CHAPTER III

METHODOLOGY

3.1 Materials

3.1.1 Silica Particles (Size 0.1 micron or 100 NM)

The silica particles used in the study have been synthesized using methods described by G.H. Bogush, M.A. Tracy and C.F. Zukoski IV[33]. The silica particles were formed by the hydrolysis of TEOS (tetraethyl orthosilicate) in an ethanol solution containing ammonia. After stirring overnight, the silica suspension was transferred from ethanol media to water media. Next, the silica suspension was centrifuged until the electron conductivity of supernatant was equal to that of pure deionized water. Then an ultrasonic waves were used to redisperse silica particles in aqueous solutions. The silica particle and the floc size were measured using Dynamic Light Scattering (DLS). The concentration of silica particles in all of the experiments was constant at 7000 ppm.

3.1.2 Salt Solution

Sodium chloride with 99% purity from EM science company were mixed with de-ionized water without further purification.

3.1.3 Polyacrylamide $[-CH_2CH(CONH_2)-]_n$

Polyacrylamide (powder) with molecular weight 5,000,000 - 6,000,000 (from Aldrich Chemical Company) are mixed with deionized water. Stir it over night to achieve completely dissolving polymer in the solution.

Polyacrylamide with molecular weight of 10,000 (aqueous solution) from Polysciences, Inc. was mixed with deionized water. Stir it over night to achieve completely dissolving polymer in the solution.

3.1.4 Glycerol [$HOCH_2CH(OH)CH_2OH$]

Glycerol with 99% purity from Aldrich Chemical was used to study the effect of viscosity on the silica and polymer system.

3.1.5 Surfactant

Cetyltrimethylammonium bromide CTAB, $C_{19}H_{42}BrN$

CTAB, 99 % purity from ACROS Company, was used in this study. Using conductivity measurements, critical micelle concentration (CMC) of CTAB was found to be 0.97 mmol/l or 435 ppm in a.[34].

Tetramethylammonium bromide TMAB, $C_4H_{12}BrN$

Short chain surfactant, Tetramethyl ammonium bromide had the purity of 98% and was obtained from Aldrich Chemical Company.

3.2 Equipment

Dynamic light scattering (DLS) :

The silica particle and the floc size were measured in the water solution. The samples were prepared by adding one drop of the sample in 3 ml water then gentle tumbling it 2 or 3 times. Care should be taken in this process to avoid impurities and bubble in the sample because it might make serious error in the measurement. A laser light scattering was passed through the sample solution. When it hit the particle, it made the light beam deviated with respect to the particle size. The size was determined from the reflective angle of the light.

Rheometer :

The mixing rate of sample solution was controlled by Rheometer. It was used for determining the exact shear applied in this experiment. The disadvantage of the rheometer comparing to the magnetic stirrer was that every time the sample was taken, the rheometer must be stopped. When the mixing was stopped, the particle could aggregate and the mixture turned to be nonuniform. Moreover, large flocs might sink to the bottom of the sample cell. The rheometer was shown in Figure 3.1.

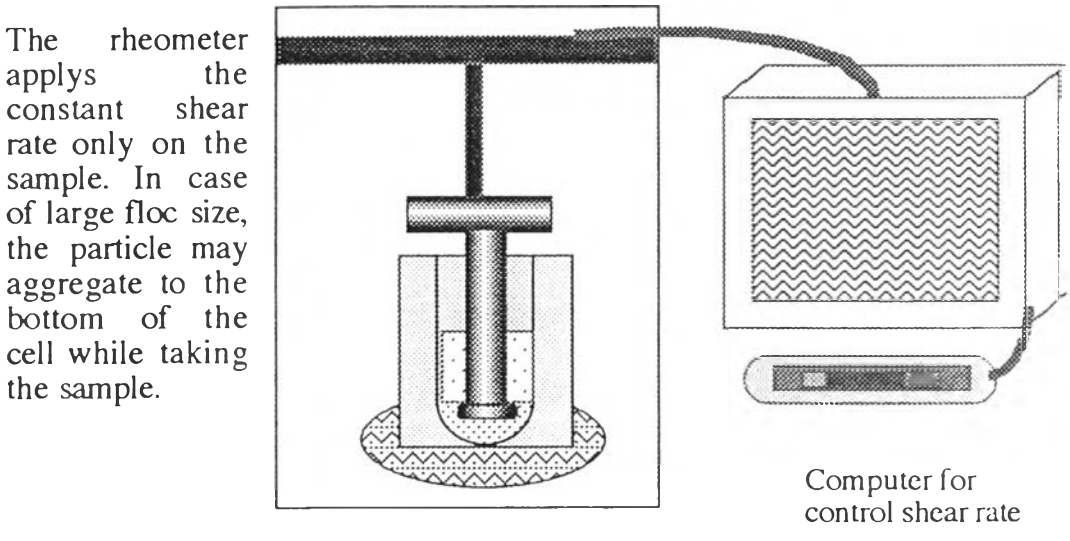

Figure 3.1 The Rheometer.

