CHAPTER III
EXPERIMENTAL

3.1 Materials

Carbon black with a specific surface area of 96 m^2/g and an average diameter of 0.24 μm was obtained from Cabot company, Inc. Sodium dodecyl sulfate (SDS) with a stated purity of 90% and octyl phenol ethoxylate (Triton X-100) with a purity more than 99% were purchased from Fluka. Three types of testing fabrics of cotton, polyester and polyester/cotton blend were purchased from Test Fabric Co. (Middlesex, VJ, USA).

3.2 Experimental methodology

There were three experimental parts in this work. The first part was surfactant adsorption isotherm experiments the second part was the zeta potential measurement and the third part was detergency experiment.

3.2.1 Adsorption Isotherm Experiments

The adsorption experiments were carried out using different concentrations of either SDS or Triton X-100. Surfactant stock solutions were diluted with deionized water to obtained different surfactant concentrations and added to a screw cap vial containing 0.25 g of carbon black at different pH values. The filled vials were allowed to equilibrate at 30°C in a shaker bath for 4 d. After equilibrium, the supernatants obtained by centrifugation at 12000 rpm for 30 min were then analyzed for surfactant concentrations by using a total organic carbon analyzer (TOC) (Shimadzu, TOC 5000) and a UV-VIS spectrophotometer (Bara Windsor). According to the surfactant adsorption experiments on fabrics, the similar procedure to carbon black was conducted except an amount of 0.5g of fabric sample was used.

3.2.2 Zeta potential measurement

An amount of 1.5 mg of carbon black powder or 0.1 mg of fabric was added into a surfactant solution having different pH values and then the mixture was stirred at 30°C for 24 h. The solution was then transferred to an electrophoretic cell of a zeta meter (Zeta Meter, 3.0+) equipped with a microscope module. After applying a suitable voltage according to the solution conductivity, the average zeta potential value of carbon black was obtained.

3.2.3 PZC measurement

An amount of 0.1 g of fabric was added into deionized water with different solution pH by adding HCl or NaOH. Then the solution was stirred at 30°C for 24 h. The initial pH value of this solution before adding a fabric was measured with a pH meter (Ultra basic DENVER). Measurements were again taken about 24 h after the addition of the fabric [Jaehyeon and John 1995].

3.2.4 Detergency experiments

- Fabric preparation

The testing fabrics of pure cotton, blended cotton-polyester, and polyester were washed with distilled water before use. The pre-washed fabrics were cut into 3x4 inches swatches.

- Soiling procedure
- Laundry procedure

A Terg-O-Tometer which is a standard testing unit for detergency was used in this study. The washing experiment was performed in 1000 mL washing solution with 20 min and followed by the rinse step using deionized water with 3 min first rinse and 2 min second rinse. The temperatures of both washing solution and rinsing water were kept at 30°C. Three swatches were washed in each bucket for on cycle as replication. The washing solution contained different concentrations of SDS and Triton X-100. To monitor the washing efficiency, the color of the testing fabrics was measured before and after washing by using a color metric

spectrophotometer (Color Flex). The percentage of detergency is calculated by the following equation:

$$\%Detergency = \left(\frac{A - B}{C_0 - B} \right) x100 \qquad (1)$$

where A is the average reflectance of the soiled swatches after washing, B is the average reflectance of the soiled swatches before washing and C0 is the average reflectance of the unsoiled swatches before washing.

3.2.5 Contact angle measurement

The contact angle measurement was carried out using the sessile drop technique by a contact angle measuring instrument (Kruss, DSA 10). The carbon black powder, blended polyester/cotton fabric or polyester fabric was first compressed into a smooth sheet. A 20 μL drop of surfactant solution which contained different surfactant concentrations was placed onto the carbon black surface or fabric surface and then the contact angle was measured after 20 s to allowed equilibrium. During the measurement, the sample chamber was kept at 30°C and saturated with water vapor to prevent the evaporation effect.