CHAPTER IV
RESULTS AND DISCUSSION

The characteristic of MCM-41 was determined by nitrogen physisorption at 77 K with Quantachrome Autosorb-1. The specific surface area of MCM-41 at various pore size was not significantly different as shown in Table 4-1.

Table 4-1 Charaterization of MCM-41

Mean pore size (A^0)	Specific surface area (m^2/g)
21	982
26	865
36	1116

Normally, C_nTAB has the head group areas from 30-50 A^2 and in this study C_nTAB head area is assumed to be 40 $A.^2$

The hydrophobic chain length containing C atoms (L) = 1.265n+0.2 A^o (Clint., 1992) where n is the number of carbons in the alkyl chains. These data can be calculated as shown in the Table 4-2.

Table 4-2 Characterization of CTAB and CMC

CTAB	Diameter head group	Hydrophobic Length(L) (A^o)	Total length (A^o)	CMC(M) at liq-air 25^0C (Rosen., 1989)
C8	3.56	11.62	15.2	$1.4*10^{-1}$
C12	3.56	16.68	20.2	$1.6*10^{-2}$
C16	3.56	21.74	25.3	$9.2*10^{-4}$

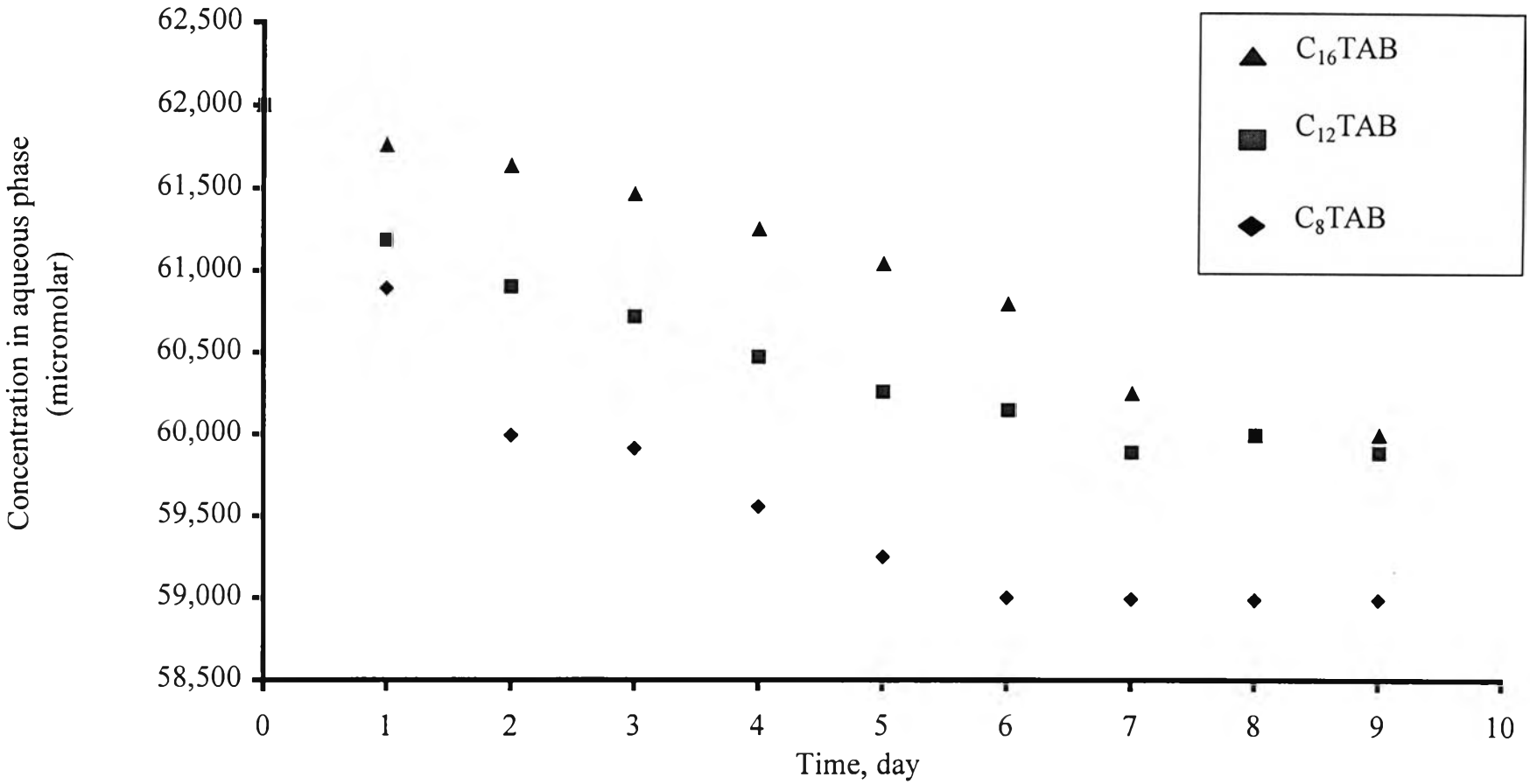

Figure 4-1 Dynamic adsorption of CTAB on MCM-41 pore size 21 A^0

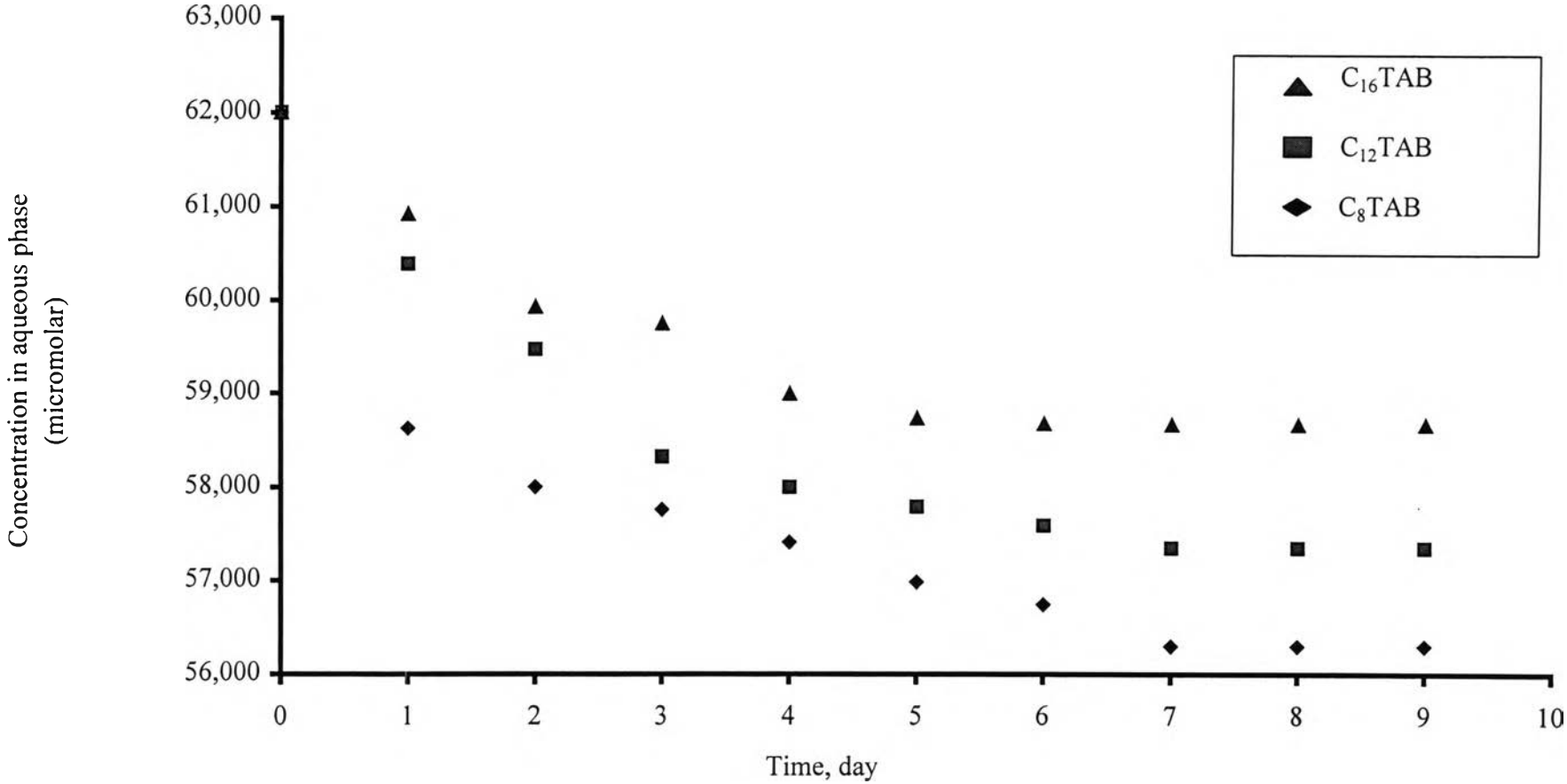

Figure 4-2 Dynamic adsorption of CTAB on MCM-41 pore size 26 A^0

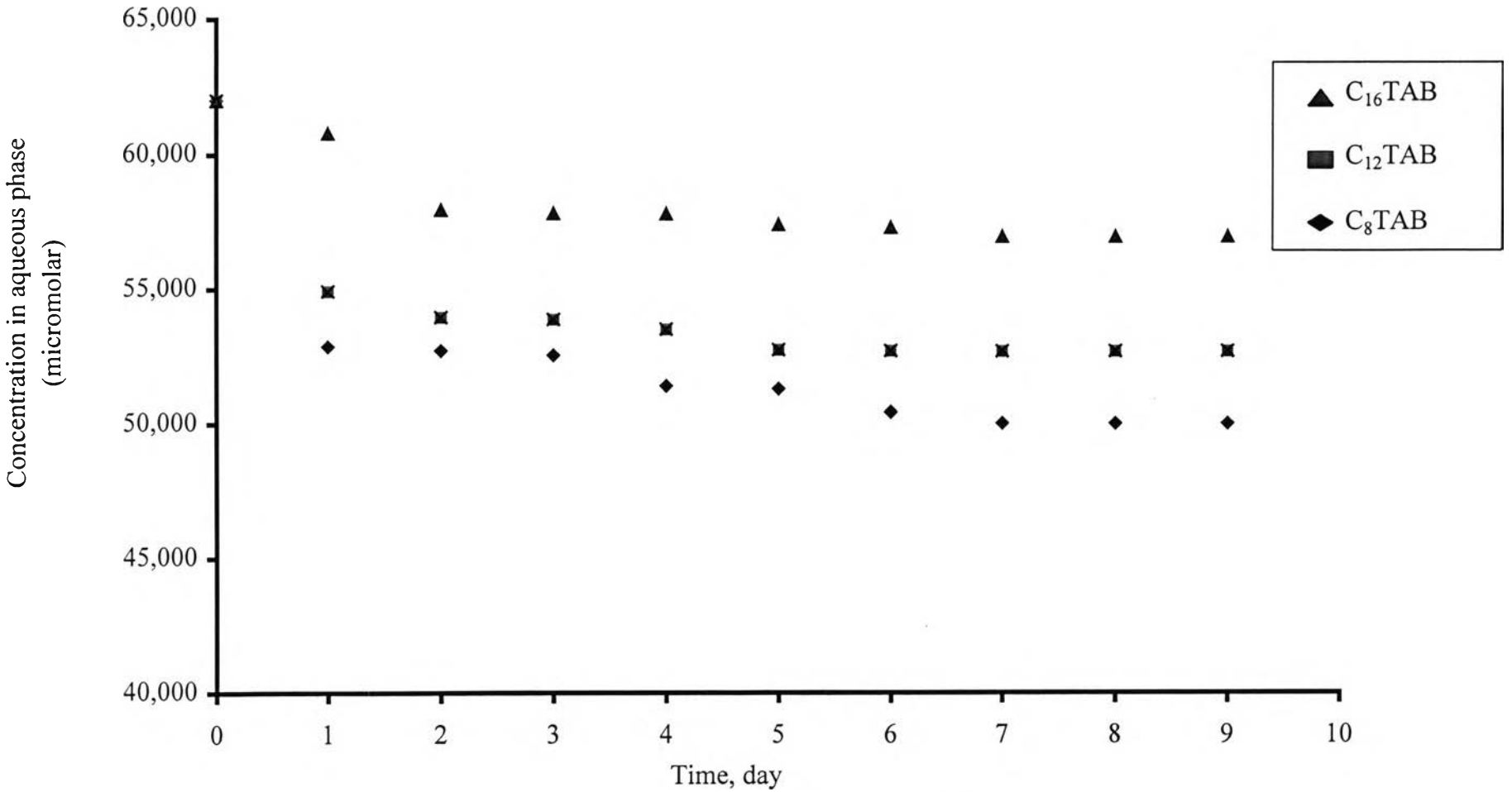

Figure 4-3 Dynamic adsorption of CTAB on MCM-41 pore size 36 A^0

4.1 Octyltrimethylammonium Bromide Adsorption Isotherm

The equilibrium time required for Octyltrimethylammonium bromide to be adsorbed on MCM-41 having different pore size 21 A^0, and 26 A^0 were found to be approximately 7 days as shown in Figure 4.1 to 4.3. These results are similar to the previous work which obtained the equilibrium time of 7 days (Srisaichua, 1997). The adsorption isotherm clearly show S-shaped isotherm at various pore size as shown in Figures 4-4 to 4-6. It can be seen that the adsorbed amount of surfactant increases with the surfactant concentration and levels off at a high surfactant concentration for each pore size. The slope of the isotherm in region II decreases somewhat with increasing pore size. The region II slopes were 0.061, 0.041, and 0.034 on the $21A^0$, $26A^0$, and 36 A^0 materials, respectively. This is surprising since the electrostatic effects would have the opposite effect. The critical admicelle concentration (cac) value of C8TAB on MCM-41 is slightly decreased when the pore size increased. The maximum adsorption of octyltrimethylammonium bromide on MCM-41 pore size $21A^0$, $26A^0$, and 36 A^0 was 370 μ mol/g, 650 μ mol/g, and 980 μ mol/g respectively. Although the specific surface area of the 21 A^0 material is 88% of the 36 A^0 material, the maximum adsorption of C8TAB is only 38% of the maximum C8TAB adsorption on the 36 A^0 material. These results strongly suggest that the pore size affects adsorption.

