Chapter 3

Adsorption Dynamic Experiments

3.1 System of Adsorption Experiments

3.1.1 Choices of Adsorbents

With the availibility for adsorption of light hydrocarbon and domestic available, activated carbon is selected to be the adsorbent used in the present study. Three kind of activated carbons, i.e. YAO, HRO and PHO are supplied by Carbokarn Co,Ltd. All of these are coconutshell based activated carbon produced by steam activation process. They are availabled for gas or air purification. Specification of these carbon are represented in Table 3.1.

Table 3.1: The specification of YAO, HRO and PHO.

Specification	Activated Carbon		
	YAO	HRO	PHO
Apparent density (g/cm^3)	0.48 - 0.52	Min. 0.5	Min. 0.53
Approximated Surface Area (cm^2/g)	1.15×10^{-7}	1.10×10^{-7}	1.00×10^{-7}
Moisture content (%)	Max. 8	Max. 5	Max. 5
pH	9-10	9-10	9-10

A micromeritic BET model ASAP 2000 is used to determined the characteris-

tics of these activated carbon. The specific surface area is the important criteria for selection one of these carbon.

3.1.2 Choices of Adsorbate

The mixed C_2 produced from ethane crackers consist of ethane, ethylene and acetylene. The last is selected to be the adsorbate in the present study due to its commercial and domestic available. This gas is manufactured by Thai Industrial Gas Company. It is a colourless gas, garlic-like odor and flamable. Its physical properties are represented in Table 3.2 [23, 24].

Table 3.2: The physical properties of acetylene.

Physical properties	
Molecular weight	26.03
Density at 323 K (g/cm^3)	9.81×10^{-4}
Vicosity at 323 K (g/cm.s)	0.011
Boiling point (K)	189
Autoignition Temp.(K)	578
Critical temperature (K)	309.5
Critical pressure (atm)	61.6

3.1.3 Inert Diluent Gas

Helium of 99.995%V/V purity is used as the diluent gas for providing the desired feed concentration, and as a purge gas for cleaning the adsorption column between each experiment. This gas is produced from Thai Industrial Gas company. The physical proerties of helium at 323 K is represented in Table 3.3 [24].

Table 3.3: The physical properties of helium.

Physical properties.	
Molecular weight	4.00
Density at 323 K(g/cm^3)	1.78×10^{-4}
Vicosity at 323 K (g/cm.s)	0.019
Boiling point(K)	4.1
Critical temperature (K)	5.26
Critical pressure (atm)	2.26

3.2 Experimental Apparatus

The schematic flow diagram of the experimental set up is shown in Figure 3.1.

