CHAPTER III
EXPERIMENTAL

3.1 Chemicals

Ferric oxide with a purity of 99% was obtained from Sigma-Aldrich Co. Four types of surfactants used in this study were methyl ester sulfonate (MES) with 90% purity obtained from PTT-Chem, alcohol ethoxylate with 9 ethoxyl groups (AE9) with 99.9% purity supplied by Thai Ethoxylate, linear alkylbenzene sulfonate (LAS), $R(CH)C_6H_5SO_3$, with 80% purity purchased from East Asiatic (Thailand) Public Co., Ltd. and Alfotera, $R((CH)CH_3CH_2O)_3SO_4$, in 28.6% solution supplied by Sasol company. Commercial detergent (Breeze excel) from Unilever Company was perchased from a supermarket in Bangkok. Sodium hydroxide (NaOH), analytical purity grade, and Hydrochloric (HCl), analytical purity grade were used for pH adjustment. Three types of fabrics for detergency testing; pure polyester, pure cotton and blend polyester/cotton were obtained from Test Fabrics Inc.

3.2 Experimental Methodology

There were three experimental parts in this work. The first part was studie of adsorption isotherms on carbon black and fibers, the second part was the zeta potential measurement and the third part was detergency experiment.

3.2.1 Adsorption isotherm experiments

The experiments were carried out to find the amount of surfactant adsorbed on the solid surface and on fiber as a function of surfactant concentration. The adsorption experiments were carried out using different concentrations of surfactants; MES and AE9. Surfactant stock solutions were diluted with deionized water to obtained different surfactant concentrations and added to screw cap vials containing 0.25 g of ferric oxide at different pH values. The filled vials were allowed to equilibrate at 30°C in a shaker bath for 4 days. After equilibrium, the supernatants ware separated from mixtures by centrifugation at 1200 rpm for 30 minutes. The filtered supernatant samples were then analyzed for bulk phase concentrations of

surfactant by using a total organic carbon analyzer (TOC) (Shimadzu, TOC 5000). For the adsorption isotherm experiments on fibers, (cotton and polyester), the similar procedure was conducted but an amount of 0.5g of fiber sample was used.

3.2.2 Zeta Potential measurement

An amount of 1.5 mg of ferric oxide powder or 0.1 mg of fabric was added into a surfactant solution having different concentrations and solution pH values and then the mixture was stirred at 30°C for 24 hours. The solution was then transferred to an electrophoretic cell of a zeta meter (Zeta Meter 3.0+ unit) equipped with a microscope module.

3.2.3 PZC measurement

An amount of 0.1 g of fabric was added into deionized water with a pH of about 5.7 and the solution pH was varied with 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 Instrument). Measurements were again taken about 24 h after the addition of the fabric to obtain equilibrium pHs. [Jaehyeon and John, 1995].

3.2.4 Detergency Experiments

- 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 cotton or polyester was first compressed into a smooth sheet. A 20 μL drop of the surfactant solution which contained different surfactant concentrations was then placed onto a compressed fabric sheet and the contact angle was measured after 20 s. During the measurement, the sample chamber was kept at a constant temperature of 30°C.

- Fabric preparation

The three test fabrics of pure cotton, pure polyester and blend polyester/cotton were washed with distilled water before use. The pre-washed fabrics were cut into 5x5 cm swatches in the warp and weft directions

- Soiling procedure

The ferric oxide was used to soil the test fabrics. An amount of 20 pieces of the pre-washed fabric specimens were soiled in 0.2 g/L of ferric oxide/water dispersion liquid using a Terg–O–Tometer (Copley Scientific, DIS 8000). The dispersion liquid was prepared by ultrasonic treatment for 20 min. Soiling time was 3 min and the temperature of soiling was 30°C with an agitation speed of 100 rpm. After that, the soiled fabric swatch was dried at room temperature for 1 day.

- Laundry procedure

The washing experiments were performed in 1000 mL of a washing solution using the same Terg-O-Tometer at 20 minute wash, 3 minute first rinse and 2 minute second rinse with de-ionized water. The temperatures of both washing solution and rinse water were kept constant at 30°C. Three swatches were washed in each bucket for on cycle as replication. The washing solution contained different concentrations of MES, AE, LAS or Commercial detergent.

- Determination of soil

Soil removal efficiency was determined by reflectance measurement of before washed and after washed swatches and the calculation was expressed in terms of the percentage of detergency (%D). The reflectance measurements of unsoiled swatchs, the soiled swatches and post-washed swatches were conducted by using a colorimetric spectrophotometer (Hunter Lab, Color Flex). The percentage of detergency is calculated by the following equation:

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

where A is the average reflectance of the soiled swatches after washing, B is the average reflectance of the soiled swatches before washing and C_0 is the average reflectance of the unsoiled swatches before washing.

3.2.5 Determination surface area of ferric oxide and fabrics

A ferric oxide weighing 1 g was degassed at 200°C overnight, and pure polyester or pure cotton fabric, or polyester/cotton blend weighing 1 g was cut into very small pieces and degassed at 100°C overnight. Its surface area was then determined by nitrogen adsorption BET measurement by a surface area analyzer (Quanta Chrome, Autosorb-1).