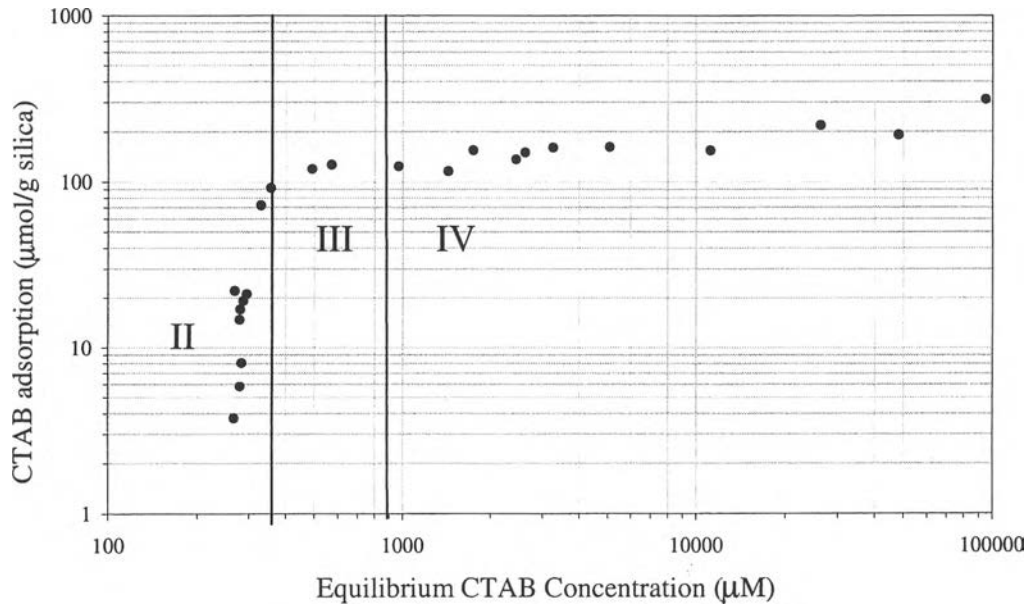




## CHAPTER IV RESULTS AND DISCUSSION

### 4.1 Adsorption Isotherm of CTAB on Aerosil®OX50

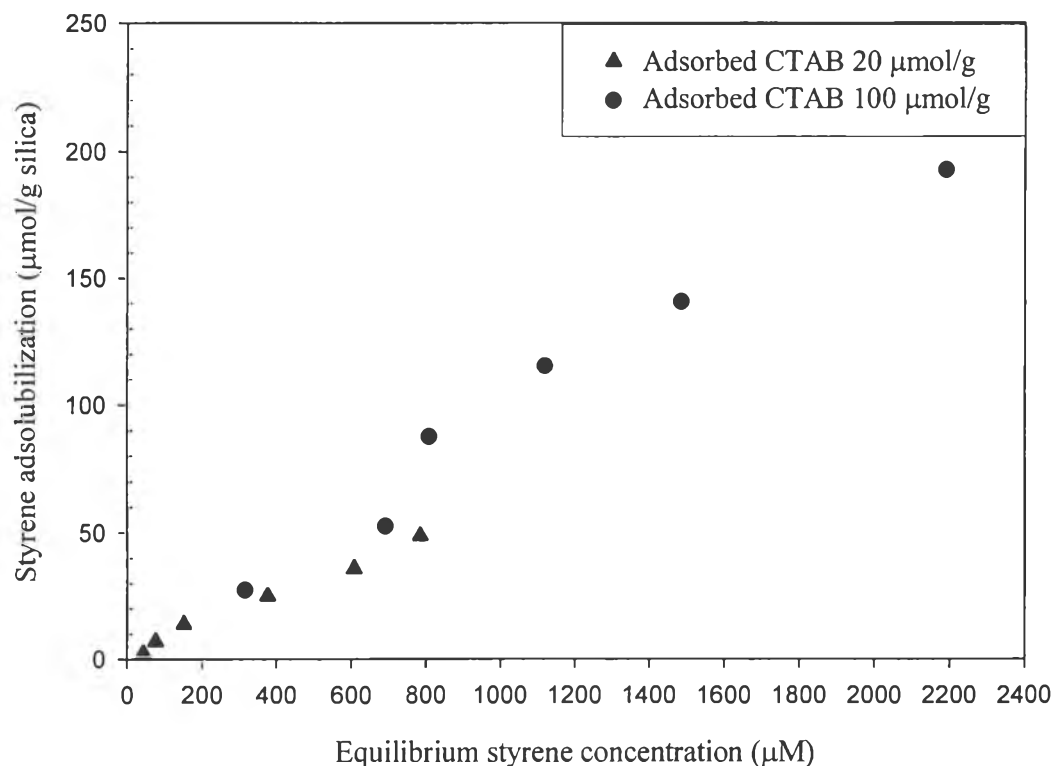


**Figure 4.1** CTAB adsorption isotherm on Aerosil®OX50.

The adsorption isotherm of CTAB on nonporous silica (Aerosil®OX50) is shown in Figure 4.1. The CTAB adsorption isotherm illustrates three regions (II, III and IV) of the standard adsorption isotherm (Rosen, 1989). The plateau region shows the maximum amount of CTAB on silica of approximately 130  $\mu\text{mol}$  of CTAB per gram of silica. As suggested, the silica had a measured  $\text{N}_2$  BET surface of 53.8  $\text{m}^2/\text{g}$ . If it is assumed that CTAB forms a perfect bilayer and completely covers the silica surface, the head of CTAB will cover 1.37  $\text{nm}^2$  on silica surface. However, the head group area of CTAB was 0.5  $\text{nm}^2$ . (Thakulsukanant, 1989) Therefore it is possible that there was electrostatic repulsion between adjacent CTAB head groups or CTAB does not completely cover the silica surface.

## 4.2 Styrene Adsolubilization Measurement

Figure 4.2 shows styrene adsolubilization versus the bulk equilibrium of styrene at two CTAB concentrations (20 and 100  $\mu\text{mol/g}$ ).



**Figure 4.2** Adsolubilization isotherm of styrene at two CTAB adsorption concentrations (20  $\mu\text{mol/g}$  and 100  $\mu\text{mol/g}$ ).

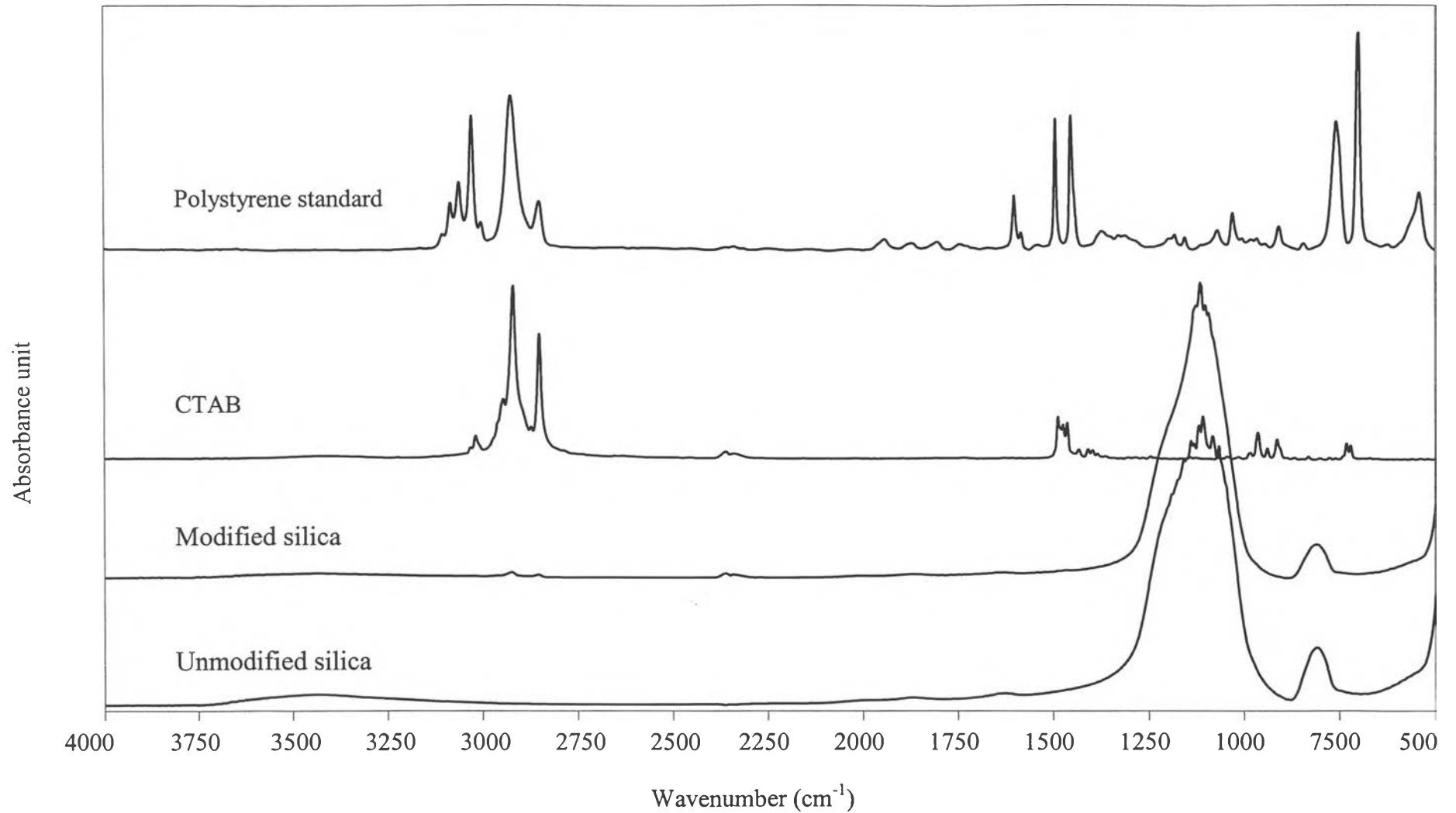
As expected, the adsolubilization of styrene increased with increasing styrene equilibration concentration at both CTAB adsorption levels. Comparing the styrene adsolubilization at the two CTAB levels, the adsolubilization levels were approximately equal for bulk styrene concentrations below 800  $\mu\text{M}$ . As expected styrene adsolubilization of CTAB 100  $\mu\text{mol/g}$  can be studied in the higher equilibration styrene concentration comparing with styrene adsolubilization of CTAB 20  $\mu\text{mol/g}$ . As the maximum amount of added styrene is a function of the capacity of the feed solution, styrene adsolubilization could not be studied at high level in the lower concentration system.