Zeta potentiometer :

The zeta potential could be measured by a zeta potentiometer. Samples of silica particles were prepared at concentration of 500 ppm in aqueous solution. The principal of this depended on the ions in the particle whether they moved to the positive or the negative side. The potential value (mv) was the value of the opposite force that made particles stop moving in the electric field.

3.3 Experimental Procedure

Following the procedure described, first the silica particles 7000 ppm were mixed with polymer (polyacrylamide) or surfactant (CTAB) using magnetic stirrer until the equilibrium was reached, normally one night. Salt solutions were prepared by adding sodium chloride in deionized water. After that the salt solution was added to the mixture of silica particles and polymer or surfactant, stirred it continuously at constant shear rate by using the magnetic stirrer.

The hydrodynamic diameter of the floc size was measured as a function of time beginning with the time the salt added. Then it was measured every 15 minutes for 3 hours, and was left overnight before measuring the final particle size. All of the experiments were performed at room temperature (23^0C).

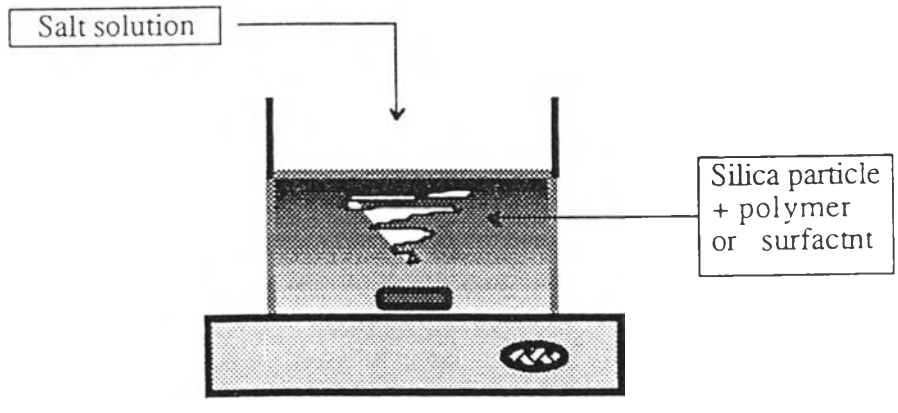

Figure 3.2 Diagram of orthokinetic experiment.

Effects of the following parameters were studied :

- Shear rate :

Magnetic stirrer (high shear rate), rotator (low shear rate), and rheometer were used to investigate the effect of shear force in the stability of silica suspension.

- Viscosity :

The high viscosity could be a hinder for the particle movement. This experiment were performed to show the effect of viscosity in stabilizing the mixture. The silica particles were mixed with polymer or other chemical at the same viscosity. The viscosity of the solution was measured by using capillary tube method and was kept at constant temperature (18 ^{0}C). The experiment was followed the procedure explained above. The examples of sample preparation were shown in Table 3.1. The sample were kept at constant viscosity such as 9 cp. The first sample, silica particle were mixed with a polymer to obtain the viscosity of 9 cp. The second one, pure glycerol were mixed with glycerol instead of polymer at the same viscosity. Finally, the third sample, the silica particle was mixed with both polymer and glycerol.

Table 3.1 Composition of sample in viscosity test

No.	*Silica Particle*	*Polymer*	*Glycerol*	*Viscosity*
1	√	√	-	9
2	√	-	√	9
3	√	√	√	9

- Molecular weight and chain length :

The molecular weight of polymer and the chain length of surfactant were varied. The polymer were - Polyacrylamide (high molecular weight)
- Polyacrylamide (low molecular weight)

The surfactant was - CTAB ($C_{19}H_{42}NBr$)
- Tetramethylammonium bromide ($C_4H_{12}BrN$)

- pH :

pH was adjusted by using HCl or NaCl in the range from pH 4 to pH 10.