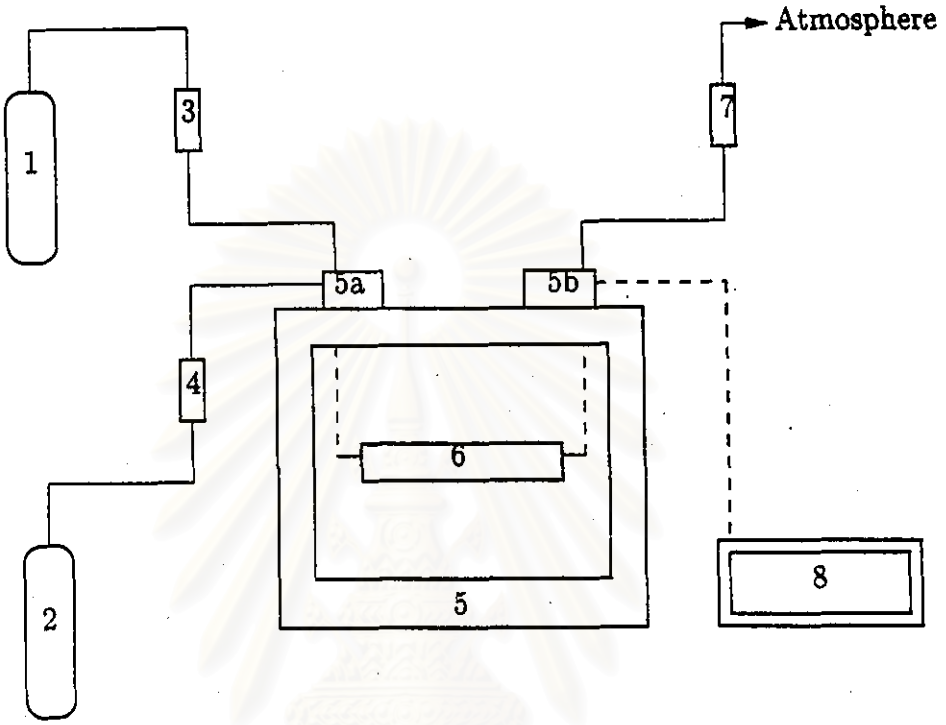
3.2.1 Gas Chromatograph

The main part of the experimental set up for the measurement of breakthrough curves is a Shimadzu 8A gas chromatograph. The outlet concentration of acetylene corresponding to the step inputs are detected by the thermal conductivity detector (TCD).

3.2.2 Column of Adsorbent

The adsorption columns are made of 0.46 cm in diameter stainless steel tube and are packed with activated carbon at various column lengths between 2 and 10 cm. The remaining sections at both end of each column were 1 cm each long and are filled with glass wool to prevent the carryover of the activated carbon particles.

The porosity of a packed column, ϵ, can be determined from the following Blake-Kozeny equation [22].



1 = Acetylene gas cylinder

2 = Helium gas cylinder

3 , 4 = Flowmeter with built-in needle valve

5 = Gas chromatography. 5a = Injection Port , 5b = Detector

6 = Adsorption column

7 = Bubble flow meter

8 = Integrator

Figure 3.1: The schematic flow diagram of the experimental setup.

$$log\left(\frac{\Delta P}{\rho_b v^2}\right)\left(\frac{D_p}{L}\right) = -log\left(\frac{D_p v \rho_b}{\mu}\right) + log\left(\frac{150(1-\epsilon)^2}{\epsilon^3}\right) \quad (3.1)$$

where ΔP is pressure drop across the bed, ρ_b is bulk density, v is superficial velocity, D_p is particle diameter and L is bed length.

The bed porosity can then be obtained from the intercepts of the plots between $log\left(\frac{\Delta P}{\rho_b v^2}\right)\left(\frac{D_p}{L}\right)$ against $log\left(\frac{D_p v \rho_b}{\mu}\right)$.

In addition, the pellet density (ρ_s) of the activated carbon can be estimated from the corresponding bed porosity by the use of the following correlation.

$$\rho_s = \frac{\rho_b}{1-\epsilon} \quad (3.2)$$

The characteristics of packed columns are summarized in Table 3.4

Table 3.4: The characteristics of YAO packed column.

Mesh size	Bed Length (cm)	Wieght (g)	Bulk density (g/cm^3)	Bed porosity
60 - 80	2	0.174	0.529	0.490
	4	0.333	0.507	0.510
	6	0.512	0.520	0.498
	8	0.690	0.525	0.493
	10	0.886	0.540	0.478
40 - 60	6	0.494	0.501	0.516
30 - 40	6	0.492	0.499	0.518

The adsorption column is installed in the gas chromatograph in order to maintain a uniform column temperature. The column inlet is connected to the injection port and the outlet is connected to the detector.

3.2.3 Integrator

The integrator used for monitoring and calculating the output signal from the detector of a gas chromatograph is a chromatopac C-R3A from Shimadzu Corporation. The outlet concentration of acetylene are printed out continuously on the chart paper.

3.3 Breakthrough Curve Measurement

The experimental procedure involve heating the adsorption column to 323 K while helium is flowed continuously from the gas cylinder through the column. The velocity of helium is maintained constant at a desired rate by adjusting the needle valve built-in the flowmeter and periodically measure with a soap bubble flowmeter at the outlet of gas chromatograph. Similarly, acetylene is then flowed continuously from the gas cylinder to the injection port of the gas chromatograph to mix with helium stream. Therefore, acetylene-helium mixture are entranced to the adsorption column. The output signal from the detector are then monitored and calculated to give the breakthrough curve by chromatopac integrator. After steady state condition is obtained. Acetylene stream is switched off and the remaining of acetylene in the column are then swept out by helium stream.