### 4.3 Characterization of Modified Silica, Extracted Polystyrene, and Extracted Silica

#### 4.3.1 Fourier Transform Infrared Spectroscopy Result

The presence of polystyrene on silica particles after modifications was confirmed by FTIR. Figure 4.3 shows the spectra of polystyrene standard, CTAB, modified silica and unmodified silica. The silica was modified by feed conditions of CTAB 100  $\mu\text{mol/g}$  : styrene 200  $\mu\text{mol/g}$  (1:2) at six hours reaction time. As a result the spectrum of modified silica in Figure 4.3 shows small absorbency peaks of methylene C–H stretching in the range of 2980-2840  $\text{cm}^{-1}$ , suggesting the presence of CTAB or polystyrene on silica particles. However peaks of polystyrene were not clearly represented on spectrum of the modified silica. Because polystyrene peaks were interfered by the high intensity of silica adsorption bands and the amount of polystyrene coated on silica particles was very low when compared to the amount of silica, it is difficult to detect the polymer. Therefore, after modified silica was passed through the polymer extraction process, extracted material was characterized by FTIR technique.

The extracted material was characterized by FTIR. Figure 4.4 shows the comparison between FTIR spectra of extracted material and polystyrene standard. The extracted material was extracted from modified silica conducted in the condition of CTAB 100  $\mu\text{mol/g}$  : styrene 200  $\mu\text{mol/g}$  (1:2) in six hours reaction time. The result shows that all peaks observed on spectra of extracted material were consistent to peaks on polystyrene standard spectra. And it clearly showed important peaks that represented benzene ring which are aromatic C–H stretching at 3100-3000  $\text{cm}^{-1}$ , aromatic C=C stretching at 1600  $\text{cm}^{-1}$ , 1495  $\text{cm}^{-1}$ , 1454  $\text{cm}^{-1}$ , and out-of-plane aromatic C–H bending at 700  $\text{cm}^{-1}$ . This result can confirm that polystyrene was formed on the silica particles by admicellar polymerization.



**Figure 4.3** FTIR spectra of polystyrene standard, CTAB, modified silica, and pure silica.

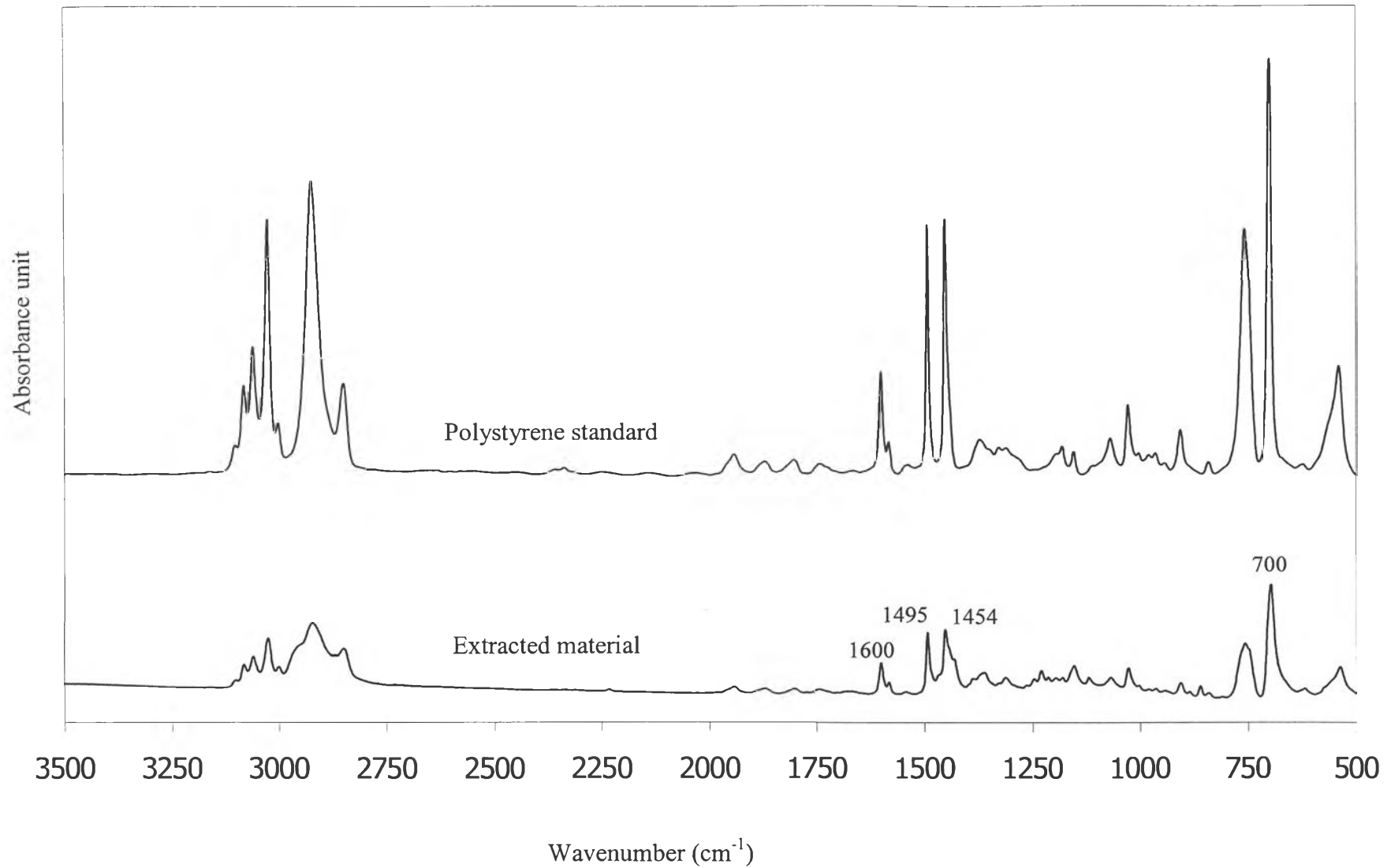


Figure 4.4 FTIR spectra of polystyrene standard and extracted material.

### 4.3.2 Thermogravimetric Analysis Result

TGA technique was used to study thermal stability of extracted polystyrene and to quantify amount of polystyrene formed on the silica particles.

Figure 4.5 shows thermogram of extracted polystyrene extracted in the condition of CTAB 100  $\mu\text{mol/g}$  : styrene 100  $\mu\text{mol/g}$  (1:1) in four hours reaction time. The thermogram of extracted polystyrene shows that extracted polystyrene gradually degrades in the temperature range of 120-350°C and rapidly degrades in the temperature range between 350-450°C. From the thermogram of extracted polystyrene the result suggested that the degradation below 350°C was caused by degradation of low molecular weight polystyrene (Anja Ilse Roland and Gudrun Schmidt-Naake, 2000) as well as CTAB trapped in the extracted polystyrene.

