## **CHAPTER IV**

## RESULTS AND DISCUSSION

### 4.1 Characterization of the functional groups of the copolymers by FTIR

The synthesized copolymers were characterized for the functional groups using FTIR technique. The FTIR spectrum of silica, polyacrylamide, poly[acrylamide-co-(itaconic acid)], polyacrylamide/silica composites, poly[acrylamide-co-(itaconic acid)]/silica composites are shown in Figures 4.1-4.7 and demonstrated in Tables 4.1-4.2. The spectrum of the copolymer confirmed that silica was intercalated onto both acrylamide and itaconic acid.

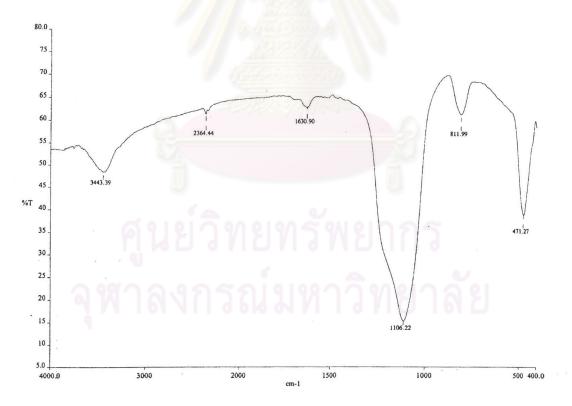


Figure 4.1 Infrared spectrum of silica aerosil 90

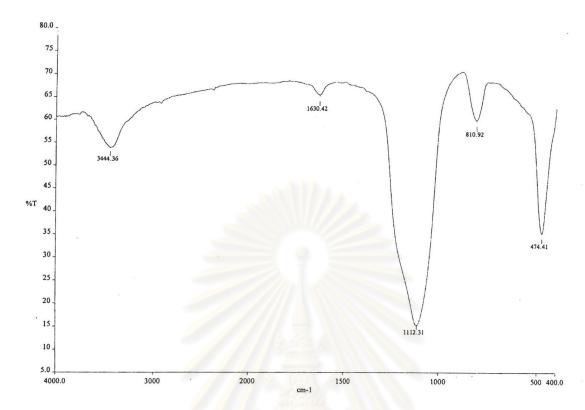


Figure 4.2 Infrared spectrum of silica aerosil 200

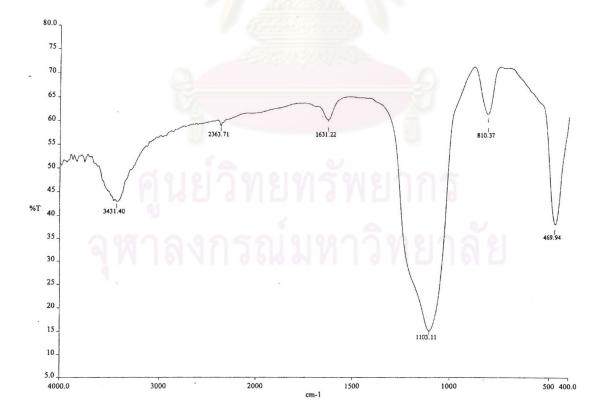


Figure 4.3 Infrared spectrum of silica aerosil 300

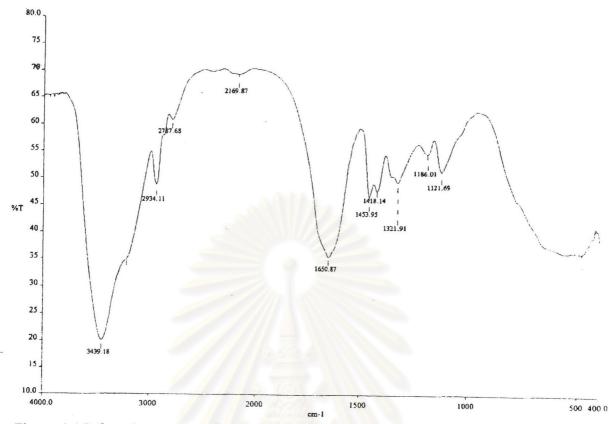


Figure 4.4 Infrared spectrum of polyacrylamide

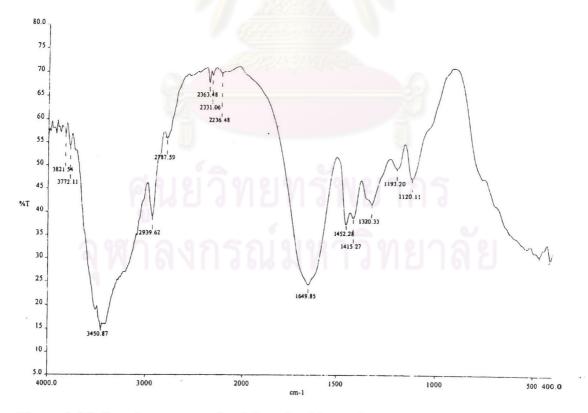


Figure 4.5 Infrared spectrum of poly[acrylamide-co-(itaconic acid)]