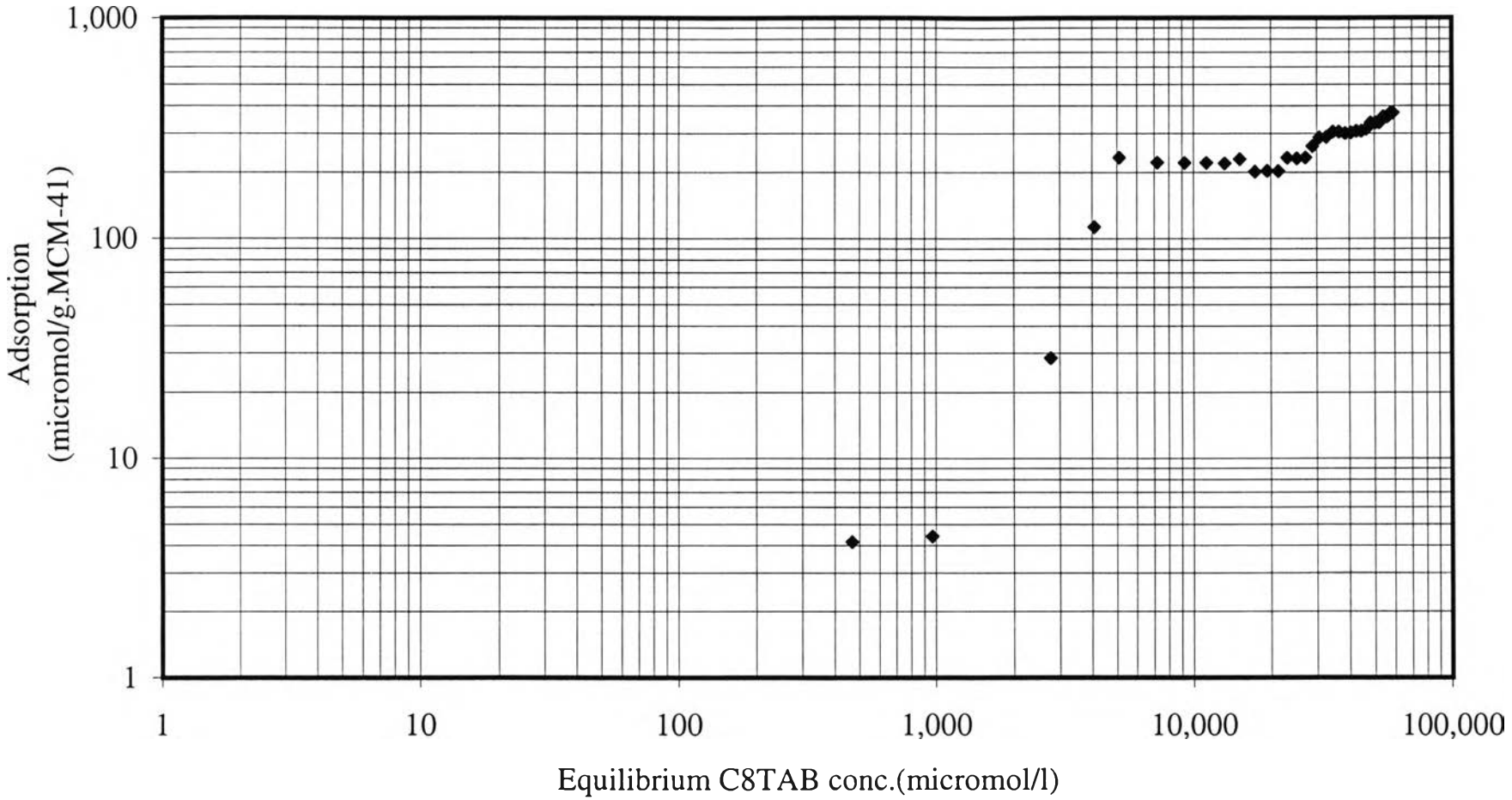

Figure 4-4 C8TAB Adsorption isotherm on MCM-41 pore size 21 A^0

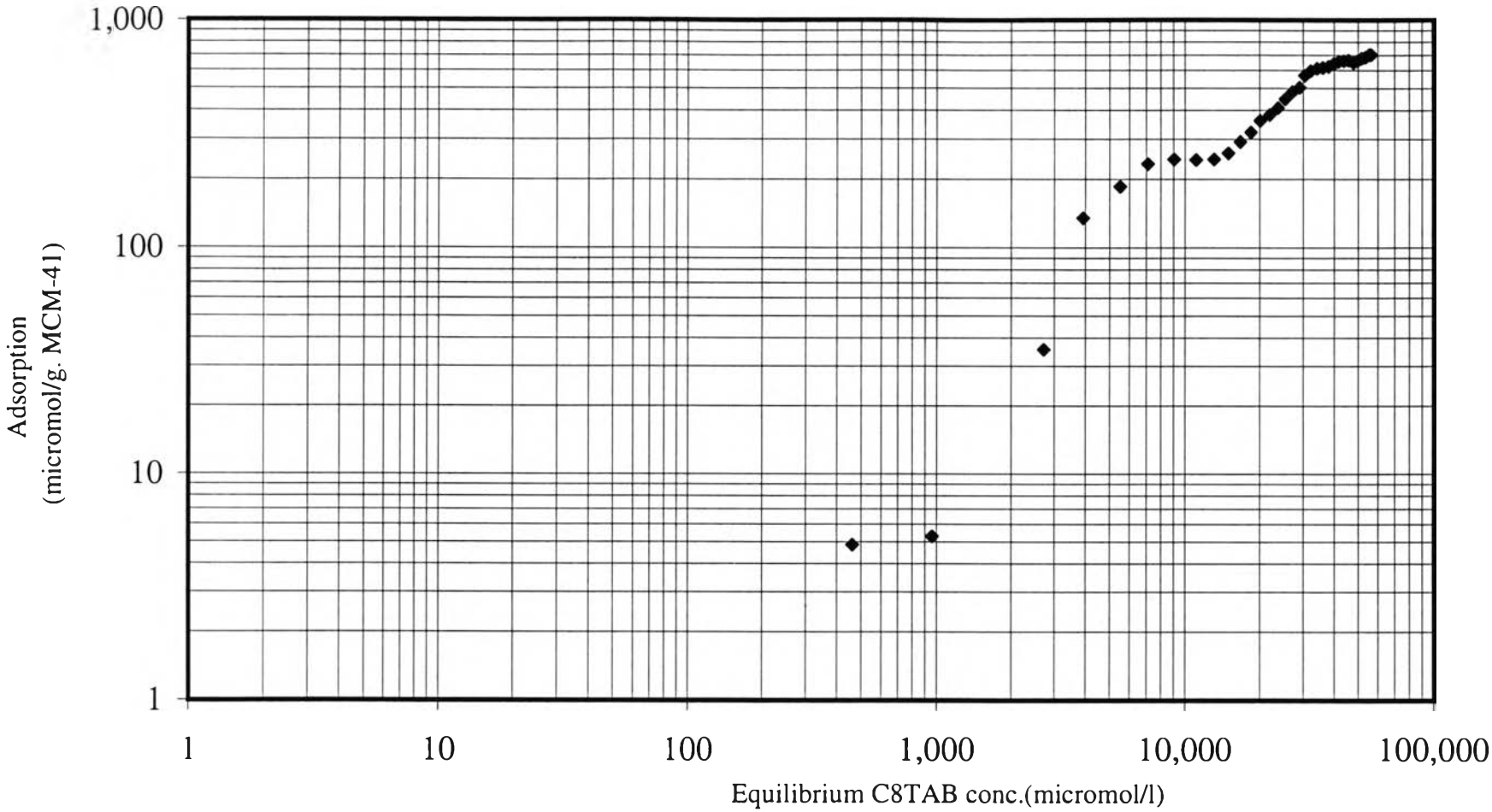

Figure 4-5 C8TAB Adsorption isotherm on MCM-41 pore size $26A^0$

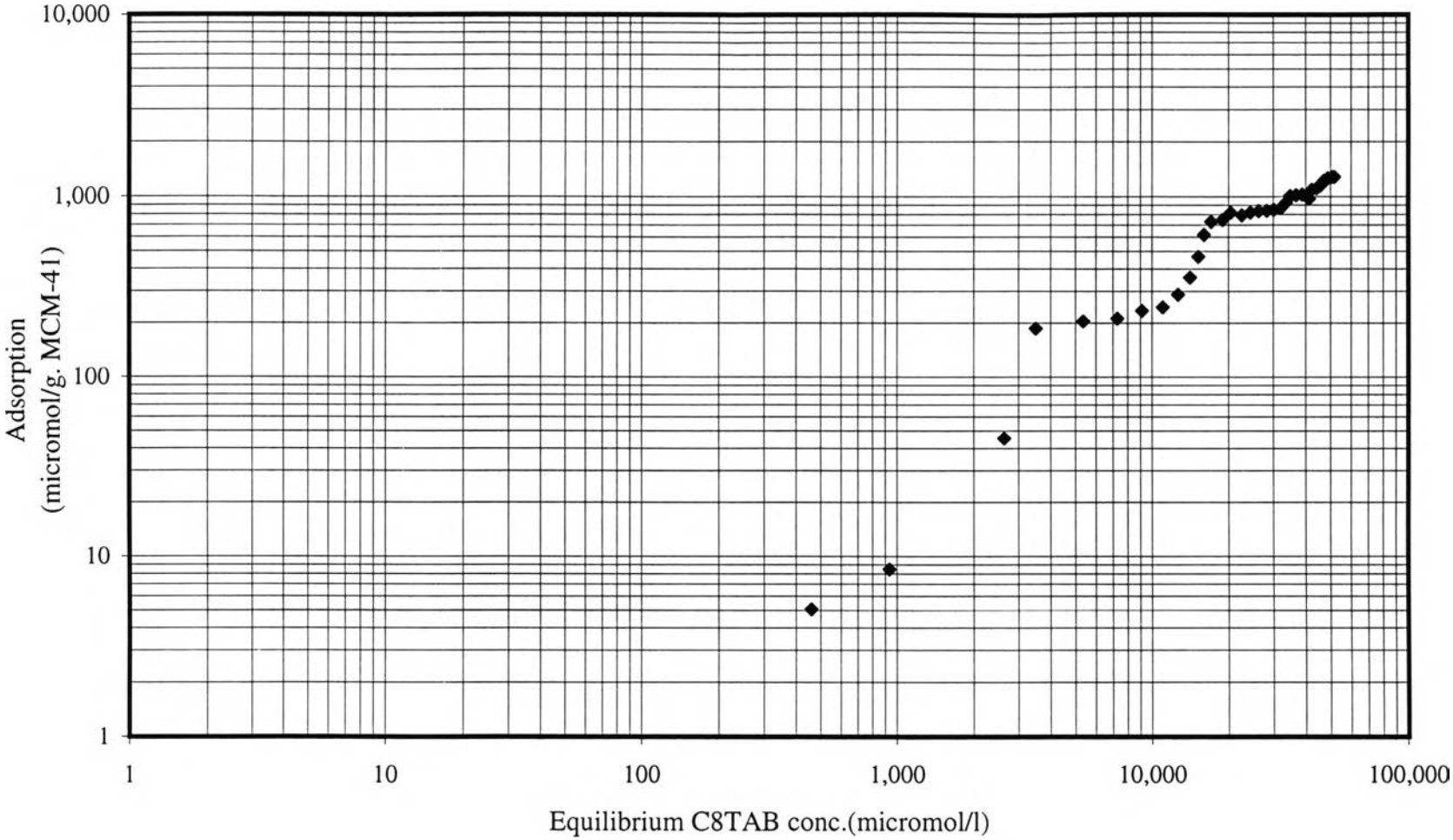

Figure 4-6 C8TAB Adsorption isotherm on MCM-41 pore size $36A^0$

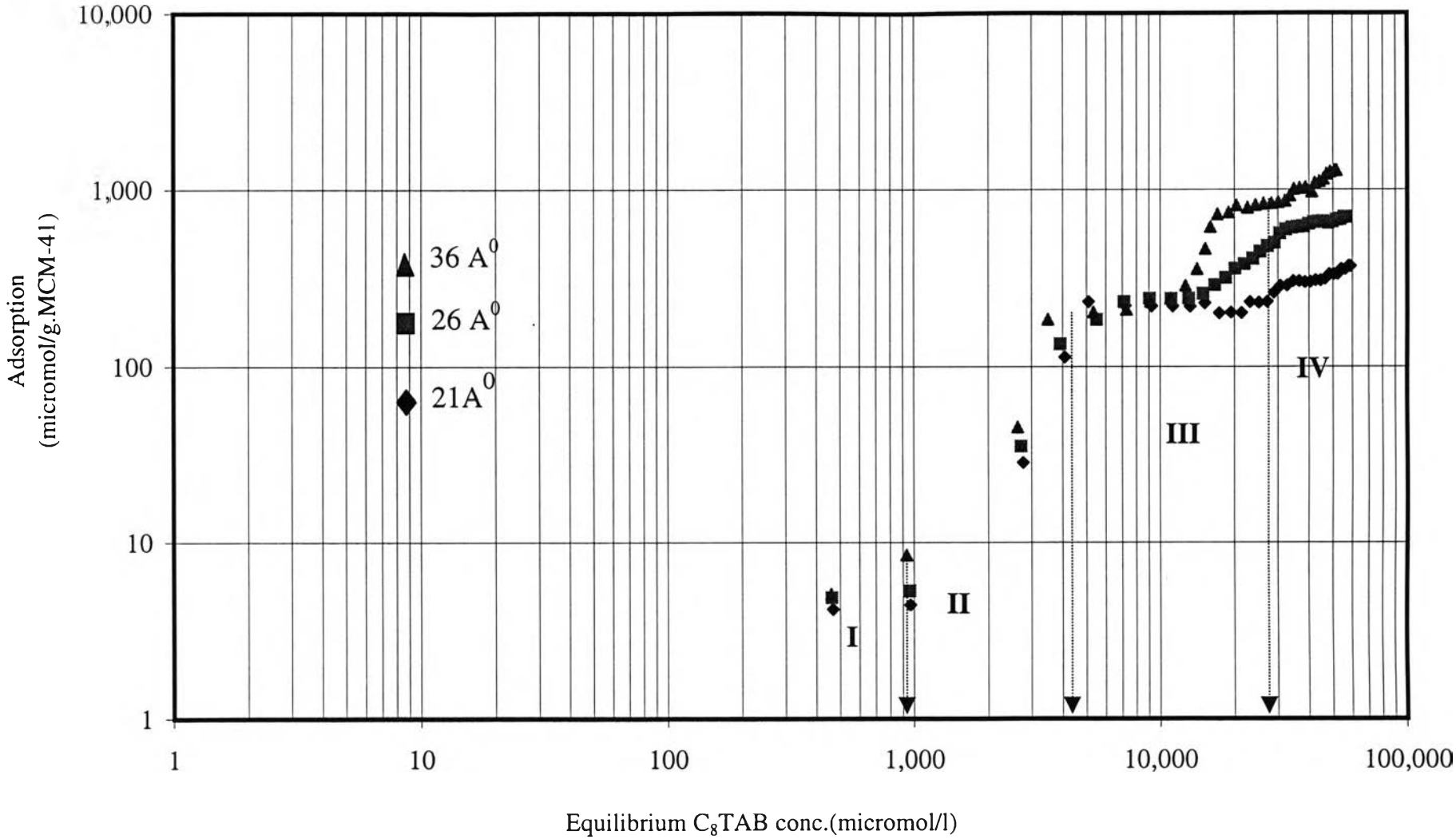

Figure 4-7 C_8TAB Adsorption isotherm at various pore size on MCM-41

4.2 Dodecyltrimethylammonium Bromide Adsorption Isotherm

Dodecyltrimethylammonuim bromide absorbed on MCM-41 pore size 21 A^0, 26 A^0, 36 A^0 took the equilibrium time about 7 days as shown in Figures 4-1 to 4-3. The adsorption dynamics of dodecyltrimethylammonuim bromide on MCM-41 were similar to C8TAB but the adsorption isotherms of pore size 36A^0 show obviously difference from the isotherm of pore size 21 and 26 A^o as shown in Figure 4-11. The amount of adsorbed surfactant at low concentration increased dramatically when the pore size MCM-41 increased. The isotherm slope in the region II on MCM-41 pore size 21 A^0, 26 A^0, 36 A^0 was approximately 0.03, 0.046, and 0.154 respectively. This characteristics illustrate that isotherm slope increased when the pore size MCM-41 increased in contrast to the results observed with C8TAB. The maximum adsorption of dodecyltrimethylammonuim bromide on MCM-41 pore size 21 A^0, 26 A^0, 36 A^0 was estimately 270 μ mol/g, 550 μ mol/g, and 930 μ mol/g respectively. The maximum adsorption of C12TAB is 29% of the maximum C12TAB adsorption on the36 A^0 material.

