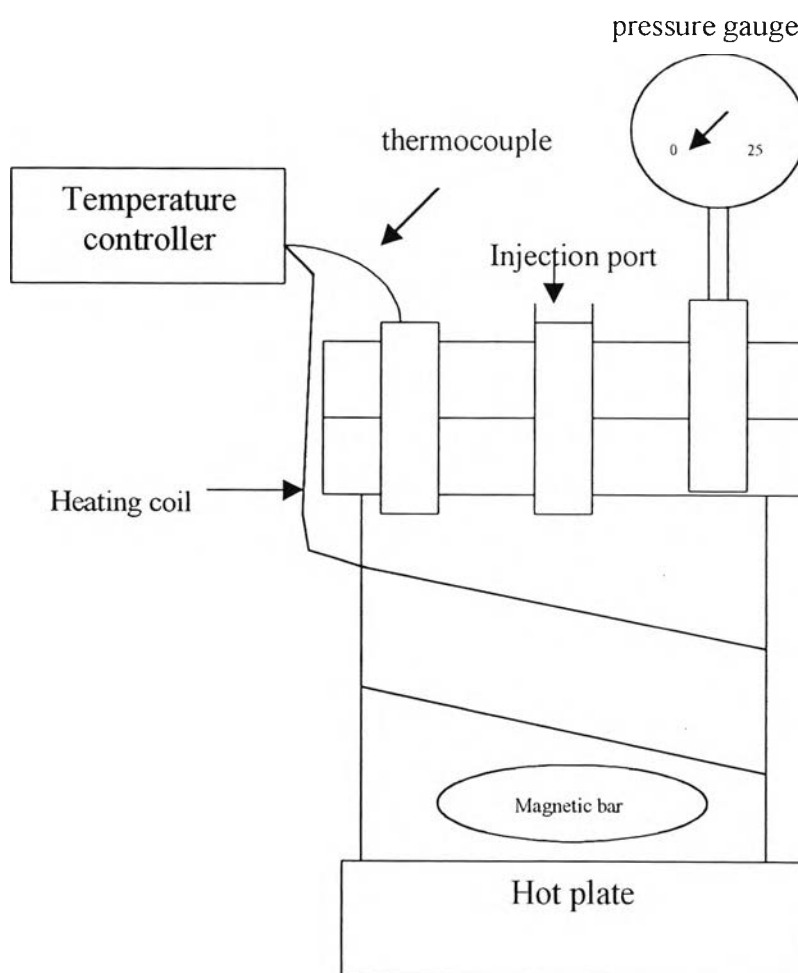


Figure 3.1 The experimental setup.

3.1 Experimental Setup

The reactor was built with 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 a PID controller with $\pm 1^{\circ}\text{C}$ in the temperature range of 30-700 $^{\circ}\text{C}$. The reactor was connected with a pressure gauge and a type-K 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 an 1-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. The sample was then injected into a Fison gas chromatography and mass spectrography 800 with RSL-150 capillary column for the analysis. The GC/MS used He with 99.995 % purity as a carrier gas. The gas was supplied by the Thai Industrial Gas Co., Ltd. Each cycle of the analysis required about 8 minutes to complete before the next injection can start. The conditions used for the analysis in this research were summarized below:

Injection Temperature:	220 $^{\circ}\text{C}$
Oven Temperature:	60 $^{\circ}\text{C}$ for 1 minute; goes up to 100 $^{\circ}\text{C}$ with the rate of 20 $^{\circ}\text{C}/\text{min}$; stays at 100 $^{\circ}\text{C}$ for 1 minute; goes up to 120 $^{\circ}\text{C}$ with the rate of 10 $^{\circ}\text{C}/\text{min}$ and stays there for 1 minute
Detector Temperature:	180 $^{\circ}\text{C}$
Interface Temperature:	170 $^{\circ}\text{C}$
Carrier Gas:	He (99.995%)

3.2 Experimental Procedure

3.2.1 Sample Preparation

Five sample sets of each aromatic and toluene were prepared according to Tables 3.1 to 3.4. *P*-xylene with 99 % purity was obtained from the Fluka Co., Ltd. *M*-xylene, *o*-xylene and toluene were purchased from the Merck Chemical Co., Ltd. Toluene, which was used as a desorbent along with *n*-nonane, which was used as a tracer were obtained from the Lab-Scan Co., Ltd. All chemicals were used without further purification. Two zeolites, *KBaX* and *KY* (LOI 2.5%), were supplied by the UOP LLC. Properties of the *KBaX* and *KY* zeolites from the BET analysis were shown in Table 3.5.

Table 3.1 Samples preparation for *p*-xylene.

Sample	Toluene (ml.)	<i>p</i> -xylene (ml.)	<i>n</i> -nonane (ml.)	Zeolite (g)
1	35.159	8.841	2.650	9.515
2	39.577	4.423	2.652	9.521
3	41.788	2.212	2.653	9.523
4	42.894	1.106	2.653	9.524
5	43.447	0.553	2.653	9.525

Table 3.2 Samples preparation for *m*-xylene.

Sample	Toluene (ml.)	<i>m</i> -xylene (ml.)	<i>n</i> -nonane (ml.)	Zeolite (g)
1	35.184	8.816	2.652	9.522
2	39.591	4.409	2.653	9.524
3	41.795	2.205	2.653	9.525
4	42.898	1.102	2.653	9.525
5	43.449	0.551	2.653	9.526

Table 3.3 Samples preparation of *o*-xylene.

Sample	Toluene	<i>o</i> -xylene	<i>n</i> -nonane	Zeolite
	(ml.)	(ml.)	(ml.)	(g)
1	35.320	8.679	2.663	9.559
2	39.668	4.333	2.658	9.542
3	41.836	2.164	2.656	9.534
4	42.918	1.082	2.655	9.530
5	43.459	0.541	2.654	9.528

Table 3.4 Samples preparation of ethylbenzene.

Sample	Toluene	ethylbenzene	<i>n</i> -nonane	Zeolite
	(ml.)	(ml.)	(ml.)	(g)
1	35.208	8.792	2.654	9.528
2	39.604	4.396	2.654	9.527
3	41.802	2.198	2.654	9.527
4	42.901	1.099	2.654	9.526
5	43.451	0.549	2.654	9.526

Table 3.5 Properties of the *KBaX* and *KY* zeolites from the BET analysis*.

	<i>KBaX</i> zeolite	<i>KY</i> zeolite
Composition	<i>Ba</i> 20%wt <i>K</i> 4.96%wt	<i>K</i> 12%wt
Langmiur surface area (m ² /g)	684.14	813.69
Micropore volume (cc/g)	0.23	0.27
Average pore diameter (°A)	16.32	17.29

*Provided by Kulprathipanja (2000)

3.2.2 Adsorption Experiment

The experiment was started with adding all the components according to Tables 3.1-3.4 and the zeolite into the reactor. 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 by the liquid solution with the GC/MS until constant concentration of each species was obtained. After that, the solution was heated to 65 and 90 °C. Once the system reached its equilibrium, the amount of each species presented in the solution was defined by calibration curve.