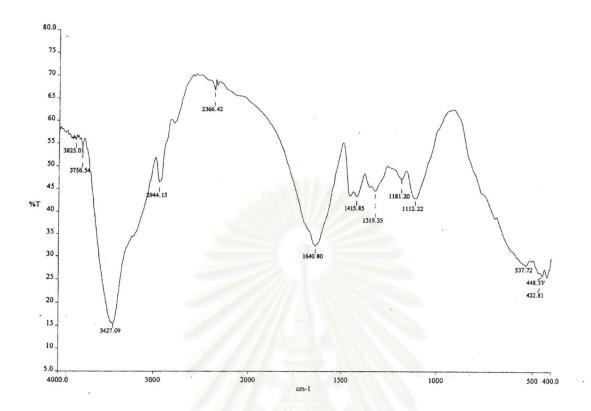


Figure 4.6 Infrared spectrum of polyacrylamide/silica composites

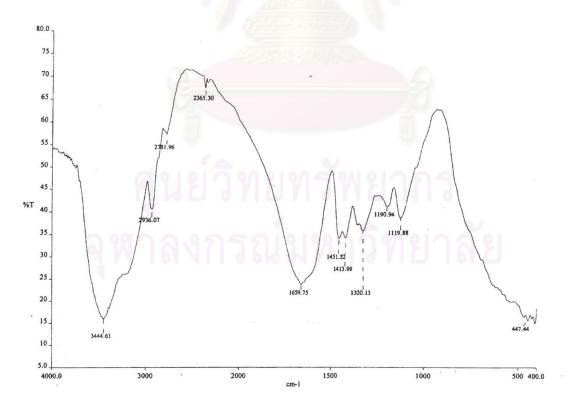


Figure 4.7 Infrared spectrum of poly[acrylamide-co-(itaconic acid)]/silica composites

Table 4.1The infrared absorption of functional groups in silica aerosil 90, silica aerosil 200, and silica aerosil 300

Absorption wavenumber, cm <sup>-1</sup> for								
Silica aerosil 90	Silica aerosil 200	Silica aerosil 300	Assignment					
3443 (m)	3444 (m)	3431 (m)	O-H stretching					
1631 (m)	1630 (m)	1631 (m)	C=O stretching					
471 (s)	474 (s)	470 (s)	Si-O					

s: strong; m: medium; w: weak.



Table 4.2 The infrared absorption of functional groups in polyacrylamide, poly[acrylamide-co-(itaconic acid)], polyacrylamide/silica composites, and poly[acrylamide-co-(itaconic acid)]/silica composites

	Absorption wavenumber, cm <sup>-1</sup> for								
(AM)	(AM-co-IA)	(AM)/silica	(AM-co-IA)/silica	Assignment					
3782 (m)	3772 (w)	3757 (w)	3752 (w)	O-H stretching					
3439 (w)	3450 (s)	3427 (s)	3445 (s)	N-H stretching					
2934 (m)	2940 (m)	2944 (m)	2936 (m)	C-H stretching					
1650 (s)	1650 (s)	1641 (s)	1660 (s)	C=O stretching					
1418 (s)	1452 (m)	1416 (m)	1452 (m)	C-H bending					
-	2500-3300 (m)	1000 (1000) 1000 (1000) 1000 (1000)	2500-3300 (m)	O-H stretching of COOH					
1122 (m)	1120 (m)	1112 (m)	1120 (m)	N-H bending					
-		448 (w)	447 (w)	Si-O					

(AM): polyacrylamide

(AM-co-IA): poly[acrylamide-co-(itaconic acid)]

(AM)/silica: polyacrylamide/silica composites

(AM-co-IA)/silica: poly[acrylamide-co-(itaconic acid)]/silica composites

s: strong; m: medium; w: weak.

#### 4.2 Surface morphology of the synthesized copolymers

The synthesized copolymers were investigated by scanning electron microscopy technique to observe the surface appearance. The electron micrographs of the synthesized copolymers are shown in Figures 4.8-4.10.

SEM photograph of the synthesized copolymers of poly[acrylamide-co-(itaconic acid)], which are the polymers of good absorbency, illustrated in Figure 4.8 shows that a porous structure and a broad network are found in the polymer. It is supposed that these pores are the regions of water permeation. The interaction of the hydrophilic groups with water makes the copolymer high water absorption.

The SEM photograph of the polyacrylamide/silica composites in Figure 4.9 has fine network and smooth surface. On the other hand, the SEM photograph of poly[acrylamide-co-(itaconic acid)]/silica composites that give less water absorption, illustrated in Figure 4.10, show the smooth surface. The water absorbency of the polyacrylamide/silica composites was lower than that of poly[acrylamide-co-(itaconic acid)]/silica composites.

The number of hydrophilic groups of poly[acrylamide-co-(itaconic acid)]/silica composites is higher than that of the polyacrylamide/silica composites, so the swelling capacity of polyacrylamide/silica composites is therefore lower than the copolymer composites.



Figure 4.8 SEM photograph of poly[acrylamide-co-(itaconic acid)] (WA= 233±8 g/g)



Figure 4.9 SEM photograph of polyacrylamide/silica composites (WA=51±2 g/g)

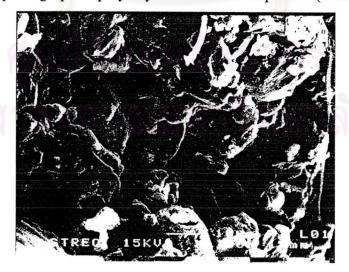


Figure 4.10 SEM photograph of poly[acrylamide-co-(itaconic acid)]/silica composites (WA=199 $\pm$ 4 g/g)

#### 4.3 Effect of itaconic acid concentration

## 4.3.1 Effect of itaconic acid concentration (mole percent) on the water absorbency of the synthesized copolymers

The water absorbency of the synthesized copolymer, synthesized by solution polymerization of various concentrations of itaconic acid (mole percent) with 1.0%wt APS, 0.5%wt N-MBA, at 45°C, 250 rpm, 30 min is shown in Table 4.3 and Figure 4.11.

