CHAPTER V
ADSORBENT CHARACTERIZATION

After adsorbent preparation, the characterization of the dried algal sorbent was done in order to investigate properties of the adsorbent that could play an important role in the adsorption process. The adsorbent characterizations were carried out by Fourier Transform Infrared Spectrophotometer, Scanning Electron Microscopy, Laser Particle size Analyzer, Surface Area Analyzer, and Zeta Meter Electrophoresis. The micrographs of dried *C. lentillifera* were taken by Scanning Electron Microscopy (SEM), Jeol, JSM-5800LV, and are shown in Fig. 5.1

5.1 Chemical functional groups on the surface of the algal sorbent

FTIR spectrum was collected using Fourier Transform Infrared Spectrophotometer (FTIR), Perkin Elmer, Model 1760X (Fig. 5.2). The possible functional groups on algal surface are shown in Table 1 which illustrated that the most abundant function groups included carboxyl and amine. C-O and sulfonyl were second most abundant followed by carbonyl, S-O, and alkyl, respectively.

5.2 Particle size distribution

The ground algal sorbent was further investigated for the distribution of particle size using Laser Particle size Analyser, Malvern, Mastersizer-S long bed Ver 2.19. The particle size distribution curve (PSD curve) is shown in Fig. 5.3. The two separated peaks implied the shape of ground alga. The rod-shape (Fig. 5.4) was suggested because this kind of shape could give 2 different ranges (length and width).

5.3 BET specific surface area

The surface area, the pore specific volume, and the median pore width of the algal sorbent measured by N_2-BET technique are shown in Table 5.2 and the BET adsorption isotherms of both ground and unground algal sorbent are shown in Fig. 5.5. It was found from the median pore width that the pores on the surface of algal sorbent can be classified as mesopore (Dubinin, 1966; Gregg and Sing, 1982).

Among 6 types of isotherms (Fig. 5.6), the isotherms of ground and unground algal sorbents fitted best with type III isotherm. This class of isotherm is the characteristics of weak adsorbate-adsorbent interactions (Kiselev, 1968) and is most commonly associated with both non-porous and microporous adsorbents. The weak interactions between the adsorbate and the adsorbent lead to low uptakes at low relative pressures. However, once a molecule has become adsorbed at a primary adsorption site, the adsorbate-adsorbate interaction, which is much stronger, becomes the driving force of the adsorption process, resulting in accelerated uptakes at higher relative pressure.

5.4 Point of Zero Charge

The relationship between the zeta potential and and solution pH is illustrated in Fig. 5.7. The result indicated that the alga surface had negative charge at any pH higher than 2. Hence, the alga sorbent could be used as biosorbent for positive charged contaminants such as basic dyes above this pH range. This also implied that the PZC (Point of Zero Charge) of the alga was less than 2. So the alga exhibited negative charge on its surface at any pH higher than 2.

Table 5.1 Functional groups in dried *C. lentillifera*

Functional group	Standard Wavenumber (Skoog and Leary, 1992) (cm^{-1})	Wavenumber from the results (cm^{-1})	Relative quantity*
Hydroxyl; O-H	3250-3700	3200-3600	1
Carboxyl; COOH	2400-3300	3200-3600	1
Amine; NH2	3300-3500	3200-3600	1
C-O	1050-1300	1000-1200	2
Sulfonyl; S=O	1040-1200	1000-1200	2
Carbonyl; C=O	1670-1780	1600-1750	3
S-O	550-650	500	4
Alkyl; C-H	Carboxylic acid: 2500-3100 Alcohol: 3400-3640	2900-3000	5

* The quantity in the order from large to small (1 is the most abundant and 5 is the least.)

