CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Chemicals

Carbon black, was obtained from Cabot Company, Inc. Its surface area is 96 m^2/g and the average diameter is about 0.24 micron.

Two types of surfactants were used in this study Sodium dodecyl sulphate (SDS). Cetyltrimetyl ammonium bromide (CTAB) was obtained from Fluka Chemical Company.

Sodium hydroxide (NaOH), analytical purity grade, and hydrochloric (HCL), analytical purity grade, were purchased from Labscan Asia Co,Ltd. All chemicals were used as received without further purification.

Distilled water which was used throughout this work for preparing aqueous surfactant solution, as rinsing water was purchased from the government pharmaceutical organization, Bangkok, Thailand.

Two types of fabric for detergency tests, a standard unsoiled pure polyester and pure cotton were purchased from Test Fabrics Co. (Middlesex,VJ, USA).

3.2 Experimental Methodology

There were two experimental parts in this work. The first part was the study of adsorption isotherm on carbon black and fiber and the second part is the detergency experiment.

3.2.1 Adsorption Isotherm Experiment

This experiment was carried out in order to find the amount of surfactant adsorbed on the solid surface and on fiber as a function of surfactant concentration.Adsorption experiments were carried out using different

concentrations of surfactants of cetyltrimethylammonium bromide (CTAB) and sodium dodecyl sulphate (SDS). Surfactant stock solutions were diluted with deionized water to obtain different surfactant concentrations and added to screw cap vials contaning 2.5 g of carbon black. The filled vials were allowed to equilibrate at 30°C in a shaker bath for 4 days. After equilibrium, the supernatant was separated from the mixtures by centrifugation at 12000 rpm for 30 minutes. The concentrate samples were then analysed for the bulk phase concentrations of surfactant using a total organic carbon analyzer (TOC) (Shimadzu, TOC 5000) For the adsorption isotherm experiment on fibers, (cotton and polyester) the similar procedure was conducted but an amount of 1 g of fiber sample was used. In addition, the effect of solution pH on surfactant adsorption was also studied.

3.2.2 Zeta Potential Measurement

A very small amount of carbon black powder was added into a surfactant solution. The solution pH was adjusted at different values and the mixture was stirred at 30 °C for 24 hours. The solution was then transferred to an electrophoretic cell of a zeta meter (ZM3.0+) equipped with a microscope module. After applying a suitable voltage according to the solution conductivity, the time for any observable carbon black to move for a certain distance was measured. For a given data set, at least 20 carbon black particles were monitored. The average time was used to calculate the average zeta potential value of carbon black under particular conditions.

3.2.3 Fabric Preparation

The two testing fabrics of cotton and polyester were washed with distilled water before use. The pre-washed fabrics are cut into 3 × 4 inches swatches in the warp and weft directions.

3.2.4 Soiling Procedure

An amount of 0.02g of carbon black was added into 80g of distilled water. The mixture was well mixed by using a magnetic stirrer before pouring on a pre-washed fabric specimen which was placed in a plastic container. The plastic container was placed in a shaker bath for 1 hour. After that, the stained fabric swatch was dried on a glass plate at 100°C for 1 day.

3.2.5 Laundry Procedure

A Terg-O-Tometer which is a standard testing unit for detergency was used in this study. The testing experiment was performed in 1000 mL washing solution, 20 min wash, 3 min first rinse and 2 min second rinse with de-ionized water. Temperatures of both washing solution and rinse water was varied from 20-40°C. Three swatches were washed in each bucket for on cycle as replication. The washing solution contained different concentrations of SDS or CTAB.

3.2.6 Soil Removal Method

To simplify the interpretation of test results, the excess (loose) soil is removed from the fabric by two methods 1) Gravimetric Method and 2) Refraction Method.

Calculation of Efficiency

Detergency performance is determined by reflectance measurement of pre-washed and post-washed swatches and calculated in terms of the percentage of detergency (%D). Reflectance measurements of the unsoiled swatches, the soiled swatches and post-washed swatches were conducted by Color Flex (Hunter Lab). The percentage of detergency is calculated by the following equation:

$$\% \text{ Detergency} = [(A-B)/(C_0-B)]\times 100$$

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.

Calculation of Soil removal

Apart from determination of detergency efficiency, soil removal percentage is determined from the quantity of residual soil on the swatches. The quantity of residual soil is measured from the difference of the dried weight of swatch before and after washing.

3.2.7 Contact Angle Measurement

The contact angle measurement was carried out using sessile drop technique by a contact angle measuring instrument (Kruss, DSA 10). The carbon black powder was first compressed into a smooth sheet. A 5 μL drop of surfactant solution which contained different surfactant concentrations was then placed onto the carbon black sheet and the contact angle was measured after few minutes to allow equilibrium. During the measurement, the sample chamber was kept at 30 °C and saturated with water vapor to prevent drop evaporation effect.

3.2.8 Surface Tension Measurement

The measurement of surface tensions of solutions containing different surfactant concentrations of CTAB and SDS was conducted by using a bubble pressure tensiometer (Kruss, BP2). The rate of bubble generation was varied during measurement of the bubble pressure. The saturated value of the bubble pressure obtained from the plot of the bubble pressure and time was used to calculated the equilibrium surface tension of any surfactant solution.

3.2.9 Determination of Surface Area of Carbon Black and Fabric

A carbon black weighing 1 g degassed at 200 °C overnight, and polyester and cotton fabrics 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 on a Quanta Chrome model Autosorb-1.

3.2.10 Scanning Electron Microscope (SEM)

The surface morphology of the fiber before washed and after washed were observed by SEM (JEOL LS002). They were coated by thin film of gold for 4 minutes prior to analysis.