Table 4.3 Effect of itaconic acid concentration (mole percent) on the water absorbency

Itaconic acid concentration (mole percent)	Water absorbency (g/g)
0	51 <u>+</u> 2
1	157 <u>+</u> 9
2	191 <u>+</u> 6
3	233 <u>+</u> 8

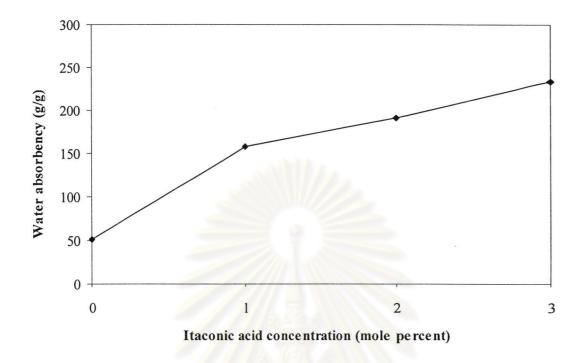


Figure 4.11 Effect of itaconic acid concentration (mole percent) on the water absorbency

The water absorbency increases with increasing concentration of the hydrophilic and ionic functional groups. The structure of the copolymer should contain more AM units than IA units owing to the higher acrylamide concentration. The reactivity ratios of AM and IA are 0.77 and 1.36, respectively [42], *i.e.* acrylamide monomer molecules are used up earlier than itaconic acid. The reaction is short of AM molecules as the reaction proceeds. The ultimate chain end contains most of the itaconic acid molecules. The synthesized copolymer must have an optimum ratio of AM-to-IA in order to balance hydrophilic or ionic functional groups and chain flexibility to produce the copolymer with the highest water absorption capacity. The copolymer synthesized by AM: IA ratio of 97:3 with 1.0%wt APS, 0.5%wt N-MBA, at 45°C, 250 rpm, 30 min, can absorb distilled water at the highest value of 233± 8 g/g.

## 4.3.2 Effect of itaconic acid concentration (mole percent) on the absorbency under load of the synthesized copolymers

The water absorbency under load (0.28 psi and 0.70 psi) of the copolymer, synthesized by solution polymerization of various concentrations of itaconic acid (mole percent) with 1.0%wt APS, 0.5%wt N-MBA, at 45°C, 250 rpm, 30 min is shown in Table 4.4 and Figure 4.12.

Table 4.4 Effect of itaconic acid concentration (mole percent) on the absorbency under load

Itaconic acid concentration	Water absorption under load (AUL) (g/g) at			
(mole percent)	0.28 psi	0.70 psi		
0	14.2	12.0		
1	13.5	11.5		
2	12.9	11.1		
3	11.6	10.7		

AUL = absorbency under load;

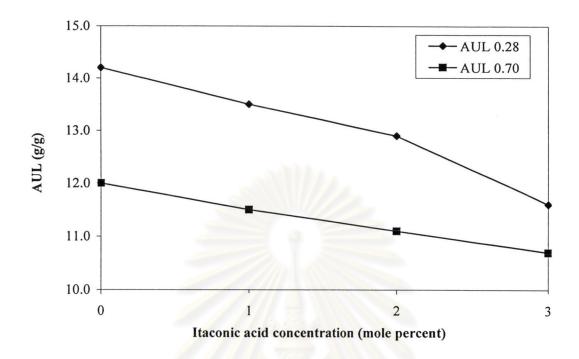


Figure 4.12 Effect of itaconic acid concentration (mole percent) on the absorbency under load

The highest AUL of 0.28 psi was found in the work at the IA concentration at 0%, which gave a value of 14.2 g/g. It decreased to 13.5, 12.9, and 11.6 g/g when IA concentrations were set at 1, 2, and 3 %, respectively. Likewise, the highest AUL of 0.70 psi was found in the work at the IA concentration at 0%, which gave a value of 12.0 g/g and it decreased to 11.5, 11.1, and 10.7 g/g when IA concentrations at 1, 2, and 3 %, respectively were used. The absorbency under load decreases with increasing the concentration of the hydrophilic or ionic functional groups and chain flexibility.

# 4.3.3 Effect of itaconic acid concentration (mole percent) on the swelling kinetics of the synthesized copolymers

The swelling kinetics of the copolymer, synthesized by solution polymerization of various concentrations of itaconic acid (mole percent) with 1.0%wt APS, 0.5%wt N-MBA, at 45°C, 250 rpm, 30 min is shown in Table 4.5 and Figure 4.13.

Table 4.5 Effect of itaconic acid concentration (mole percent) on the swelling kinetics

Itaconic acid concentration	W	ater absorb	ency (g/g) a	at absorptio	n time (mii	1)
(mole percent)	15	45	105	225	1125	1605
0	43 <u>+</u> 3	47 <u>+</u> 7	51 <u>+</u> 9	52 <u>+</u> 7	56 <u>+</u> 1	64 <u>+</u> 7
1	54 <u>+</u> 5	107 <u>+</u> 6	128 <u>+</u> 7	142 <u>+</u> 7	149 <u>+</u> 7	155 <u>+</u> 7
2	92 <u>+</u> 3	131 <u>+</u> 3	149 <u>+</u> 7	152 <u>+</u> 6	176 <u>+</u> 1	179 <u>+</u> 5
3	149 <u>+</u> 2	205 <u>+</u> 4	221 <u>+</u> 8	236 <u>+</u> 7	237 <u>+</u> 4	239 <u>+</u> 6

