# Chapter 4

# ADSORPTION EXPERIMENTS

## 4.1  Determination of Adsorbent Characteristics.

Two types of volcanic rocks, i.e. expanded perlites and pumices, were used in adsorption experiments. Both types were seived to obtain 3 ranges of particle sizes, which were 40-60 mesh, 60-80 mesh, and 80-100 mesh size. Surface characteristic of each adsorbent was inspected with a scanning electron microscope (SEM). While an x-ray diffraction (XRD) was employed to examine the existence of known crystals, as well as types of the crystals. In addition, specific surface area of each adsorbent was measured by nitrogen adsorption at the normal boiling point of nitrogen (77K apprroximately). Subsequently, the pore size distribution of each adsorbent could be obtained from the equilibrium adsorption of nitrogen. Both measurements were carried out with Micromeritics model ASAP2000 (at Analysis Centre of Department of Chemical Engineering Faculty of Engineering, Chulalongkorn University). Besides physical characteristics mentioned above, chemical compositions of both volcanic rocks were also analysed by wet chemical analysis consisting of determination of humidity, loss of ignition and measurement
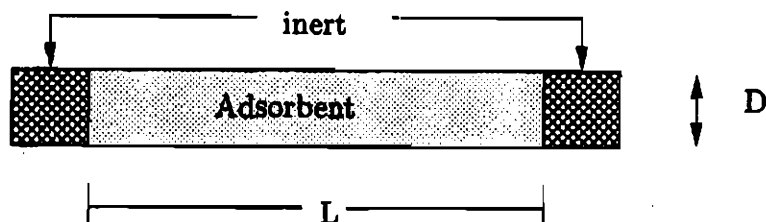
Figure 4.1: A typical packed column of an adsorbent for adsorption experiments of metal oxides contents with Atomic Absorption Spectroscopy (AAS).

## 4.2 Experiments on Gas Chromatography

### 4.2.1 Determination of Packed Bed Characteristics

Each adsorbent was packed in a stainless steel tube with 1/4 inch in diameter. Both ends of the adsorbent bed were packed with inert material, i.e. glass wool, in order to prevent the entainment of adsorbent, as illustrated in Figure 4.1. The bed length ($L$) of each adsorbent had to be adjusted in order to obtain proper shape of chromatograms. Then the amount of adsorbent for each packed bed had to be recorded. If the adsorbent density was known, the corresponding bed porosity would be able to be determined from the volume ratio of the adsorbent to the packed bed.

If the density of an adsorbent had not been known, it was able to be determined from the Blake-Kozeny equation. In that circumstance, an additional packed column with sufficient length of packed bed might be required for measurement of the variation of pressure drop across the bed with the flowrates. With an appropriate form of linear plot, such as an expression in Equation 4.2 in a log-log

scale, the bed porosity was able to be determined from the intercept.

$$\left(\frac{\Delta P}{L}\right)\left(\frac{d_p}{\rho \bar{v}_0^2}\right) = 150 \left(\frac{(1-\epsilon)^2}{\epsilon^3}\right)\left(\frac{1}{(d_p \bar{v}_0 \rho / \mu)}\right) \qquad (4.1)$$

$$\log\left(\frac{\Delta P}{L}\right)\left(\frac{d_p}{\rho \bar{v}_0^2}\right) = -\log\left(\frac{d_p \bar{v}_0 \rho}{\mu}\right) + \log\left(\frac{150(1-\epsilon)^2}{\epsilon^3}\right) \qquad (4.2)$$

Finally, the adsorbent density was also able to be determined from the relationship between the bed density and the bed porosity, as given in equation 4.3:

$$\rho_p = \frac{\rho_b}{1-\epsilon} \qquad (4.3)$$

Subsequently, the resulted adsorbent density was used to determine the bed porosity of the corresponding packed bed for adsorption experiments.

## 4.2.2 Adsorbate Preparation

Toluene and acetone vapors were prepared in a closed volumetric flask filled as illustrated in Figure 4.2 with a certain amount of liquid toluene and acetone, respectively. They were sealed carefully with paraffin film and had been kept at room temperature for a few days to obtain sufficient amount of the corresponding adsorbate vapors or to achieve vapor-liquid equilibrium.