Table 5.2 Surface area, pore specific volume, and median pore width of algal sorbent

Sample size	Specific surface area ($m^2\ g^{-1}$)	Pore specific volume ($cm^3\ g^{-1}$)	Median pore width (Å)
Ground	4.69±0.28	0.130±0.0012	99.77±9.1
Unground	3.91±0.19	0.115±0.0008	65.66±2.1

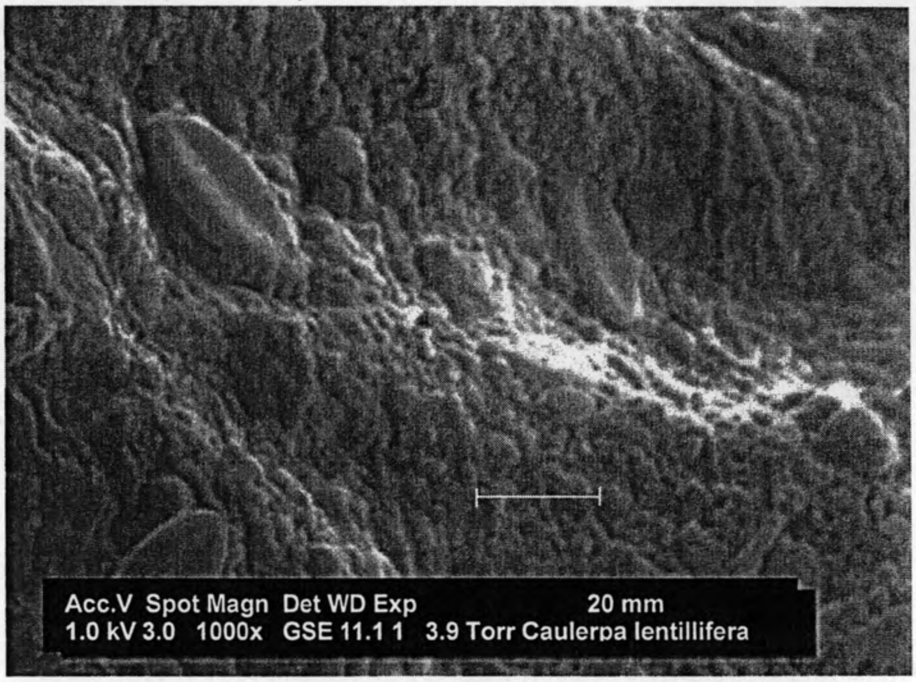

Fig. 5.1 SEM micrograph of the dried *C. lentillifera*