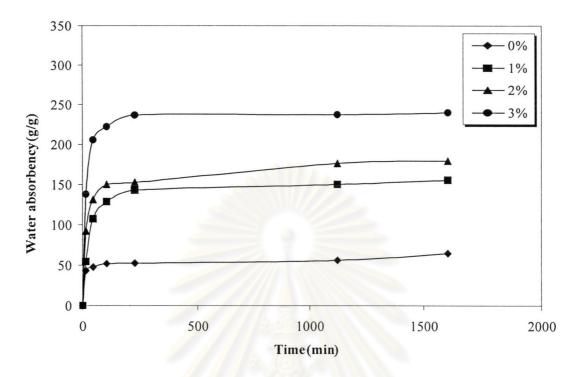


Figure 4.13 Effect of itaconic acid concentration (mole percent) on the swelling kinetics

The swelling behavior of the superabsorbent polymer is a diffusive process. After water has diffused into the polymer, the polymer chains relax and diffuse outwardly into the liquid. The polymer chains must diffuse and more volume of the polymer increases. Because of the diffusive mechanism, a principal factor of the swelling kinetics is the size of polymer particle, its bulk morphology, and its shape.

In practical applications, not only is a higher absorption capacity required of the absorbent, but also characterization by its high swelling kinetics. The synthesized copolymer can absorb water up to 149±2 g/g of the dry copolymer within 15 min for the polymer synthesized by AM-to-IA ratio of 97:3. The dependence of swelling time on equilibrium swelling arises because the mass of liquid absorbed during the swelling rate measurement depends on the equilibrium swelling capacity of the polymer. As the

equilibrium swelling capacity increases, less polymer amount is used in the test so that each sample reaches the same relative swelling extent at the specific time.

### 4.4 Effect of type of silica

# 4.4.1 Effect of type of silica on the water absorbency of the synthesized copolymers

The water absorbency of the copolymer, synthesized by the AM-to-IA ratio of 97:3 with 1.0%wt APS, 0.5%wt N-MBA, at 45°C, 250 rpm, 30 min and various types of 2.0%wt silica is shown in Table 4.6 and Figure 4.14.

Table 4.6 Effect of type of silica on the water absorbency

Type of silica	Water absorbency (g/g)
no silica	233 <u>+</u> 8
aerosil 90	199 <u>+</u> 4
aerosil 200	193 <u>+</u> 6
aerosil 300	188 <u>+</u> 1

AM-to-IA ratio, 97:3; silica, 2.0%wt;

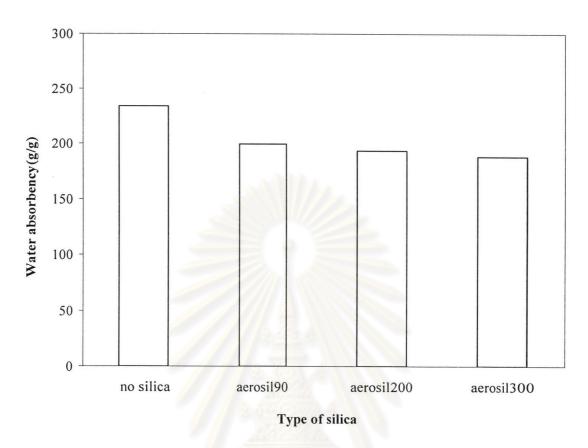


Figure 4.14 Effect of type of silica on the water absorbency

The water absorbency amount for the synthesized copolymer with no silica at the AM-to-IA ratio of 97:3 is 233±8 g/g and decreases to 199±4, 193±6, and 188±1 g/g (reduced by 14.6, 17.2, and 19.3% based on the non-silica superabsorbent polymer) in the case of copolymer containing silica aerosil 90, silica aerosil 200, and silica aerosil 30, respectively. The water absorbency of the copolymers in the presence of silica aerosil 300 is somewhat smaller than those of silica aerosil 90 and silica aerosil 200. The reason is related to higher hydrophilic properties of the hydrophilic chain and the silica particles. The BET surface areas (m²/g) of silica aerosil 90, silica aerosil 200, and silica aerosil 300 are 90±15, 200±25, and 300±30, respectively. Their surface areas are quite much different but the surface area does not influence the water absorption of the copolymers.

# 4.4.2 Effect of type of silica on the absorbency under load of the synthesized copolymers

The water absorbency under load (0.28 and 0.70 psi) of the copolymer, synthesized by the AM-to-IA ratio of 97:3 with 1.0%wt APS, 0.5%wt N-MBA, at 45°C, 250 rpm, 30 min and various types of 2.0%wt silica is shown in Table 4.7 and Figure 4.15.

Table 4.7 Effect of type of silica on the absorbency under load

Toma of siling	Water absorption under load (AUL) (g/g) at			
Type of silica	0.28 psi	0.70 psi		
no silica	11.6	10.7		
aerosil 90	12.7	11.8		
aerosil 200	13.2	12.0		
aerosil 300	13.2	12.1		

AUL = absorbency under load; AM-to-IA ratio, 97:3; silica, 2.0%wt; APS, 1.0%wt; N-MBA, 0.5%wt; at 45°C, 250 rpm, 30 min.