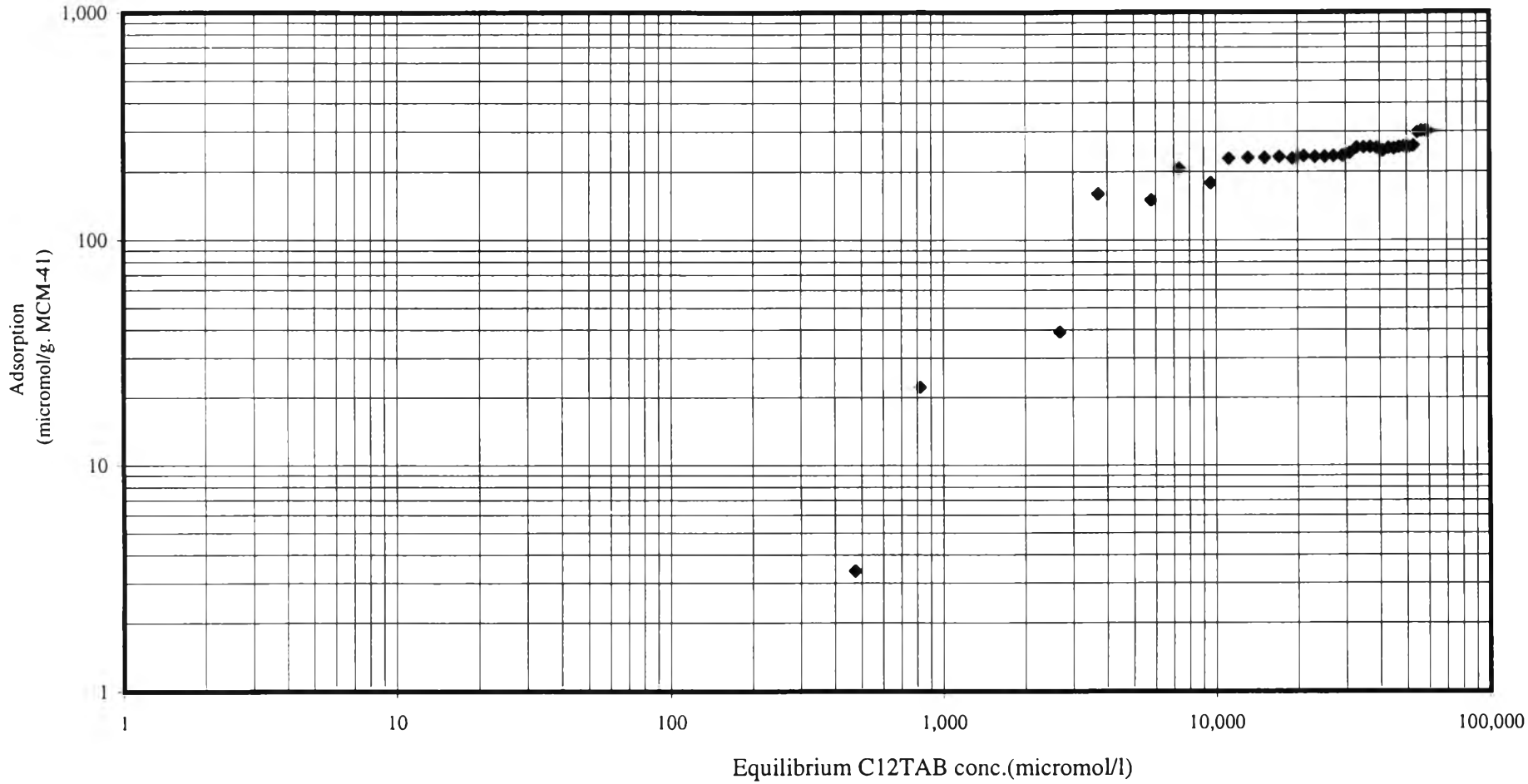

Figure 4-8 C12TAB Adsorption isotherm on MCM-41 pore size 21 A^0

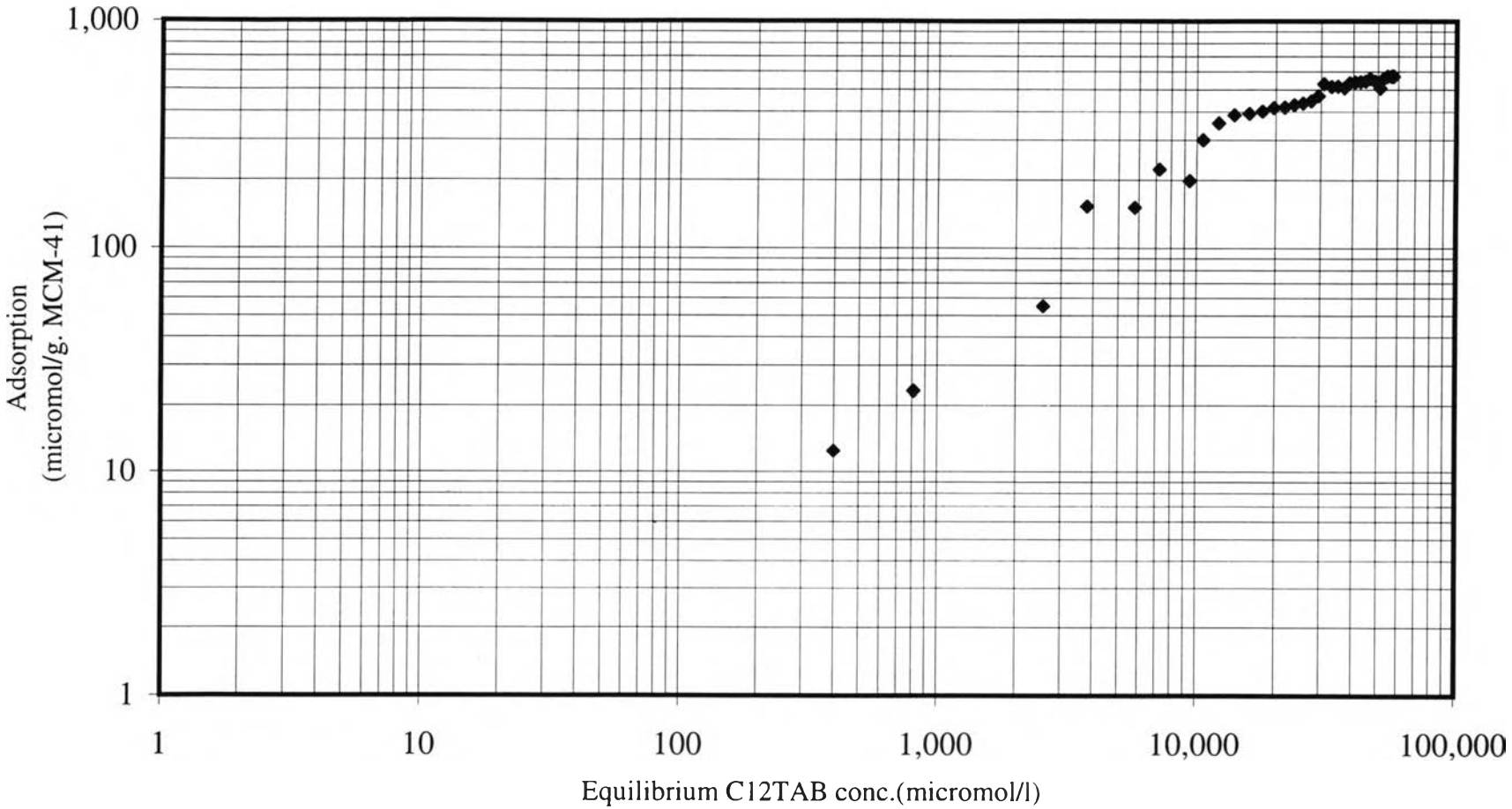

Figure 4-9 C12TAB Adsorption isotherm on MCM-41 pore size $26A^0$

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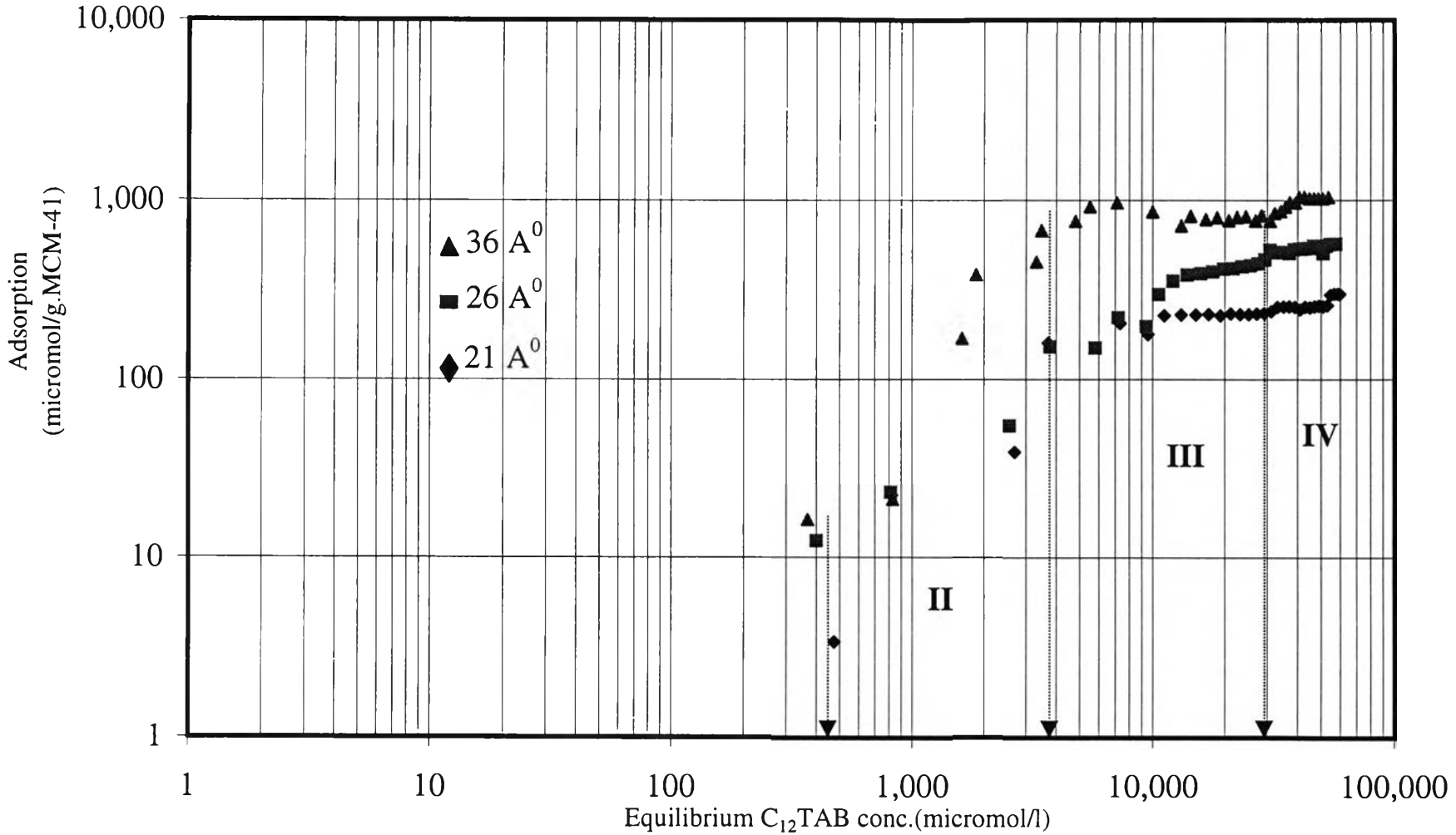

Figure 4-11 C_{12}TAB Adsorption isotherm at various pore size on MCM-41

4.3 Hexadecyltrimethylammonuim Bromide Adsorption Isotherm

The equilibrium time for Hexadecyltrimethylammonuim bromide adsorption on MCM-41 pore size 21 A^0, 26 A^0, 36 A^0 was approximately 7 days. The adsorption dynamics of Hexadecyltrimethylammonuim bromide on MCM-41 are presented in Figure 4-15. The isotherm slope of region II and plateau values of region IV increase slightly with increasing the pore size. And the isotherm slope of MCM-41 pore size 21 A^0, 26 A^0, 36 A^0 was about 0.026, 0.046, and 0.078 respectively.

