

CHAPTER IV RESULTS AND DISCUSSION

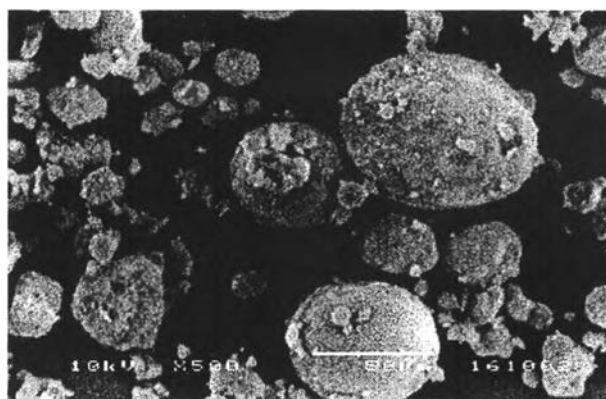
4.1 Surface Characterization of Modified Silicas

The admicellar polymerization process has been known to affect a variety of physical characteristics of silicas, including the morphology, BET surface area and mean agglomerate particle size of modified silicas. All samples were given a designation consisting of a number indicating the surfactant loadings (146, 175 and 200 g of surfactant) per kg silica, and a letter representing the initiator loadings (0.4, 0.8 and 1.65 g of initiator) is denoted by A, B and C, respectively.

4.1.1 Morphology of Modified Silicas

The scanning electron micrographs of the unmodified silica and modified silicas with different in surfactant and initiator loadings are shown in Figures 4.1 to 4.4.

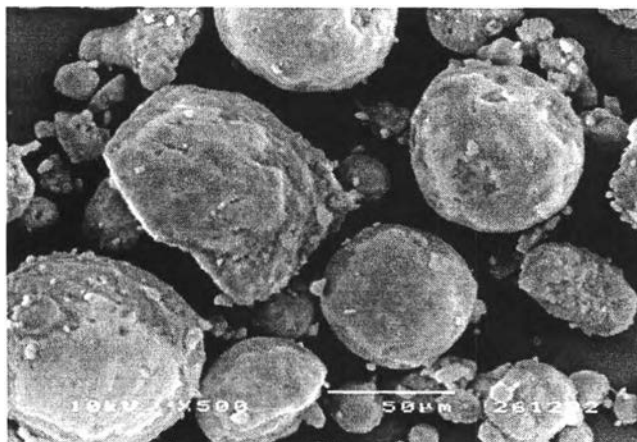
By comparing the unmodified and modified silica micrographs, the particle size of any modified silica increased after the modification. The surfaces of the modified silica samples appeared rough when compared with the surface of the unmodified silica. The polymerization of the co-monomers styrene-isoprene on the silica surface results in the rough appearance, possibly due to agglomeration of particles following modification.



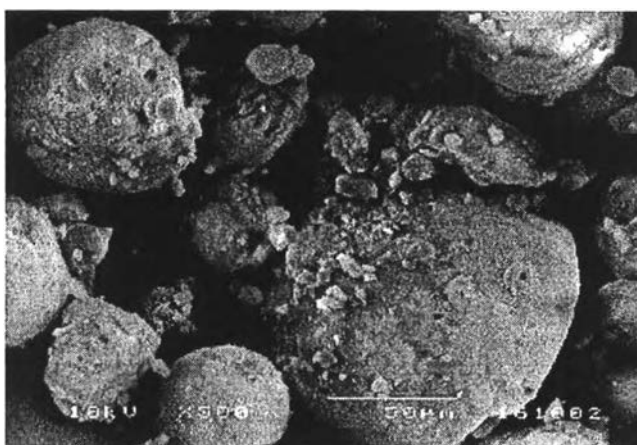
————— 50 μ m

Figure 4.1 Scanning electron micrograph of unmodified silica, Hi-Sil[®]255.

A. 146/A



B. 146/B



C. 146/C

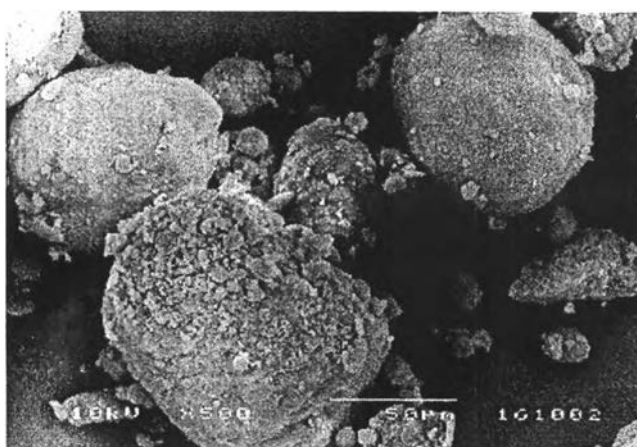
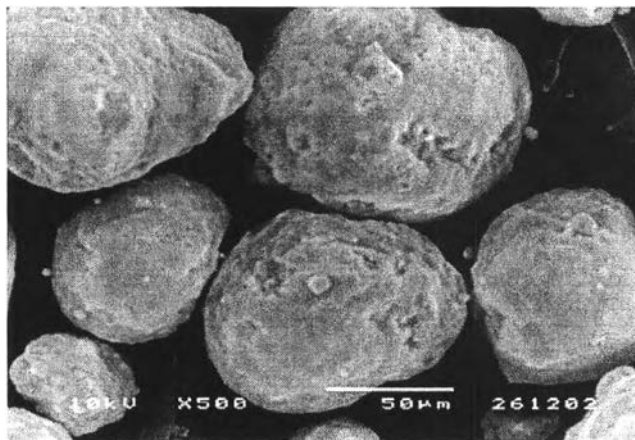
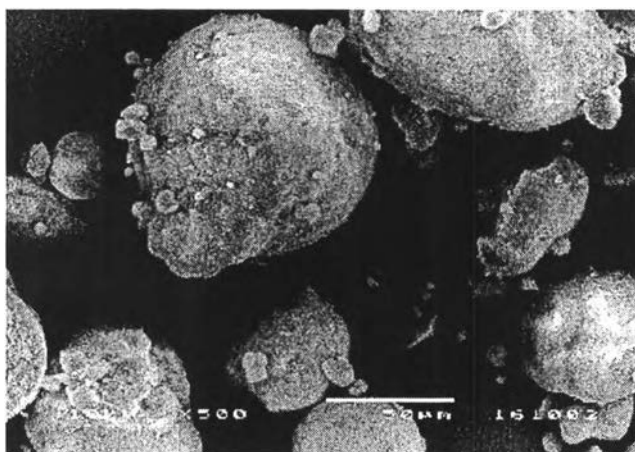


Figure 4.2 Scanning electron micrographs of modified silica, 146 g of surfactant loading and three initiator loadings, at 500X magnification.

A. 175/A



B. 175/B



C. 175/C

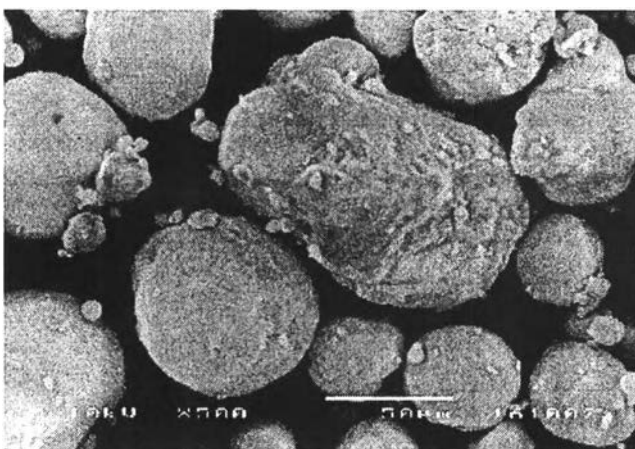
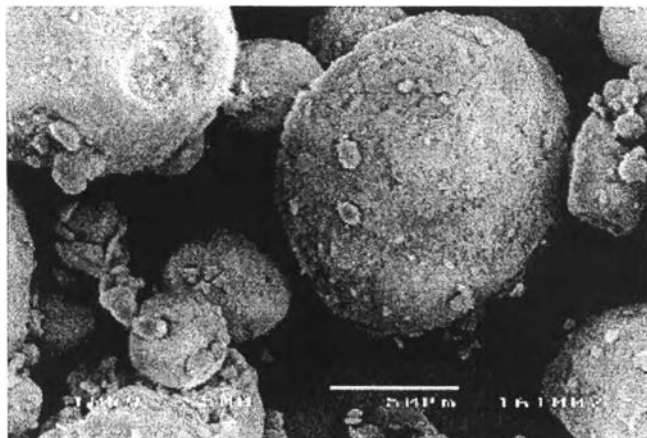
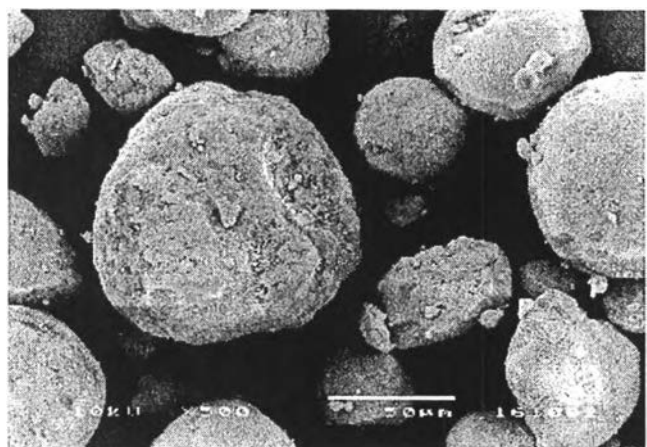


Figure 4.3 Scanning electron micrographs of modified silica, 175 g of surfactant loading and three initiator loadings, at 500X magnification.

A. 200/A



B. 200/B



C. 200/C

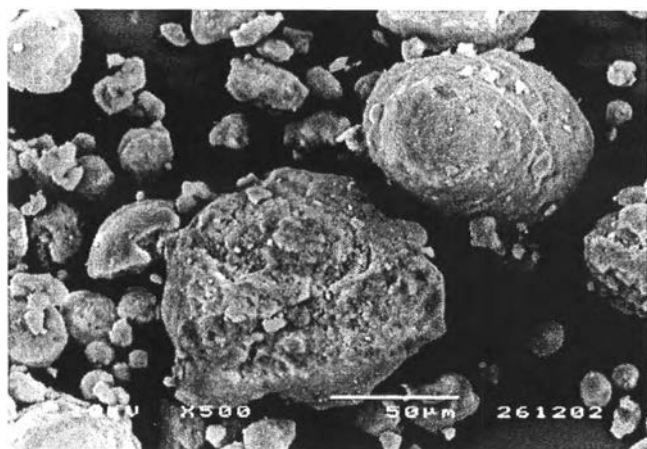


Figure 4.4 Scanning electron micrographs of modified silica, 200 g of surfactant loading and three initiator loadings, at 500X magnification.

4.1.2 BET Surface Area

All modified silica samples had the reduced The BET nitrogen surface areas of all modified silicas, some by as much as 50% (Figure 4.5). The change in surface area is most likely due to the blocking of the micropores in the precipitated silica by the formed polymer. As can be seen from the figure, all modified silica, except 200/A, made only a slightly difference in BET surface area values. Comparing the present result with the results of Nontasorn (2002) at the same surfactant and initiator loadings, modified silica (200/C) has a lower BET surface area than the one previously found.

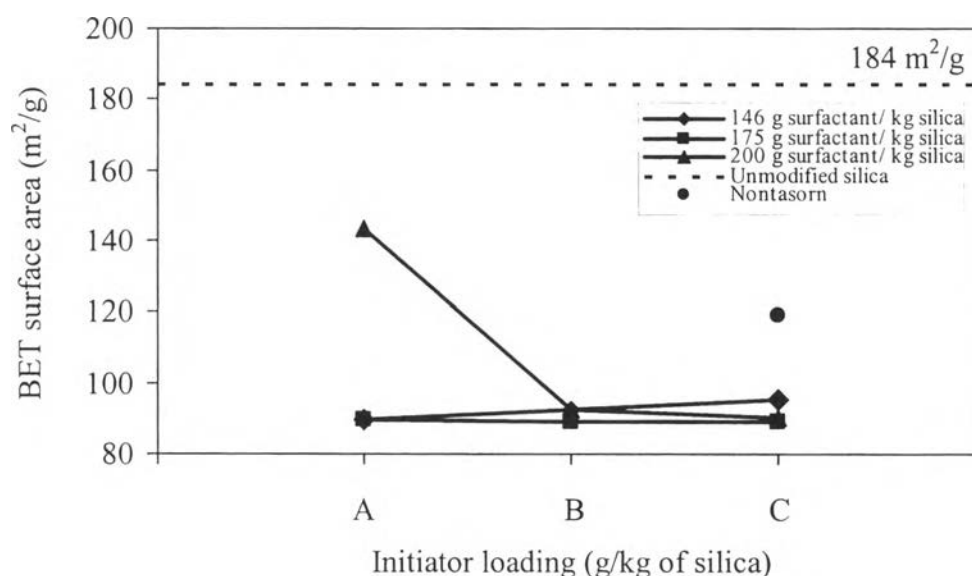


Figure 4.5 BET surface areas of modified silicas as a function of surfactant and initiator loadings.

4.1.3 Mean Agglomerate Particle Size

The increase in mean agglomerate particle size may simply be attributed to the subsequent reprocessing of the modified silicas, it could be a result of the organic polymer-forming process. Figure 4.6 shows the effect of surfactant and initiator loadings on the mean agglomerate particle size. As seen from the figure, the mean agglomerate particle size of all modified silicas increased with increasing initiator loading. The increase in the mean agglomerate particle size may result from the development of polymer bridges between silica particles

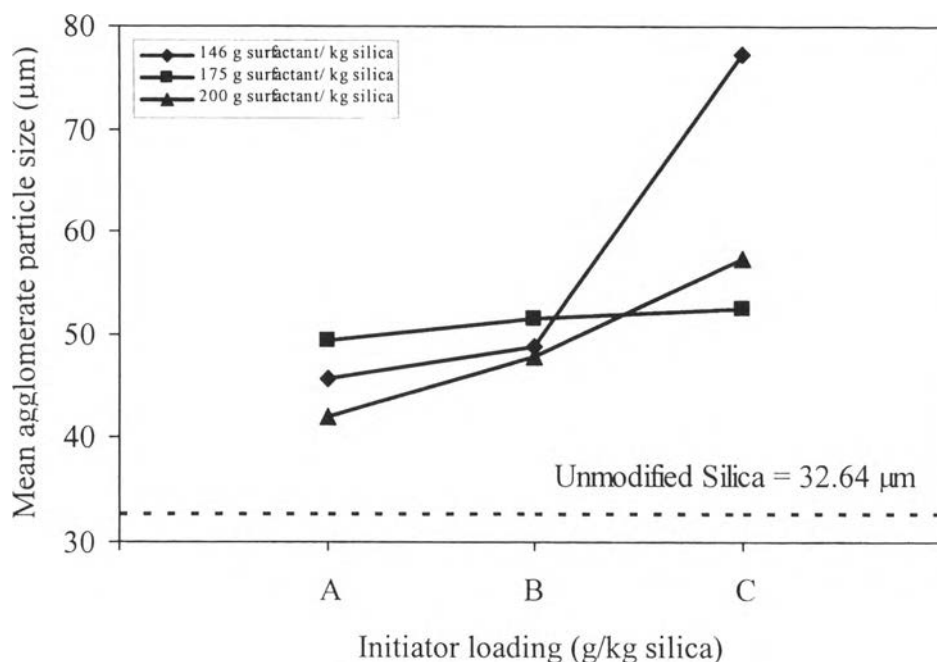


Figure 4.6 Mean agglomerate particle size of modified silicas as a function of surfactant and initiator loadings.

(Chaisirimahamorakot, 2001). The effect of surfactant loading on the mean agglomerate particle size was pronounced than that of initiator loading. The largest mean agglomerate particle size was found at the highest initiator loading and the lowest surfactant loading.

4.1.4 Verification of the Existence of polymeric Material on Silica

All samples were examined by thermogravimetric analysis in order to verify the existence of poly(styrene-isoprene) forming on the silica surfaces.

The water loss from the unmodified silica below 150°C is shown in Figure 4.7. Consequently, the weight change above 150°C must be the result of the surface modification of modified silicas.