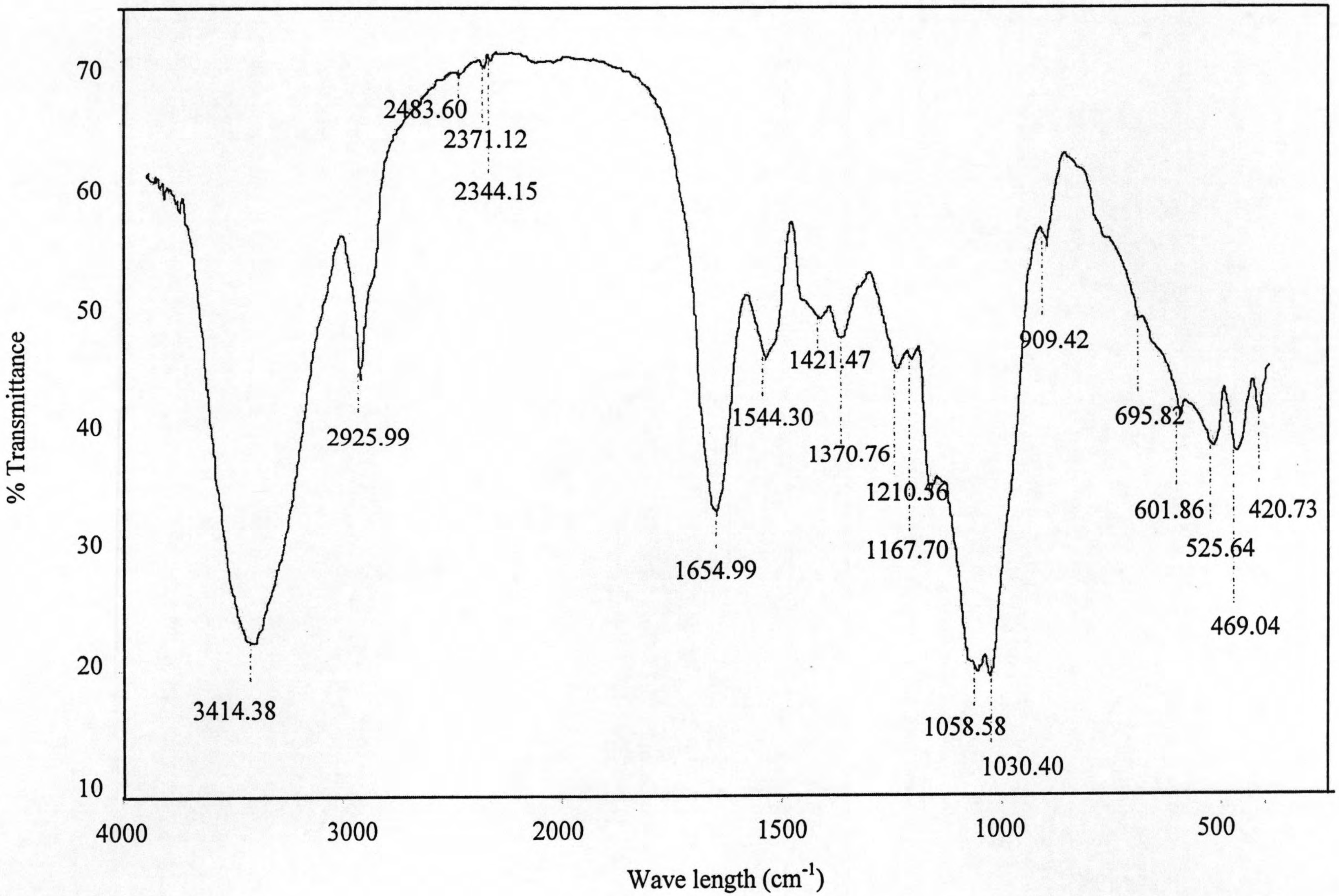

Fig. 5.2 FTIR spectrum of dried *C. lentillifera*

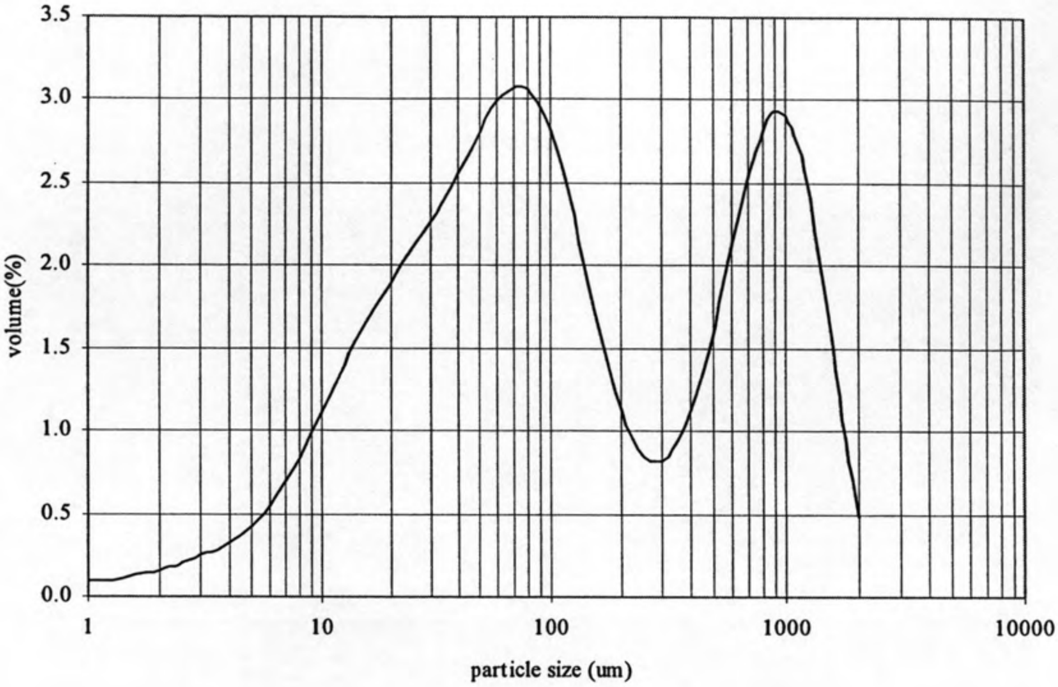

Fig. 5.3 PSD curve of dried *C. lentillifera*

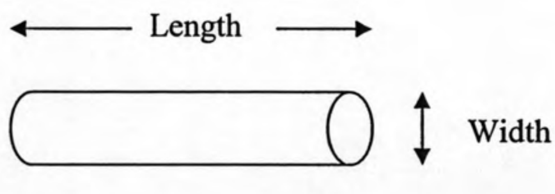