From the plateau region data, the amount adsorbed at saturation of C16TAB on MCM-41 pore size 21 A^0, 26 A^0, 36 A^0 was approximately 200 μ mol/g, 360 μ mol/g, 500 μ mol/g. This result indicated that C16TAB has the lowest measured equilibrium concentration.

4.4 The Relationship between Number of Carbons in the Surfactant Tails and Maximum Adsorption Density.

The density of surfactant adsorption will be a function of the orientation of the adsorbate at the adsorbent-solution interface (Dobias, 1984; Hough and Rendall , 1983; Zajac and Partyka,1966; and Koopal, 1993). If adsorbate molecules are oriented perdicular to the surface in a close-packed arrangement, the effectiveness of adsorption will be determined only by the size of the hydrated head group. In a parallel orientation, or orientation titled away from the perpendicular, the arrangement will be far from being close-packed and the effectiveness will be greatly reduced in consequence. From Figure 4-16 , the relationship between number of carbons in the surfactant tail (n) and the maximum adsorption density (M) (molecule/ nm^2) to give

$$M_{36} = -0.0438n + 1.055$$

$$M_{26} = -0.025n + 0.6867$$

$$M_{21} = -0.0096n + 0.3023$$

Where subscript number is the mean pore size of MCM-41 in angstrom.

As can be observed in Figure 4-16, the maximum adsorption density decreases with increasing the number of carbon atoms (n) in the alkyl chain of the surfactant in the order C16TAB < C12TAB < C8TAB and then it increases with increasing pore size.