- Adsorption :

The amount of surfactant adsorbed on the silica particles was measured using the following procedure. First, surfactant was added to silica suspension, which was continuously rotated for one night or until the equilibrium reached. The amount of surfactant adsorbed on the silica particle was calculated by the difference in the concentration of CTAB suspended in the solution before and after centrifuging. The concentration in the supernatant was measured by two phase titration method (35). Methylene blue was used as an indicator and SDS (Sodium dodecyl sulfonate) was used as the antagonistic substance. The end point was taken as the first appearance of the blue color of the complex formed between excess cationic tritrant and the indicator anionic in the organic phase.

- Redispersion experiment for CTAB :

Silica flocs were formed by the mixing of 100 ppm CTAB and 7000 ppm silica particles in aqueous solution. The result solution was placed in a solution of 2000 ppm CTAB and was sheared in a Rheometer. The particle size was measured from very low shear rate (0.01 1/s) to very high shear rate

(1460 1/s). The shear rate was increased every one hour until the highest shear rate was reached.

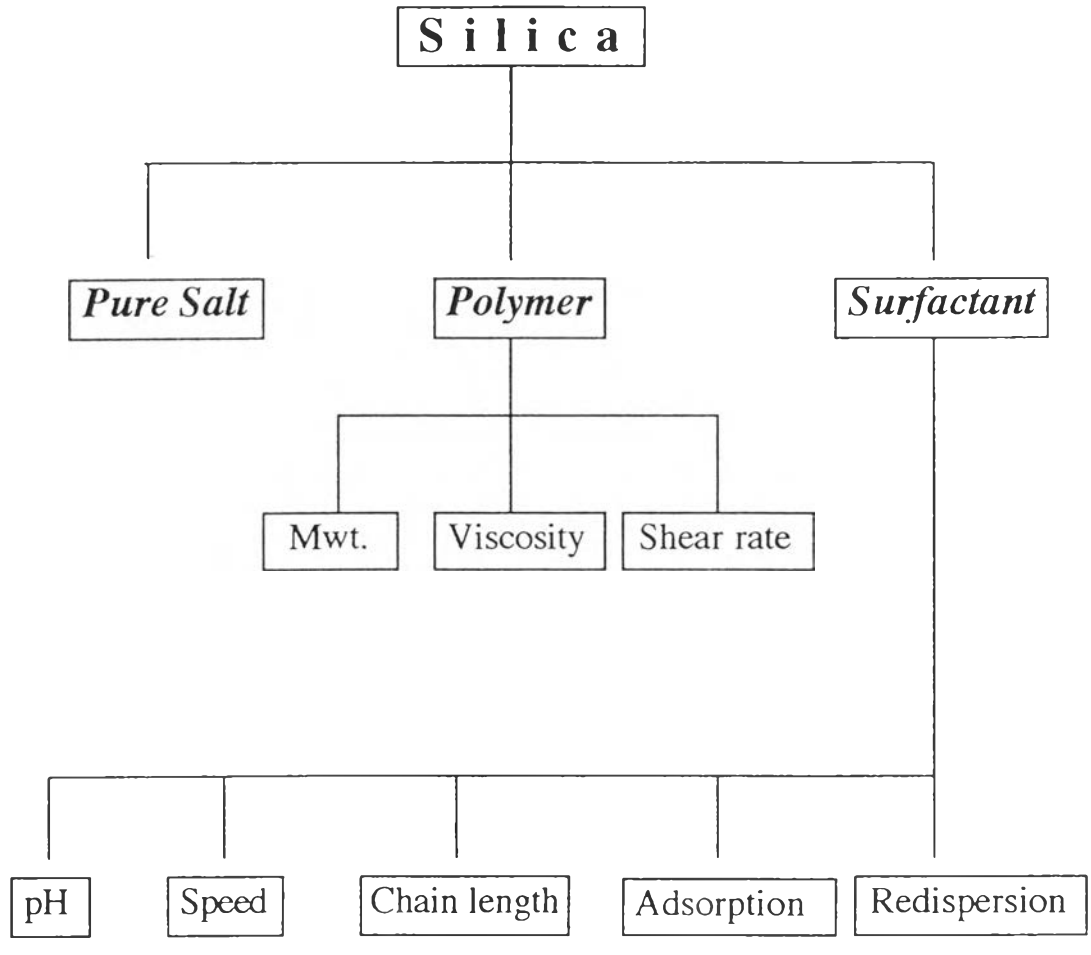

Figure 3.3 Parameters Diagram.