Fig. 5.4 Suggested shape of the ground algal sorbent

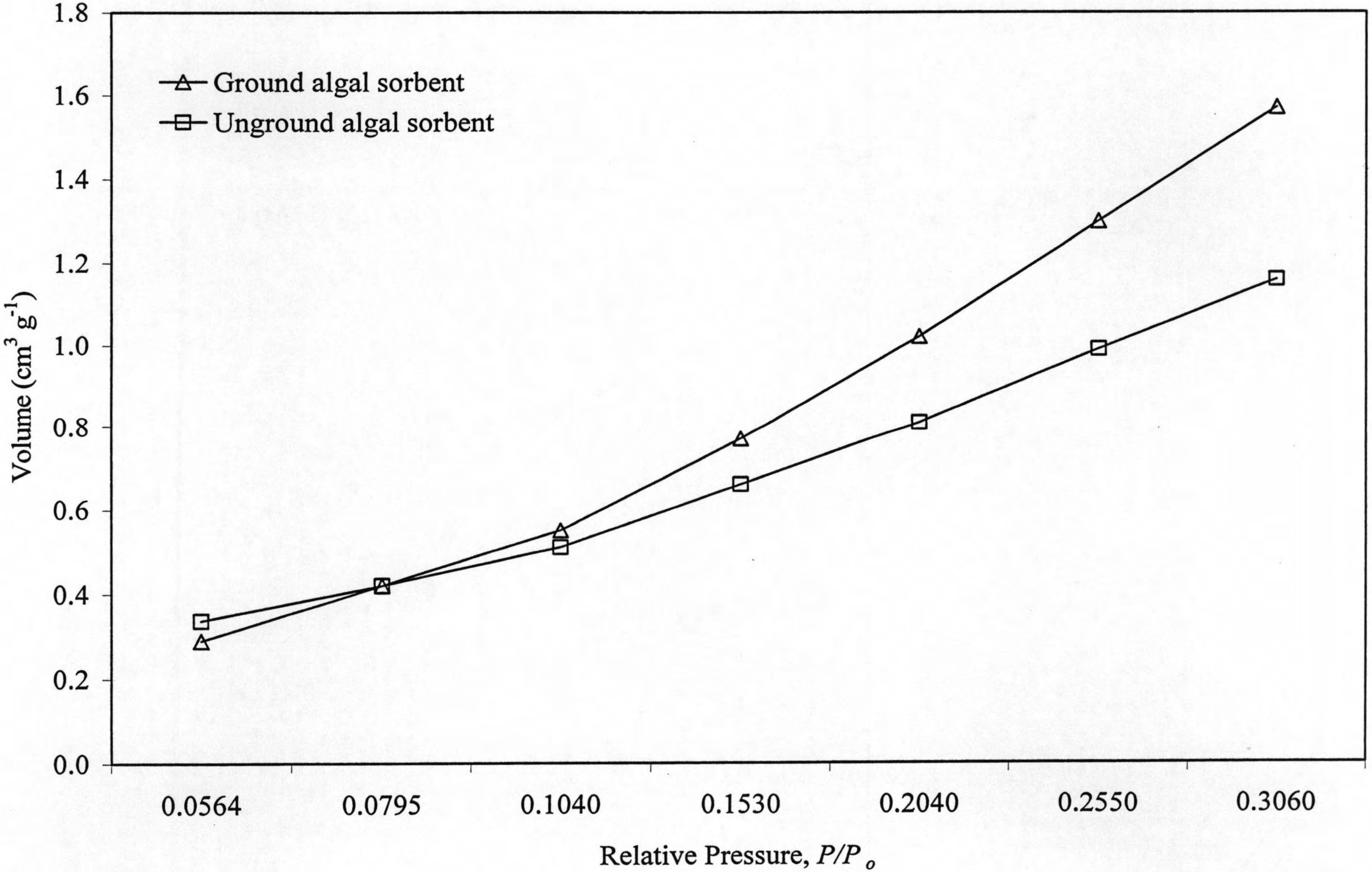

Fig. 5.5 BET adsorption isotherms of both ground and unground algal sorbent

