สำนักงานวิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย

CHAPTER III
EXPERIMENTAL

3.1 Equipment:

- Terg-o-tometer
- Colorimetric spectrometer (hunter Lab, Color Flex).
- Spinning drop tensiometer (Krüss, DSA10)
- UV/VIS Spectrophotometer
- Video camera (Sony, SSC-DC58AP /1) with the image processing software (Image-Pro Plus 5.1).
- Contact angle tester (Krüss, DSA10).
- Total organic carbon analyzer (Shimadzu, TOC 5000)

3.2 Chemicals:

Oil-red-O soluble dye (solvent Red 27, CI. NO.26125) was purchased from Aldrich Chemical Company, Inc. It was preparing dyed oil solution before being applied on the fabric.

2-propanol, analytical grade, was used to extract the oil from the fabric samples in detergency tests for determining the oil removal after washing.

Sodium chloride (NaCl), used as and electrolyte was purchased from Labscan Asia Co., Ltd.

Dichloromethane, analytical reagent grade, was used for diluting dyed oil before applied on fabrics. It was purchased from Italmar (Thailand) Co., Ltd.

Distilled water was used throughout this research for preparing aquwous surfactant solution, as rinsing water and cleaning glassware.it was purchased from Government Pharmaceutical Organization, Bangkok, Thailand.

3.3 Material

Fabric:

Polyester/cotton blend [65/35] was purchased from Test Fabrics Co.

Surfactant:

There are two type of surfactant will be used in this research work which are branched alcohol propoxylate sulfate sodium salt with 14-15 carbon and 3

propylene oxides (Alflotera 145-3PO) in 28.6% solution which is an anionic surfactant which is pretty long hydrophobic portion, was supplied by Sasol company. And an ionic surfactant and secondary alcohol exthoxylate nonionic surfactant (Tergital 15S5), was purchased from Union Cabide Co., Ltd.

Oil:

motor oil commercially avaiable for use in gasoline engines, Castrol GTX type SAE 10W-30. The motor oil used in this research is a commercial product and can vary in composition. The oil was kept in refrigerator at 4 degree celsius under close system.

3.4 Experimental Procedures

3.4.1 Phase study

Phase studies was performed by preparing different aqueous surfactant concentrations. The aqueous surfactant solution and oil was added to flat-bottomed screw-capped tubes. The volumetric ratio is unity. Sample shaken well for 3 min and left in a water bath for attainment of equilibrium at 30°C which could be reached within a few days to a month, depending on the combination of the system. After equilibration, the interfacial tension values between the two equilibrated phases were measured by a spinning drop tension meter, Model 500; University of Texas, Austin, TX.

3.4.2 Detergency performance

3.4.2.1 Fabric preparation and soiling procedure

The fabric Pre-wash to eliminate residues of mill finishing agents that might influence the oil removal results. The pre-washing method was followed ASTM standard guide D4265-98 (Annual Book of ASTM Standards, 2000). The oil was dyed by the oil-soluble Oil-Red-O dye using the standard method (Goel, 1998).

For soiling, apply the oil-soluble dye on the fabrics. Approximately 0.1 g of the oil-soluble dye, having λ_{max} around 520 nm in 100 mL of the oil, is prepared for

use as colored soil for the detergency experiments. The colored oil was filtered until clear. The soiling procedure was done by diluting 10 mL of the clear dyed oil with dichloromethane (or dimethyl chloride) to 100 mL. The fabric was folded and put in a glass container into which the dyed oil solution was poured until the fabric is completely submerged. It was allowed to stand for 1 min. The soiled fabric was then unfolded and laid on a flat plate in a ventilated hood at room temperature overnight in order to dry the soiled fabric. After drying, the soiled fabric was cut into 3x4 inch swatches in the warp and weft directions. It must be marked to assure that all specimens in the same test series was stained and graded with the same fabric orientation. All soiled swatches was kept in a sealed glass container before use. By using this soiling method, the average weight ratio of oil-to-fabric is approximately 0.15. All soiled swatches was freshly prepared for each set of laundry experiments since a different aging of the soil on the textile has an effect on detergency results

3.4.2.2 The laundry procedure

The laundry procedure was carried out by using a terg-o-tometer (Copley, Model DIS 8000), which simulates home washing-machine action in a bench scale unit. The laundry procedure has three steps: first of all washing, solution for 20 min; secondly rinse1, a 3-min rinse and finally rinse2, a 2-min rinse. All three steps are performed with deionized water (DI-water or distilled water). Agitation speed of every step is 110 rpm. Temperatures of both the washing solution and the rinse water are varied from 40-50°C.

3.4.2.3 Detergency measurement

Detergency performance was measured by reflectance measurement of pre-wash and post-wash swatches to determine the percentage of detergency (%D). Sample measurements of the unsoiled swatche, the pre-wash soiled swatches and post-wash soiled swatches are obtained by Color Flex (Hunter Lab). The percentage of detergency was calculated by the following equation (Annual Book of ASTM Standards, 2000):

$$\%Detergency = [(A - B)/(C_0 - B)]x100 ,$$

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.

3.4.2.4 Oil removal measurement

Oil removal is quantified by percentage of oil removal, which is a portion of residual oil on the swatches to be washed during the detergency process. The residual oil was extracted by submerging a swatch in isopropanol overnight at room temperature, and then the absorbance of the extracted solution was measured at 520 nm of wavelength by an ultraviolet/visible spectrophotometer (UV/VIS spectrophotometer Model 8452A; Hewlett-Packard). The residual concentration of oil was calculated from the calibration curve of control oil solution, which plots between colored oil concentration and absorbance. The oil removal (%) is obtained from the values of oil levels on the swatch before and after washing and compared with a commercial liquid detergent product (CP) was also selected for the detergency experiments under the same laundering conditions.

3.4.2.5 Interfacial tension measurement

Dynamic interfacial tension was measured by a spinning drop tension meter (model 500; University of Texas, Austin, TX). The heavy phase (or dense phase) is the aqueous washing solution and the light phase is the dyed oil. The volumetric ratio of the aqueous solution to the oil of (100:1) was used to measure IFT values. The diameters the oil drop was measured as a function of time. The interfacial tension was calculated from the following equation:

$$IFT = e(Vd)^3 n^2 \Delta\rho ,$$

where IFT is interfacial tension (mN/m), e is 3.427×10^{-4} (mN cm^3 min^2/m g mm^3), V is 0.31 (mm/sdv), d is diameter of the measured droplet (sdv), n is number of revolution or speed (rpm), and $\Delta\rho$ is the density difference between the heavy phase and the light phase (g/cm^3).

3.4.2.6 Adsorption Isotherm measurement:

The adsorption isotherm was measured the amount of surfactant adsorbed on a fabric by using a total organic carbon analyzer (TOC) Shimadzu, TOC 5000.

3.4.2.7 Contact angle measurement:

Contact angle of the oil drops at the fabric/water interface are determined using plates which are made by fusing the fabric (polyester) used in the washing experiments onto clean glass slides. After separation of the glass and fabrics, the fabric surface is smoother than that of standard Mylar plate, giving more reproducible contact angles.

3.4.2.8 Oil Detachment:

A 1μl drop of Motor oil on a dry compressed fabric surface for about 30 s after that, 20 ml of surfactant solution was added to cover up the compressed fabric surface. The oil detachment rate was investigated by using a video camera (Sony, SSc-DC58AP /1) with the image processing software (Image-Pro Plus 5.1).