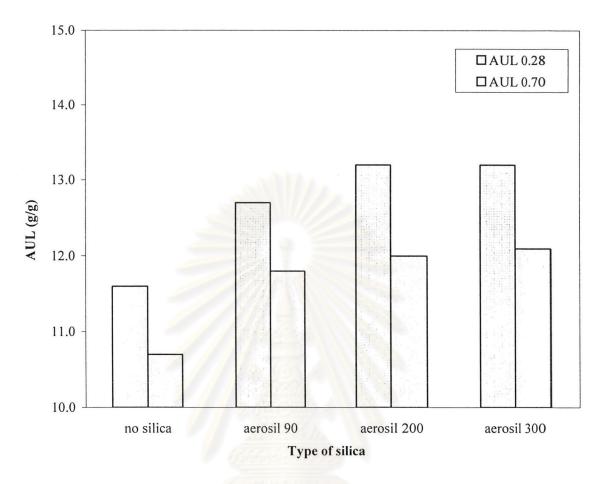


Figure 4.15 Effect of type of silica on the absorbency under load

The absorbency under load of 0.28 psi for the synthesized copolymer with no silica at the AM-to-IA ratio of 97:3 is 11.6 g/g and increases to 12.7, 13.2, and 13.2 g/g (reduced by 9.5, 13.8, and 13.8% based on the non-silica superabsorbent polymer) in the case of copolymer containing silica aerosil 90, silica aerosil 200, and silica aerosil 300, respectively. Likewise, the absorbency under load of 0.70 psi for the synthesized copolymer with no silica at the AM-to-IA ratio of 97:3 is 10.7 g/g and increases to 11.8, 12.0, and 12.1 g/g (reduced by 10.3, 12.2, and 12.1% based on the non-silica superabsorbent polymer) in the case of copolymer containing silica aerosil 90, silica aerosil 200, and silica aerosil 300, respectively. It may be attributed to the fact that fine silica particles act as additional junctions. Therefore, crosslink density increased by the increasing silica amount led to increase gel strength. The absorbency under load then

increases slightly with the added silica. Therefore, silica improves gel strength or absorbency under load of the polymer although the value of the synthesized copolymer with silica aerosil 90, silica aerosil 200, and silica aerosil 300 are not much different.

### 4.4.3 Effect of type of silica on the swelling kinetics

The swelling kinetics of the copolymer, synthesized by the AM-to-IA ratio of 97:3 with 1.0%wt APS, 0.5%wt N-MBA, at 45°C, 250 rpm, 30 min and various types of 2.0%wt silica is shown in Table 4.8 and Figure 4.16.

Table 4.8 Effect of type of silica on the swelling kinetics

Type of		Water a	absorbency	(g/g) at tim	e (min)	
silica	15	45	105	225	1125	1605
no silica	149 <u>+</u> 2	205 <u>+</u> 4	221 <u>+</u> 8	236 <u>+</u> 7	237 <u>+</u> 4	239 <u>+</u> 6
aerosil 90	121 <u>+</u> 8	167 <u>+</u> 9	183 <u>+</u> 5	185 <u>+</u> 6	186 <u>+</u> 3	188 <u>+</u> 6
aerosil 200	119 <u>+</u> 8	165 <u>+</u> 7	181 <u>+</u> 7	182 <u>+</u> 1	182 <u>+</u> 3	184 <u>+</u> 2
aerosil 300	118 <u>+</u> 8	164 <u>+</u> 6	178 <u>+</u> 6	179 <u>+</u> 9	181 <u>+</u> 6	183 <u>+</u> 4

AM-to-IA ratio, 97:3; silica, 2.0%wt;

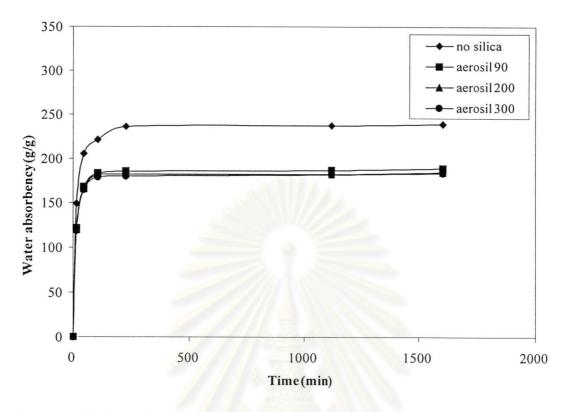


Figure 4.16 Effect of type of silica on the swelling kinetics

The copolymer synthesized with the AM-to-IA ratio of 97:3 and 2.0%wt silica with 1.0%wt APS, 0.5%wt N-MBA, can absorb water up to 121±8, 119±8, and 118±8 g/g of the dry polymer within 15 min for silica aerosil 90, silica aerosil 200, and silica aerosil 300, respectively. The dependence of swelling time on equilibrium swelling arises because the mass of liquid absorbed during the swelling rate increases throughout the measurement. It depends on the equilibrium swelling capacity of the polymer. The swelling absorbency value of the synthesized copolymer with silica aerosil 90, silica aerosil 200, and silica aerosil 300 are not much different.

#### 4.5 Effect of silica concentration

# 4.5.1 Effect of silica concentration on the water absorbency of the synthesized copolymers

The water absorbency of the copolymer, synthesized by the AM-to-IA ratio of 97:3 with 1.0%wt APS, 0.5%wt N-MBA, at 45°C, 250 rpm, 30 min and various silica concentrations (%) is shown in Table 4.9 and Figure 4.17.

Table 4.9 Effect of silica concentration on the water absorbency

Silica concentration	Water absorbency (g/g) of			
(%)	aerosil 90	aerosil 200	aerosil 300	
0.1	228 <u>+</u> 1	228 <u>+</u> 7	225 <u>+</u> 2	
0.2	220 <u>+</u> 1	218 <u>+</u> 2	216 <u>+</u> 3	
0.5	218 <u>+</u> 1	210 <u>+</u> 5	208 <u>+</u> 5	
2.0	199 <u>+</u> 4	193 <u>+</u> 6	188 <u>+</u> 1	

AM-to-IA ratio, 97:3; APS, 1.0%wt; N-MBA, 0.5%wt; at 45°C, 250 rpm, 30 min.

