

CHARPER III

EXPERIMENTAL

3.1 Materials

Kaolinite (purum; natural) was supplied by ITALMAR Co., Ltd. (BKK, Thailand). Motor oil was supplied by Castrol Co., Ltd. (BKK, Thailand). Two studied fabrics; pure cotton, and pure polyester were obtained from Testfabrics, Inc. (Middlesex, NJ, USA). Two types of surfactants used in this study were methyl ester sulfonate (MES, 89% purity) and alcohol ethoxylate with 3, 5, 7, and 9 groups (99% purity) were obtained from PTT Co., Ltd., Commercial detergent (Breeze excel) from Unilever Company was purchased from a supermarket in Bangkok. For other chemicals, Oily-Red-O soluble dye (99% dye content) and hydrofluoric acid were purchased from Sigma-Aldrich (BKK, Thailand). Nitric acid (HNO₃, analytical purity grade) was supplied by Lab-Scan Analytical Sciences (BKK, Thailand). Dichloromethane and 2-propanol (analytical purity grade) were purchased from RCI Labscan Ltd.

3.2 Experimental Methodology

3.2.1 Cloud Point Experiment

Firstly, 1 wt% of mixed surfactant solutions of MES and AE at different MES weight fraction was added into a series of flat bottom test tubes. The solutions were first heated in a water bath close to 100 °C and after that the temperature was slowly decreased to 0 °C with a cooling rate of 1 °C/ 30 min. The lowest temperature when the solution turned cloudy was used to indicate the cloud point.

3.2.3 CMC Determination

A mixed surfactant solution with different weight fractions of MES was prepared at different total surfactant concentration. The surface tension measurement was carried out by using a tensiometer (Kruss, EasyDyne) with the Wil-

helmy Plate method. The reflect point of the plot of surface tension vs. the log of surfactant concentration was used to indicate the critical micelle concentration (CMC).

3.2.4 Detergency Experiments

The test fabric (either pure polyester or pure cotton) was pre-washed with distilled water in order to remove the residues of mill finishing agents. After that, the pre-washed fabrics were cut into 3x4 inch. The prepared test fabric samples were first soiled by soaking into an aqueous solution containing the kaolinite at a concentration of 0.5 wt%. The mixing solution was suspended homogeneously for 20 min by using a Terg-O-Tometer (Copley, DIS8000). The fabric sample was taken to dry at room temperature for overnight. The dried soiled fabric sample was then added into 2 mL of the motor oil stained by the Oil-Red-O dye at room temperature. The soil fabric sample was finally dried at room temperature overnight. For washing process, 1,000 mL of washing solution at different total surfactant concentrations using a selected formulation. The selected formulation was based on the lowest CMC which was found at a weight ratio of MES to AE7= 1:9. The detergency experiments were conducted using a Terg-O-Tometer (Copley, DIS8000). The washing experiment was carried out under following conditions: 20 min for washing step with 120 rpm, 3 min for the first rinse and 2 min for the second rinse. The temperature of washing was set constant at 30°C. For investigating the re-deposition, unsoiled test fabric was introduced additionally in the washing bucket. In addition, a commercial liquid detergent (Breeze, Excel) which is available in Thai market was also tested for a comparison by conducting with the same method as mentioned above.

3.3 Measurement and Analysis Methods

3.3.1 Oil Concentration Analysis

For measurement of oil removal, the residue oil on the test fabric sample was extracted by using 2-propanol with 2 times of extraction overnight at room temperature. The absorbance of extracted solution was measured using an Ultraviolet/Visible spectrophotometer (Shimadzu, UV1800). In addition, an indirect mea-

surement for oil removal was carried out by using a colorimetric spectrophotometer (Hunter Lab, Color Flex).

3.3.2 Silica Analysis

For particulate soil removal, the soil fabric samples before and after the washing experiment were digested with concentrated HF and HNO₃ acids to dissolve the kaolinite. The mixture was filtrated and diluted before subjected to analyze for silica concentration by using an atomic absorption spectrophotometer (Varian, SpectrAA300).

3.3.3 Surfactant Analysis

To measure the residual surfactants on the soiled test fabrics after washing, the surfactant solutions before and after the washing step, and two rinses were taken for surfactant analysis using a HPLC with an evaporative light scattering detector (ELSD) (Alltech, 2000EX).

3.3.4 Detergency Efficiency Calculation

The percentage of detergency is calculated by the following equation:

$$Detergency(\%) = \left[\frac{(A - B)}{(C - B)} \right] \times 100$$

where, A, B, and C are the average reflectance of post-wash, pre-wash soiled fabric and unsoiled fabric, respectively.