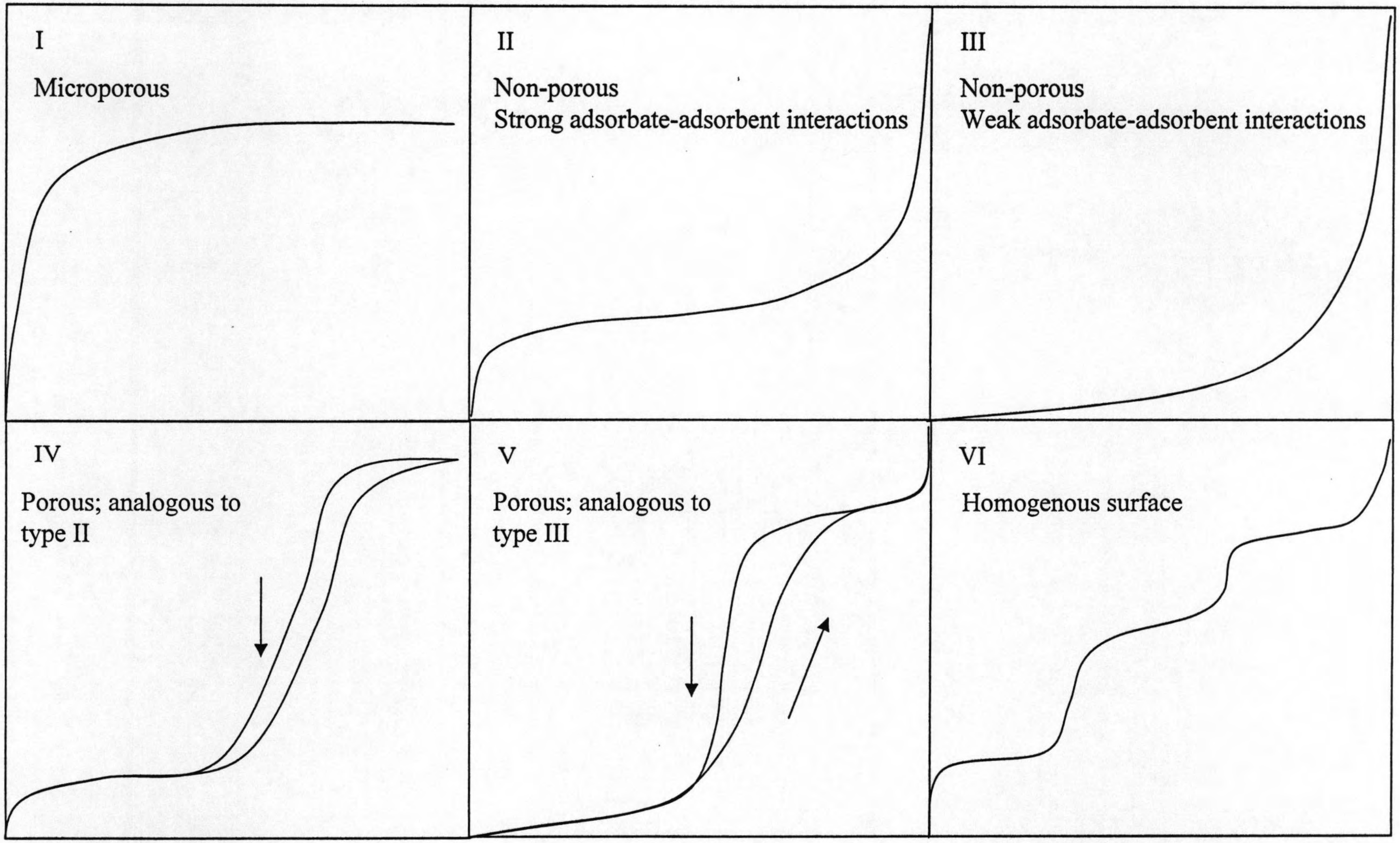

Fig. 5.6 Diagrammatic representation of isotherm classification (Brunauer et al., 1940)

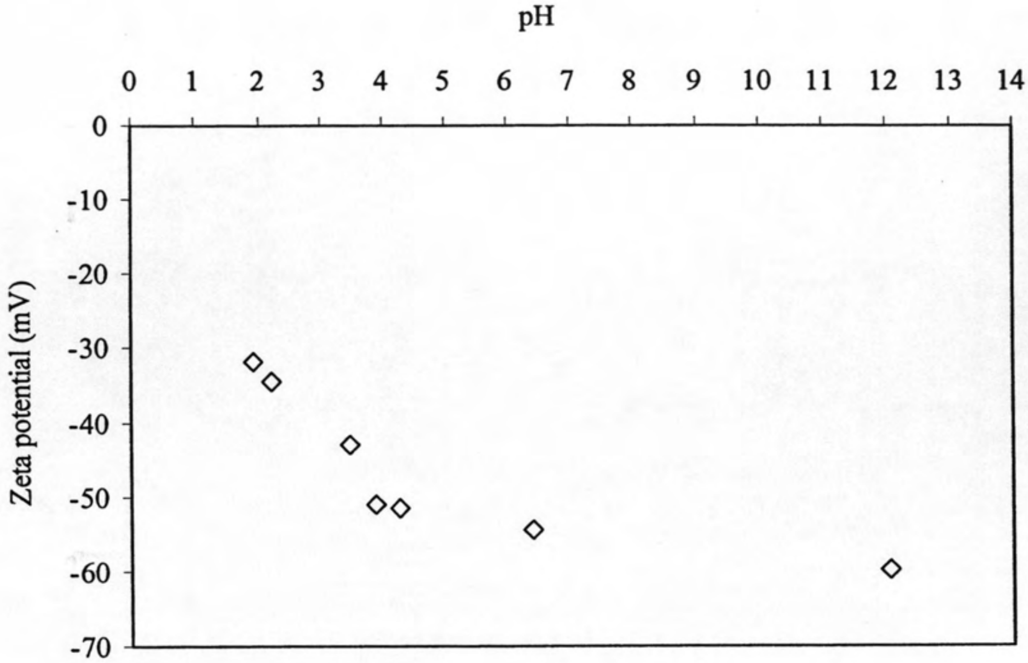

Fig. 5.7 Plots of pH versus zeta potential