Figure 4.8 shows the decomposition of CTAB between 200 to 300°C. Interestingly, for CTAB adsorbed onto the silica, there are two major peaks of weight loss; the first peak from 170 to 300°C and the second peak from 300 to 450°C, as shown in Figures 4.9. The second peak of the weight loss is likely resulted from the stronger bonding between silica and CTAB.

All samples were prepared by depositing polystyrene dissolved with THF onto the silica surface in order to predict the decomposition temperature of poly(styrene-isoprene) of the modified silicas. The decomposition of polystyrene from 350 to 480°C is shown in Figure 4.10. Poly(styrene-isoprene) is decomposed from the admicellar polymerization modified silica as shown in Figure 4.11. The graphs evidently show the decomposition of CTAB takes place between 200 to 280°C and 300 to 450°C while that of the polymer is started from 280 to 400°C.

Figures 4.12 to 4.20 show the TGA results of modified silicas before and after the THF extraction in order to predict the decomposition of poly(styrene-isoprene) of modified silicas. The results show a two-step decomposition process, CTAB deformation is decomposed at 150 to 300°C in the first-step and the second one is CTAB chemisorbed on the silica at 300 to 400°C. Figure 4.21 shows the amount of % carbon of polymer of modified silicas in numerous of surfactant and initiator loadings. It shows the result related with the BET surface area result. As shown in Table 4.1, all modified silica provided the polymer formed on the surface. In order to calculate the amount of polymer formed, the CTAB decomposition amount is calculated from the first weight drop, and this is deducted from the weight loss at the second. Table 4.2 shows we cannot extract all polymer on the silica surface indicating that there is a significant amount of poly(styrene-isoprene) locating in the microporous structure of the silica. O'Haver and co-workers (1994) found that not all polymers could be extracted from the modified silica.

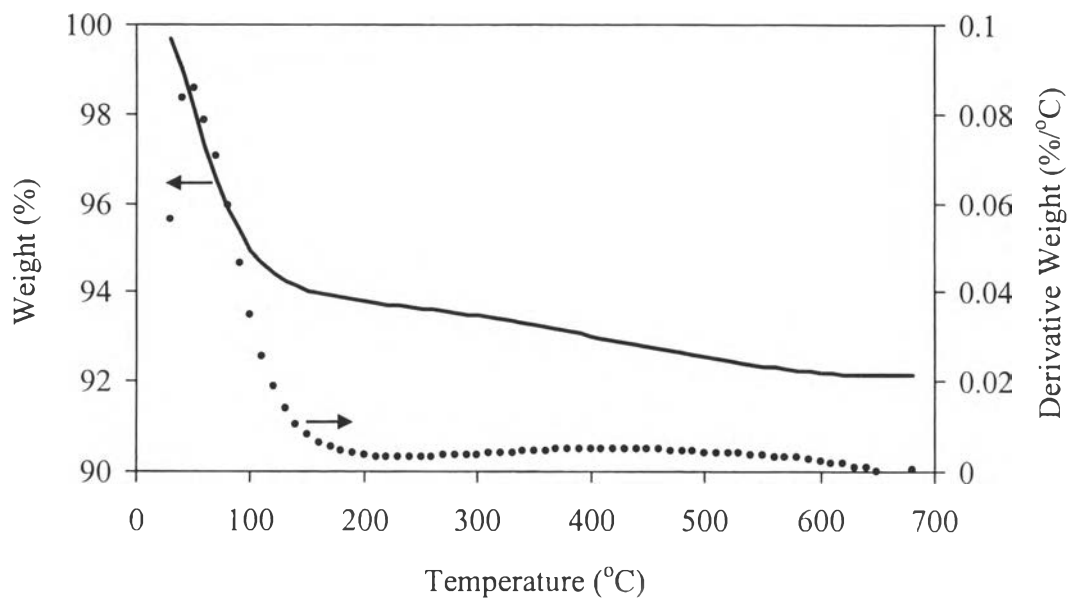


Figure 4.7 TGA results of unmodified silica Hi-Sil[®]255.

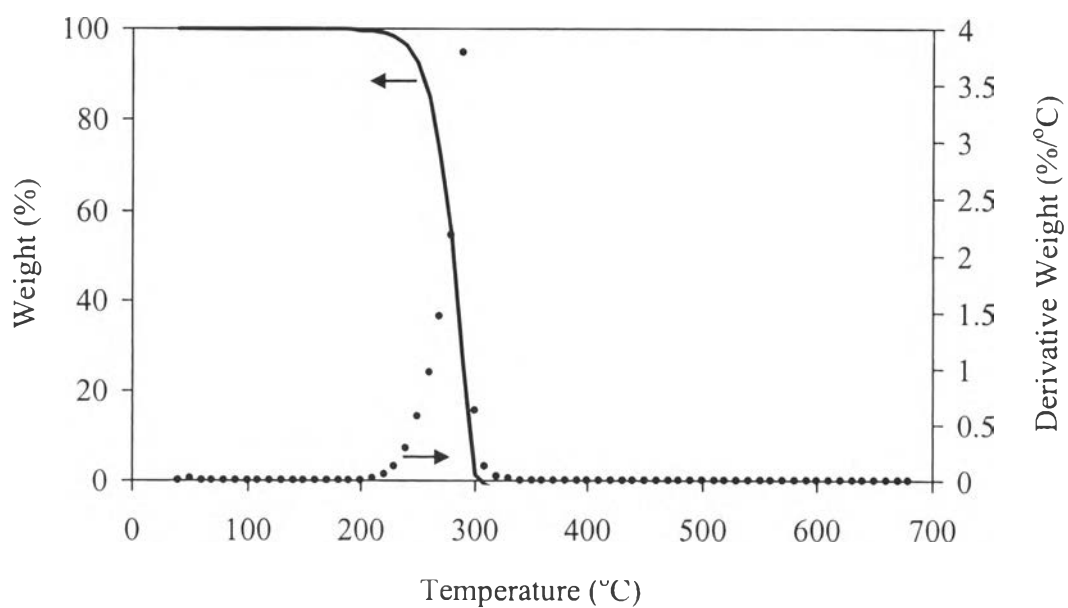


Figure 4.8 TGA results of Hexadecyltrimethylammonium bromide (CTAB).

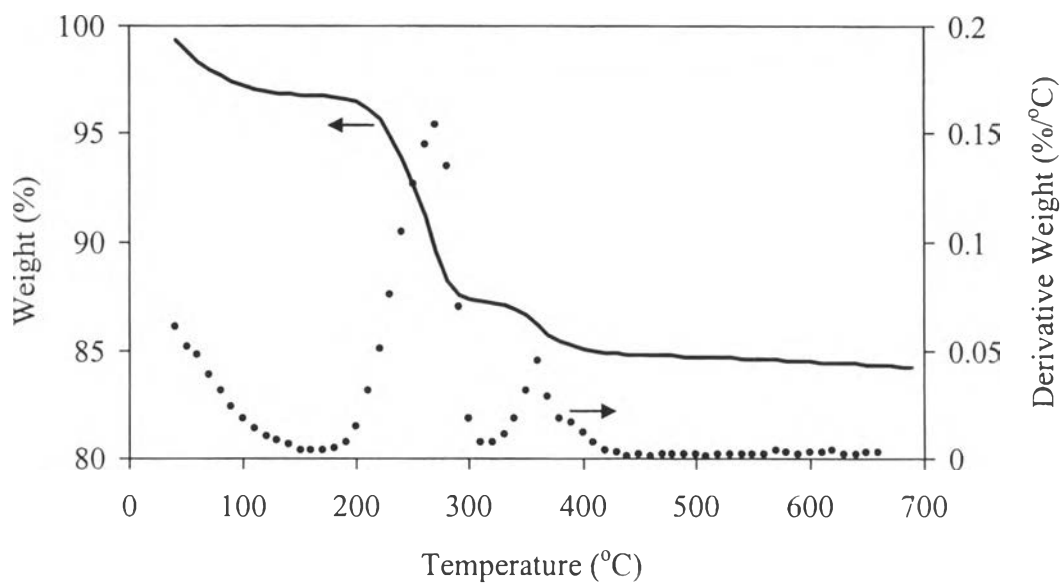


Figure 4.9 TGA results of silica Hi-Sil[®]255 adsorbed with CTAB (Chaisirimahamorakot, 2001).

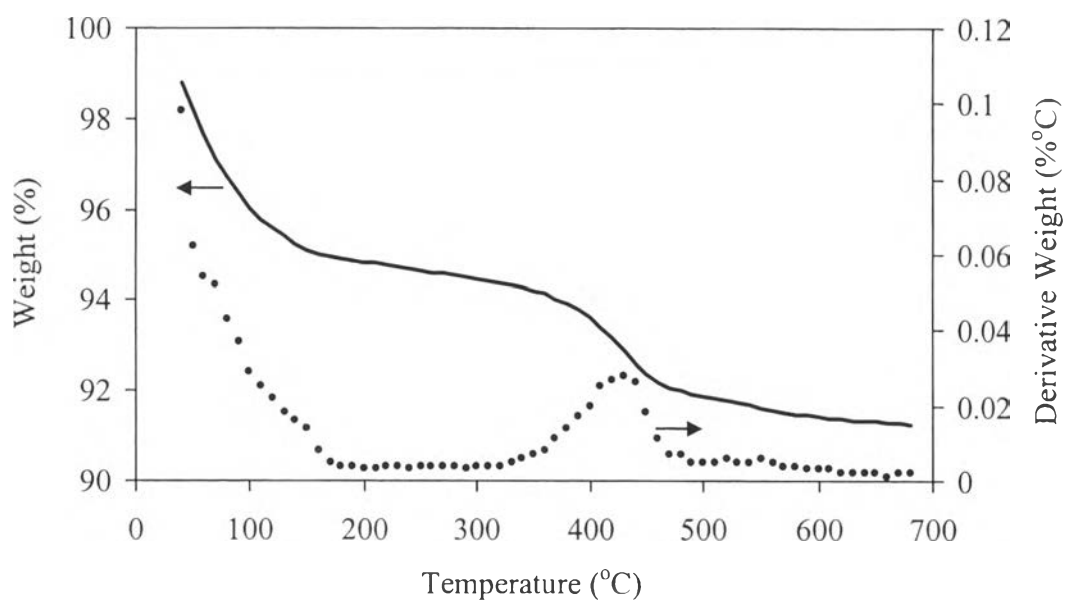


Figure 4.10 TGA results of silica Hi-Sil[®]255 adsorbed with polystyrene (Chaisirimahamorakot, 2001).

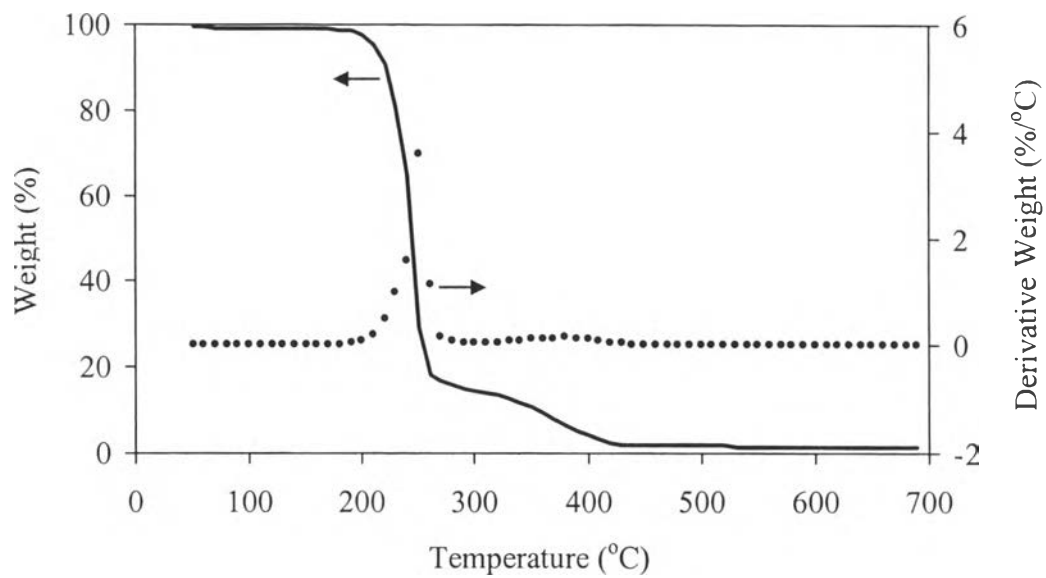
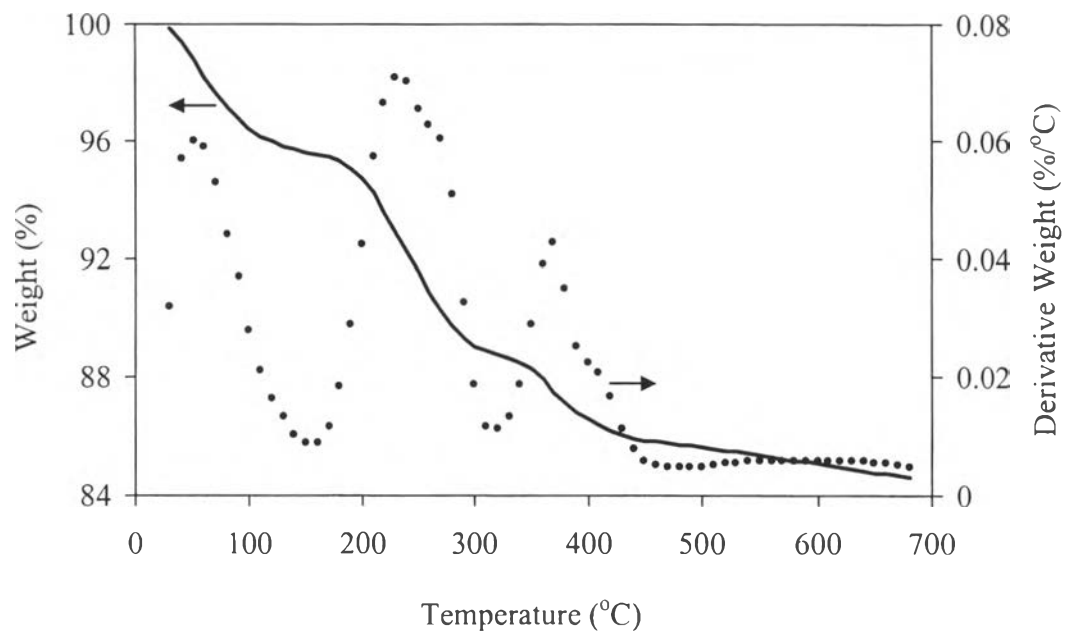
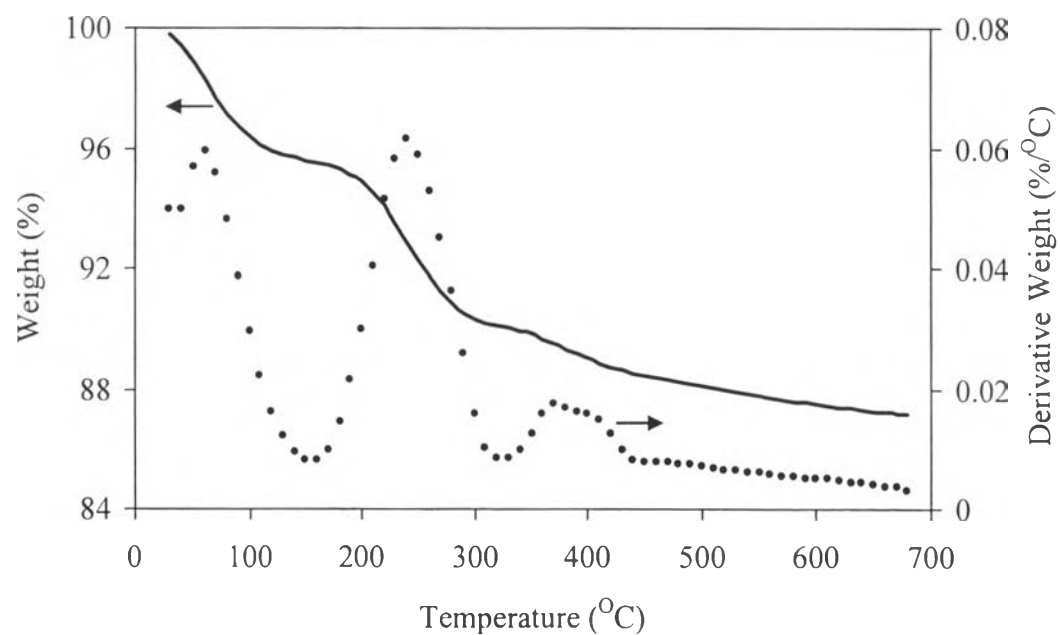


Figure 4.11 TGA results of poly(styrene-isoprene) polymerized in CTAB (Chaisirimahamorakot, 2001).