3.4 Experimental Procedure

Prior to an experiment, the adsorbent is pretreated by passing helium through the packed column for 3 hours at 393 K in order to purge all adsorbed species. The velocity of helium is maintained constant at 10 cm/s . Also between each experiment, the adsorption column is purged under this condition for a suffiiently long time allowed for the thermal conductivity detector baseline to stabilize and the column temperature to become uniform.

3.4.1 Adsorption Isotherm of Acetylene

The adsorption isotherms for acetylene on activated carbon are measured at 323 K by a dynamic equilibrium technique. The adsorption column used in this experiment is 8 cm long bed of 60-80 mesh activated carbon. The bed pressure is maintained constant by setting the pressure regulator on helium and acetylene at the values of 0.99 atm. The total velocity are maintained at 15 cm/s. The measurement procedure involve varying the concentration of acetylene in the combined stream by adjusting the velocity of each stream as summarized in Table 3.5.

The experiments are performed twiced in order to verify the reproducibility of the results.

In order to investigated the effects of bed lenths, superficial velocity, feed concentration and the size of an adsorbent particles on the concentration profile, the experiments are devided into 3 section.

Table 3.5: The velocity of acetylene and helium used in the experiment for the measurment of the adsorption isotherm.

%V/V of acetylene	Superficial Velocity (cm/s)	
	Acetylene	Helium
13	1.95	13.05
20	3.00	12.00
27	4.05	10.95
33	4.95	10.05
40	6.00	9.00
47	7.05	7.95
53	7.95	7.05
60	9.00	6.00
67	10.05	4.95
73	10.95	4.05
80	12.00	3.00
87	13.05	1.95

3.4.2 Effects of Bed Length and Feed Concentration

The experimental breakthrough curve of 20, 33 and 60%V/V acetylene in feed are conduct at the total velocity of 15 cm/s. The column used in this experimental section are 2, 4, 6, 8, and 10 cm long beds of 60-80 mesh activated carbon. The velocity of acetylene and helium are varied to obtain the desired feed concentration and total velocity as illustrated in Table 3.6.

Table 3.6: The velocity of acetylene and helium corresponding to the total velocity 15 cm/s.

%V/V of acetylene	Superficial Velocity (cm/s)	
	Acetylene	Helium
20	3.00	12.00
33	4.95	10.05
60	9.00	6.00

3.4.3 Effects of Adsorbent Particle Size

The experimental breakthrough curve of 60-80, 40-60 and 30-40 mesh activated carbon column with 6 cm long are investigated. The total velocity is maintained constant at 15 cm/s. The concentration of acetylene in the combined stream is altered to be 20, 33 and 60%V/V.

3.4.4 Effects of Superficial Velocity

- **Effects of Superficial Velocity on the Shape of the Profile**

  The experimental breakthrough of 60%V/V acetylene in feed are conducted at various velocity. The column used in this experiment is 6 cm long bed of activated carbon. The velocity of each stream are varied in order to obtain the desire total velocity and feed concentration as represent in Table 3.7.

Table 3.7: The velocity of acetylene and helium corresponding to the total velocity 5, 10, 15, 20, 25 and 30 cm/s.

Total Velocity	Superficial velocity (cm/s)	
	Acetylene	Helium
5	3	2
10	6	4
15	9	6
20	12	8
25	15	10
30	18	12

- **Effects of Superficial Velocity on the Propagation of the Profile**

In this section, the experiment are conducted in the same manner as in the experiments for investing the influence of bed length and feed concentration on the concentration profile, except for the total velocity used, which is reduced from 150 cm/s to 5 cm/s. The velocity of each stream are also altered to obtain the desired operating condition as represent in Table 3.8.

Table 3.8: The velocity of acetylene and helium corresponding to the total velocity 5 cm/s.

%V/V of acetylene	Superficial Velocity (cm/s)	
	Acetylene	Helium
20	1.00	4.00
33	1.65	3.35
60	3.00	2.00

All experiment are performed twice in order to verify the reproducibilty of the results.