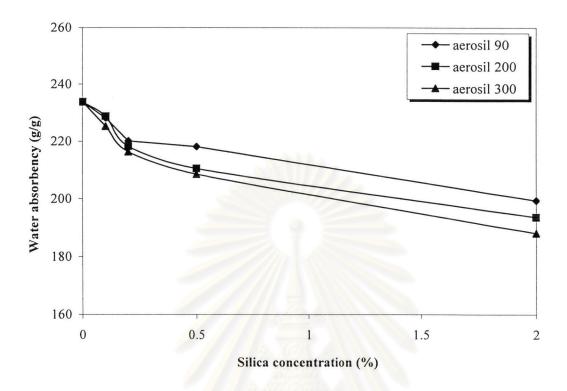


Figure 4.17 Effect of silica concentration on the water absorbency

The water absorbency of the synthesized copolymer at the AM-to-IA ratio of 97:3 with 0.1%wt silica is 228±1, 228±7, and 225±2 g/g for silica aerosil 90, silica aerosil 200, and silica aerosil 300, respectively. It decreased to 199±4, 193±6, and 188±1 for silica aerosil 90, silica aerosil 200, and silica aerosil 300, respectively, at the AM-to-IA ratio of 97:3 with 2.0%wt silica. The water absorbency decreased with increasing concentration of silica. It may be attributed to the fact that fine silica particles act as additional junctions. Therefore, crosslink density increases by increasing silica amount, which led to decreased swelling.

# 4.5.2 Effect of silica concentration on the absorbency under load of the synthesized copolymers

The absorbency under load (0.28 psi and 0.70 psi) of the copolymer synthesized by the AM-to-IA ratio of 97:3 with 1.0%wt APS, 0.5%wt N-MBA, at 45°C, 250 rpm, 30 min and various silica concentrations (%) is shown in Tables 4.10-4.11 and Figures 4.18-4.19.

Table 4.10 Effect of silica concentration on the absorbency under load 0.28 psi

011	A P	AUL 0.28 psi (g/g) (	of
Silica concentration (%)	aerosil 90	aerosil 200	aerosil 300
0.1	11.9	12.2	12.3
0.2	12.2	12.4	12.6
0.5	12.5	12.8	12.9
2.0	12.7	13.2	13.2

AM-to-IA ratio, 97:3; APS, 1.0%wt; N-MBA, 0.5%wt; at 45°C, 250 rpm, 30 min.

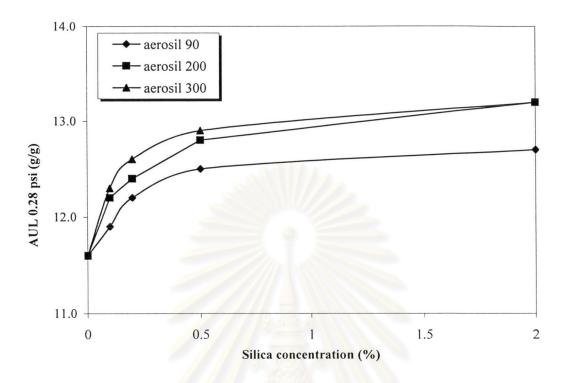


Figure 4.18 Effect of silica concentration on the absorbency under load 0.28 psi

Table 4.11 Effect of silica concentration on the absorbency under load 0.70 psi

Siling concentration (94)	AUL 0.70 psi (g/g)				
Silica concentration (%)	aerosil 90	aerosil 200	aerosil 300		
0.1	10.9	11.0	11.3		
0.2	11.0	วิ <sup>11.1</sup> าล	11.5		
0.5	11.6	11.6	11.8		
2.0	11.8	12.0	12.1		

AM-to-IA ratio, 97:3; APS, 1.0%wt; N-MBA, 0.5%wt; at 45°C, 250 rpm, 30 min.

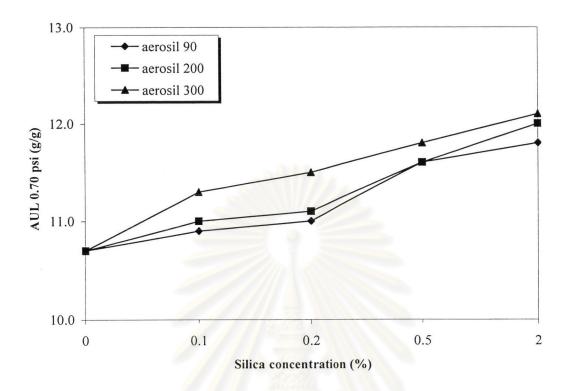


Figure 4.19 Effect of silica concentration on the absorbency under load 0.70 psi

The highest absorbency under load was measured to be 13.2 g/g and 12.1 g/g for 0.28 psi and 0.70 psi, respectively, at the AM-to-IA ratio of 97:3 with 2.0%wt silica aerosil 300. In general, silica has been employed to help improve the gel strength or absorbency under load of the swollen gel but it is very effective to reduce the water absorbency. The swelling in the present work decreases slightly with increasing silica percentage. The additional amounts of silica probably act as non-reactive filler dispersed in the network. It may be attributed to the fact that fine silica particles act as additional junctions. Therefore, silica has an effect on gel strength or absorbency under load improvement, so that the higher the silica percentage, the higher the gel strength will be.

# 4.5.3 Effect of silica concentration on the swelling kinetics of the synthesized copolymers

The swelling kinetics of the copolymer, synthesized by the AM-to-IA ratio of 97:3 with 1.0%wt APS, 0.5%wt N-MBA, at 45°C, 250 rpm, 30 min and various silica concentrations (%) is shown in Table 4.12 and Figure 4.20.