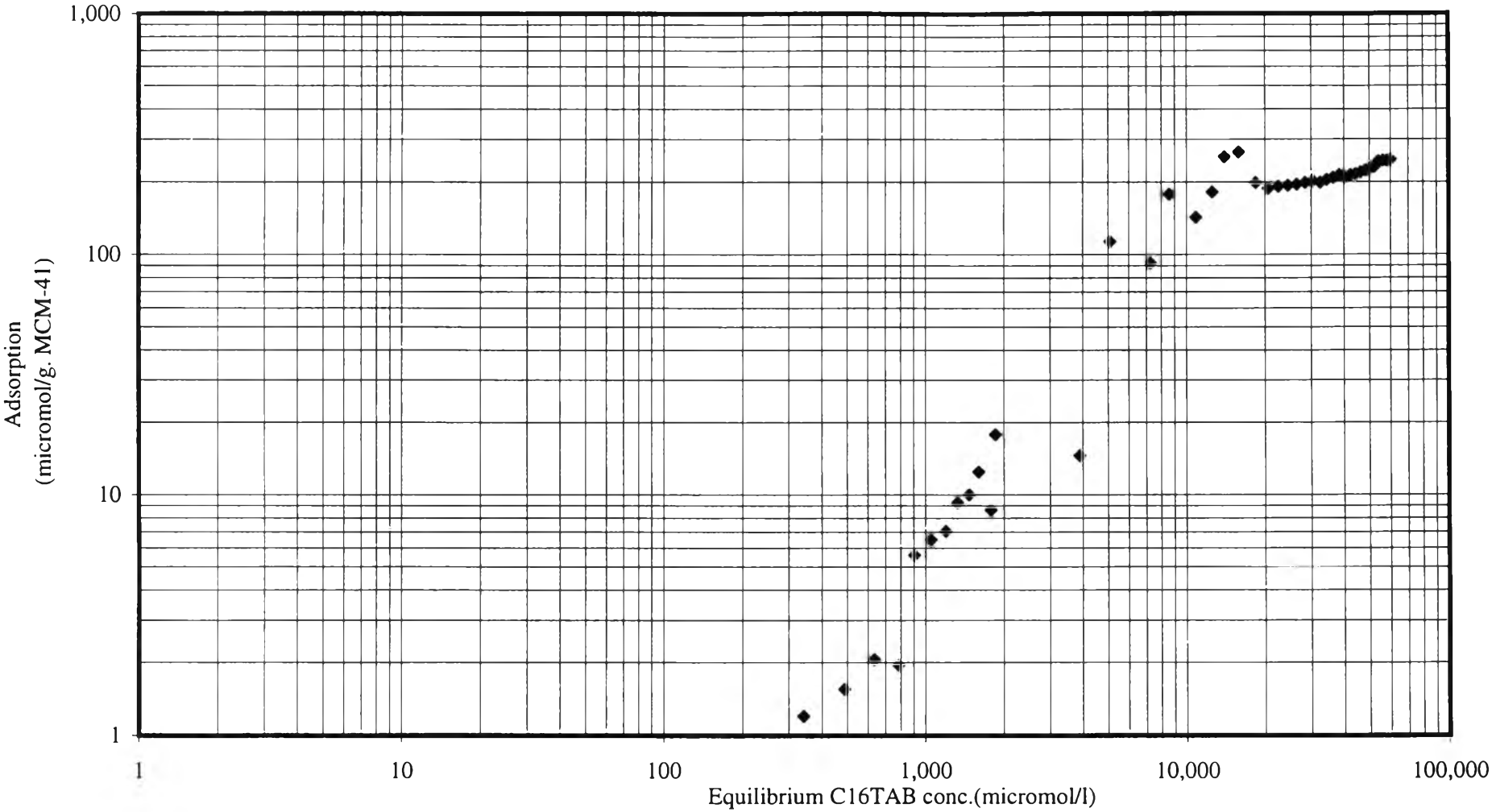

Figure 4-12 C16TAB Adsorption isotherm on MCM-41 pore size 21 A^0

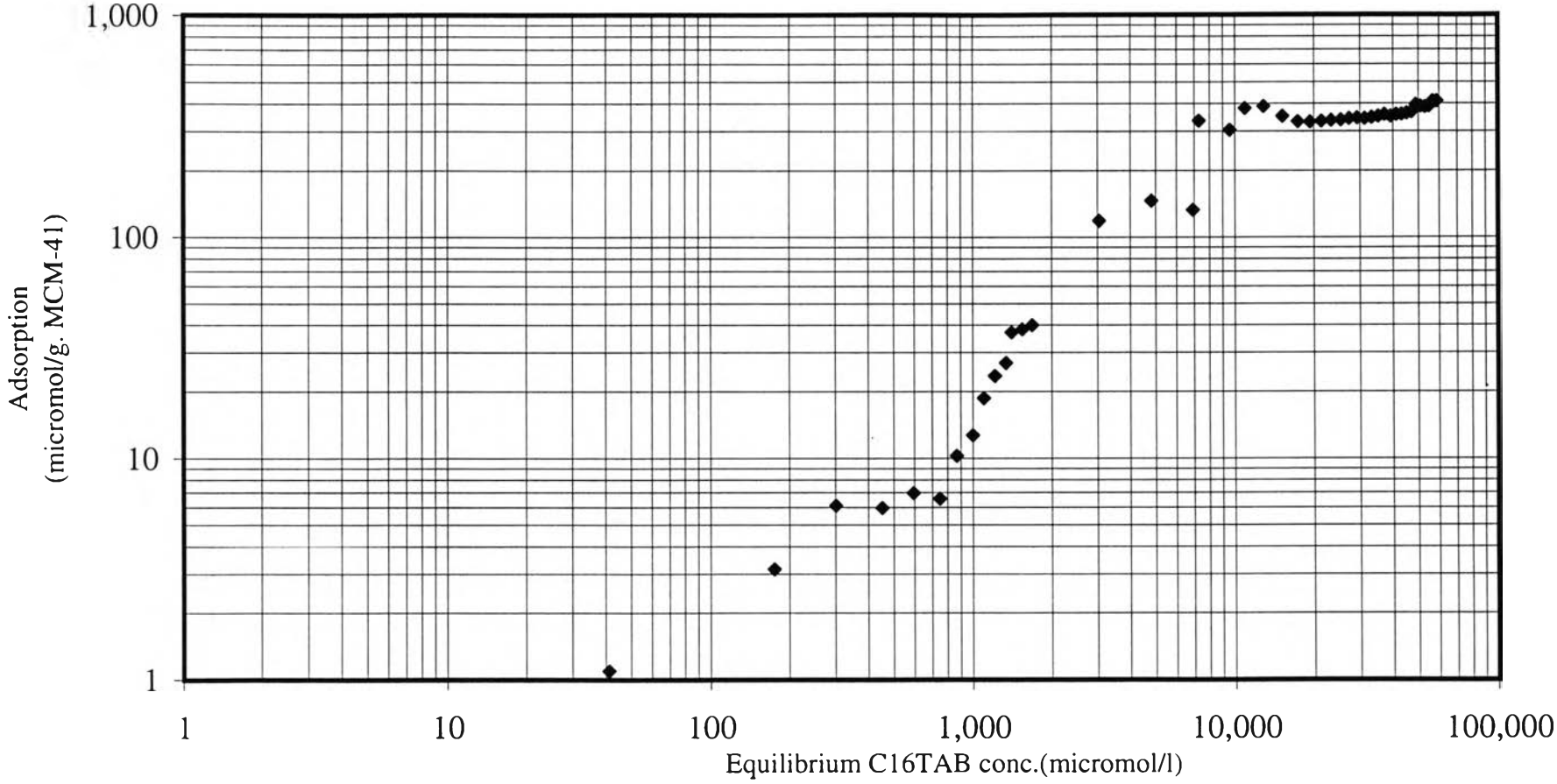

Figure 4-13 C16TAB Adsorption isotherm on MCM-41 pore size $26A^0$

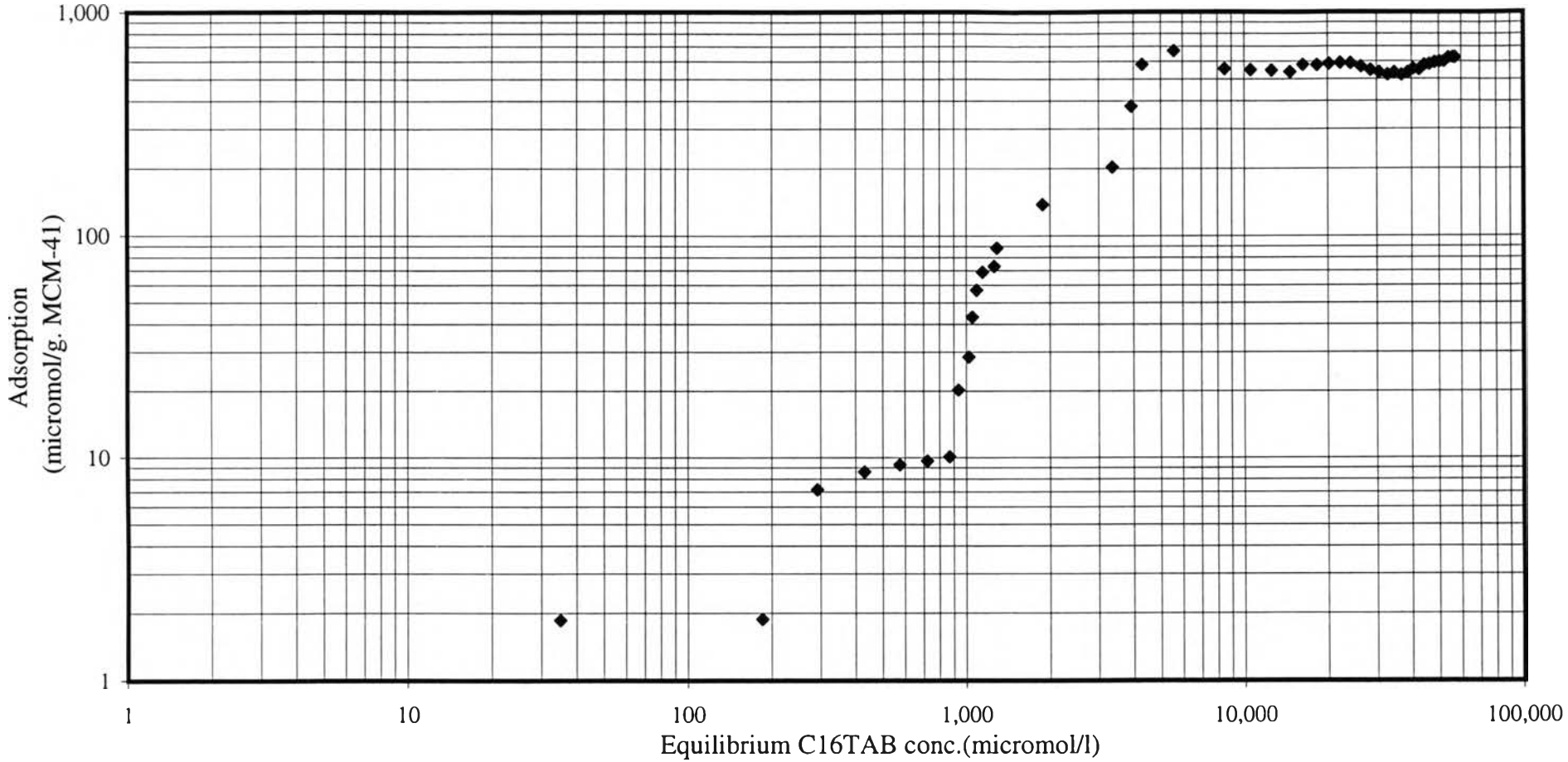

