CHAPTER III

EXPERIMENTAL

3.1 Chemicals, equipment and glassware

3.1.1 Chemicals

a) Acrylamide (AM, Siam Chemicals, Thailand)

b) Itaconic acid (Merck, Hohenbrunn, Germany)

c) *N,N'*-methylenebisacrylamide (N-MBA, Fluka, Buchs, Switzerland)

d) Ammonium persulfate (APS, Merck, Hohenbrunn, Germany)

e) *N,N,N',N'*-tetramethylethylenediamine (TMED, Fluka, Buchs, Switzerland)

f) Silicon dioxide (SiO_2, JJ-Degussa-Huts, Thailand)

Table 3.1 Silica type

ITEM	aerosil 90	aerosil 200	aerosil 300
BET surface area, m^2/g	90±15	200±25	300±30
Average primary particle size, nm	20	12	7
pH	3.7-4.7	3.7-4.7	3.7-4.7
SiO_2, %	>99.8	>99.8	>99.8
Al_2O_3, %	<0.05	<0.05	<0.05
Fe_2O_3, %	<0.003	<0.003	<0.003
HCl, %	<0.025	<0.025	<0.025

3.1.2 Equipment and glassware

a) Mechanical stirrer: Ika-Ruhrwerke RW20, Staufen, Germany

b) Water bath circulator: WBU 45 Memmert, Schwabach, W-Germany

c) Magnetic stirrer: Snijders, Tiburg, Holland

d) Analytical balance: Mettler AE260, Switzerland

e) Oven: Hotpack, U.S.A.

f) Muffle furnace: VULCAN 3-550 PD, U.S.A.

g) Other general laboratory glassware and equipment: flat bottomed flask, steel wire filtering with 100-mesh aluminium screen, reduced pressure filtering system, four-necked round bottomed flask, spiral condenser, desiccator, porcelain crucible 100 ml.

3.2 Analytical instruments

a) Fourier transform infrared spectroscopy (FTIR), model Nicolet Impact 410, U.S.A.

b) Scanning electron microscope (SEM), model JSM-6400, JEOL, Japan.

c) Differential scanning calorimetry (DSC), NETZSCH, model DSC 200, U.S.A.

3.3 Procedures

3.3.1 Optimum concentration of itaconic acid

A series of the copolymer superabsorbent were prepared by following procedure:

Copolymerization: A mixture (100 ml) of the AM-to-IA ratios of 100:0, 99:1, 98:2, and 97:3 with 0.5 %wt monomer of the crosslinker (N-MBA) and an initiator (1.0%wt monomer of ammonium persulfate: 0.20 cm^3 of *N,N,N',N'*-tetramethylethylenediamine) was prepared in a 500 ml four-necked round bottomed flask equipped with a mechanical stirrer (with a rate of 250 rpm), a spiral condenser, and a thermometer at 45°C (in a water bath) under nitrogen atmosphere for 30 minutes. The resulting polymer was dewatered with methanol, cut into small pieces, dried in a vacuum oven for 24 h to a constant weight and then milled.

3.3.2 Optimum concentration of inorganic component (silica)

A series of the copolymer superabsorbent were prepared by following procedure:

Copolymerization: A mixture (100 ml) of the AM-to-IA ratios of 97:3 with 0.5%wt monomer of the crosslinker (N-MBA), an initiator (1.0%wt monomer of ammonium persulfate: 0.20 cm^3 of *N,N,N',N'*-tetramethylethylenediamine), and various silica concentrations (0.1, 0.2, 0.5, and 2.0 %wt of the total monomer) was prepared in a 500 ml four-necked round bottomed flask equipped with a mechanical stirrer (with a rate of 250 rpm), a spiral condenser, and a thermometer at 45°C (in a water bath) under nitrogen atmosphere for 30 minutes. The resulting polymer was dewatered with methanol, cut into small pieces, dried in a vacuum oven for 24 h to a constant weight and then milled.