Table 4.12 Effect of silica concentration on the swelling kinetics

Silica concentration		Water absorbency (g/g) at time (min)						
(%)	15	45	105	225	1125	1605		
0.1	135 <u>+</u> 1	202 <u>+</u> 4	222 <u>+</u> 5	232 <u>+</u> 4	234 <u>+</u> 5	235 <u>+</u> 6		
0.2	133 <u>+</u> 3	203 <u>+</u> 6	220 <u>+</u> 3	225 <u>+</u> 4	226 <u>+</u> 6	227 <u>+</u> 1		
0.5	124 <u>+</u> 3	190 <u>+</u> 3	210 <u>+</u> 7	212 <u>+</u> 3	213 <u>+</u> 4	214 <u>+</u> 6		
2.0	121 <u>+</u> 8	167 <u>+</u> 9	183 <u>+</u> 5	185 <u>+</u> 6	186 <u>+</u> 3	188 <u>+</u> 6		

AM-to-IA ratio, 97:3; silica type, aerosil 90;

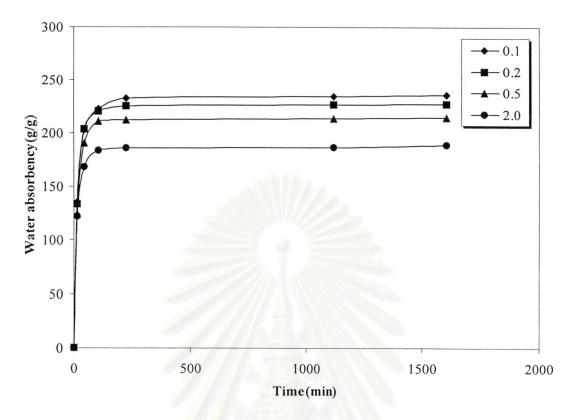


Figure 4.20 Effect of silica concentration on the swelling kinetics

The synthesized copolymer can absorb water up to 135±1 g/g of the dry copolymer within 15 min for the AM-to-IA ratio 97:3 and silica aerosil 90. Therefore, the swelling decreases with increasing silica percentage up to 2.0%wt. The dependence of swelling time on equilibrium swelling arises because the mass of liquid absorbed during the swelling rate increases during the measurement, depending on the equilibrium swelling capacity of the polymer.

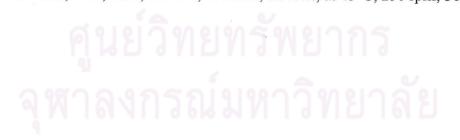
### 4.6 Effect of water temperature on water absorbency

The water absorbency of the copolymer, synthesized by the AM-to-IA ratio of 97:3 with 1.0%wt APS, 0.5%wt N-MBA, at 45°C, 250 rpm, 30 min, and various temperatures of water is shown in Table 4.13 and Figure 4.21.

Table 4.13 Effect of water temperature on the water absorbency

Water temperature (°C)	Water absorbency (g/g)
30	228 <u>+</u> 8
40	214 <u>+</u> 7
50	189 <u>+</u> 8
60	178 <u>+</u> 5
70	167 <u>+</u> 4

AM-to-IA ratio, 97:3; APS, 1.0%wt; N-MBA, 0.5%wt; at 45°C, 250 rpm, 30 min.



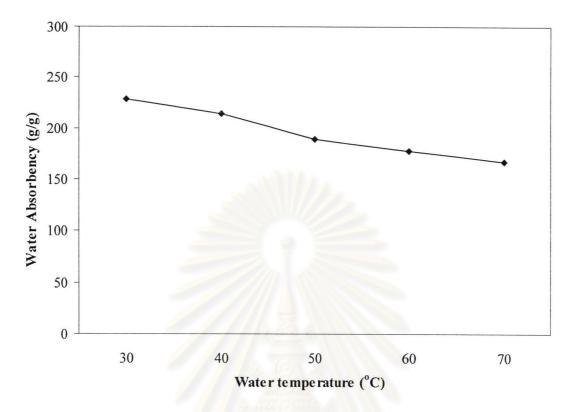


Figure 4.21 Effect of water temperature on the water absorbency

Figure 4.21 shows that at the water temperature higher than 30 °C, the water absorption decreases steadily. This indicates a release of water-soluble materials, *i.e.*, the non-crosslinked copolymers or a lower crosslinking degree. When the gel is cool, the swollen state is thermodynamically stable. The gel formed by the synthesized copolymers seems to be a thermosensitive polymer gel.

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## 4.7 Thermal properties of the synthesized copolymers

The synthesized copolymers were investigated by DSC technique under identical conditions to identify endothermic peaks of copolymer. The DSC thermograms of the synthesized copolymers of poly[acrylamide-co-(itaconic acid)], and poly[acrylamide-co-(itaconic acid)]/silica composites are shown in Figure 4.22.

The reference T<sub>g</sub> value of polyacrylamide is 165°C [43]. The T<sub>g</sub> of poly[acrylamide-co-(itaconic acid)] and poly[acrylamide-co-(itaconic acid)]/silica composites are far higher than the reference T<sub>g</sub> value of polyacrylamide (165°C). It can be found that crystalline of copolymer are increased with the added silica led to increase gel strength. The T<sub>g</sub> of the poly[acrylamide-co-(itaconic acid)]/silica composites, synthesized with different types of silica (aerosil 90 and aerosil 300) are approximately equal. The BET surface areas (m²/g) of silica aerosil 90 and silica aerosil 300 are 90±15 and 300±30, respectively. Their surface areas are quite much different but the surface area does not influence thermal property.