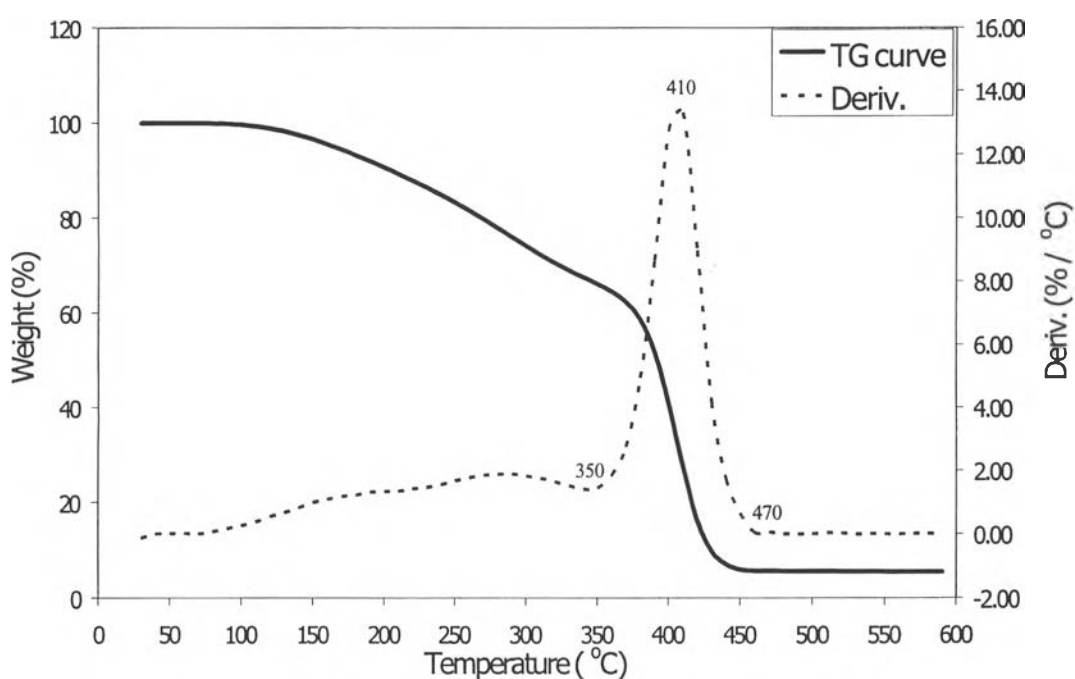
Figure 4.6 shows thermogram of CTAB, it was clearly shows that the decomposition of CTAB was at temperatures between 200-300°C. Figure 4.7 shows the thermogram of CTAB adsorbed on silica via condition of CTAB 100  $\mu\text{mol/g}$ . Decomposition of CTAB adsorbed on silica shows weight loss occurring in two steps, the first step was between 150-290°C and the second step was from 290-370°C. It was possible that the weight loss in the second step was a result from the delay degradation of CTAB head groups that were adsorbed on silica surface.

Figures 4.8 and 4.9 show thermogram of modified silica obtained at reaction condition of CTAB 100  $\mu\text{mol/g}$  : styrene 100  $\mu\text{mol/g}$  (1:1) in four hours reaction time. The thermogram of modified silica clearly shows three steps degradation in the temperature ranges between 150-300°C, 300-370°C, and 300-470°C. From the thermogram of modified silica, the result suggested that the components which degraded in the temperature between 150-370°C consisted mainly of CTAB and some low molecular weight polystyrene. And the weight loss between 300-470°C was due the decomposition of relatively high molecular weight polystyrene which was confirmed by thermogram of extracted polystyrene in Figure 4.5.

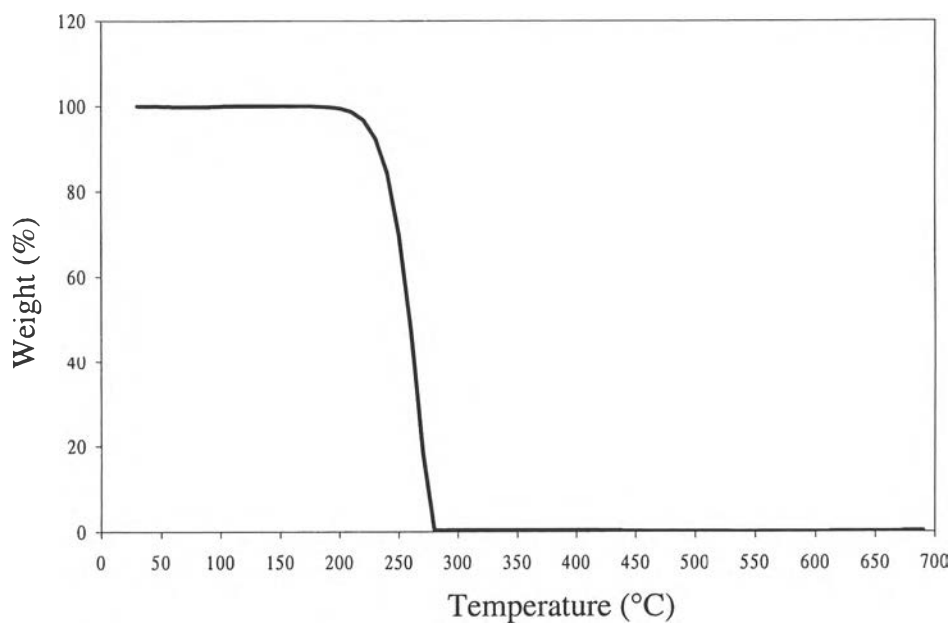
Figure 4.9 shows thermogram of modified silica after extraction. The extracted silica was obtained from silica which was modified from condition of CTAB 100  $\mu\text{mol/g}$  : styrene 100  $\mu\text{mol/g}$  (1:1) in four hours reaction time. When comparing the thermograms of modified silica before and after extraction (Figure 4.8

and Figure 4.9), an absence of the weight loss between 300-470°C in thermogram of extracted silica (Figure 4.9) can be confirmed that most of polymer were removed from silica particles after extraction.

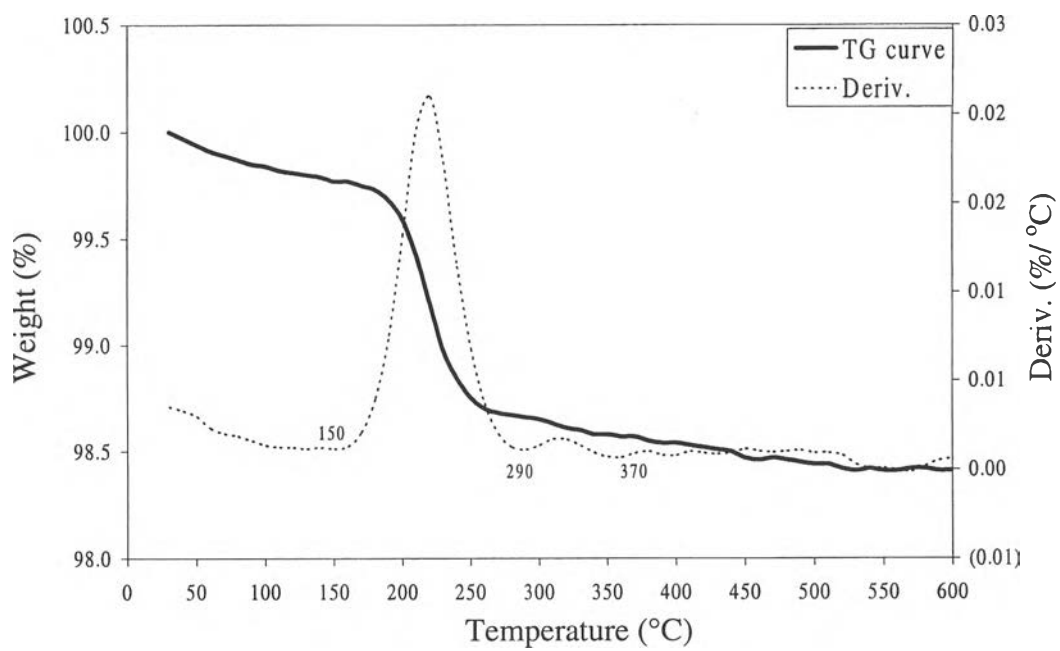
TGA analysis was used to quantify the amount of relatively high molecular weight polystyrene ( $\overline{M}_w > 9000 \text{ gmol}^{-1}$ ) formed on silica particles. All samples of modified silica was evaluated at the temperatures between 30-700°C and the mass loss detected between 300-470°C in order to determine the % weight of relatively high molecular weight polystyrene. Table 4.1 reports the % weight of relatively high molecular weight polystyrene coated on silica particles as determined from thermograms of the modified silica.