3.3.3 Type of inorganic component (silica)

A series of the copolymer superabsorbent were prepared by following procedure:

Copolymerization: A mixture (100 ml) of the AM-to-IA ratios of 97:3 with 0.5%wt monomer of the crosslinker (N-MBA), an initiator (1.0%wt monomer of ammonium persulfate: 0.20 cm^3 of *N,N,N',N'*-tetramethylethylenediamine), and various the type of silica (aerosil 90, aerosil 200, and aerosil 300) was prepared in a 500 ml four-necked round bottomed flask equipped with a mechanical stirrer (with a rate of 250 rpm), a spiral condenser, and a thermometer at 45°C (in a water bath) under nitrogen atmosphere for 30 minutes. The resulting polymer was dewatered with methanol, cut into small pieces, dried in a vacuum oven for 24 h to a constant weight and then milled.

3.4 Characterization of the copolymers

The synthesized copolymers were investigated for their entities as below:

3.4.1 Identification of the functional groups of the synthesized copolymers

The functional groups of the copolymers were investigated by using a Fourier transform infrared spectroscopy using a KBr pellet. The interface software (Nicolet Omnic) was connected to the Nicolet FTIR in a data acquisition system.

3.4.2 Determination of surface morphology of the synthesized copolymers

The surface morphology of the copolymers was investigated using a scanning electron microscope without cross-section. The thickness of gold coated on the copolymer was 25 nm, and SEM was operated with a 15 or 20 kV accelerating voltage.

3.4.3 Determination of the thermal properties of the synthesized copolymers

The copolymers were investigated with a differential scanning calorimetry for their thermal property. The measurements were carried out over a temperature range of 50 to 300°C with a heating rate of 10°C/min, under the nitrogen flow rate of 10 cm^3/min; the weight of sample used was 5-10 mg.

3.5 Water absorbency of the copolymers

3.5.1 In distilled water

Distilled water (150 g) was added to 0.1 g of the dry copolymer (A) in a 250 cm^3 glass beaker and it was allowed to swell for 30 min. The completely swollen copolymer gel was filtered through 100 mesh aluminium screen for 3 hr and the remaining swollen copolymer gel was weighed (B). The water absorbency (g of water/g of dry copolymer) was calculated by the equation shown below:

$$\text{Water absorbency of copolymer (g/g)} = (B-A)/A \qquad 3.1$$

3.5.2 Absorbency under load

Distilled water (25 ml) was placed in a Petri dish. The dry copolymer weighed 0.16 g (A) was carefully scattered onto the filter screen of the test device (a flexiglass cylinder with 100 meshes made of stainless steel cloth in the bottom: the cylinder diameter 26 mm, height 35 mm). A piston assembly, including an additional weight to achieve a load of 0.28 psi was placed on the top of the dry copolymer. After weighing the assembly device (B), it was placed on the Petri dish, and absorption was allowed for 1 hr. After 1 hr, the entire device was reweighed (C). The similar experiments were each carried out for a load of 0.70 psi. The absorbency under load (g of water/g of dry copolymer) was calculated by:

$$\text{Absorbency under load (g/g)} = (C-B)/A \tag{3.2}$$

3.5.3 Kinetics of absorption of the copolymers

Distilled water (1000 ml) was placed in a 2 liter beaker. The dry copolymer of 0.1 g was sealed in a teabag. The seal teabag was placed in a beaker and it was allowed to swell for 15 min. The soaked teabag was removed from the beaker and the remaining swollen copolymer was weighed. The similar experiments were each carried out for the gel swelling times of 15, 45, 105, 225, 1125, and 1605 min, respectively.

3.6 Determination of silica retained in the gel by sinteration

The dry copolymer weighed 1.00±0.01 g (A) was placed in a 100 ml porcelain crucible. The crucible was placed on a hot plate and was allowed to heat the copolymer for 45 min. After 45 min, the crucible was placed in the muffle furnace at 600°C for 90 min (until combustion of all the remaining white silica powder has occurred). The crucible was removed from the furnace and cool. The remaining content was weighed (B). The remaining content was calculated by:

$$\text{Silica retained in the gel} = (B-A)/A \tag{3.3}$$