Figure 4-14 C16TAB Adsorption isotherm on MCM-41 pore size $36A^0$

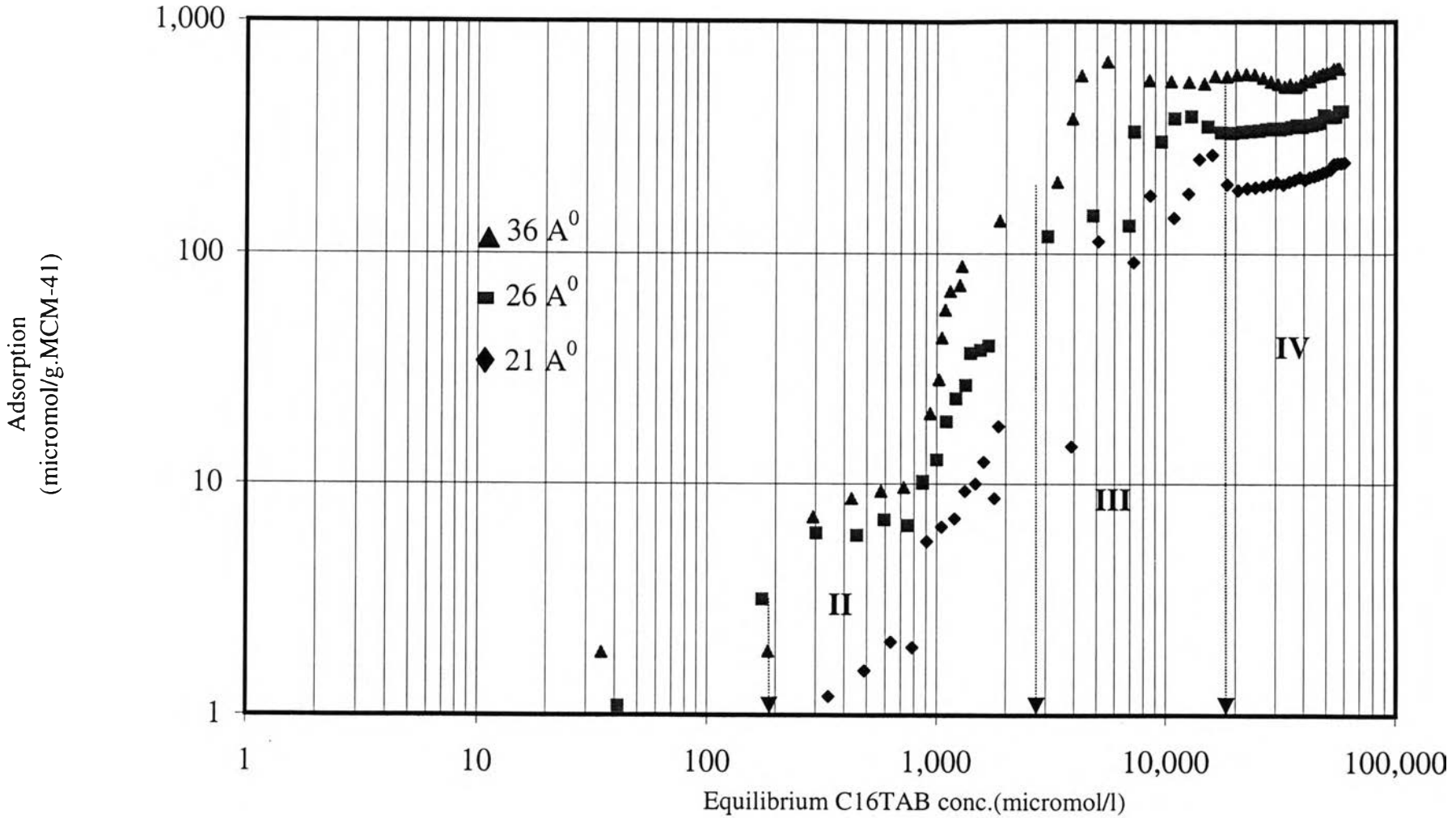

Figure 4-15 C16TAB Adsorption isotherm at various pore size on MCM-41

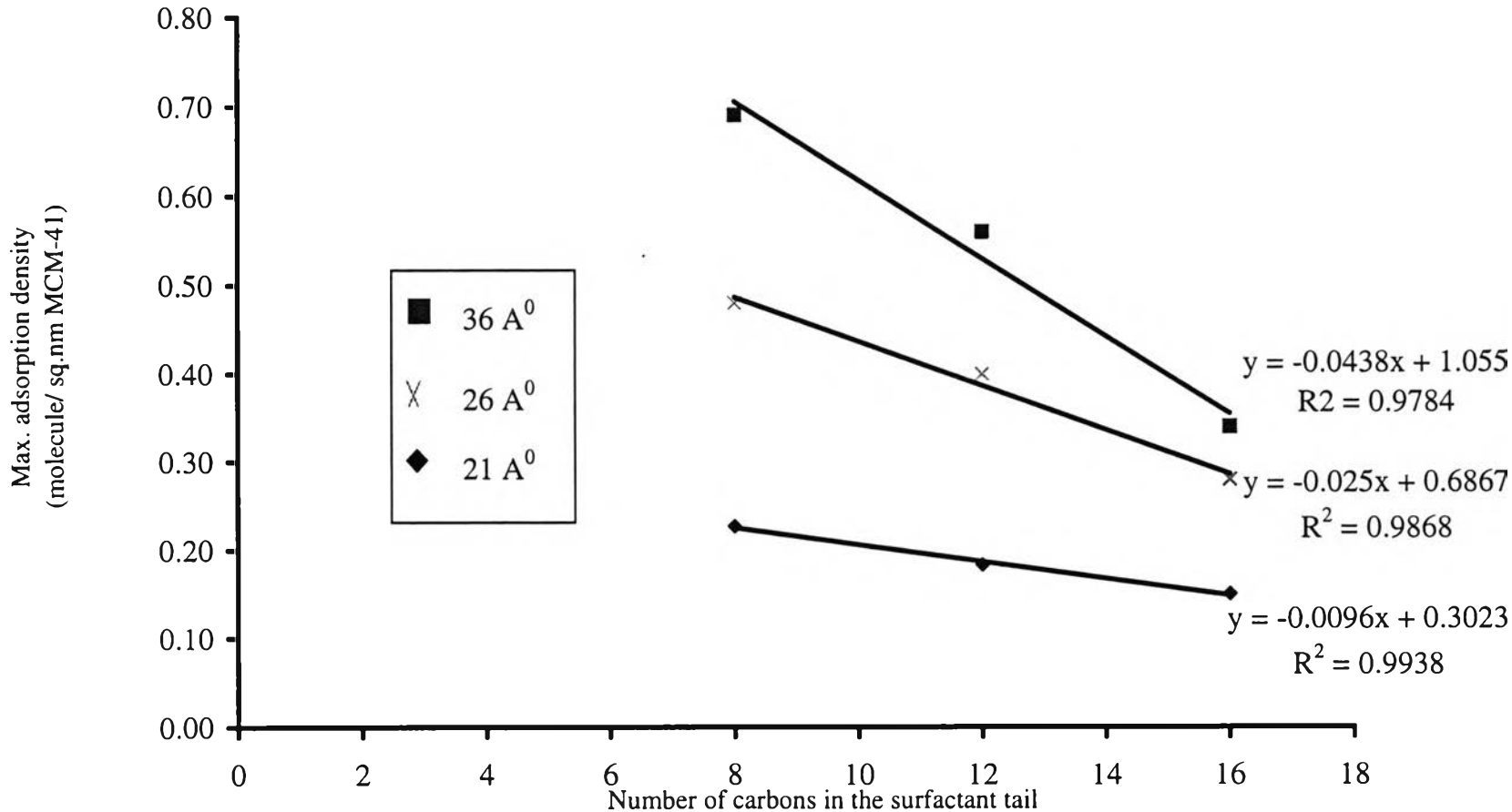