**Figure 4.5** Thermogram of extracted polystyrene.

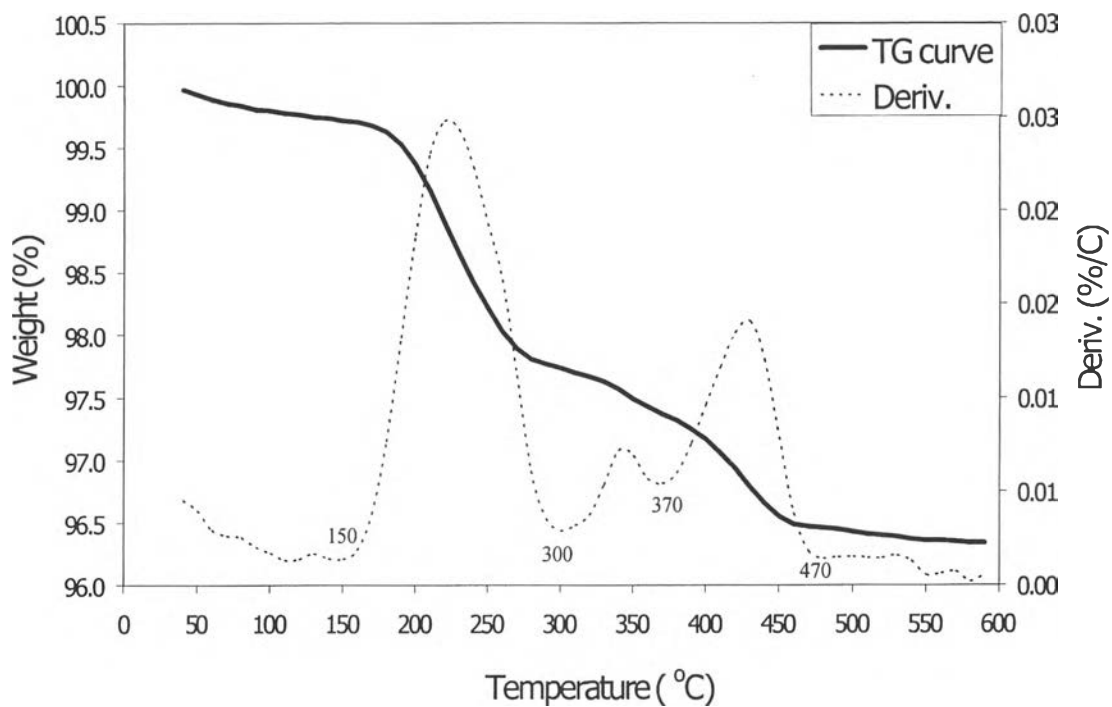


**Figure 4.6** Thermogram of CTAB.

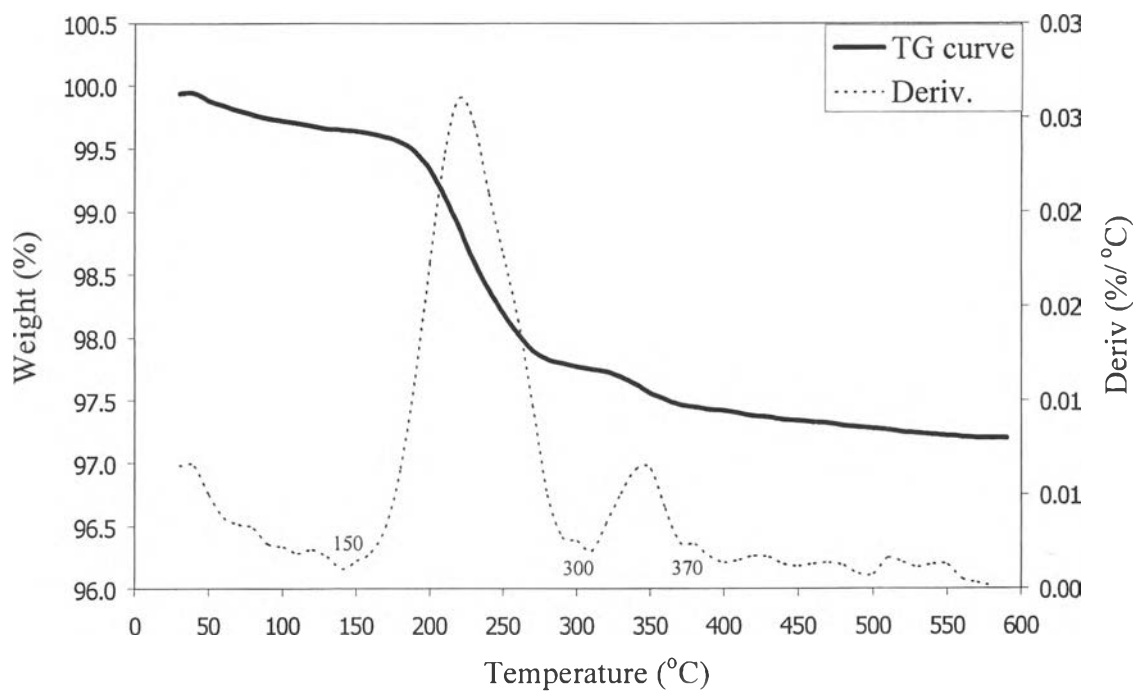


**Figure 4.7** Thermogram of adsorbed CTAB on silica (100  $\mu\text{mol/g}$ ).





**Figure 4.8** Thermogram of modified silica.



**Figure 4.9** Thermogram of modified silica after extraction.

**Table 4.1** % weight of polystyrene produced in adsorbed CTAB 20  $\mu\text{mol/g}$  on nonporous silica with vary monomer loading and reaction time

CTAB <sub>adsorp</sub> : Styrene <sub>adsol</sub>	Reaction time (h)	Weight of polystyrene (% w/w of modified silica)	
		CTAB 20 $\mu\text{mol/g}$	CTAB 100 $\mu\text{mol/g}$
1:0.5	1	-	0.28
	2	-	0.39
	4	-	0.29
	6	-	0.71
1:1	1	0.10	0.35
	2	0.14	0.87
	4	0.21	0.97
	6	0.09	1.14
1:2	1	-	1.57
	2	0.28	1.88
	4	0.43	1.79
	6	0.49	1.94

CTAB<sub>adsorp</sub> = Adsorption of CTAB, ( $\mu\text{mol/g}$ ), Styrene<sub>adsol</sub> = Adsolubilization of styrene, ( $\mu\text{mol/g}$ )

- = weight loss lower than 0.1%

Table 4.1 shows that polystyrene prepared from reaction condition of CTAB 20  $\mu\text{mol/g}$  : styrene 10  $\mu\text{mol/g}$  in one to six hours reaction time was low molecular weight. Results from other reaction conditions suggested that when reaction time increased, the amount of polystyrene on silica particles slightly increased. In addition, at the same reaction time, amount of polystyrene on silica particles was clearly increased when ratio of styrene loading increased.

### 4.3.3 Gel Permeation Chromatography Result

GPC was used to study effects of styrene loading and reaction time on the molecular weight of the polystyrene obtained.

Tables 4.2 and 4.3 show the number average molecular weight ( $\overline{M}_n$ ), weight average molecular weight ( $\overline{M}_w$ ), and molecular weight distribution (MWD) of extracted materials which were extracted from modified silica. These modified silica were produced from two different CTAB concentrations (100 and 20  $\mu\text{mol/g}$ ) at different monomer loading and reaction time.

The GPC curve of extracted materials showed three peaks of different retention times which indicated that three different ranges of molecular weight polymer were obtained. The three peaks observed were in the ranges of weight average molecular weight ( $\overline{M}_w$ ) between 66000–9000  $\text{gmol}^{-1}$ , 1000–600  $\text{gmol}^{-1}$ , and 270–310  $\text{gmol}^{-1}$ . The value observed in the third peak is consistent with the molecular weight of CTAB, thus the third peak was probably due to CTAB trapped in the extracted polystyrene. It could also be possible that the upper CTAB layer was incompletely removed from modified silica in the washing step and some CTAB was extracted during the extraction process. Therefore the results suggest that the first peak and second peaks of the extracted material were polystyrene peaks. The low molecular weight of extracted polystyrene observed in the range of 600–1000  $\text{gmol}^{-1}$  (second peak) could be due to growing chains or cyclic chains of polystyrene.