## 4.2.3 Adsorption Experiments

All adsorption experiments were carried out on a gas chromatography model Perkin-Elmer 8700 with flame ionization detector as illustrated in Figure 4.3. A conventional analysis column, was replaced with another packed column containing a selected adsorbent. The adsorbents was purged with helium, which was
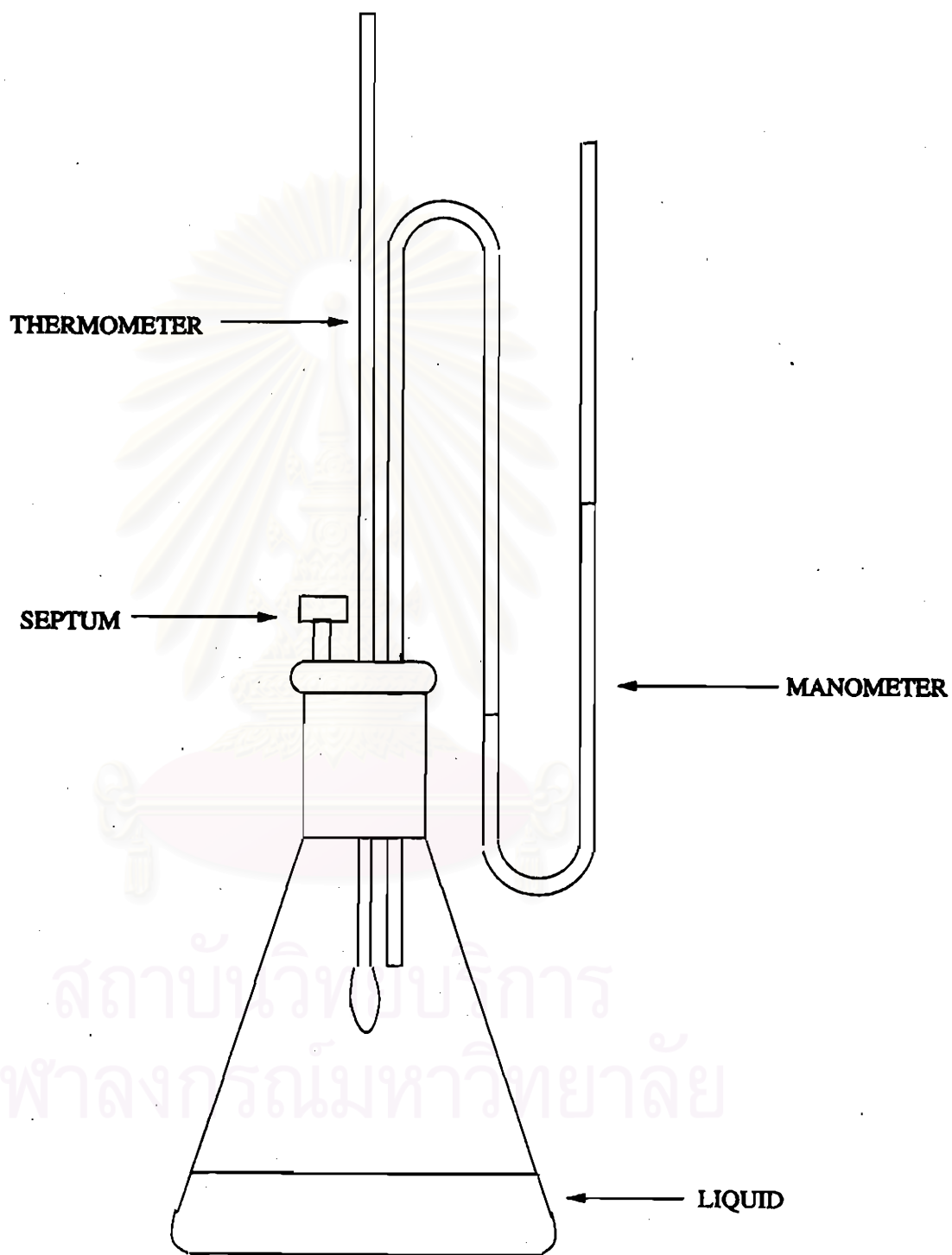
Figure 4.2: Closed volumetric flask used for adsorbate vapors preparation

used as a carrier gas, at temperature of 250°C and flowrate of 50 cm³/min for at least 2 hours to ensure that no other adsorbates remained on the adsorbent surface. The flowrate of carrier gas was measured carefully with a bubble flow meter before and after each experiment to verify the consistance of the flowrate. The adsorption experiments were carried out with pulse injection as a conventional chromatographic condition. In order to ensure the assumption of dilute solution, an injected volume of gas contained a selected adsorbate vapor is less than $100\mu$l. While the flowrate of carrier gas was varied in a range of 10-50 cm³/min at a given temperature. The adsorption experiments were repeated at other temperatures from 40°C upto 110°C with 10°C interval. For another type of adsorbates, the same experiments were repeated except the injected gas containing another type of selected adsorbates. Similarly, the adsorption experiments on another type of adsorbents were also carried out in the same procedure except the packed column containing the desired adsorbent that have changed in block of column as illustrated in Figure 4.3.