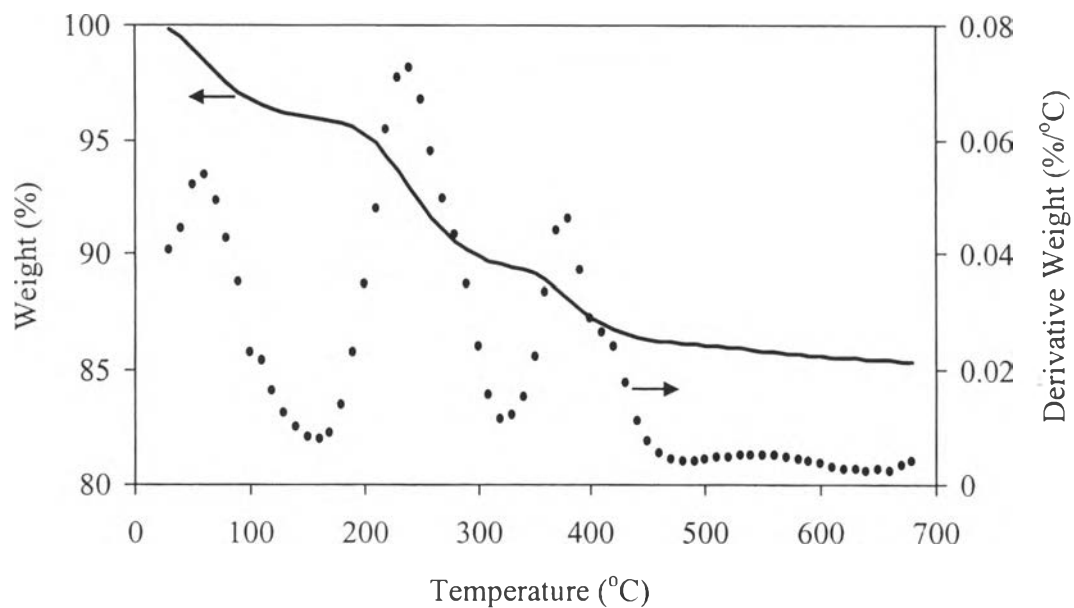
A. Modified silica before extraction



B. Modified silica after extraction

**Figure 4.12** TGA results of modified silica 146/A.

A. Modified silica before extraction



B. Modified silica after extraction

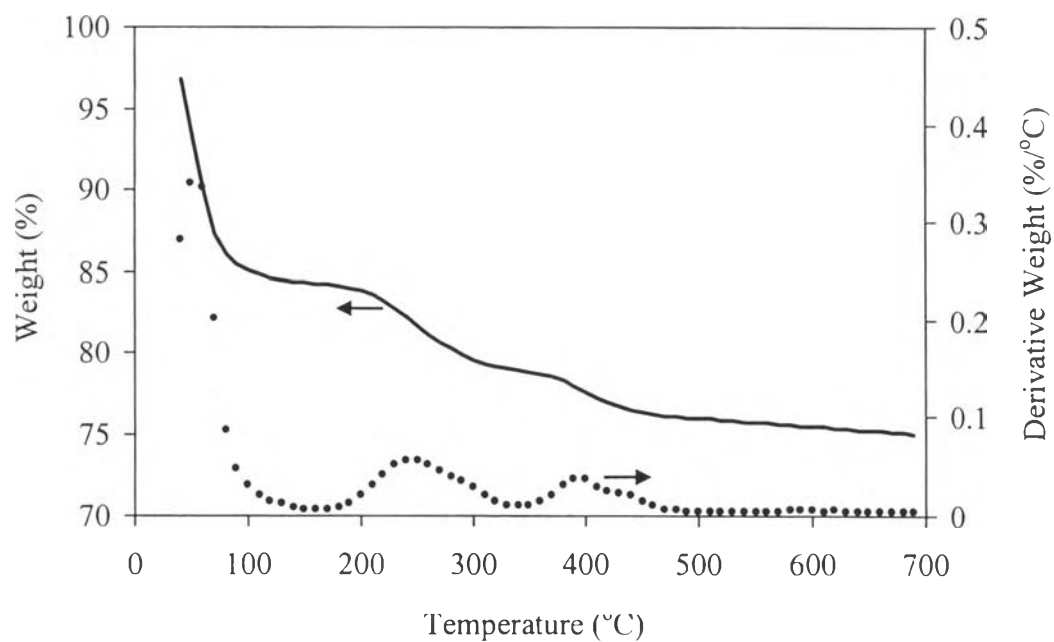
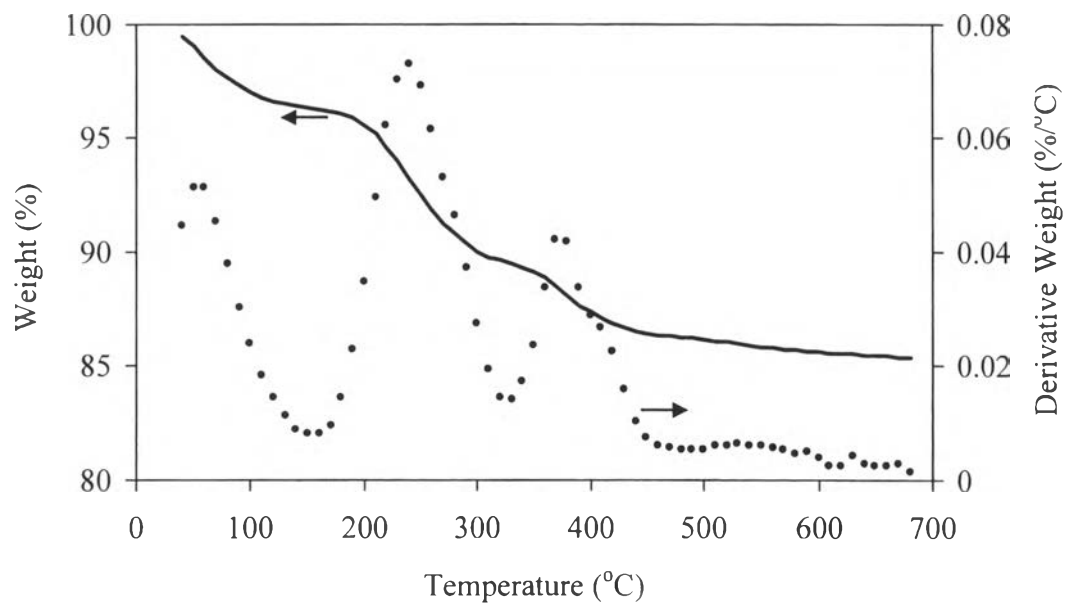


Figure 4.13 TGA results of modified silica 146/B.

A. Modified silica before extraction



B. Modified silica after extraction

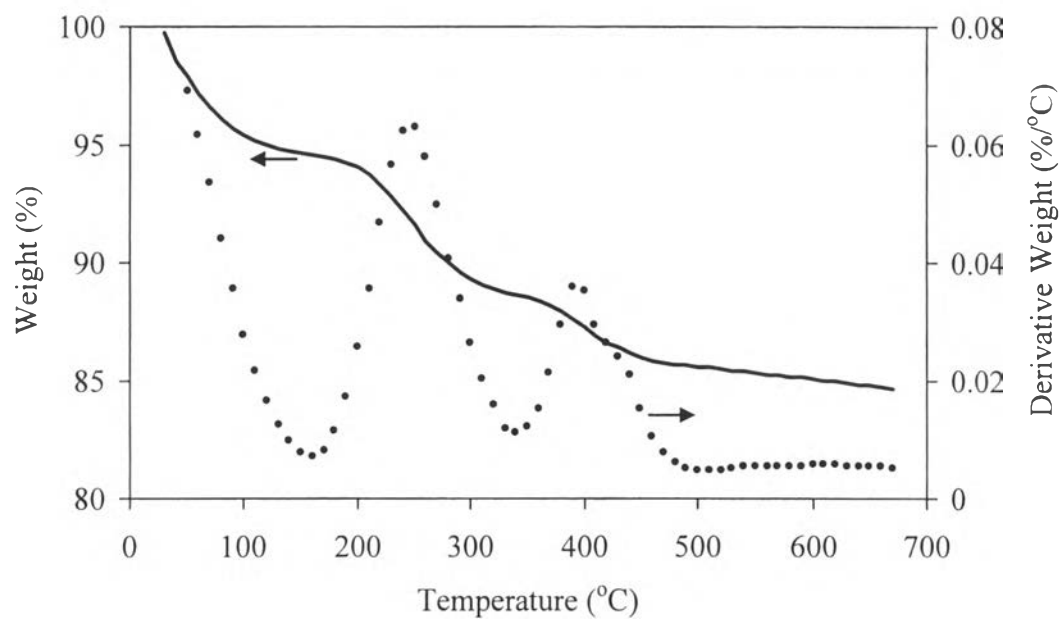
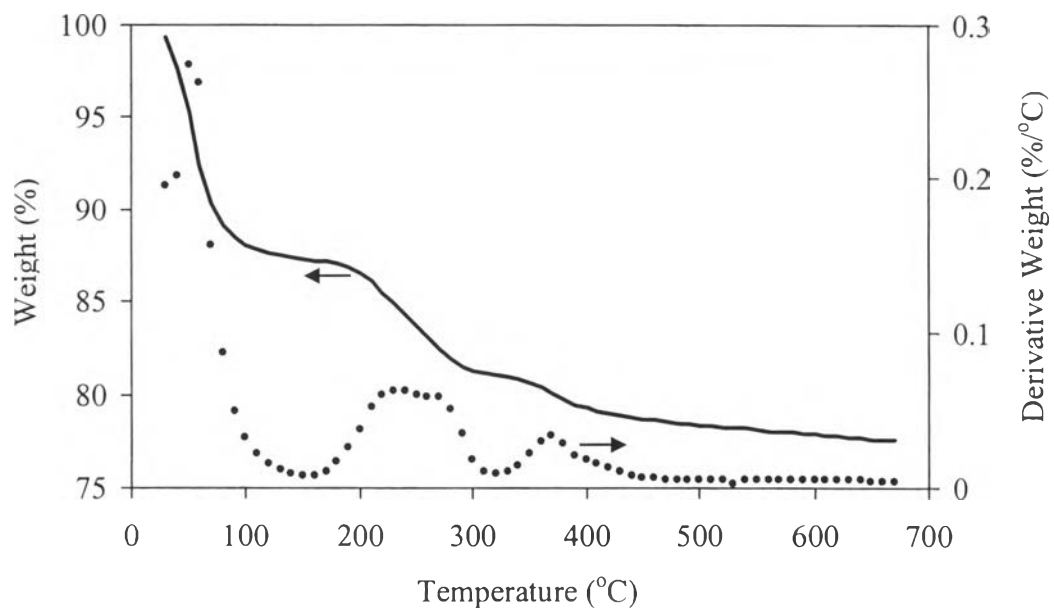
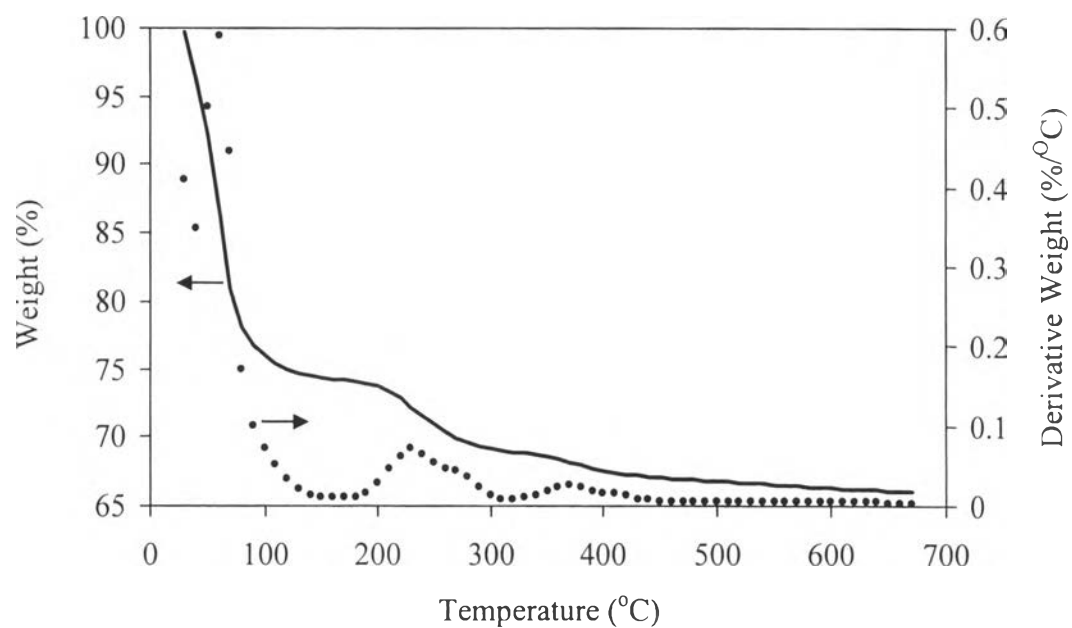


Figure 4.14 TGA results of modified silica 146/C.