**Table 4.2**  $\overline{M}_w$ ,  $\overline{M}_n$ , and MWD of extracted materials produced in CTAB 20  $\mu\text{mol/g}$

CTAB <sub>adsorp</sub> : Styrene <sub>adsol</sub> ( $\mu\text{mol/g}$ ) : ( $\mu\text{mol/g}$ )	Reaction time (h)	First peak			Second peak			Third peak		
		$\overline{M}_n$	$\overline{M}_w$ , MWD		$\overline{M}_n$	$\overline{M}_w$ , MWD		$\overline{M}_n$	$\overline{M}_w$ , MWD	
20:10 (1:0.5)	1	-	-	-	790	1019	1.30	291	306	1.05
	2	-	-	-	613	727	1.17	258	277	1.07
	4	-	-	-	713	801	1.12	294	310	1.05
	6	-	-	-	659	727	1.10	288	304	1.06.
20:20 (1:1)	1	-	-	-	750	1773	2.36	270	292	1.08
	2	5677	9154	1.61	501	704	1.41	-	-	-
	4	6554	10468	1.6	526	754	1.43	-	-	-
	6	4826	9046	1.87	469	665	1.42	-	-	-
20:40 (1:2)	1	504	2427	4.82	-	-	-	-	-	-
	2	10997	27839	2.53	715	854	1.19	282	301	1.07
	4	4888	22480	4.6	736	868	1.18	270	289	1.07
	6	9306	28294	3.04	798	936	1.17	292	310	1.06

CTAB<sub>adsorp</sub> = Adsorption of CTAB, ( $\mu\text{mol/g}$ ), Styrene<sub>adsol</sub> = Adsolubilization of styrene, ( $\mu\text{mol/g}$ )

**Table 4.3**  $\overline{M}_w$ ,  $\overline{M}_n$ , and MWD of extracted materials produced in CTAB 100  $\mu\text{mol/g}$

CTAB <sub>adsorp</sub> : Styrene <sub>adsol</sub> ( $\mu\text{mol/g}$ ) : ( $\mu\text{mol/g}$ )	Reaction time (h)	First peak			Second peak			Third peak		
		$\overline{M}_n$	$\overline{M}_w$	MWD	$\overline{M}_n$	$\overline{M}_w$	MWD	$\overline{M}_n$	$\overline{M}_w$	MWD
100:50 (1:0.5)	1	-	-	-	675	776	1.15	291	304	1.05
	2	6352	24020	3.78	468	691	1.48	-	-	-
	4	6397	19749	3.09	424	588	1.39	-	-	-
	6	6350	12530	1.97	761	912	1.20	278	301	1.09
100:100 (1:1)	1	9419	14695	1.56	350	526	1.50	-	-	-
	2	9956	35397	3.56	720	835	1.16	282	298	1.06
	4	9784	39726	4.06	691	783	1.13	277	296	1.07
	6	10300	48666	4.72	736	820	1.11	282	296	1.05
100:200 (1:2)	1	10963	64526	5.89	683	725	1.06	285	299	1.05
	2	12515	53916	4.31	641	694	1.08	282	298	1.06
	4	10943	66149	6.04	682	737	1.08	292	306	1.05
	6	9639	48728	5.06	736	807	1.10	291	306	1.05

CTAB<sub>adsorp</sub> = Adsorption of CTAB, ( $\mu\text{mol/g}$ ), Styrene<sub>adsol</sub> = Adsolubilization of styrene, ( $\mu\text{mol/g}$ )

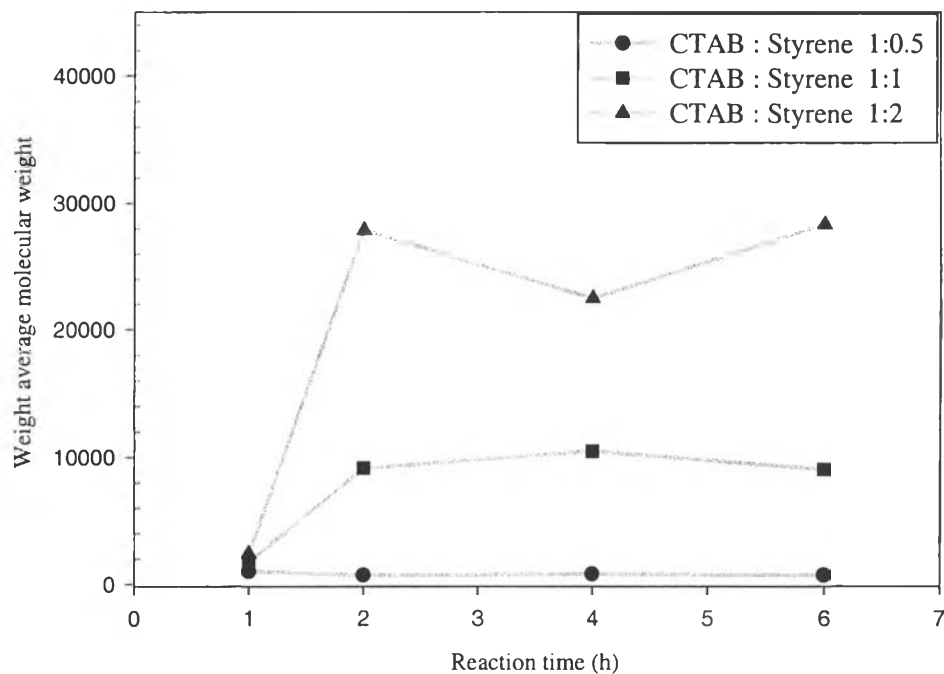
#### 4.3.3.1 Effect of reaction time

Figure 4.10 and figure 4.11 shows the effects of reaction time on eight average molecular weight of polystyrene which formed in CTAB 20  $\mu\text{mol/g}$  and CTAB 100  $\mu\text{mol/g}$  respectively. The results show that the polystyrene formed in the ratios of styrene loading 1:0.5, 1:1, and 1:2 were low  $\overline{M}_w$  at one hour reaction time. This suggests that there is a relatively low degree of polymerization was occurred at one hour reaction time.

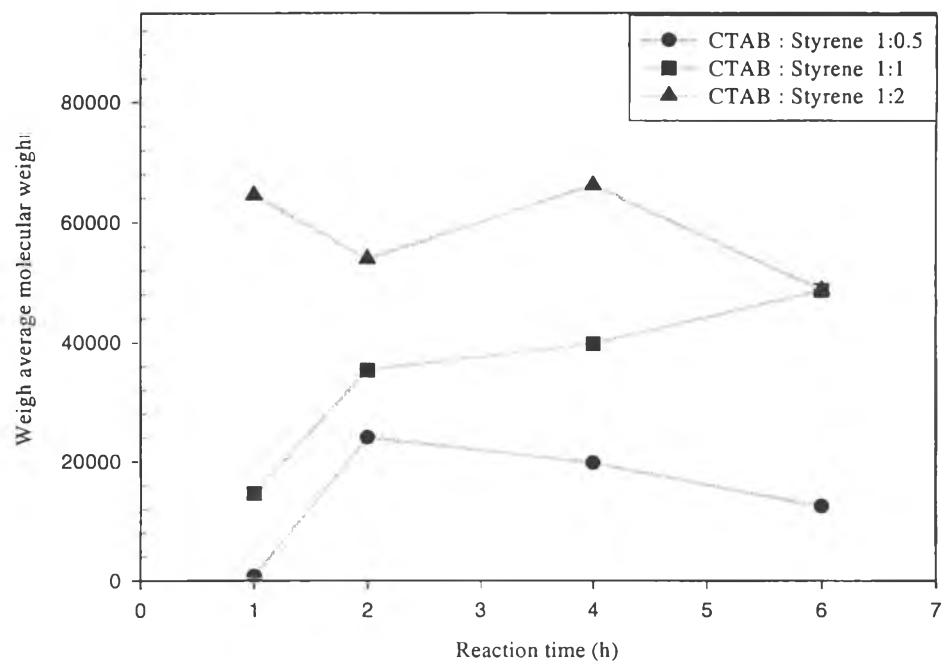
For 2–6 hours reaction time, molecular weights of polystyrene obtained were relatively high and closed to the highest molecular weight polystyrene obtained at each styrene level (Figures 4.10 and 4.11). These results were consistent except for the lowest styrene level in the 20  $\mu\text{mol/g}$  CTAB system where low  $\overline{M}_w$  was obtained at all reaction times (Figure 4.10). This might suggested that perhaps the styrene monomer were so spread out in the admicelle and the chances for two styrene monomers to collide were reduced thus, the formation of higher molecular weight polymer is not possible.

In Figure 4.11, result from polystyrene obtained at a styrene : CTAB loading ratio of 1:2 in CTAB 100  $\mu\text{mol/g}$  was relatively different from polymers obtained from different reaction conditions. High  $\overline{M}_w$  polystyrene was obtained at one hour reaction time. This result suggested that, due to the high amount of styrene (200  $\mu\text{mol/g}$ ) in CTAB bilayer, monomers were converted into polymer very quickly and high  $\overline{M}_w$  is achieved in a shorter time. This could be due to the presence of a more continuous styrene phase present at high monomer levels or of the presence of larger “pools” of styrene that have phase separated within the admicelle.

Nevertheless, the results from Figure 4.10 and 4.11 show that when the reaction time exceeded two hours, the molecular weight of polystyrene is similar for each set of conditions, suggesting that the time for complete conversion in these admicellar polymerization systems is between one and two hours.



**Figure 4.10** Effect of reaction time on weight average molecular weight of polystyrene (CTAB concentration 20  $\mu\text{mol/g}$ ).