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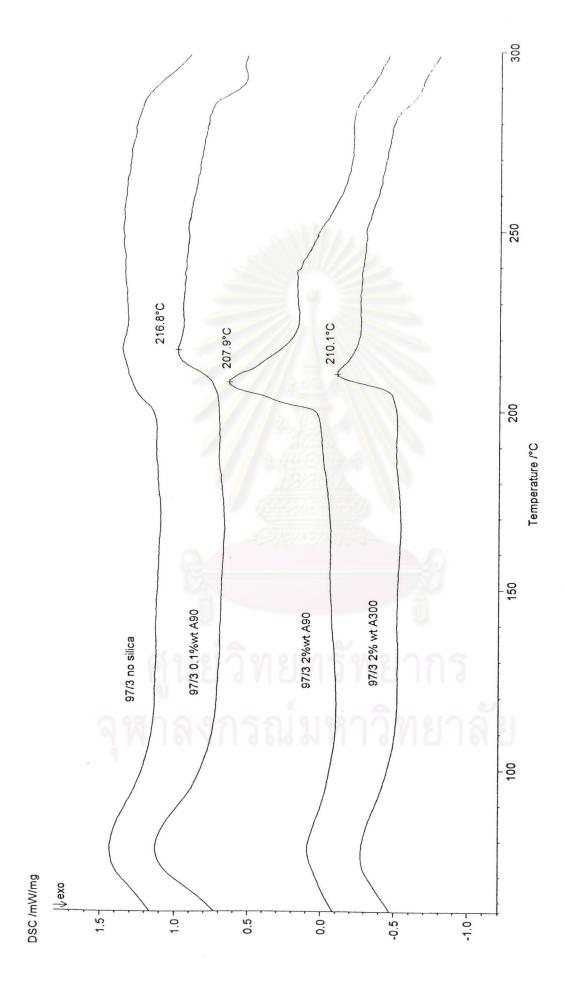


Figure 4.22 DSC thermograms of the synthesized copolymers

### 4.8 The silica retained in the gel

The silica retained in the gel, synthesized by the AM-to-IA ratio of 97:3 with 1.0%wt APS, 0.5%N-MBA, at 45°C, 250 rpm, 30 min, is shown in Tables 4.15-4.16.

Table 4.14 The silica retained in the gel, at various silica concentrations

Silica concentration added to monomer (%)	Silica concentration after sinteration <sup>a)</sup> (%)	Silica retained in the gel (%)
0.1	0.09	90
0.2	0.19	95
0.5	0.44	88
2.0	1.79	90

AM-to-IA ratio, 97:3; Silica type, aerosil 90;

APS, 1.0%wt; N-MBA, 0.5%wt; at 45°C, 250 rpm, 30 min.

Table 4.15 The silica retained in the gel, at various types of silica

Type of silica	Silica concentration after sinteration <sup>a)</sup> (%)	Silica retained in the gel (%)
aerosil 90	1.79	90
aerosil 200	1.85	93
aerosil 300	1.71	86

AM-to-IA ratio, 97:3; APS, 1.0%wt; N-MBA, 0.5%wt; at 45°C, 250 rpm, 30 min.

a) sample weight = 1 g.

a) sample weight = 1 g.

The percentage of silica retained in the gel at AM-to-IA ratio of 97:3 with 0.1, 0.2, 0.5, and 2.0%wt silica aerosil 90, is 90, 95, 88, and 90%, respectively. The percentage of silica retained in the gel, synthesized with AM-to-IA ratio of 97:3 with 2.0%wt silica is 90, 93, and 86% for silica aerosil 90, silica aerosil 200, and silica aerosil 300, respectively. The amount of silica retained in the gel is lower than the amount of silica added to monomer. It can be found that the whole amount of silica is not intercalated in to the polymer network. The BET surface areas (m²/g) of silica aerosil 90, silica aerosil 200, and silica aerosil 300 are 90±15, 200±25 and 300±30, respectively. Their surface areas are quite different but the surface cannot influence the intercalation of the copolymer.

## 4.9 The role of silica in the synthesized copolymers

When a crosslinking agent such as N-MBA was used, the water absorption decreased drastically. It is rather difficult to control the desirable water absorption through chemical crosslinking reaction, inorganic particulate filler with hydrophilic surface such as silica is normally recommended to incorporate during the polymerization of the superabsorbent as the solid crosslinking sites as the polymerized silica superabsorbent. As shown above, the silica particles are dispersed rather evenly in the polymer to give better strength. Although the presence of silica in the superabsorbent polymer reduces water absorption as a negative contribution, it increases the gel strength for water absorption under load as a compliment. When it is assembled as a baby diaper or famine napkins, the absorbed fluid is still held within the superabsorbent network. In this work, we tuned the combination between the chemical crosslinking reaction by N-MBA and the minimum silica incorporation to obtain the desirable water absorption capacity.

On the other hand, one can mix or blend the superabsorbent with the silica particle as an organic and inorganic composites, the resulting composites perhaps can perform similarly as a silica-filled superabsorbent, but not the polymerized silica superabsorbent. The performance of the silica-filled superabsorbent cannot match that the polymerized silica superabsorbent because the loose silica can leak out from the composites and its strength could be somewhat lower. Water absorption value could be in the intermediate between the non-silica superabsorbent and polymerized silica superabsorbent polymer. In the same anticipation, the strength is also in the intermediate between the polymerized silica superabsorbent and non-silica superabsorbent.