From an obtained chromatogram, in Figure 4.4, adsorption equilibrium constant, overall mass transfer coefficient were able to be determined with the first absolute moment and the second central moment of the chromatogram, respectively. In addition, the effects of temperature on the equilibrium constant and on the mass transfer coefficient were investigated.
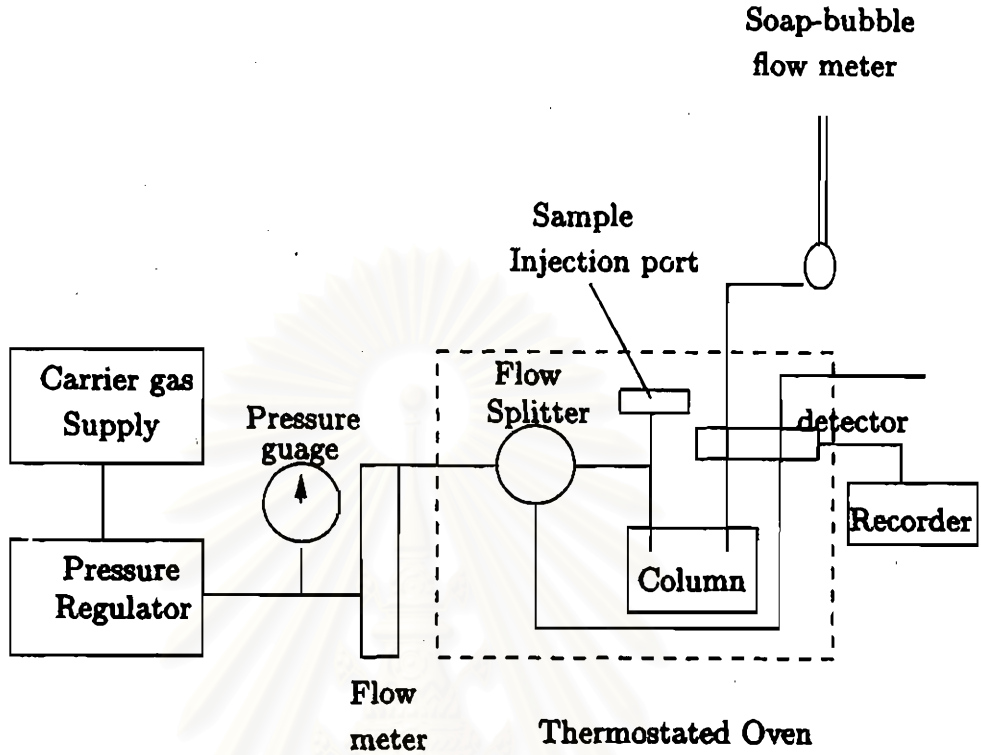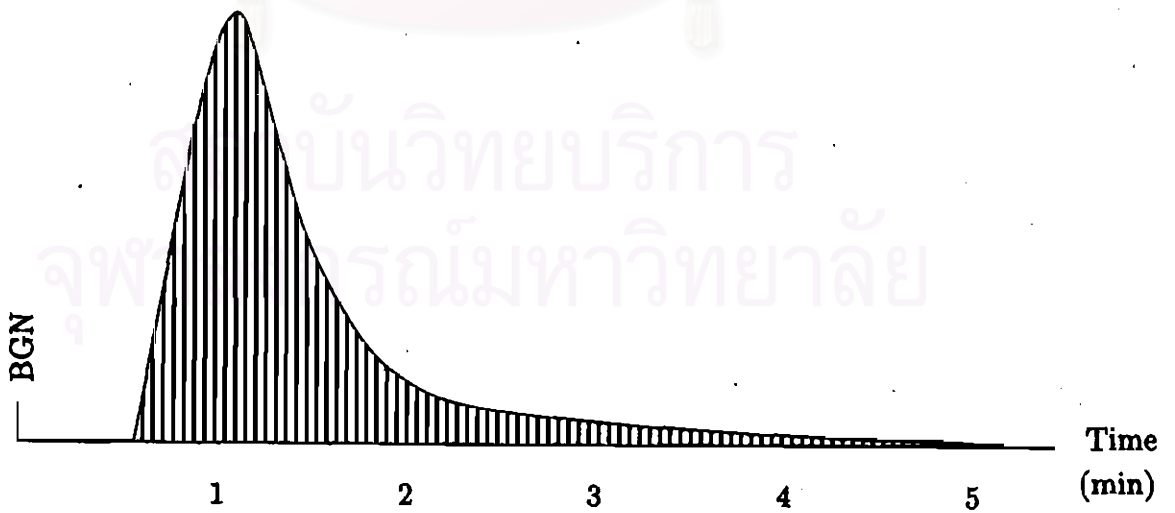
Figure 4.3: Schematic of Perkin Elmer gas chromatograph 8700



Figure 4.4: Chromatogram from the experiment result