# CHAPTER III
# EXPERIMENTAL

## 3.1 Materials

### 3.1.1 Carbon Black

The carbon black (type 400 R) used in this study was manufactured by Carbot Corporation. The carbon black was necessarily washed in order to remove the ionic salts. The carbon black was mixed with distilled or deionized water in the ratio of 1 to 4 and agitated thoroughly. After that the mixture was centrifuged at 2500 rpm for 15 minutes, and water was decanted off. This procedure was repeated 4 times which were sufficient to reduce the calcium concentration in the rinse water to less than 0.15 ppm. Finally the washed carbon black was dried at 50°C for 5 days. The surface area of the washed carbon black determined by BET surface area was 96 $m^2/g$.

### 3.1.2 Paper Fiber

Fiber paper was prepared by pulping common office paper (Xerox A4 80 GSM) at 5% consistency at 3,000 rpm in disintegrate machine. The pulp slurry was then washed over a filter funnel number 0 (nominal maximum pore size of 160 – 250 μM) to remove extraneous ions especially $Ca^{2+}$. Water from pulp slurry was collected and concentration of $Ca^{2+}$ was detected by Atomic Absorption Spectrophotometer (AAS Varian 300). Washing was continued until concentration of $Ca^{2+}$was less than 0.1 ppm. Pulp slurry was then pressed to remove excess water and was dried at 50°C for 2 days.

3.1.3 Surfactant

Sodium octanoate (C8, $C_8H_{15}O_2Na$) with a purity of 99% was purchased from Sigma Chemical Company (St. Louis, MO) and used without further purification.

3.1.4 Calcium Chloride as Counterion

The reagent grade calcium chloride dihydrate ($CaCl_2.2H_2O$) obtained from Fluka Co., Ltd. (Switzerland) was used in the study. Due to the chemical hygroscopic nature, it was necessary to dry at 90 °C for 12 hours just prior to manufacturing the stock solution.

3.1.5 pH Adjustment Solution

Sodium hydroxide (NaOH) manufactured by J.T. Baker Chemicals B.V. (Deventer, Holland) was used for adjusting pH.

## 3.2 Experimental Procedure

3.2.1 Adsorption Isotherm Experiment

Adsorption isotherms were obtained at 30°C using solution depletion method: 2.5 g of washed carbon black was mixed with 20 ml of stock solution and 1.0 g of dry paper fiber was mixed with 25 ml of stock solution in the vial with screw cap. Then the condition was adjusted to pH of alkaline by NaOH. It was held until reaching equilibrium for 4 days in water bath shaker. After that it was centrifuged at 2500 rpm for 15 minutes. The surfactant and calcium concentration in supernatant liquid was analyzed after decanting off and filtering by 0.22 μM cellulose acetate filter membrane. C8 concentrations were determined by Total Organic Carbon or TOC (model 5000A). The calcium concentrations were analyzed by Atomic Absorption Spectrophotometer (AAS varian 300).

3.2.2 Zeta Potential Experiment

Zeta Potential was determined by using Zeta Meter Model 3.0+. 1.5 mg of carbon black was mixed with 40 ml of stock solution and 0.1 g of dry paper fiber was mixed with 40 ml of stock solution. After that pH was adjusted to alkaline by NaOH and the system was allowed to equilibrate in a water bath shaker at 30°C for a day. The sample was placed in electrophoresis cell. Electrodes placed at each end of the cell are connected to a power supply, which creates an electric field, causing the charged colloid to move. Individual particles are tracked as they travel under a grid in the eyepiece of the microscope. All of experiments were controlled at constant temperature (30° C).