CHAPTER III
EXPERIMENTAL

3.1 Materials

Materials:

1) Vial 10 ml, purchased from Amani Co., Ltd
2) 20 mm, PTFE/Black Butyl Molded Septa with Caps, purchased from Amani Co., Ltd
3) NaY, KY, BaX, and BaY zeolites, supplied by UOP. A Honeywell Company. USA

Chemicals:

1) *p*-Xylene ($C_6H_4(CH_3)_2$ ≥ 99% purity), purchased from Merck KGaA, Germany
2) *m*-Xylene ($C_6H_4(CH_3)_2$ ≥ 99% purity), purchased from Merck KGaA, Germany
3) *n*-Nonane ($CH_3(CH_2)_7CH_3$ ≥ 99% purity), purchased from Fluka, Sigma-Aldrich Co., Inc., Singapore
4) Toluene ($C_6H_5CH_3$ ≥ 99% purity), purchased from Carlo Erba Reagents, Italy
5) Acetone ($(CH_3)_2CO$ analytical grade), purchased from Lab Scan Analytical Sciences, Thailand

Gases:

1) Helium (He 99.99% purity), purchased from Praxair (Thailand) Co., Ltd.
2) Air (Air 99.99% purity), purchased from Praxair (Thailand) Co., Ltd.
3) Hydrogen (H_2 99.99% purity), purchased from Praxair (Thailand) Co., Ltd.
4) Nitrogen (N_2 99.99% purity), purchased from Praxair (Thailand) Co., Ltd.

3.2 Equipment

1) Headspace sampler: Model G1888, Agilent Technologies
2) Gas chromatography system: Model 6890N, Agilent Technologies
3) Column: Model Stabilwax, length 30 m, internal diameter 0.53 mm, Restek

4) Hot air oven: Model UC 30, Memmert GmbH and Co. KG., Western Germany
5) Laboratory chamber furnaces: Model CWF 1100, Carbolite, United Kingdom
6) 4-Digit precision weighting balance: Model AG 204, Mettler Toledo, Switzerland

3.3 Methodology

3.3.1 Adsorbent Preparation

Commercial zeolites were calcined to remove water, initially at 50 °C then heated at approximately 5 °C/min to 350 °C and left at this temperature for 3 hr. The water content of the zeolites was controlled at around 5 wt%. In order to determine the water content, a small amount of calcined zeolite was weighed and then calcined at 900 °C for 3 hr where water was completely eliminated and zeolite decomposed. Then, the zeolite was weighed again. The weight loss was calculated in weight percent and indicated as water content in zeolite.

3.3.2 Sample Preparation

The sample consisted of *p*-xylene, *m*-xylene, nonane, and toluene. The concentration of *p*-xylene and *m*-xylene was varied in the range of 1.25-20 wt%. The sample composition is shown in Table 3.1.

Table 3.1 Sample preparation

Sample	Concentration of *p*-xylene (wt%)	*p*-xylene (g)	*m*-xylene (g)	Toluene (g)	Nonane (g)
1	1.25	0.36	0.36	28.08	1.44
2	2.5	0.72	0.72	27.36	1.44
3	5	1.44	1.44	25.92	1.44
4	10	2.88	2.88	23.04	1.44
5	20	5.76	5.76	17.28	1.44

3.3.3 Headspace Gas Chromatography

One gram of liquid mixtures was added into a vial containing 0.5 g adsorbent. The vial was then left for 2 hr at a required temperature. The vapor phase was auto-injected to the gas chromatography system, which was connected to the headspace sampler, and its composition was determined. The headspace sampler condition is shown in Table 3.2. The GC condition for the analysis is shown in Table 3.3.

Table 3.2 Headspace sampler condition

Setting	**Condition**
Vial volume	10 mL
Vial equilibrium time	120 min
Vial temperature	40/60/80/100/120 °C
Loop temperature	50/70/90/110/130 °C
Transfer line temperature	60/80/100/120/140 °C
Carrier gas pressure	9.3 psig
Vial pressure	6.0 psig
Pressurization time	0.20 min
Loop fill time	0.16 min
Loop equilibrium time	0.01 min
Inject time	0.50 min
Shake	High
GC cycle time	30 min

The vapor phase composition obtained from experiments was used to calculate the composition of liquid phase by vapor-liquid equilibrium relationship. Then, the adsorbed phase concentration can be determined by mass balance (Torres *et al.*, 2001). Raoult's Law was used to calculate liquid phase mole fractions from vapor phase mole fractions obtained experimentally as shown in the following equation.

$$\frac{x_A}{x_B} = \frac{p_B}{p_A} \cdot \frac{y_A}{y_B} \quad , \tag{3.1}$$

where x and y represent liquid mole fraction and vapor mol fraction, respectively. P is vapor pressure.

Table 3.3 GC condition for the analysis

Setting	**Condition**
Injection temperature	220 °C
Oven temperature	60 °C for 1 min
	60-92 °C at 4 °C/min
	92 °C for 4.5 min
	92-220 °C at 20 °C/min
Detector temperature	270 °C
Interface temperature	200 °C
Carrier gas	Helium 99.99% purity