Chapter 3

Adsorption Experiments

Corncob has been selected as a material for carbon adsorbent preparation. It has been carbonized and activated with selected acid and salt solutions in a single step. The solutions used are 50 wt% sulfuric acid solution [27], 20-50 wt% phosphoric acid solution [28] and 60 wt% zinc chloride solution [29]. Characteristics of these carbon adsorbents such as moisture content, specific surface area etc., have been examined. Adsorption characteristics for some aromtic vapors on each carbon adsorbent have been determined by chromatographic method at temperature about 200°C

3.1 Carbon Adsorbent Preparation

Activation of corncob with each solution must be carried out under appropriate conditions, such as temperature. Procedure of preparation with each solution is described below:

3.1.1 Preparation with Sulfuric Acid

1. Adding 50 wt% sulfuric acid solution to corncob (10-20 mesh) in ratio 1:1, based on the dry weight of corncob
2. Dry the mixture at 110°C
3. Heating the powder residue at 250°C for 1 1/2 hours

4. The dry powder is washed with hot water to remove any unreacted acid and soluble salts
5. Dry products at 110°C for 3 hours
6. Repeat the preparation procedure except changing heating the powder residue for step increase 50°C from 150°C to 350°C

3.1.2 Preparation with Phosphoric Acid

1. Impregnated 10-20 mesh corncob into 20 wt% phosphoric acid solution the volume ratio of corncob to acid of 1 : 2.5
2. Dry the mixture at 110°C for 1 hour
3. Put the powder residue into crucible
4. Crucibles are activated at 300°C for 3 hours
5. The cooled carbonization products were washed repeatedly with hot water
6. Dry products at 110°C for 3 hours
7. Repeat the preparation procedure by changing to 30, 40 and 50 wt% phosphoric acid solution

3.1.3 Preparation with Zinc chloride solution

1. Impregnated 60 wt% Zinc chloride solution into 10-20 mesh corncob (solid/acid = 1 : 4)
2. Dry the mixture to dryness at 110°C for 1 hour
3. Put the powder residue into crucible
4. Put powder contained crucible in the 600°C furnace and left them for 3 hours
5. The cooled products are washed repeatly with dilute HCl and hot water
6. Dry products at 110°C for 3 hours

3.2 Characteristics Examination

- Moisture (ASTM D 2867) [30].
- Iodine Number (ASTM D 4607) [31].
- BET specific surface area and average pore diameter by micrometrics instrument (ASAP 2000).
- Functional groups of carbon adsorbents by Fourier Transform Infrared Spectroscopy (FTIR).
- Pore structure at the external surface of carbon adsorbent by Scanning Electron Microscope (SEM).
- Element composition of carbon adsorbent by Electron Diffractron X-ray (EDX).

3.3 Adsorption Experiments

3.3.1 Determination of packed Bed Characteristic

Each carbon adsorbent (40-60 mesh) was packed in a stainless steel tube with 1/4 in. in diameter and with appropriate length of carbon bed in order to obtain suitable chromatograms for analysis with method of moment. The bed porosity was estimated using Blake-Kozeny (Equation 3.1). The summarized bed characteristics of packed bed is illustrated in Table 3.1.

$$log\frac{\Delta P \rho D_p^2}{\mu L} = -logRe + log\left(\frac{150(1-\varepsilon)^2}{\varepsilon^3}\right) \qquad (3.1)$$

3.3.2 Adsorbate vapor preparation

Preparing adsorbate vapors in the volumetric flask is filled with 200-250 ml of liquid adsorbate(benzene/toluene/o-xylene). The flask is sealed and is placed in the room temperature for a few days to achieve vapor-liquid equilibrium. The

Table 3.1: Bed characteristics of packed column

type of column	mass of adsorbent(g)	L (cm)	ε
H_2SO_4-benzene	0.2806	5.3	0.2997
20%H_3PO_4-benzene	0.0432	1.0	0.4253
50%H_3PO_4-benzene	0.0228	0.5	0.4998
$ZnCl_2$-benzene	0.0185	0.5	0.379
H_2SO_4-toluene	0.2806	5.3	0.2997
20%H_3PO_4-toluene	0.0432	1.0	0.4253
50%H_3PO_4-toluene	0.0228	0.5	0.4998
$ZnCl_2$-toluene	0.0115	0.4	0.5174
H_2SO_4-o-xylene	0.2806	5.3	0.2997
20%H_3PO_4-o-xylene	0.0432	1.0	0.4253
50%H_3PO_4-o-xylene	0.0228	0.5	0.4998
$ZnCl_2$-o-xylene	0.2492	6.0	0.3029

Table 3.2: Physical parameter of benzene, toluene and o-xylene

	BENZENE	TOLUENE	O-XYLENE
Formula	C_6H_6	$C_6H_5(CH_3)$	$C_6H_4(CH_3)_2$
Molecular weight	78.11	92.14	106.2
Boiling point(°C)	80.1	110.6	144.4
Antoine equation constants	A = 6.90565	A = 6.95464	A = 6.99891
	B = 1211.033	B = 1344.800	B = 1474.679
	C = 220.79	C = 219.482	C = 213.686
Heat of condensation (kJ/mol, at n.b.p.)	30.76	33.5	36.8

mole fraction of the adsorbate can be determined from the vapor pressure of the adsorbate at the given temperature. The properties of benzene, toluene and o-xylene necessary for calculation purpose are list in Table 3.2

3.4 Experiment on Gas Chromatograph

Chromatographic condition:

- Gas chromatograph GC-8A Shimadzu
- Flame Ionized Detector (FID)

- Range of temperatures of 150-210°C
- Vary carrier gas 4-5 flows in a range of flows of 20- 100 ml/min
- (+) polarity