**Figure 4.11** Effect of reaction time on weight average molecular weight of polystyrene (CTAB concentration 100  $\mu\text{mol/g}$ ).

#### 4.3.3.2 Effect of styrene loading

Figures 4.12 and 4.13 show the effects of styrene loading on weight average molecular weight of extracted polystyrene formed in CTAB concentrations of 20  $\mu\text{mol/g}$  and 100  $\mu\text{mol/g}$  respectively.

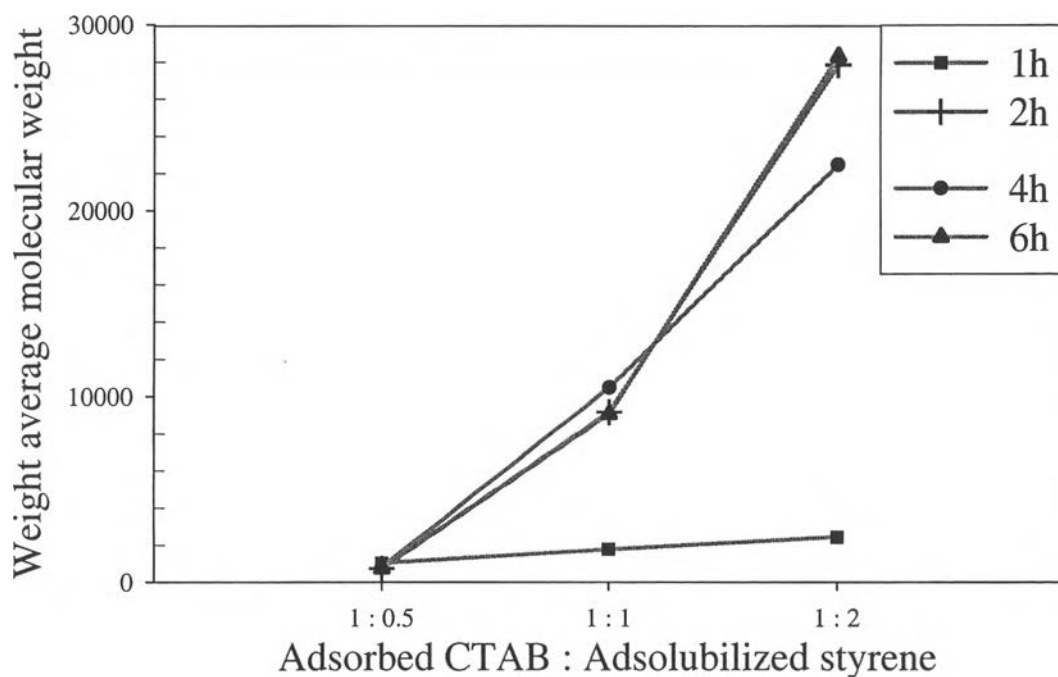
In Figure 4.12, results show that, as styrene adsolubilization within the bilayer increased, molecular weights of polystyrene also increased. These results were observed in all system except for when the reaction time is at one hour in ratio of adsorbed CTAB and adsolubilized styrene at 1:0.5, 1:1, and 1:2. This suggested that a relatively low styrene monomer in CTAB bilayer was not completely converted into relatively high molecular weight polystyrene at one hour reaction time.

For the CTAB concentration of 100  $\mu\text{mol/g}$  in Figure 4.13, the results show that for every reaction time, when ratio of styrene concentration increased, molecular weight of polystyrene obtained also increased. Only exception was polystyrene from six hours reaction,  $M_w$  of polystyrene remained constant when ratios of CTAB adsorption and styrene adsolubilization were 1:1 and 1:2. It was possible that the six hours reaction time is an anomaly and could be due to difficulties in mixing large amounts of styrene into the system.

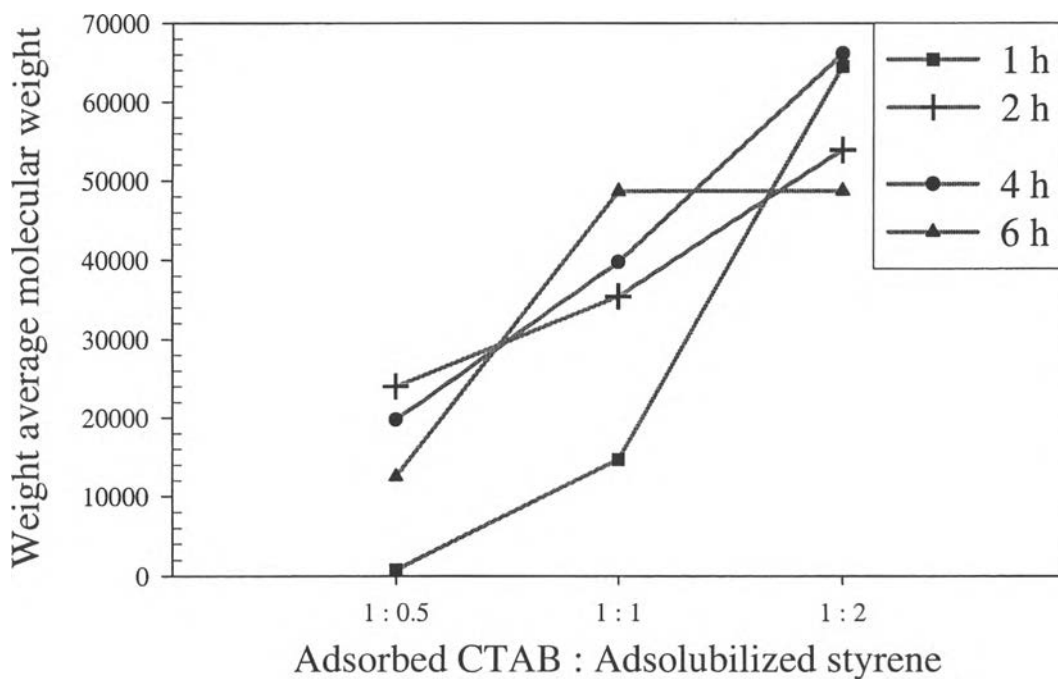
It was interesting to note that the  $\overline{M}_w$  of polystyrene increased with increasing styrene loading, this was a constant initiator loading ratio. These results could suggest that when adsolubilized styrene loading increased, the CTAB bilayer swells and the density of styrene per adsorbed CTAB molecule increases. Thus, the monomers have greater mobility and are in closer proximity, leading to relatively high  $\overline{M}_w$  polystyrene.

However, from Figures 4.12 and 4.13, the results suggested that, when adsolubilized styrene concentration increased, molecular weight of polystyrene formed on silica particles also increased.





**Figure 4.12** Effect of styrene loading on weight average molecular weight on polystyrene formed in CTAB concentration  $20 \mu\text{mol/g}$ .



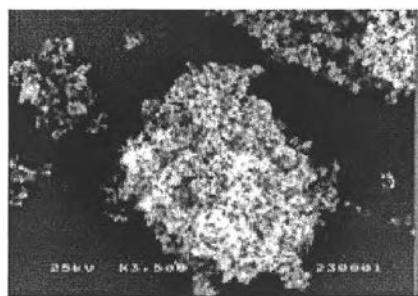
**Figure 4.13** Effect of styrene loading on weight average molecular weight on polystyrene formed in CTAB concentration  $100 \mu\text{mol/g}$ .

#### 4.3.4 Scanning Electron Micrographs of Unmodified Silica, and Modified Silica

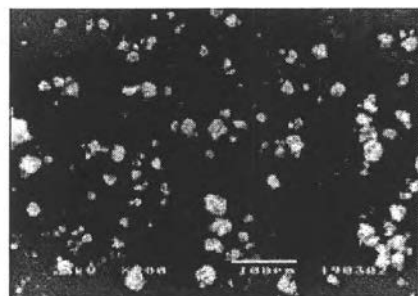
Figure 4.14 shows the scanning electron micrographs of unmodified silica in the X3500 and X200 magnifications. The scanning electron micrographs of unmodified silica shows silica particles were loose aggregates with diameter of about 15  $\mu\text{m}$ .

Figures 4.15–4.19 show the scanning electron micrographs of modified silica conducted in the condition of CTAB 100  $\mu\text{mol/g}$  : styrene 100  $\mu\text{mol/g}$  (1:1) in the reaction time from 30 minutes to 24 hours. The results show that after modification, the aggregation sizes and packing of modified silica were increased with increased reaction time. These clearly show in the scanning electron micrographs of modified silica at X200 magnification (Figures 4.15A, 4.16A, and 4.17A). It was possible that the polystyrene formed by admicellar polymerization on the silica particles joined the silica particles together and when the amount of polymer formed slightly increased with reaction time, the aggregation size was also increased.

Morphology of polystyrene formed on the silica particles could not be observed by scanning electron micrographs due to the small particle size of silica (reported average primary particles size = 40 nm).