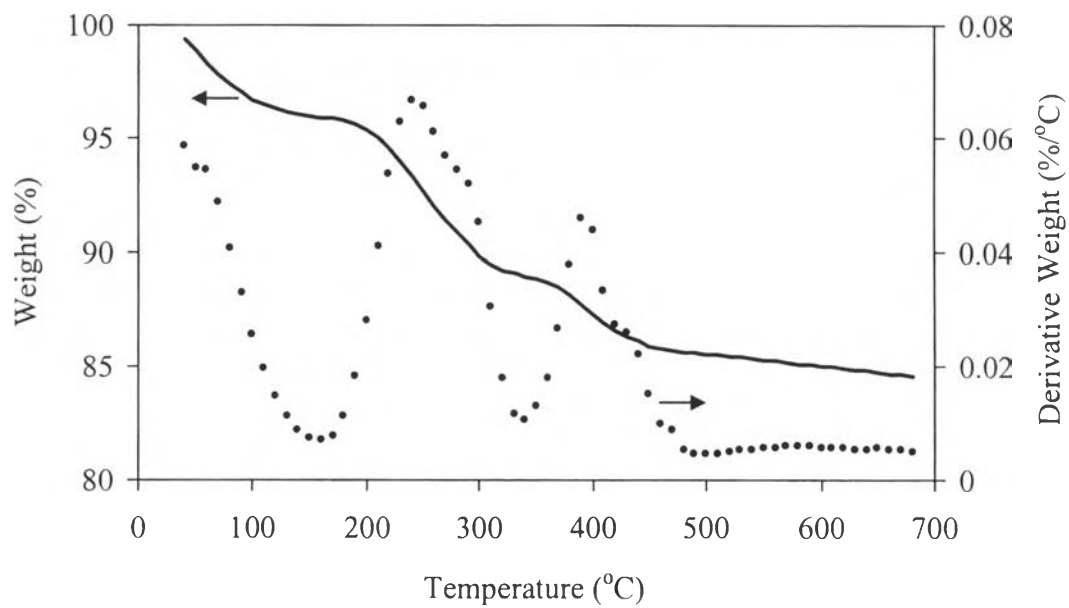
A. Modified silica before extraction



B. Modified silica after extraction

**Figure 4.15** TGA results of modified silica 175/A.

A. Modified silica before extraction



B. Modified silica after extraction

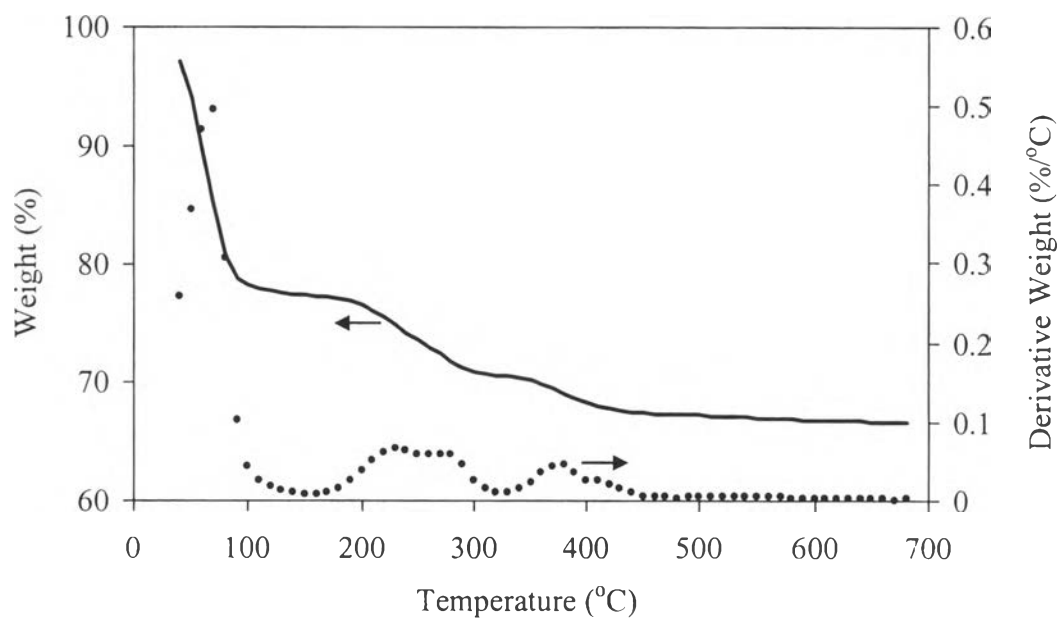
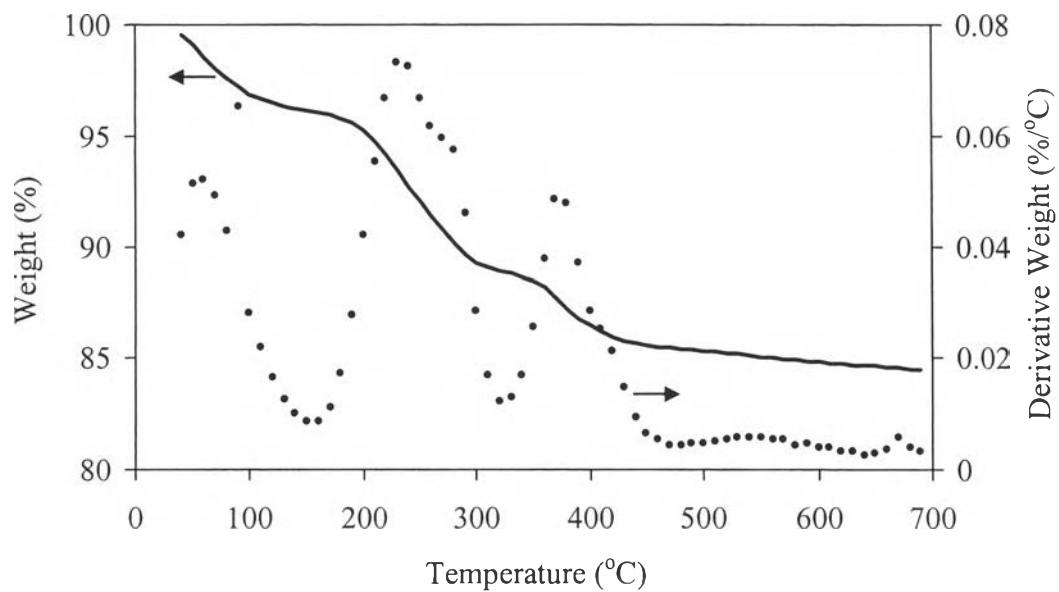


Figure 4.16 TGA results of modified silica 175/B.

A. Modified silica before extraction



B. Modified silica after extraction

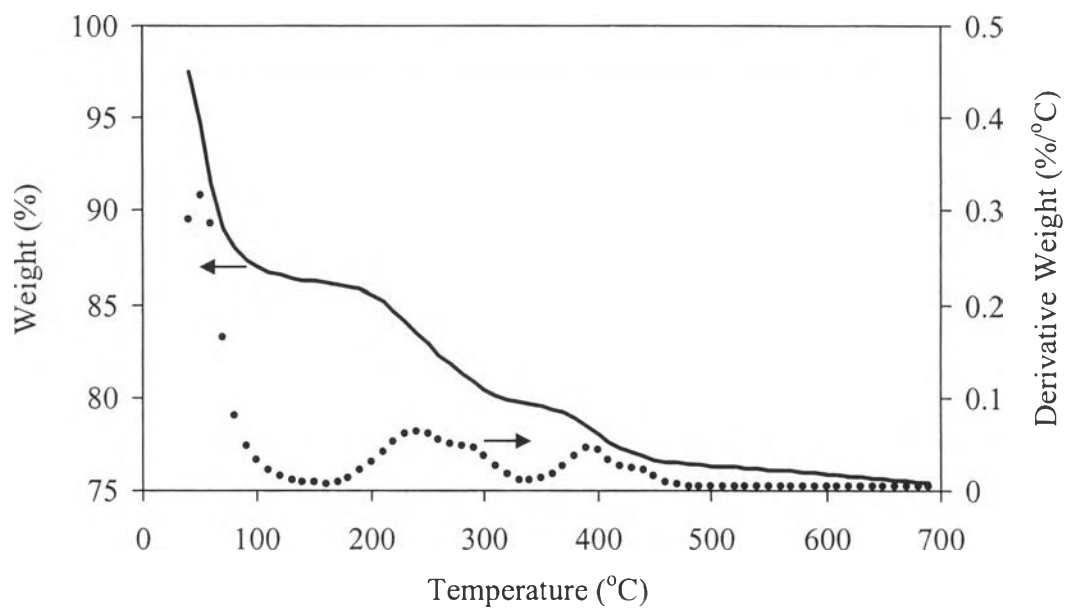
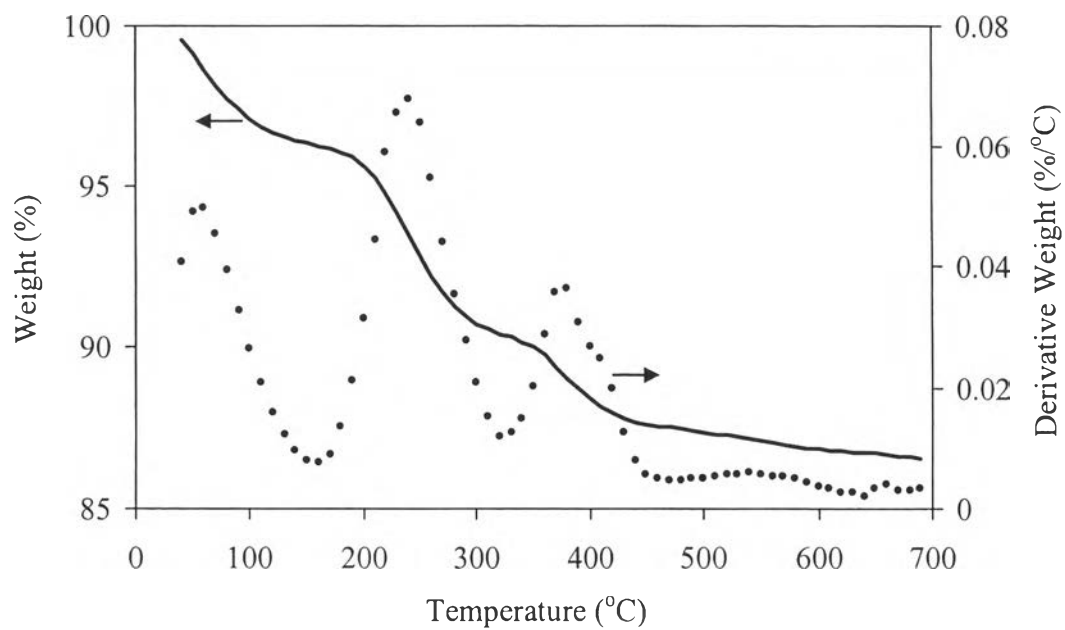
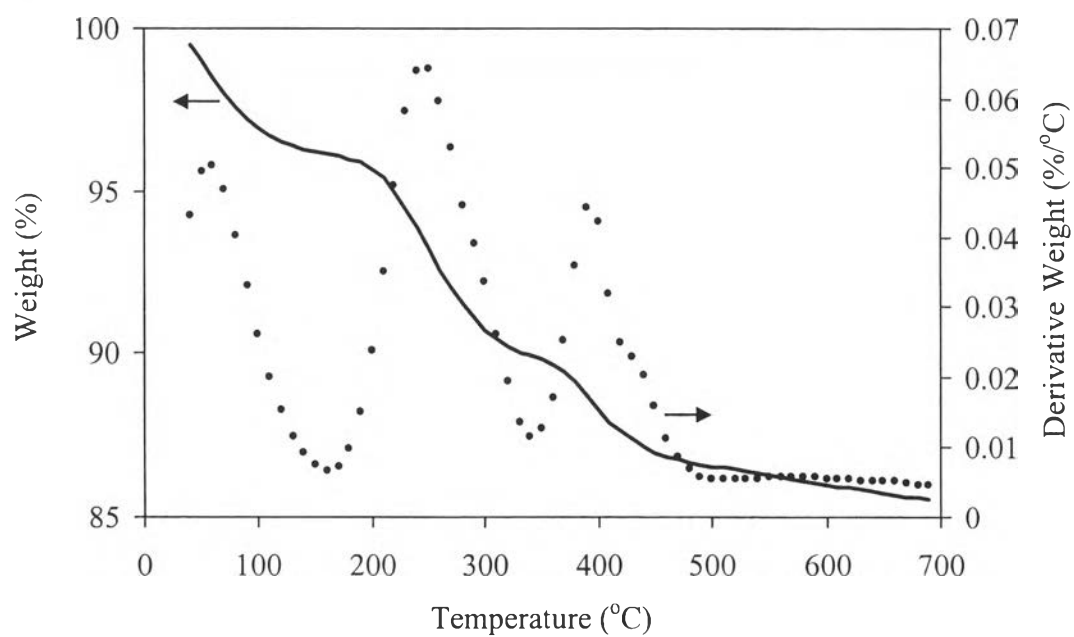


Figure 4.17 TGA results of modified silica 175/C.

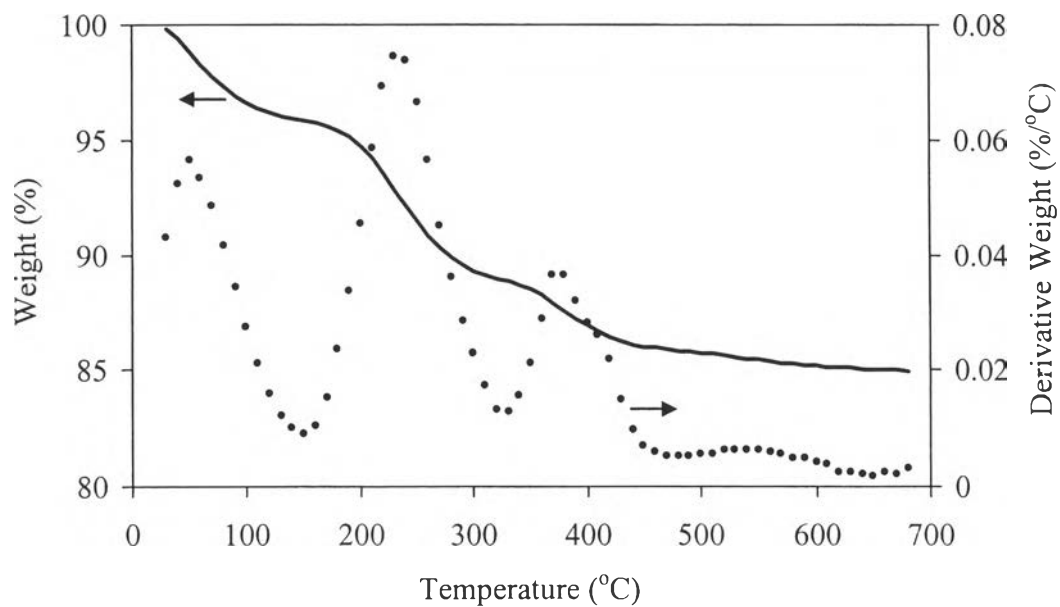
A. Modified silica before extraction



B. Modified silica after extraction

**Figure 4.18** TGA results of modified silica 200/A.

A. Modified silica before extraction



B. Modified silica after extraction

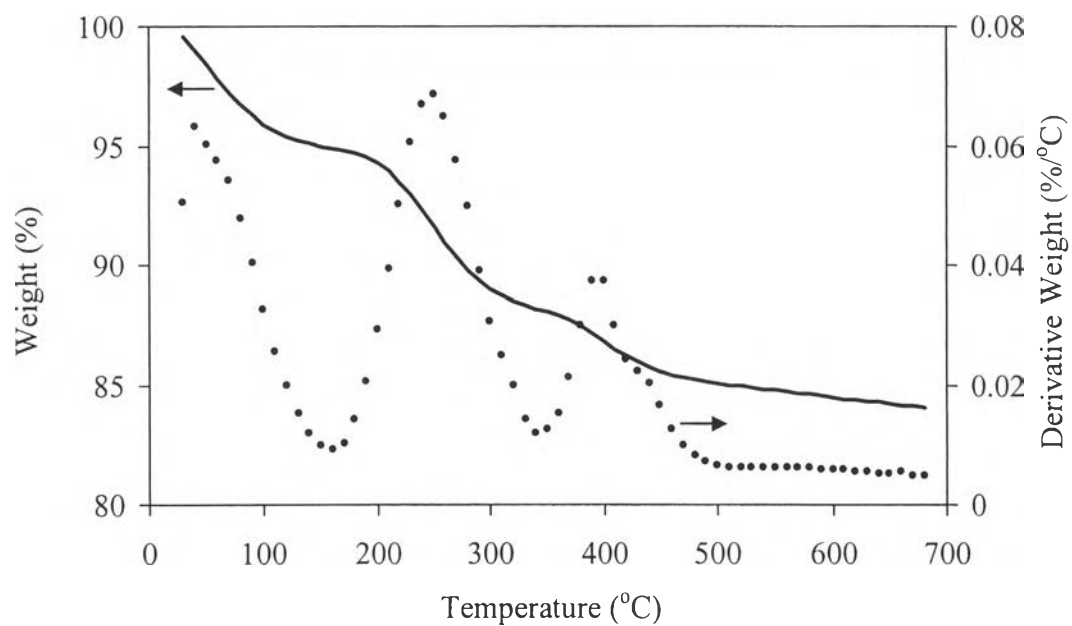
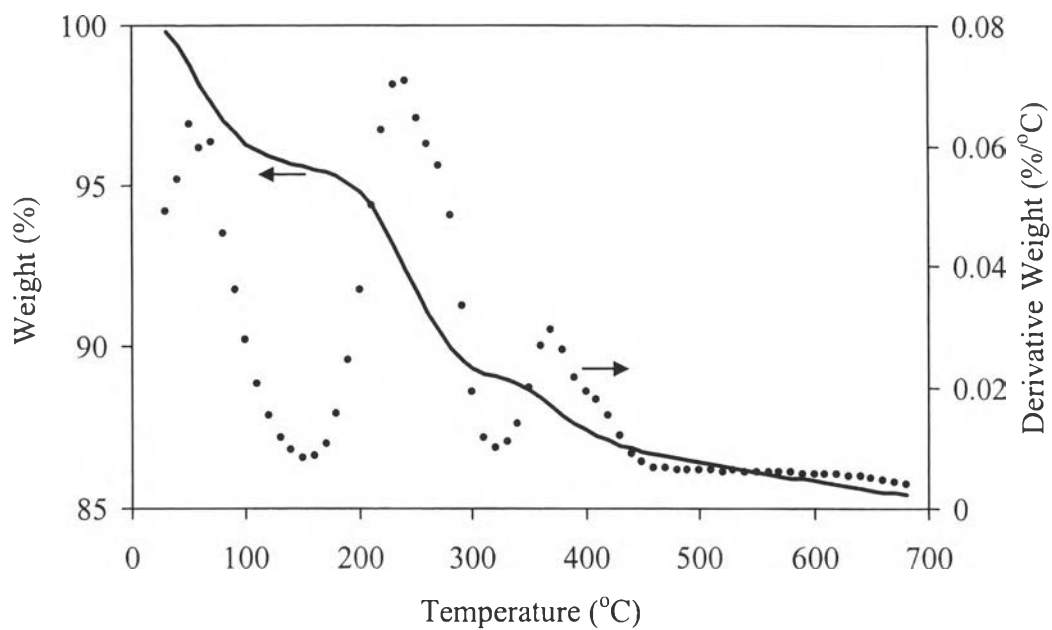
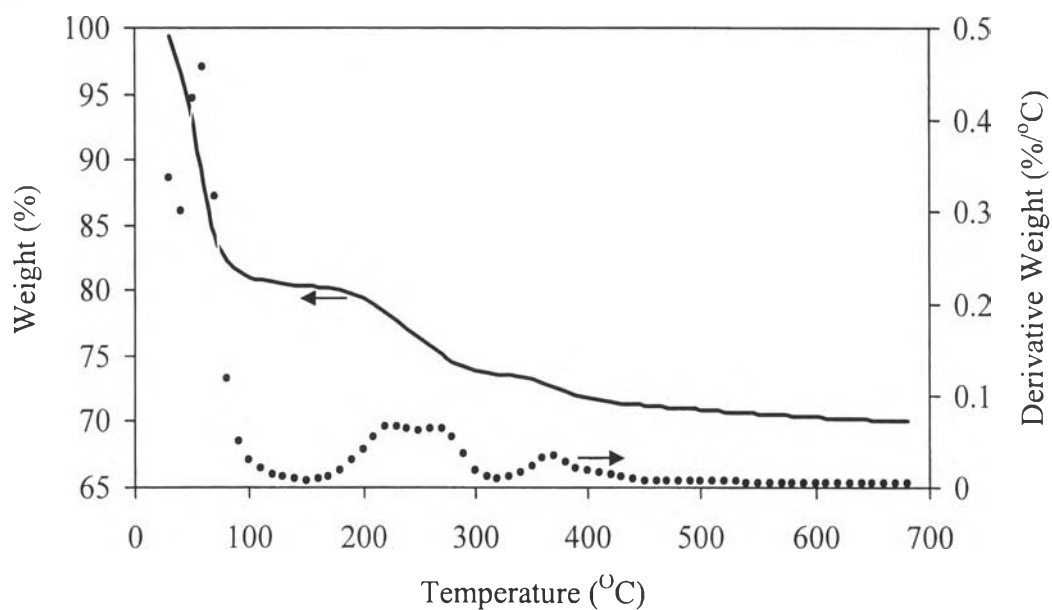


Figure 4.19 TGA results of modified silica 200/B.

