CHAPTER 3

EXPERIMENTAL

Two techniques may be used to determine liquid adsorption rates. Both techniques are presented in figure 3.1 with the corresponding concentration measurements that are obtained using such techniques and from which information on adsorption rates may be obtained.

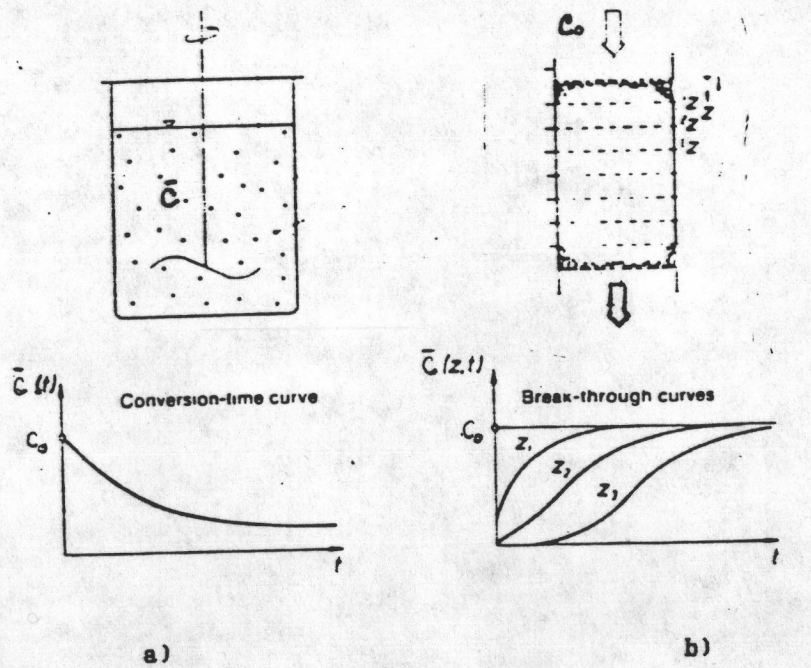

Figure 3.1 Experimental methods for measuring the adsorption rates in liquid systems, a) Batch experiments my be used to obtains adsorption isotherms and determine internal diffusion coefficient, b) continuous flow experiments where the outlet concentration of a solution with initial concentration C_o flowing through a column is measured as a function of time.

In this study experiments will follow the above techniques and details are explained as follows.

3.1 Apparatus and procedures.

The apparatus used in this work involves a system used to determine liquid adsorption isotherms , a liquid adsorption column and a gas chromatograph used to measure liquid concentrations.

3.1.1 The liquid adsorption isotherm determination system.

The equipments used in this system were as follows : a cooling bath (Grant model L20 , England) equipped with an accurate water temperature control system ; a glassware shaker (GFL German) and a set of 10 ml glass cylindrical vials equipped with screw caps which were further sealed with a piece of aluminum foil between glass and screw caps. The vials containing solution and adsorbent were immersed in a circulating cold water bath placed in the shaker. Usually about 2 g of coconut shell based activated carbon of 25x40 mesh size was added to the glass vials with 5 ml solution of binary mixtures of n-hexane with cyclohexane at 15 C ($\pm$ 1 C) and shaken for 10-12 hours. The initial liquid concentration as well as the final equilibrium liquid concentration was then determined by gas chromatography. The isotherms were expressed by a surface excess , n_t^* and also in terms of a Freundlich equation.

In order to measure intraparticle diffusion coefficients the experimental procedure is the same except that the

concentration of the mixture is measure every hour until equilibrium is reached.

For the case of liquid phase adsorption it is of course not possible to directly measure the amount of liquid adsorbed.

3.1.2 The adsorption column.

The dual pyrex tube adsorption column used is shown in figure 3.2. 15 C water from the cold water system used to maintain a constant column temperature was circulated in the annulus . The column used was 12 mm. I.D. and 100 cm.in length.The column was filled with 20 g of activated carbon soaked in cyclohexane. The top and bottom of the activated carbon layer were fitted with glass wool plugs. Several experimental conditions were then explored for the n-hexane-cyclohexane solution separation in the column at 15 C. Different liquid velocities (1.53-3.70 cm/min) were chosen as variables. Concentrations of the binary solutions were varied from 139 to 333 mg/ml .The concentrationof the exiting solution was then determined by gas chromatography as a function of time.