Figure 4-16 Maximum adsorption density of various pore size MCM-41 at different number of carbons in the surfactant tail

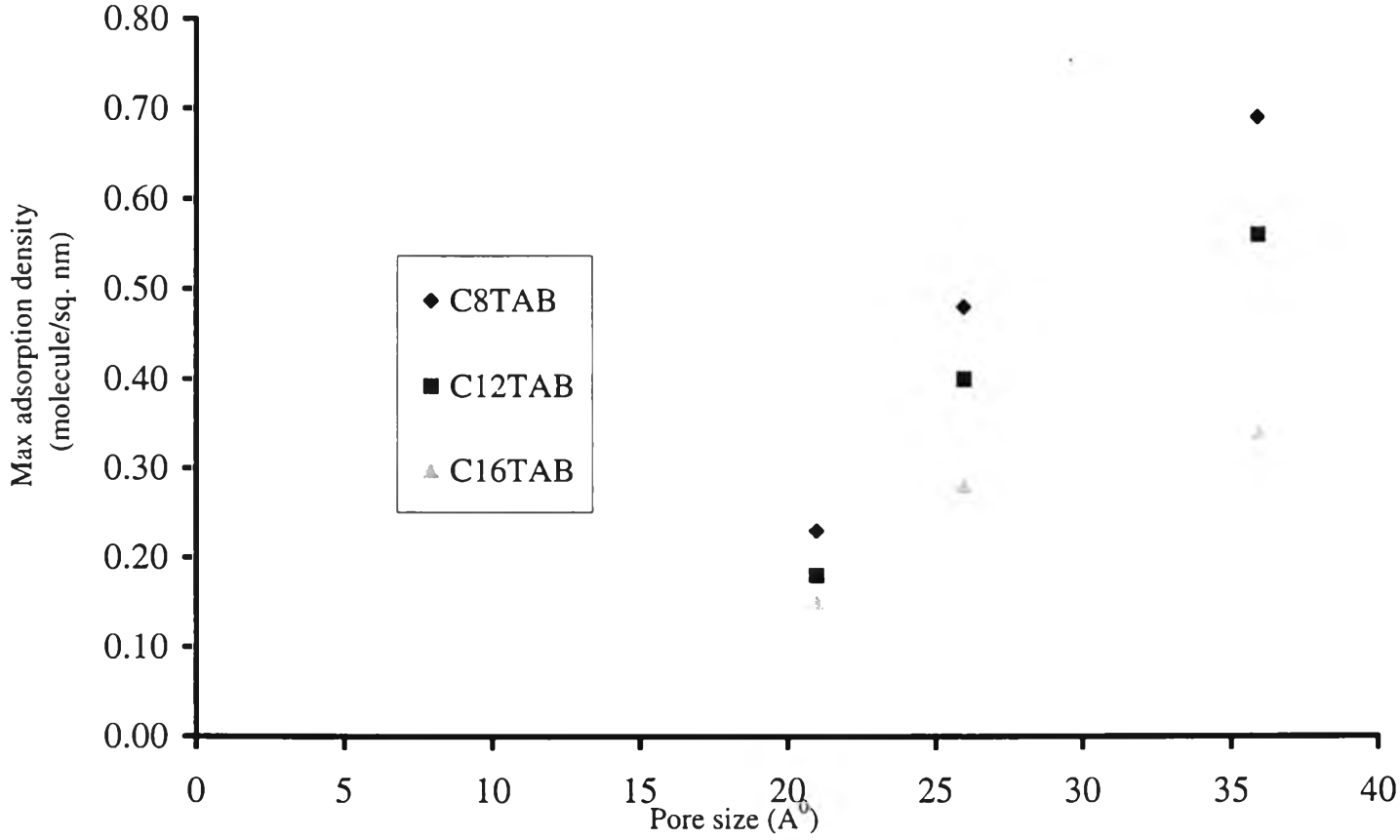

Figure 4-17 Maximum adsorption density of different number of carbons in the surfactant tail at various pore size MCM-41