A. Modified silica before extraction



B. Modified silica after extraction

**Figure 4.20** TGA results of modified silica 200/C.

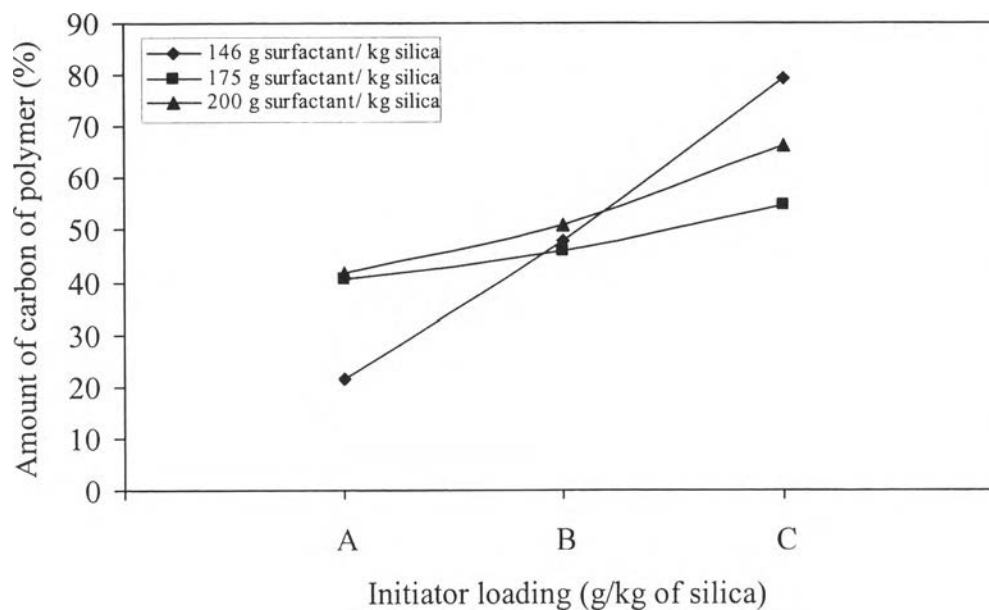


Figure 4.21 The amount of %carbon of polymer of modified silicas as a function of surfactant and initiator loadings.

Table 4.1 Amount of polymer on the silicas before and after the THF extraction

Sample	TGA				
	Surfactant Loading (g/kg silica)	Initiator Loading* (g/kg silica)	Amount of carbon of polymer (%)**		% Extracted Polymer
			Before THF Extraction	After THF Extraction	
146		A	0.347	0.133	21.43
		B	0.900	0.422	47.80
		C	0.880	0.086	79.37
175		A	0.543	0.137	40.64
		B	1.045	0.584	46.06
		C	0.681	0.133	54.82
200		A	0.927	0.508	41.89
		B	0.593	0.085	50.83
		C	0.761	0.098	66.28

* A = 0.4 g, B = 0.8 g, C = 1.65 g

** with respect to the weight loss of SI_CTAB

The apparent decrease in surface area of modified silica probably results from blocking of some of the micro-pores in the precipitated silica by the organic polymer

formed. The % extracted polymer evidence as shown in Table 4.1 supports the presence of polymer on the silica.

4.3.4 Verification of the Existence of Poly(styrene-isoprene)

The result of the FT-IR spectrum of Hi-Sil[®]255 (unmodified silica) is shown in Figure 4.22. Figure 4.23 shows the FT-IR spectrum of CTAB. The extracted material and the silicas after the extraction were analyzed by FT-IR to confirm the existence of poly(styrene-isoprene) on the silica surface and the spectra compared to the standard reference spectrum of poly(styrene-*co*-isoprene) (Aldrich 18, 292-3) (Figure 4.24). The functional group of benzene ring at the wave number of 700 cm^{-1} proves the existence of styrene while aliphatic carbon double bond (C=C) at a number of 1600 cm^{-1} proves the presence of isoprene. Peaks at a number of 1200 and 3600 cm^{-1} show the existence of amino group of CTAB and silicon dioxide. All extracted materials and polystyrene spectra are shown in Figures 4.25 to 4.33. The spectra provide a support of the existence of polystyrene on the silica surface. The inability to fully extract the polymer is quite confidently embedded in the silica pores.

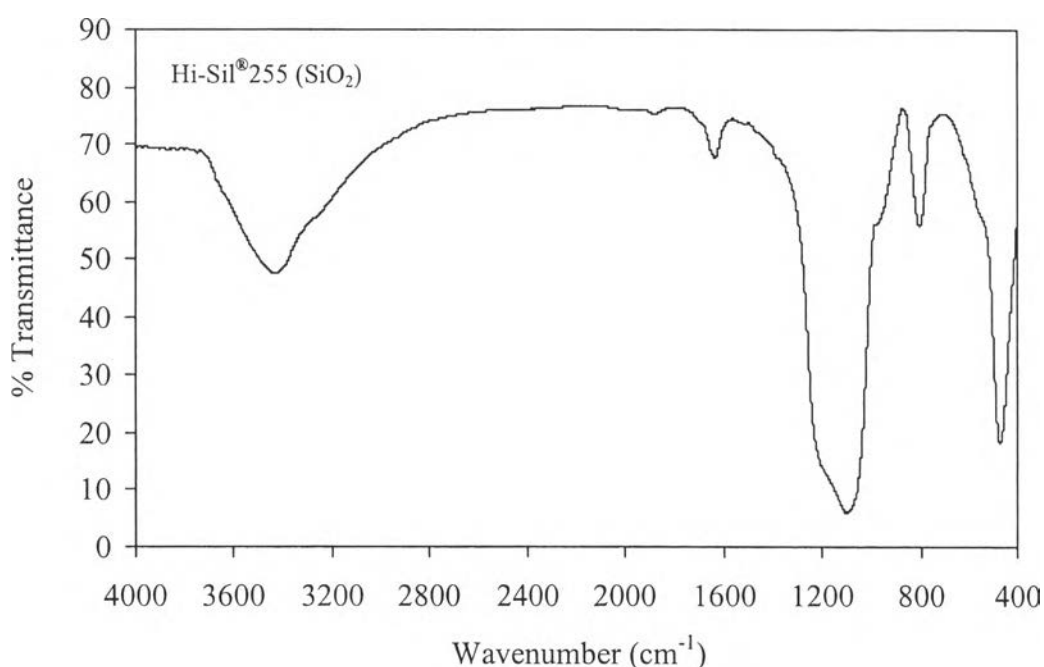


Figure 4.22 FT-IR spectrum of unmodified silica (SiO₂).

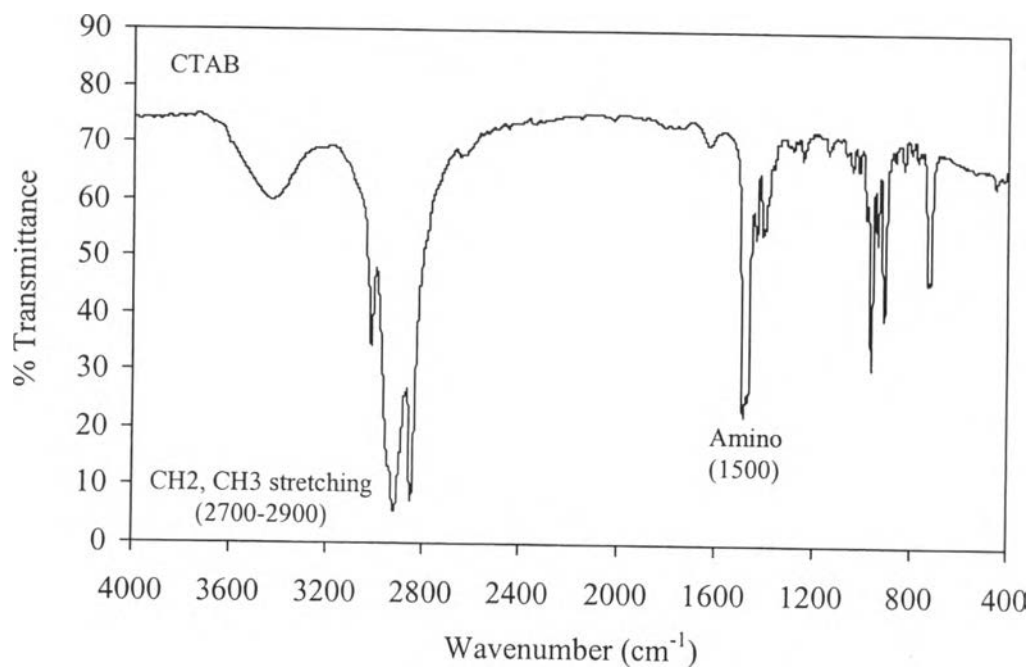


Figure 4.23 FT-IR spectrum of Hexadecyltrimethylammonium bromide (CTAB).

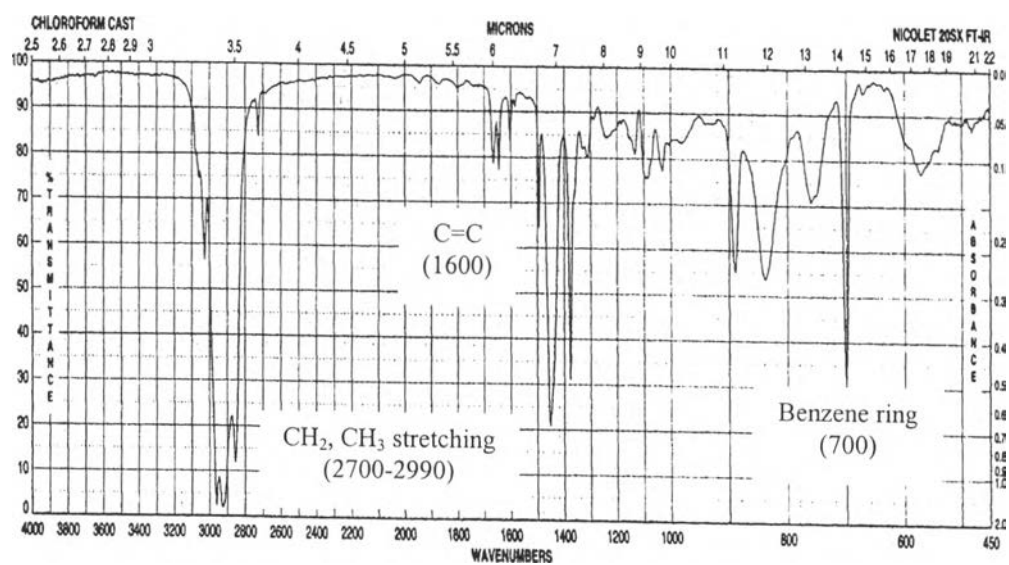


Figure 4.24 FT-IR spectrum of Poly(styrene-co-isoprene), ABA block copolymer from Aldrich 18, 292-3.

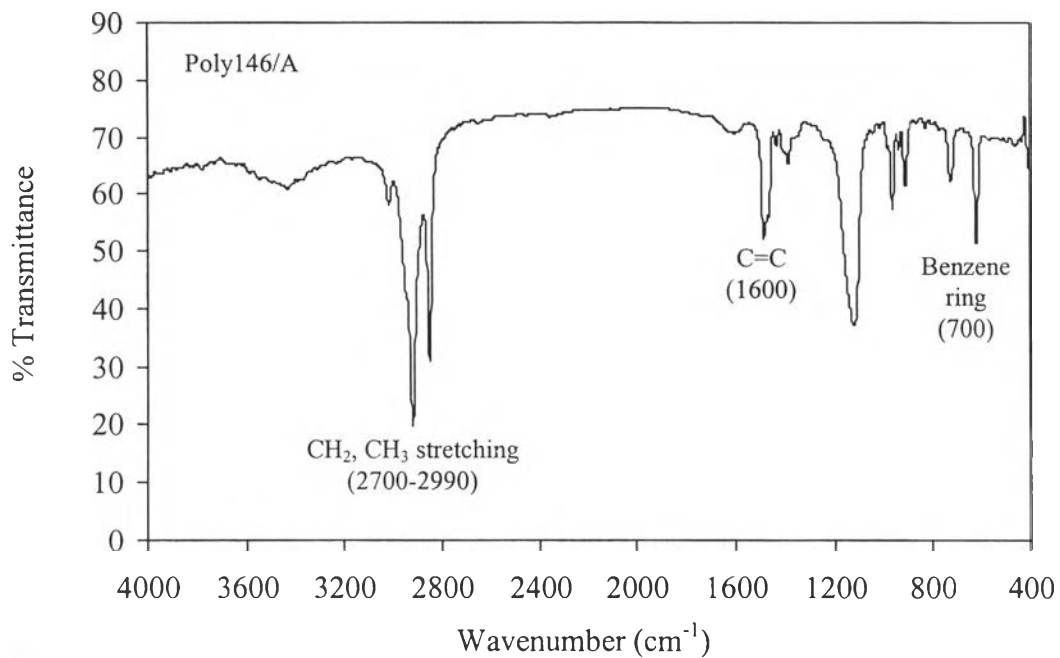


Figure 4.25 FT-IR spectrum of the extracted material (poly146/A).