The velocity (or superficial velocity) of the liquid through the column was regulated by setting the volume at the exit of the column to the desired value and maintaining a liquid level fairly constant at the top of the column.

Small samples of hydrocarbons were collected at the bottom of the column every 1 min and the concentration determined by gas chromatography. Several binary mixture concentration and velocities were then used using the same procedures as above.

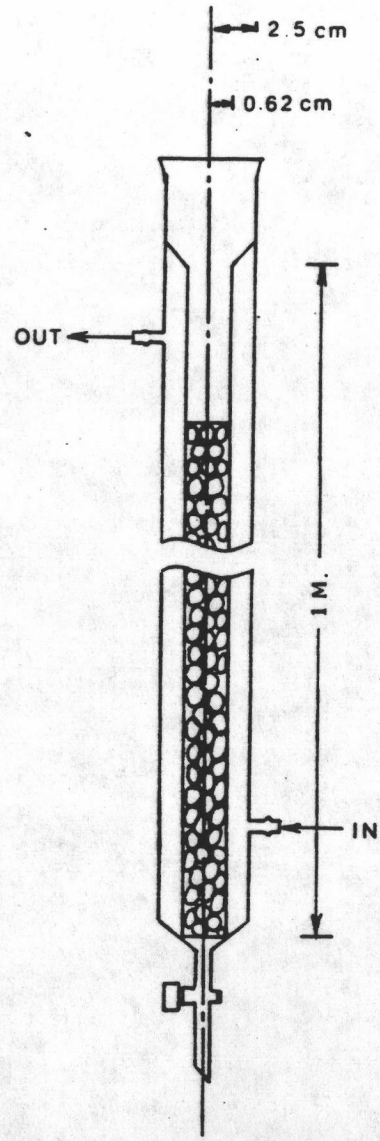

Figure 3.2 Adsorption column

3.1.3 Concentration analysis system

A Varian 3400 Gas Chromatograph with a thermal conductivity detector was used to determine the liquid concentrations. The operating conditions of the gas chromatograph are presented as follows :

Column	:	SS 1/8 in. x 20 ft. packed with 30% DC-200/50 Chromosorb P AW 60/80 mesh.
Carrier gas	:	Argon
Flow rate	:	30 ml /min
Injector temperature	:	100 °C
Column temperature	:	120 °C
Detector temperature	:	120 °C
Filament current	:	150 mA

Samples were taken with microsyringes and manually inject in the gas chromatograph were made. The concentrations were determined from the calibration curve using the height of the chromatographic peaks.

3.2 Materials and chemicals

1. The adsorbent used in this study is a coconut shell granular coconut based activated carbon manufactured by UDP.Co., Ltd. Thailand, with properties shown in table III.

Table III

Properties of coconut shell based activated carbon .

(UDP.Chemical Co.,Ltd.)

Mesh size	25-40	mesh
BET Surface area	1200-1400	cm^2/g
CTC	66	%
Bulk density	0.4430	g/cm^3
Iodine No.	1235.1354	mg/g
adsorption volume (n-hexane)	0.852	cm^3/g
particle diameter	0.110	cm
pH	9.8	
bed void fraction	0.68	
density in bed	0.35	g/cm^3
Manufacturer	UDP.Chemical Co.,Ltd.	

2. The chemicals used in this study are n-hexane and cyclohexane, the properties of which are shown in table IV.

TABLE IV

Chemical properties of n-hexane and cyclohexane

	n-Hexane	Cyclohexane
Formular	C_6H_{14}	C_6H_{12}
Molecular weight	86.17	84.16
Viscosity(cP),20 C	0.33	1.00
Boiling point(C)	68.7	80.7
Density(g/cm^3)	0.662	0.777
Polarity index	0.01	0.04
Purity(%)	98	99
Manufacturer	Carlo Erba	Fluka