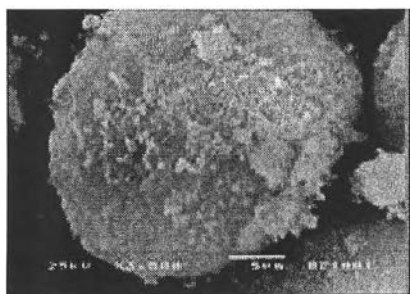


A. 3500X Magnification.

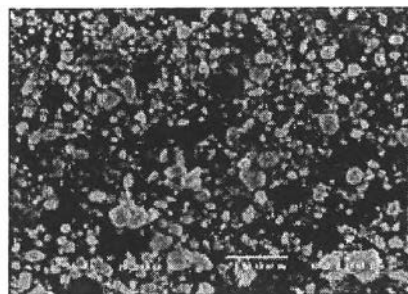


B. 200X Magnification.

**Figure 4.14** Micrographs of unmodified silica, Aerosil<sup>®</sup>OX50.

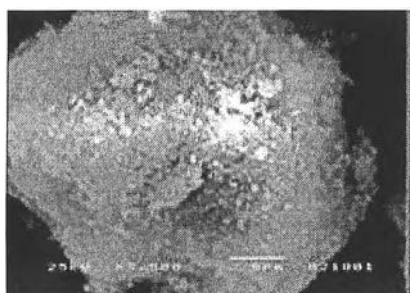


A. 3500X Magnification.

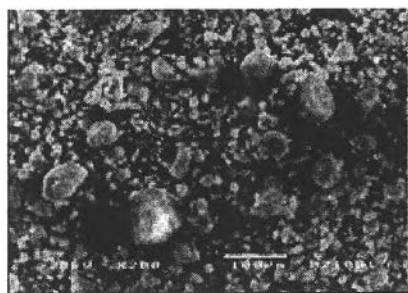


B. 200X Magnification.

**Figure 4.15** <sup>Micrographs of</sup> Modified silica with adsorbed CTAB 100  $\mu\text{mol/g}$  : adsolubilized styrene 100  $\mu\text{mol/g}$  at 30 minutes.

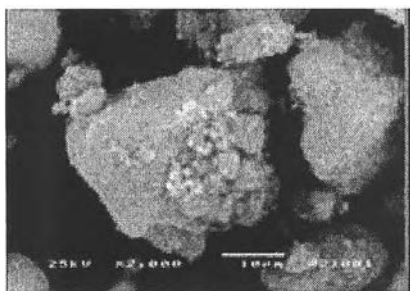


A. 3500X Magnification.

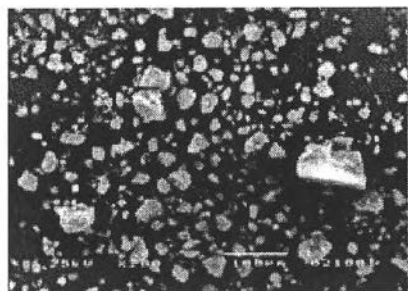


B. 200X Magnification.

**Figure 4.16** Modified silica with adsorbed CTAB 100  $\mu\text{mol/g}$  : adsolubilized styrene 100  $\mu\text{mol/g}$  at 1 hour.

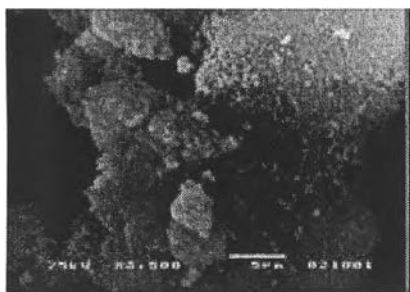


A. 3500X Magnification.

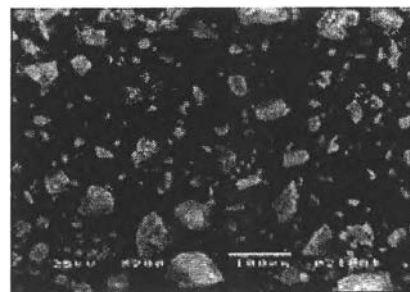


B. 200X Magnification.

**Figure 4.17** Modified silica with adsorbed CTAB 100  $\mu\text{mol/g}$  : adsolubilized styrene 100  $\mu\text{mol/g}$  at 2 hours.

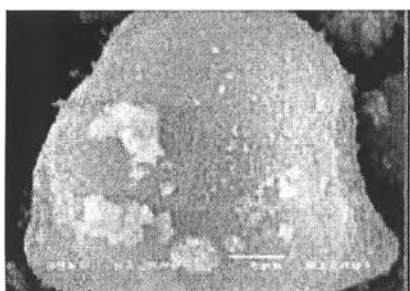


A. 200X Magnification.

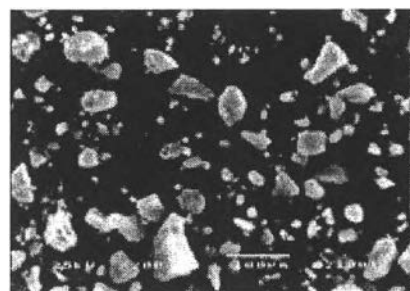


B. 3500X Magnification.

**Figure 4.18** Modified silica with adsorbed CTAB 100  $\mu\text{mol/g}$  : adsolubilized styrene 100  $\mu\text{mol/g}$  at 4 hours.



A. 200X Magnification.



B. 3500X Magnification.

**Figure 4.19** Modified silica with adsorbed CTAB 100  $\mu\text{mol/g}$  : adsolubilized styrene 100  $\mu\text{mol/g}$  at 24 hours.

#### 4.3.5 Atomic Force Micrograph of Modified Silica and Extracted Silica

Effects of styrene loading on the location and extent of the formed polystyrene on the silica particles were investigated using AFM.

In the same frame the result shows two types of image, these are topographic image and phase image. The topographic image of tapping mode image was interpreted by changing the oscillating amplitude of the tip during a tip tapped on the sample surface. The phase image can be used to interpret the hardness of sample surface. The soft sample will be seen as darker contrasts while a hard sample will appear brighter in contrast.

Figure 4.20 shows the atomic force micrograph of modified silica obtained in CTAB 20  $\mu\text{mol/g}$  : styrene 10  $\mu\text{mol/g}$  (1:0.5), six hours reaction time. The results from micrograph show that pieces of smooth polystyrene film coated on silica particles were clearly observed in the lighter regions of topographic image. The polystyrene films on silica surface appeared as the dark regions in the phase image. These images show that polystyrene films were in the form of patches, suggested that the CTAB bilayer (20 $\mu\text{mol/g}$ ) formed like islands on silica particles and they were not entirely cover the surfaces of silica particles.

Figure 4.21 shows the atomic force micrograph of modified silica obtained in CTAB 20  $\mu\text{mol/g}$ : styrene 20 $\mu\text{mol/g}$  (1:1), six hours reaction time. Patches of uneven polystyrene films coated on silica particles was observed in topographic image and the polystyrene films were clearly appeared in the darker areas contrast in the phase image.

Figure 4.22 shows the atomic force micrograph of modified silica obtained at CTAB 100  $\mu\text{mol/g}$ : styrene 50 $\mu\text{mol/g}$  (1:0.5), six hours reaction time. The micrograph shows that more polystyrene can be seen (lighter regions) on the silica surface and between particles in the topographic image. From the phase image, polystyrene which corresponded to the darker regions was formed around the edge of silica particle.

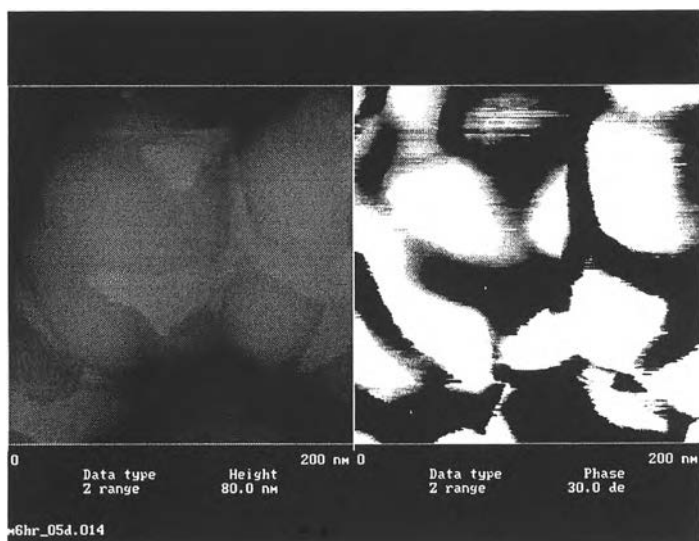
Figure 4.23 shows the atomic force micrograph of modified silica obtained at CTAB 100 $\mu\text{mol/g}$ : styrene 200 $\mu\text{mol/g}$  (1:2), six hours reaction time. Aggregates of polystyrene on the silica surface were observed by topographic image. In the phase micrograph mode, it was clearly showed that polystyrene formed into

round shape sitting on polystyrene which nearly encapsulated the silica surface. These polystyrene droplets confirm CTAB bilayer swelled at high adsorbed styrene level.