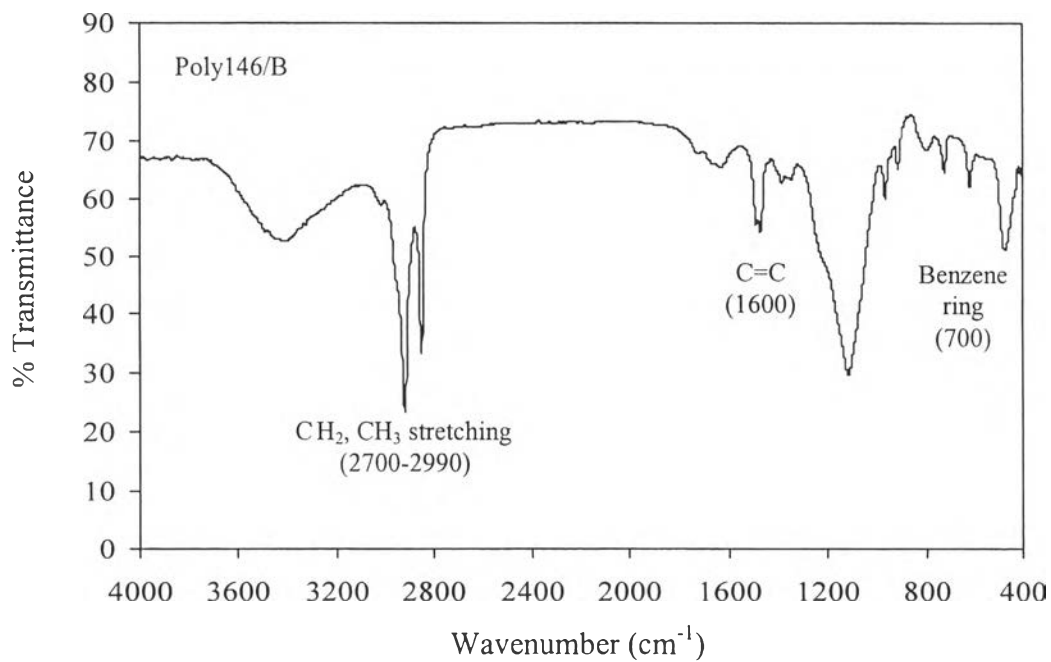


Figure 4.26 FT-IR spectrum of the extracted material (poly146/B).

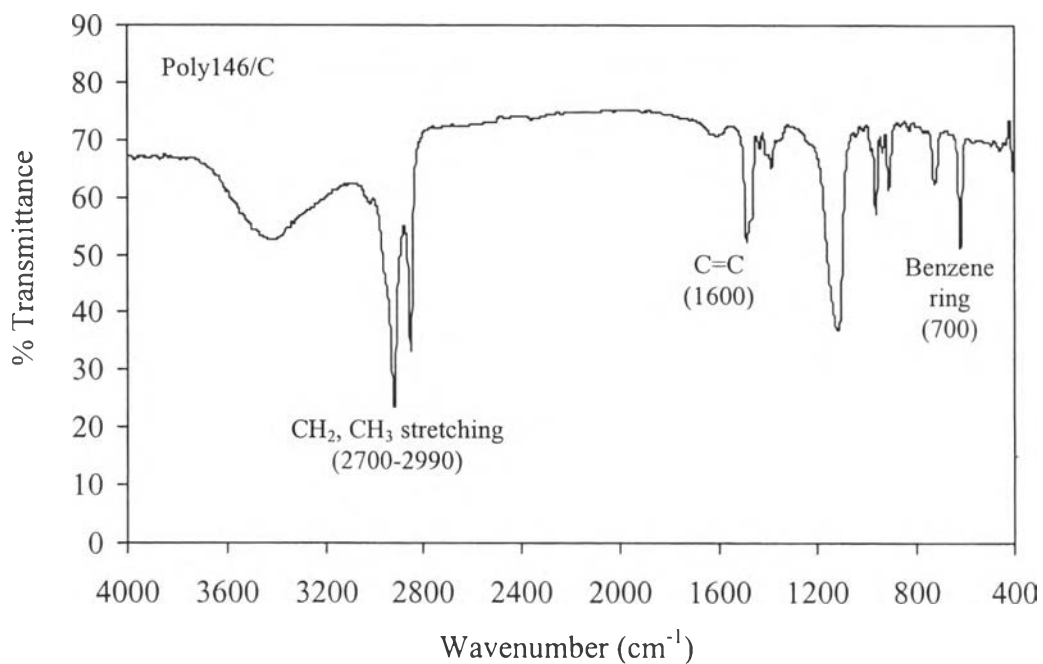


Figure 4.27 FT-IR spectrum of the extracted material (poly146/C).

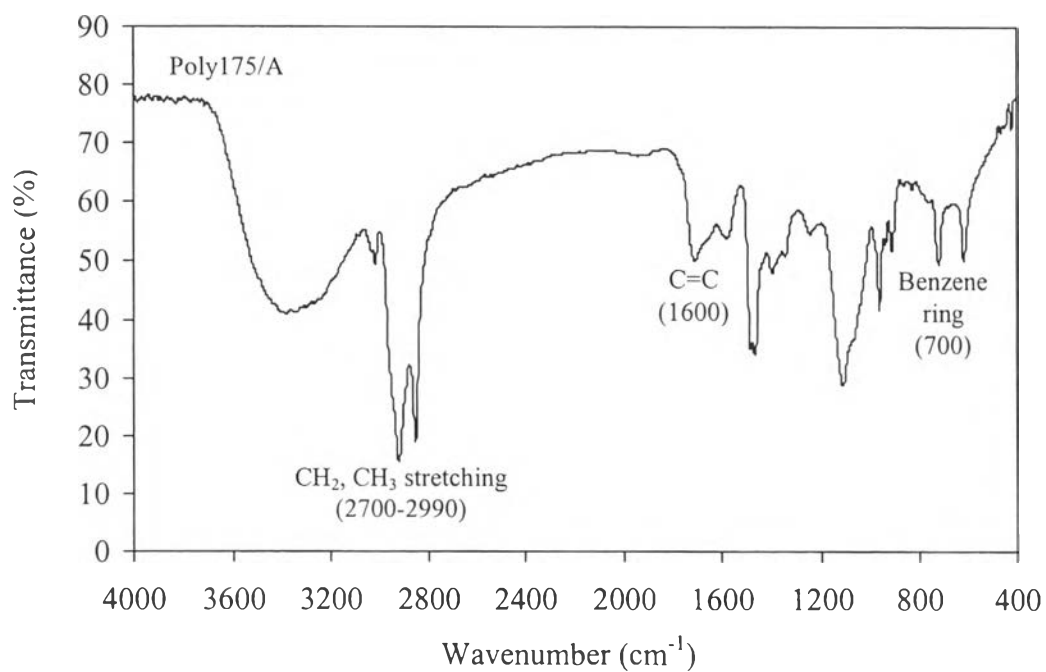


Figure 4.28 FT-IR spectrum of the extracted material (poly175/A).

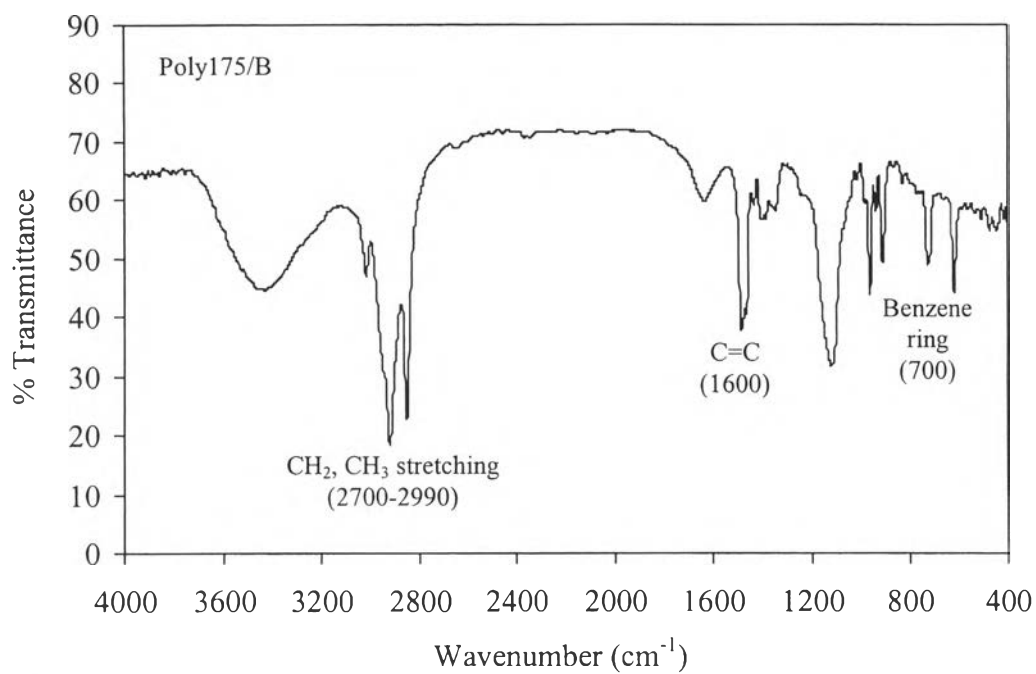


Figure 4.29 FT-IR spectrum of the extracted material (poly175/B).

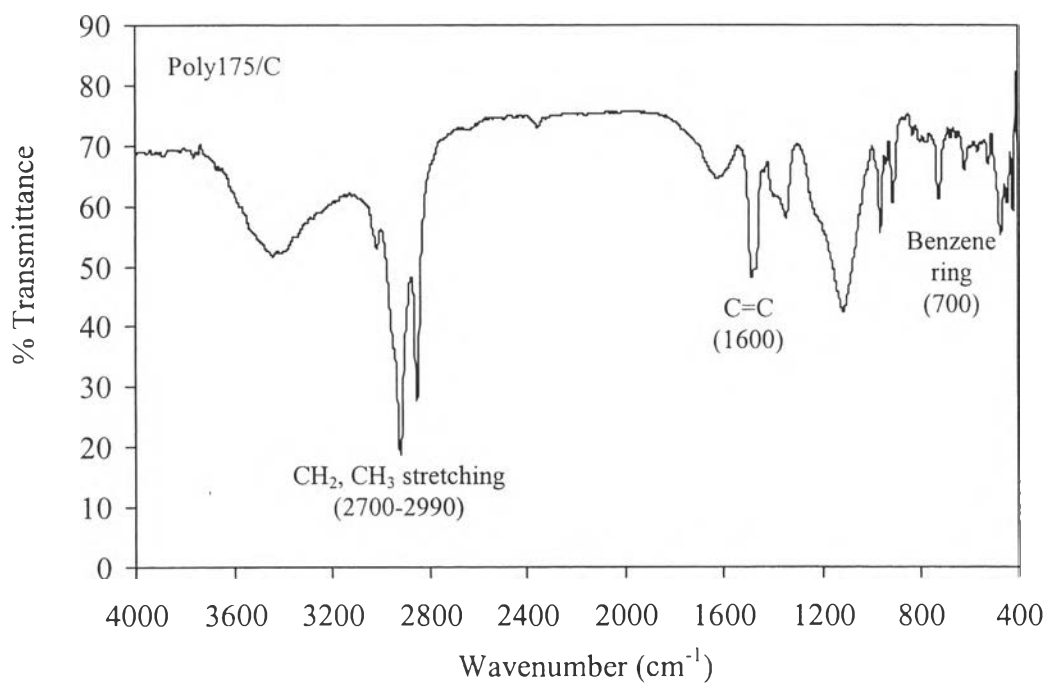


Figure 4.30 FT-IR spectrum of the extracted material (poly175/C).

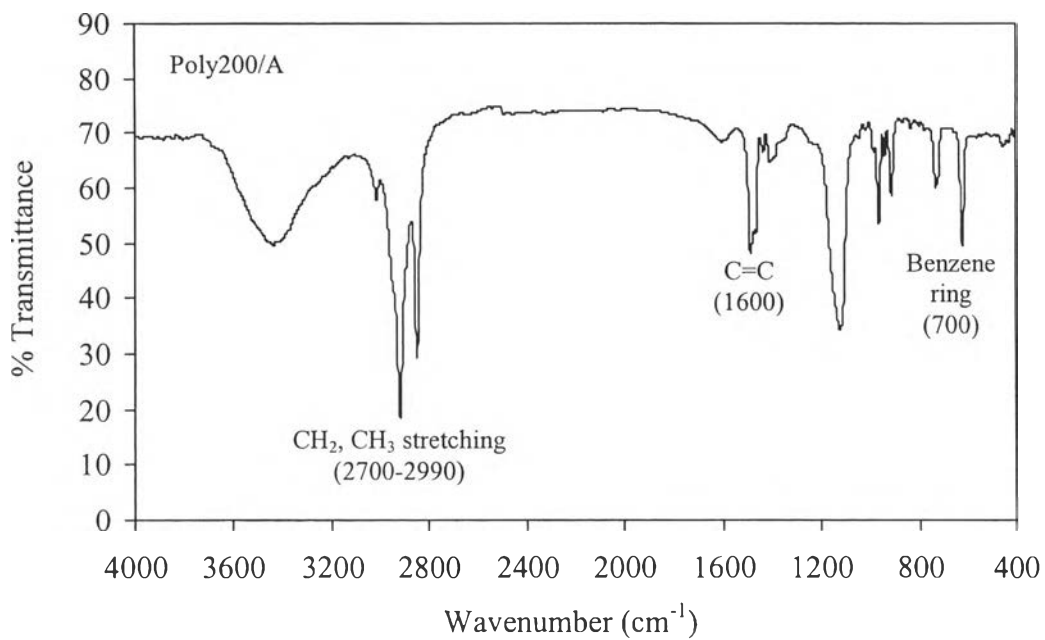


Figure 4.31 FT-IR spectrum of the extracted material (poly200/A).

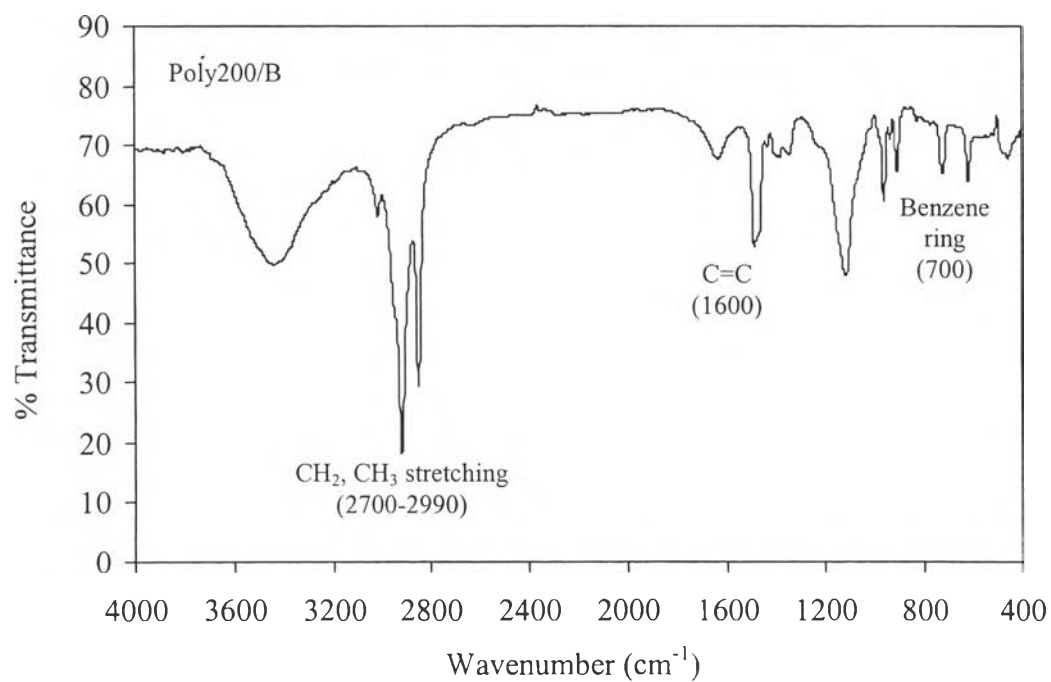


Figure 4.32 FT-IR spectrum of the extracted material (poly200/B).

