CHAPTER III
EXPERIMENTAL

3.1 Materials

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 standard atomic absorption spectroscopy (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 by oven at $50^{O}C$.

The anionic surfactant, which was selected in the experiment, was Sodium Dodecyl Sulfate (SDS). Schematic of chemical structure of the SDS is shown in Figure 3.1 SDS was purchased from Sigma Chemical Company (St. Louis, MO) with 99% purity. Sodium hydroxide, which was used for adjusting pH, was purchased from Fluka Co., Ltd. (Switzerland). Calcium chloride ($CaCl_2.H_2O$) was purchased from Sigma Chemical Company (St. Louis, MO). Because of the hygroscopic nature of $CaCl_2$ it must be dried by oven at $90^{O}C$ for 12 hours just prior to stock solution preparation. The deionization water was used through out the experiment.

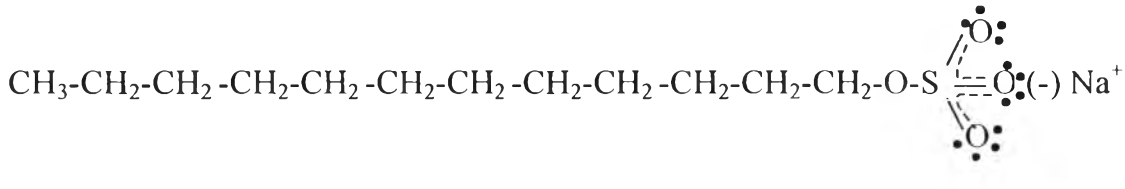

Figure 3.1 Schematic of SDS.

Methanol for HPLC was gradient grade and purchased from Merck KGaA, Germany.

3.2 Method

3.2.1 Adsorption Isotherm Experiment

Adsorption isotherms were obtained at $30^{O}C$ using solution depletion method; 1 g of dry fiber was mixed with 25 ml of stock solution and adjusted for pH of alkaline condition by NaOH in a vial with screw cap. It was held until reaching equilibrium for 4 days in water bath shaker. After that it was centrifuged at 3,000 rpm for 15 minutes. The surfactant and calcium concentration in supernatant liquid was analyzed by decanting off and filtering by 0.2 μm cellulose acetate filter membrane. Surfactant concentration was analyzed by a high performance liquid chromatograph or HPLC (Hewlett Packard series 1050) with an electrical conductivity detector (model 550 Altech Associates, Inc.). The SDS was analyzed employing a bicratic step-change solvent scheme (water-rich/methanol-rich mobile phases) on an C_{18} reverse phase silica column. The primary mobile phase was 30% by volume mixture of methanol in water. At this point, surfactant adsorbed on the reverse phase silica and chloride salt eluted from the column. During running, the composition of mobile phase was changed gradually for 2.5 minutes. After that the composition was constant at 80% by volume of methanol in water. Finally, the mobile phase was switched back to 30% methanol to complete cycle. The calcium concentrations in supernatant liquid 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+. One-tenth gram of fiber was mixed with 40 ml of stock solution and pH adjusted by

NaOH and allowed to equilibrate in water bath shaker at $30^{O}C$ for a day. The sample is placed in electrophoresis cell. Electrodes placed in 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 ($30^{O}C$).