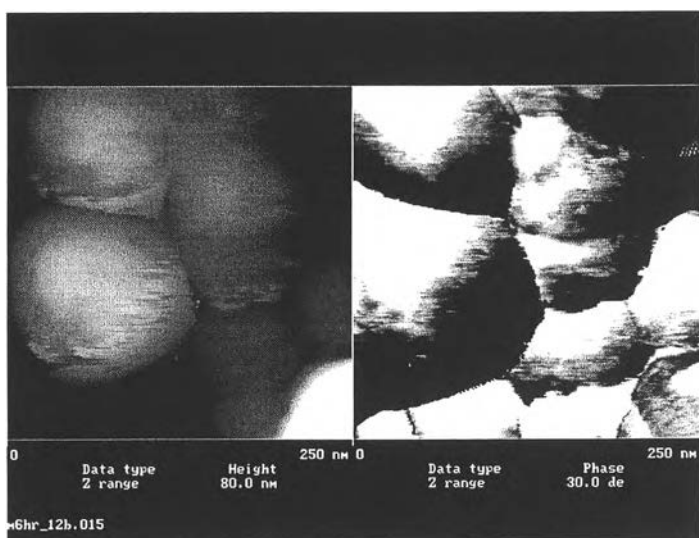
In addition the effects of styrene loading on morphologies of polystyrene formed on silica particles were investigated. At low CTAB level (20  $\mu\text{mol/g}$ ), the morphology of polystyrene formed on silica particles was compared in Figure 4.20 and 4.21. The results show that as the amount of polystyrene increased. This is clearly shown by the darker areas in the phase image (RHS Figures 4.20 and 4.21). And similar trend was observed when morphologies of polystyrene formed at high CTAB level (100  $\mu\text{mol/g}$ ) between Figure 4.22 and 4.23 were compared.

The effects of CTAB loading on morphologies of polystyrene formed on silica particles were also investigated. The morphologies of polystyrene on silica particles in the same ratio of styrene loading (1:0.5) but different CTAB levels were compared in Figure 4.20 (CTAB 20  $\mu\text{mol/g}$ ) and 4.24 (CTAB 100  $\mu\text{mol/g}$ ). When CTAB adsorption increased, area covered by the polystyrene films on silica particles also increased. This is clearly seen when the phase images of modified silica at styrene loading ratio 1:2 of different CTAB concentrations in Figure 4.21 (CTAB 20  $\mu\text{mol/g}$ ) and 4.23 (CTAB 100  $\mu\text{mol/g}$ ) were compared.

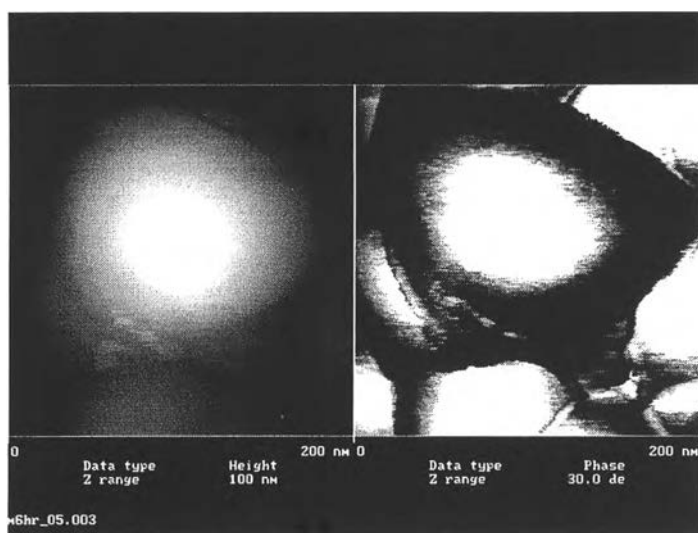
The AFM was also used to confirm that the extraction process was efficient in removing essentially the entire polymer formed. Figure 4.24 shows atomic force micrographs of modified silica before and after extraction. The result shows that the large polystyrene droplets/films which were visible on the unextracted silica surfaces were missing on the silica surfaces after extraction. This suggests that the polymer analysis is representative of all the polymer formed.



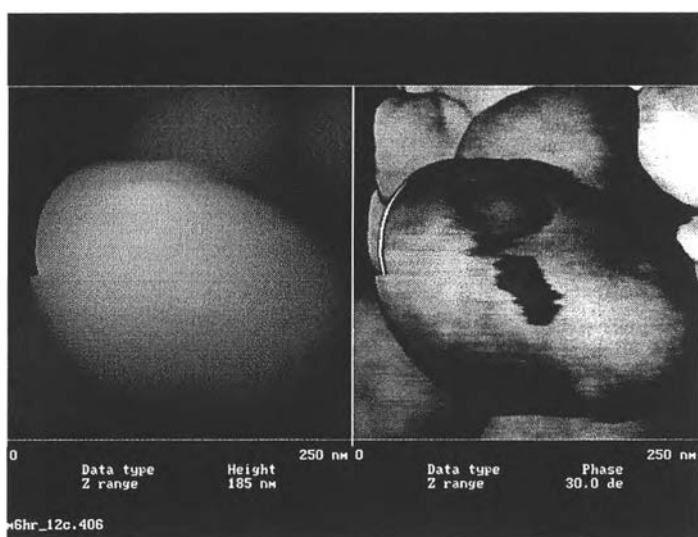
**Figure 4.20** Atomic force micrograph of modified silica prepared in CTAB  $20 \mu\text{mol/g}$  : adsolubilized styrene  $10 \mu\text{mol/g}$  (1:0.5) at 6 hours reaction time.



**Figure 4.21** Atomic force micrograph of modified silica prepared in CTAB  $20 \mu\text{mol/g}$  : adsolubilized styrene  $40 \mu\text{mol/g}$  (1:2) at 6 hours reaction time.

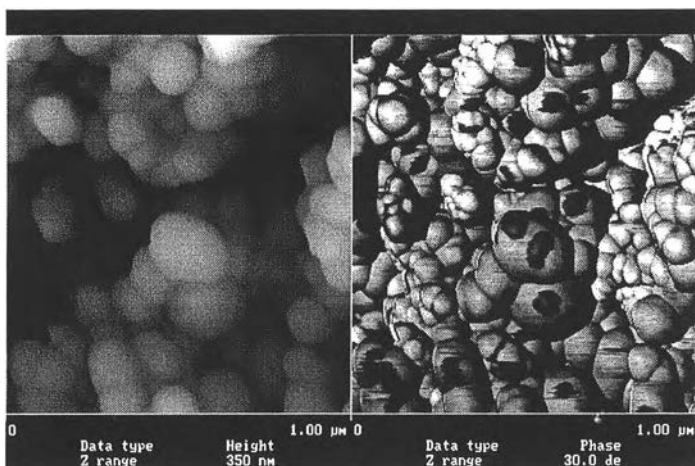


**Figure 4.22** Atomic force micrograph of modified silica prepared in CTAB 100  $\mu\text{mol/g}$  : adsolubilized styrene 50  $\mu\text{mol/g}$  (1:0.5) at 6 hours reaction time.

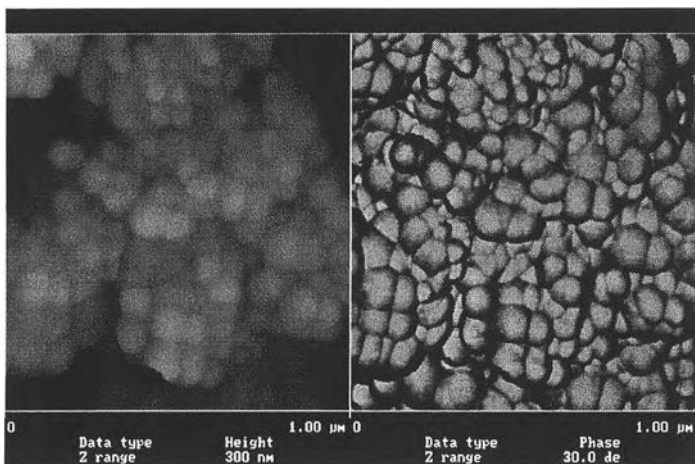


**Figure 4.23** Atomic force micrograph of modified silica prepared in CTAB 100  $\mu\text{mol/g}$  : adsolubilized styrene 200  $\mu\text{mol/g}$  (1:2) at 6 hours reaction time.





A. before extraction



B. after extraction

**Figure 4.24** Comparison of atomic force micrograph of modified silica before and after extraction.