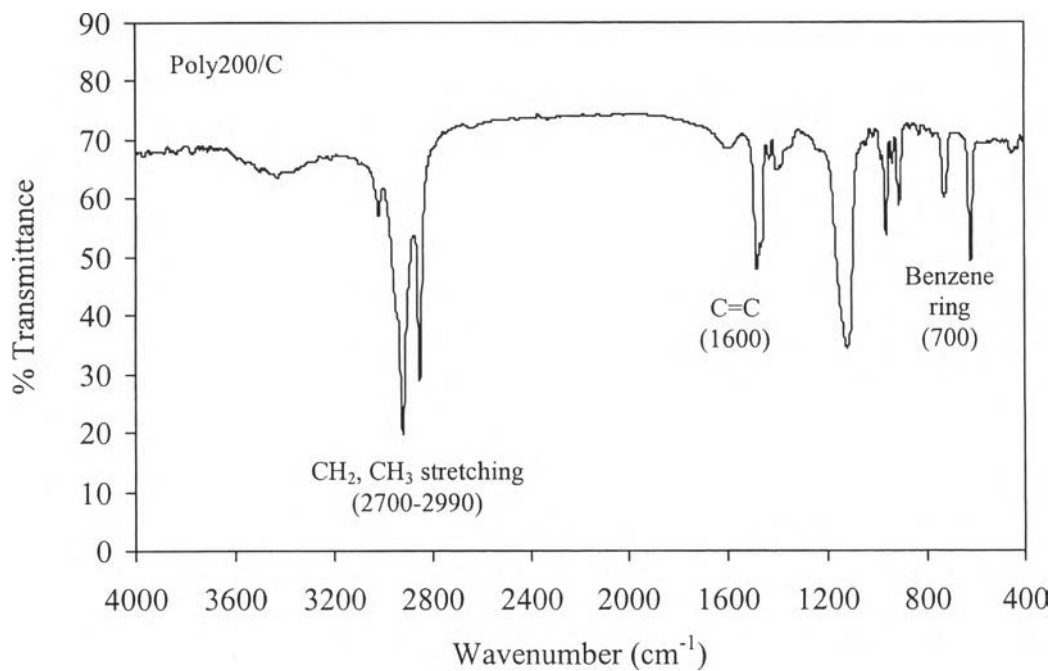


Figure 4.33 FT-IR spectrum of the extracted material (poly200/C).

4.2 Rubber Compound Physical Properties

The effects of various surface modifications with different amounts of surfactant and initiator loadings on rubber compound physical properties were investigated. The results of complete performance data of all silicas studied in rubber compound are summarized in Table 4.2. Table 4.2 also includes the results of the unmodified and modified silica from Thammathanukul *et al.* (1996).

Table 4.2 Rubber compound physical properties using different modified silicas obtained from the present study compared to the modified silicas of the previous batch system (Thammathadanukul *et al.*, 1996).

Property	Hi-Sil [®] 255*	Batch**	146A	146B	146C	175A	175B	175C	200A	200B	200C
Cure time (min)	18.63	5.98	4.75	4.14	4.79	4.64	5.02	5.26	4.65	4.93	5.07
100%Modulus @before aging (MPa)	0.77	1.33	1.85	1.81	2.04	1.53	1.79	1.97	1.83	1.64	1.77
200%Modulus @before aging (MPa)	1.57	2.54	3.15	3.03	3.47	2.45	2.98	3.28	3.11	2.66	3.02
300%Modulus @before aging (MPa)	2.84	4.19	5.30	4.85	5.84	3.74	4.84	5.51	5.12	4.23	5.01
Tensile Strength @before aging (MPa)	19.84	26.43	27.13	28.14	27.32	27.39	28.41	26.82	28.64	28.16	28.33
Tear Strength @before aging (MPa)	30.27	75.37	52.26	52.32	54.49	49.22	55.61	60.18	53.26	50.32	54.12
Abrasion (ml/kcycle)	0.96	0.66	0.48	0.50	0.46	0.47	0.48	0.43	0.50	0.57	0.45
Resilience (%)	56.7	73.6	65.75	70.53	70.10	66.10	66.00	66.40	71.50	69.00	70.80
Compression set (%)	83.11	76.02	54.96	58.57	62.27	64.67	58.75	60.32	55.28	60.15	57.46

146, 175, 200: the amount of surfactant loadings in the unit of grams per kilogram of silica

A, B, C: the amount of initiator loadings in the unit of grams

* Unmodified silica

** Styrene-isoprene co-monomer (Results from Thammathadanukul *et al.*, 1996)

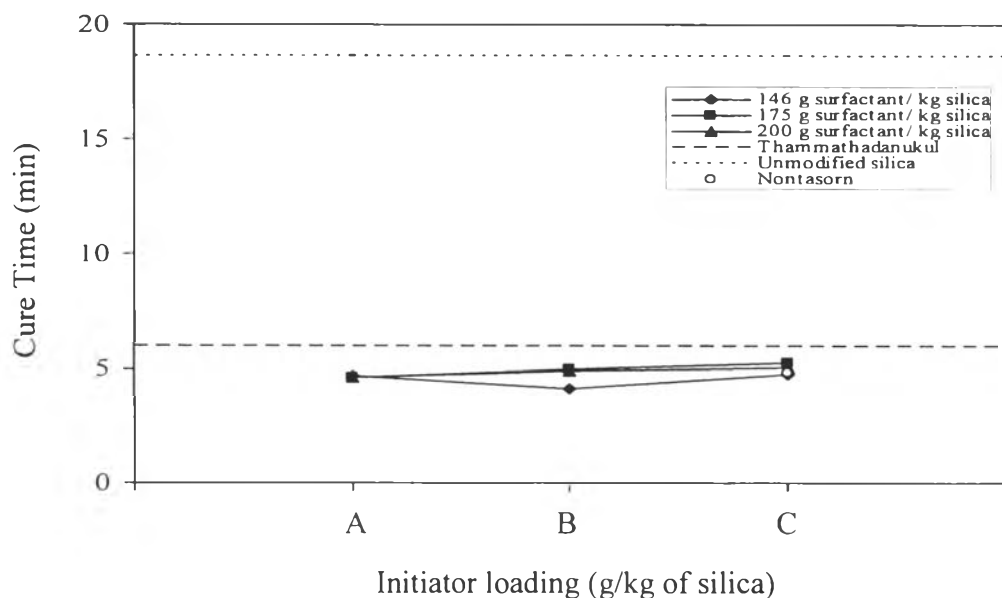


Figure 4.34 Cure time of modified silicas as a function of surfactant and initiator loadings as compared to the previous work.

The effects of various surfactant and initiator loadings on the cure time were investigated. The data show that the surfactant and initiator loadings hardly affect the cure time of the modified silicas as seen from Figure 4.34. It can be concluded from this study that the cure time of all modified silicas does not depend on both surfactant and initiator loadings. Fascinatingly, all nine modified silicas have slightly lower cure times than the previous work. This may simply be due to the better uniform layer coated on the silica surface by the continuous system as compared to that by the batch system. Thammathadanukul *et al.* (1996) concluded that the decrease in cure time is consistent with the copolymer formation within the silica pores, covering some of the surface silanol groups, thereby reduced their interaction toward the polar chemical additives used for rubber vulcanization.

Modulus values at 100% before aging of modified silicas are plotted with surfactant and initiator loadings in Figure 4.35. All nine modified silicas showed significant increases in 100% modulus values when compared to the unmodified silica, but slightly increase to those of the batch system from Thammathadanukul. However, the modified silica for 200 g surfactant loading at 1.65 g of initiator

loading was not significantly different in performance compared to Nontasorn (2002)'s work which used the same amount of surfactant loading.

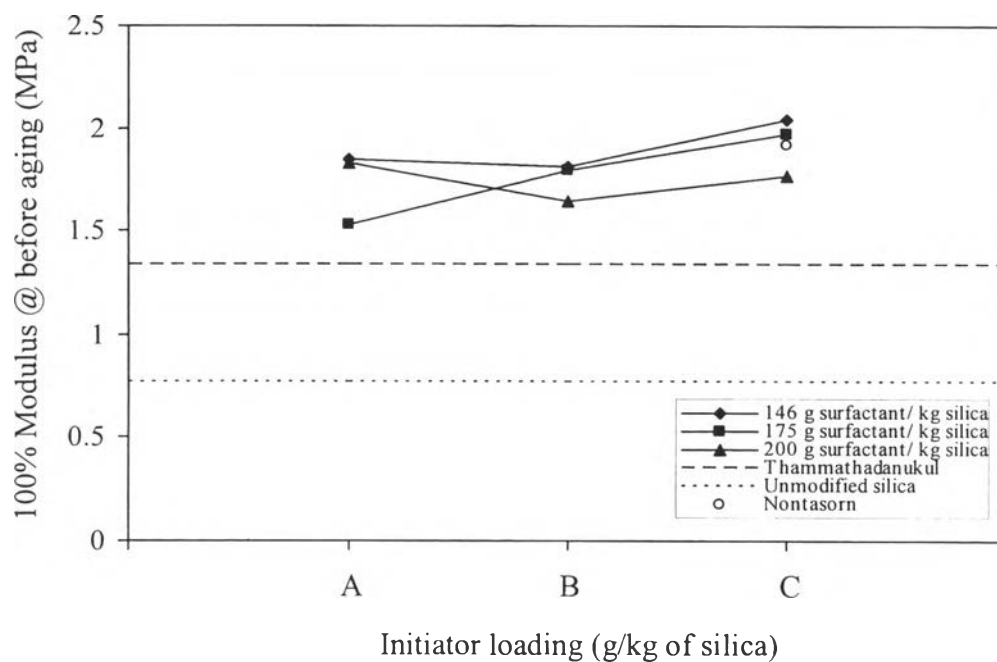


Figure 4.35 100% Modulus @ before aging of modified silicas as a function of surfactant and initiator loadings as compared to the previous work.

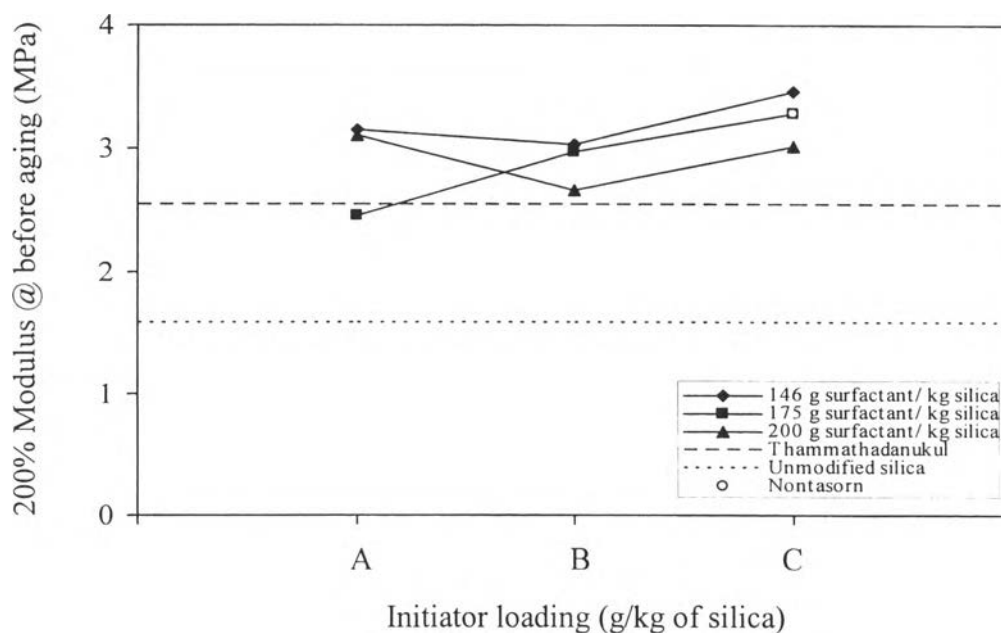


Figure 4.36 200% Modulus @ before aging of modified silicas as a function of surfactant and initiator loadings as compared to the previous work.

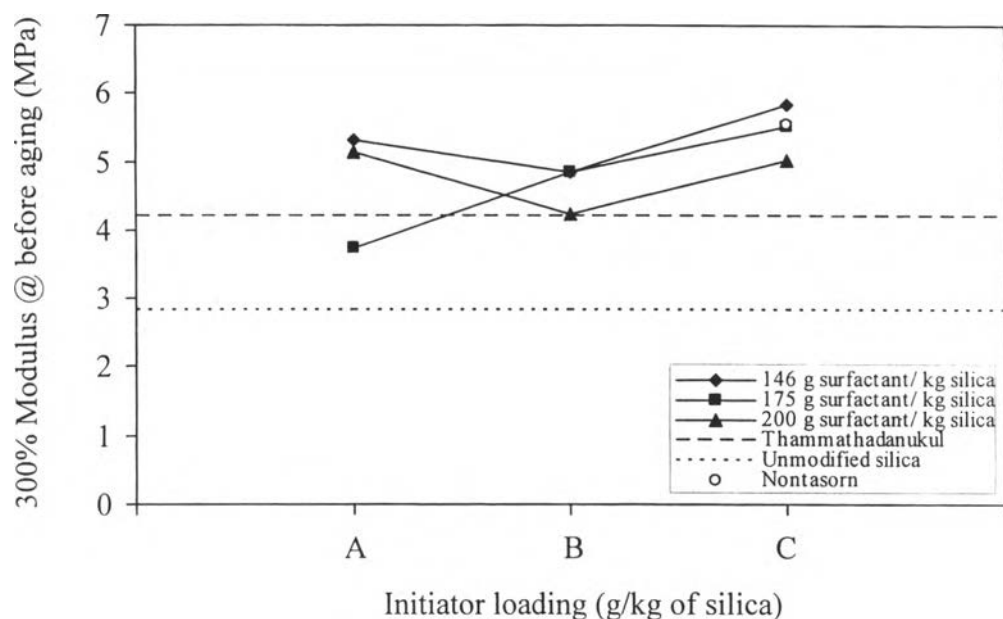


Figure 4.37 300% Modulus @ before aging of modified silicas as a function of surfactant and initiator loadings as compared to the previous work.

Figures 4.36 to 4.37 show that for any surfactant and initiator loadings, modified silicas of the present study have 200% and 300% modulus @ before aging almost greater than the batch system and also for unmodified silica. Again, modified silicas for various surfactant loadings at constant 1.65 g of initiator loading were not different in performance compared to previous work of Nontasorn (2002). The tensile strength @ before aging values of the cured rubber compounds as shown in Figure 4.38 were significantly increased when compared to that of unmodified silica and slightly increased from previous work in batch system. The modified silicas for various surfactant loadings at constant 1.65 g of initiator loading were not different in performance compared to Nontasorn (2002). The data show that the modified silicas had slightly differences in tensile values.

The tear strength @ before aging values of the rubber compounds increases for all nine modified silica compared with unmodified silica and also for the previous work as seen in Figure 4.39. As seen from Figure 4.40, the abrasion values of all nine modified silicas of the present study were decreased than previous work from batch system and significantly decreased for unmodified silica. The modified silicas

for various surfactant loadings at constant 1.65 g of initiator loading are slightly different in performance compared to Nontasorn (2002)'s work.

Again, the resilience values of all nine modified silicas as shown in Figure 4.41 were increased as compared with unmodified silica. The results indicate that all modified silicas improve the resilience values. From Figure 4.42, all modified silicas were significantly reduced the compression set values compared to that of unmodified silica and the previous work from batch system. The modified silicas for various surfactant loadings at constant 1.65 g of initiator loading provided greater performance than Nontasorn (2002)'s work.

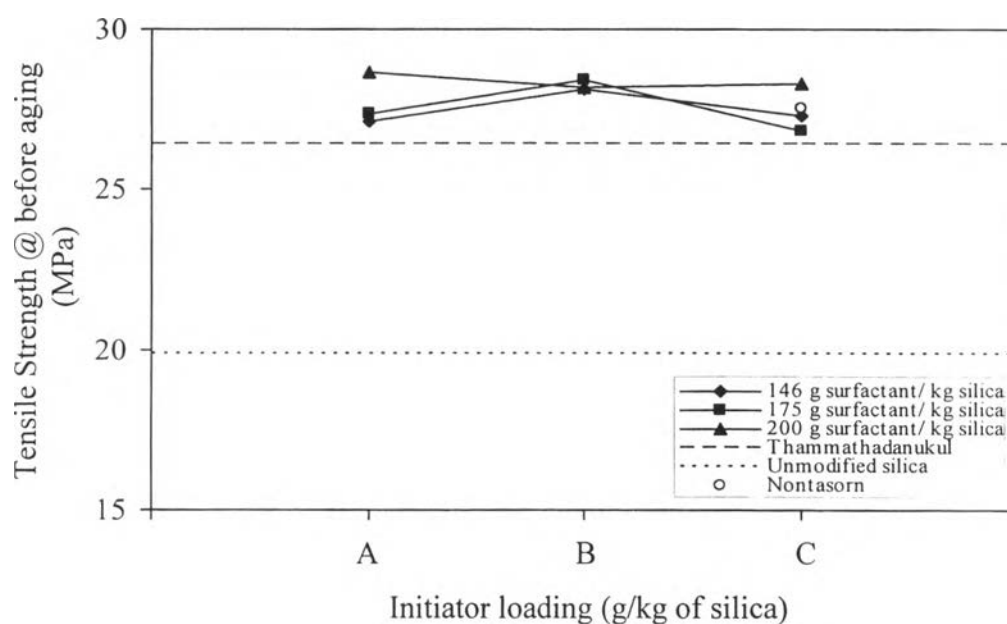


Figure 4.38 Tensile Strength @ before aging of modified silicas as a function of surfactant and initiator loadings as compared to the previous work.

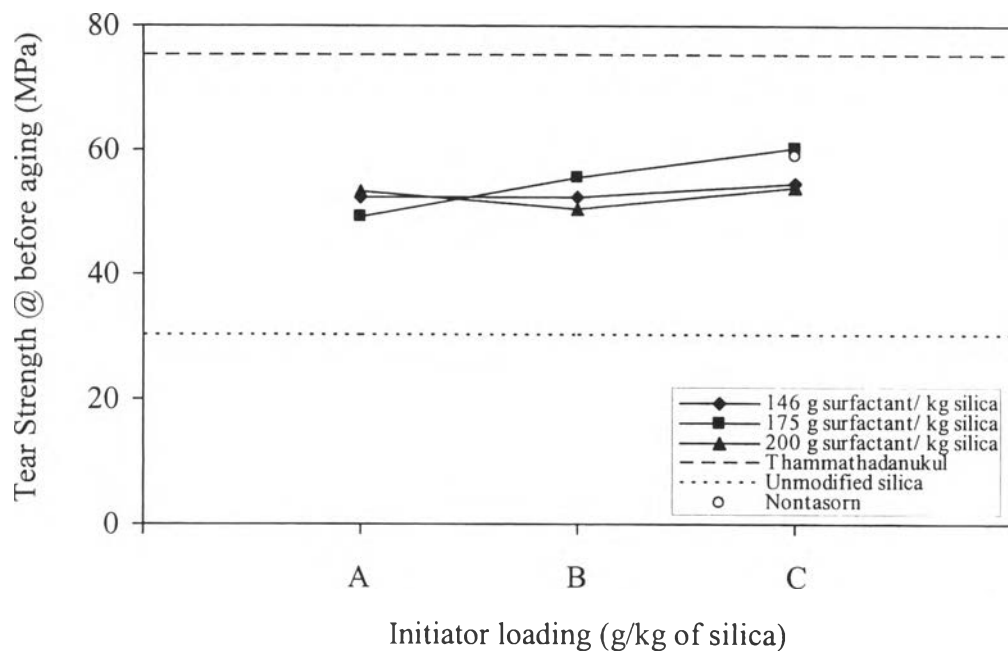


Figure 4.39 Tear Strength @ before aging of modified silicas as a function of surfactant and initiator loadings as compared to the previous work.

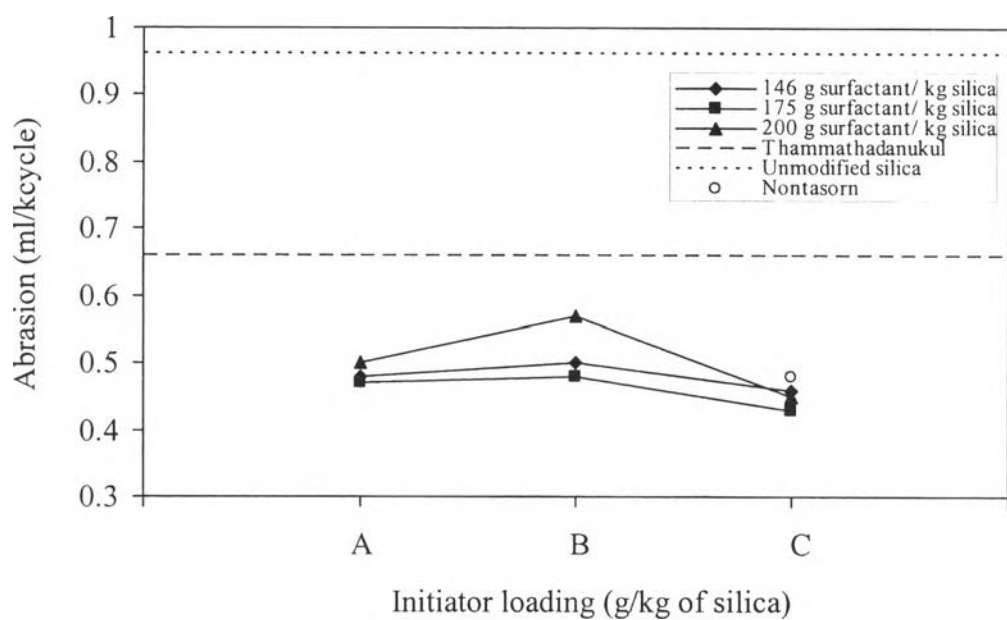


Figure 4.40 Abrasion of modified silicas as a function of surfactant and initiator loadings as compared to the previous work.

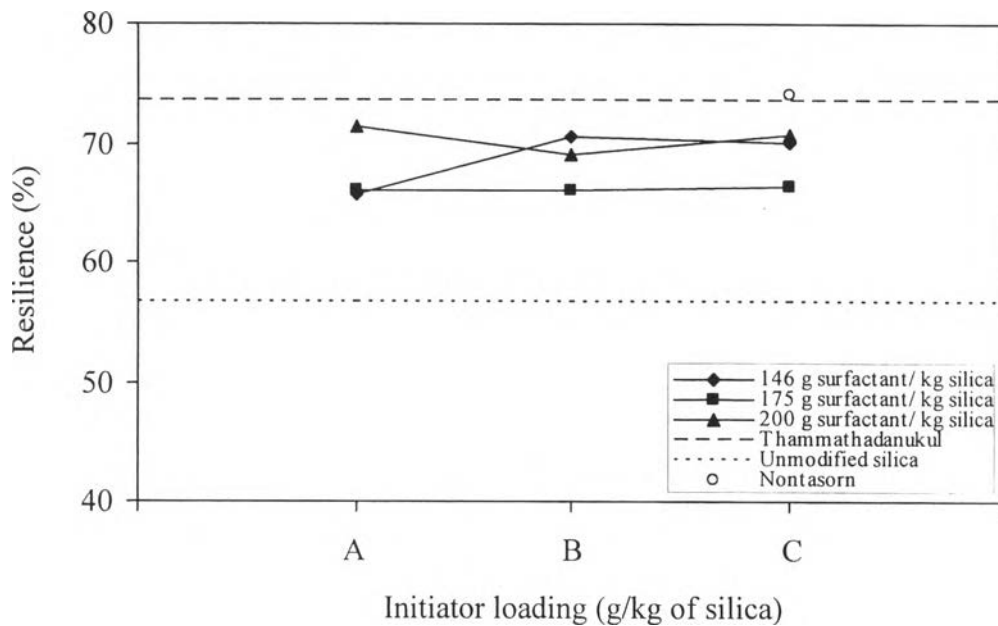


Figure 4.41 Resilience of modified silicas as a function of surfactant and initiator loadings as compared to the previous work.

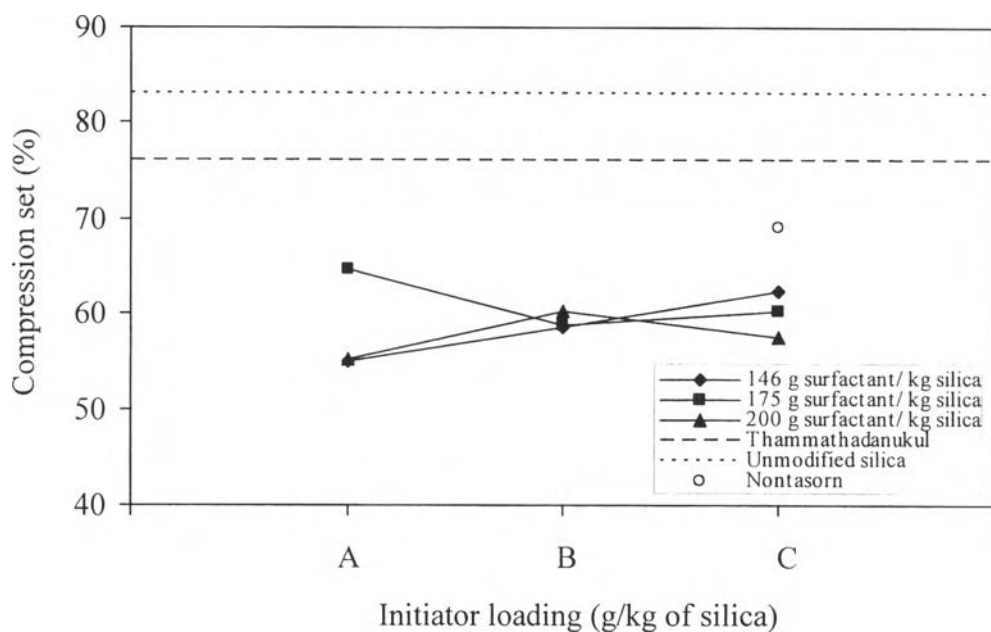


Figure 4.42 Compression Set of modified silicas as a function of surfactant and initiator loadings as compared to the previous work.

The results of the effects of surfactant and initiator loadings of the modified silica on the various rubber physical properties are shown qualitatively in Table 4.3, in which a “+” indicates greater than 10% improvement in the property over unmodified silica, a “-” indicates a greater than 10% negative impact on the property, and an “=” indicates no significant difference. A “+” is given a value of 1; a “-” is given a value of -1; and an “=” is given a 0 values for qualitative calculation of overall improvement relative to the respective unmodified silicas. Table 4.4 shows the percent improvement of surface-modified silica rubber physical properties as compared to the modified silicas of the previous batch system. As seen from the table, silicas modified by using difference in surfactant and initiator loadings show the improvements in cure rate, modulus, tensile strength, abrasion and compression set, but seem to have the negative effect on the tear strength and resilience.

Interestingly, the present work shows the improvement for surface characterization and rubber properties as well as compared to Nontasorn’s work which used the same amount of surfactant and initiator loadings (200 g surfactant and 1.65 g initiator).

However, the amounts of both surfactant and initiator loadings can be minimized whereas the rubber properties are still maintained. Comparison of the rubber compound physical properties using different modified silicas shows that one of the formulas in the most success is 146/C (see Table 4.5). According to % extracted polymer evidence is redundant supported the highest in overall rubber compound physical properties of 146/C-modified silica. Consequently, the differences in performance of the modified silicas may basically be due to the differences in their polymer contents. The results are qualitatively summarized in Table 4.6. As seen from the table, the 146/C-modified silica affords the greatest overall improvement of the rubber compound physical properties in the present study.

Table 4.3 Qualitative summary of surface-modified silica rubber physical properties as compared to the modified silicas of the previous batch system (Thammathadanukul *et al.*, 1996)

Property	146/A	146/B	146/C	175/A	175/B	175/C	200/A	200/B	200/C
Cure time	+1	+1	+1	+1	+1	+1	+1	+1	+1
100%Modulus @before aging	+1	+1	+1	+1	+1	+1	+1	+1	+1
200%Modulus @before aging	+1	+1	+1	0	+1	+1	+1	+1	+1
300%Modulus @before aging	+1	+1	+1	0	+1	+1	+1	0	+1
Tensile Strength @before	0	0	0	0	0	0	0	0	0
Tear Strength @before aging	-1	-1	-1	-1	-1	-1	-1	-1	-1
Abrasion	+1	+1	+1	+1	+1	+1	+1	+1	+1
Resilience	-1	0	0	-1	-1	0	0	0	0
Compression set	+1	+1	+1	+1	+1	+1	+1	+1	+1
Comparative Score	+4	+5	+5	+2	+4	+5	+5	+4	+5

146, 175, 200 : surfactant loadings, g per kg silica, respectively

A, B, C : initiator loadings, 0.4, 0.8, 1.65 g, respectively

Table 4.4 Percent improvement of surface-modified silica rubber physical properties as compared to the modified silicas of the previous batch system (Thammathadanukul *et al.*, 1996)

Property	146/A %	146/B %	146/C %	175/A %	175/B %	175/C %	200/A %	200/B %	200/C %
Cure time	21	31	20	22	16	12	22	18	15
100%Modulus @before aging	39	36	53	15	35	48	38	23	33
200%Modulus @before aging	24	19	37	-4	17	29	22	5	19
300%Modulus @before aging	26	16	39	-10	16	31	22	1	20
Tensile Strength @before aging	3	6	3	4	7	1	8	7	7
Tear Strength @before aging	-30	-30	-28	-35	-26	-20	-29	-33	-28
Abrasion	28	25	30	29	28	34	25	13	33
Resilience	-11	-4	-5	-10	-10	-10	-3	-6	-4
Compression set	28	23	18	15	23	21	27	21	24

146, 175, 200 : surfactant loadings, g per kg silica, respectively

A, B, C : initiator loadings, 0.4, 0.8, 1.65 g, respectively

Table 4.5 Rubber compound physical properties using different modified silicas

Property	146/A	146/B	146/C	175/A	175/B	175/C	200/A	200/B	200/C
100%Modulus @before aging (MPa)	1.85	1.81	2.04	1.53	1.79	1.97	1.83	1.64	1.77
100%Modulus @after aging (MPa)	2.18	2.25	2.77	2.08	2.29	2.52	2.40	1.90	2.28
200%Modulus @before aging (MPa)	3.15	3.03	3.47	2.45	2.98	3.28	3.11	2.66	3.02
200%Modulus @after aging (MPa)	4.09	4.12	4.95	3.83	4.11	4.59	4.39	3.44	4.22
300%Modulus @before aging (MPa)	5.30	4.85	5.84	3.74	4.84	5.51	5.12	4.23	5.01
300%Modulus @after aging (MPa)	7.09	6.89	8.30	6.67	7.15	7.93	7.38	5.74	7.20
Tensile Strength @before aging (MPa)	27.13	28.14	27.32	27.39	28.41	26.82	28.64	28.16	28.33
Tensile Strength @after aging (MPa)	26.24	27.32	27.04	29.63	29.23	28.86	28.15	30.91	27.37
Tear Strength @before aging (MPa)	52.26	52.32	54.49	49.22	55.61	60.18	53.26	50.32	54.12
Tear Strength @after aging (MPa)	54.55	50.88	55.42	50.55	53.51	55.25	51.37	47.85	54.43
Abrasion (ml/kcycle)	0.478	0.495	0.463	0.470	0.476	0.433	0.498	0.573	0.445
Resilience (%)	65.75	70.53	70.10	66.10	66.00	66.40	71.50	69.00	70.80
Compression set (%)	54.96	58.57	62.27	64.67	58.75	60.32	55.28	60.15	57.46
Hardness @before aging (shore A)	58.60	53.63	58.00	51.45	56.75	56.80	54.30	50.23	55.10
Hardness @after aging (shore A)	61.10	54.60	60.77	52.83	57.07	60.97	58.67	53.73	58.57

146, 175, 200 : surfactant loadings, g per kg silica, respectively

A, B, C : initiator loadings, 0.4, 0.8, 1.65 g, respectively

Table 4.6 Qualitative summary of rubber physical properties using different modified silicas

Property	146/A	146/B	146/C	175/A	175/B	175/C	200/A	200/B	200/C
100%Modulus @before aging (MPa)	7	5	9	1	4	8	6	2	3
100%Modulus @after aging (MPa)	3	4	9	2	6	8	7	1	5
200%Modulus @before aging (MPa)	7	5	9	1	3	8	6	2	4
200%Modulus @after aging (MPa)	3	5	9	2	4	8	7	1	6
300%Modulus @before aging (MPa)	7	4	9	1	3	8	6	2	5
300%Modulus @after aging (MPa)	4	3	9	2	5	8	7	1	6
Tensile Strength @before aging (MPa)	2	5	3	4	8	1	9	6	7
Tensile Strength @after aging (MPa)	1	3	2	8	7	6	5	9	4
Tear Strength @before aging (MPa)	3	4	7	1	8	9	5	2	6
Tear Strength @after aging (MPa)	7	3	9	2	5	8	4	1	6
Abrasion (ml/kcycle)	4	3	7	6	5	9	2	1	8
Resilience (%)	1	7	6	3	2	4	9	5	8
Compression set (%)	9	6	2	1	5	3	8	4	7
Hardness @before aging (shore A)	9	3	8	2	6	7	4	1	5
Hardness @after aging (shore A)	9	3	7	1	4	8	6	2	5
Total	76	63	105	37	75	103	91	40	85

146, 175, 200 : surfactant loadings, g per kg silica, respectively

A, B, C : initiator loadings, 0.4, 0.8, 1.65 g, respectively