



CHAPTER III

EXPERIMENTAL PROCEDURES

3.1 Materials

Alkyl diphenyl oxide disulfonate (ADPODS) or Dowfax 8390 was obtained as a 36% solution from Dow Chemical Co. (Midland, MI, USA). Dioctyl sodium sulfosuccinate (Aerosol-OT or AOT) with 98% purity was purchased from Fluka Co. Sorbitan monooleate (Span 80), a nonionic surfactant, was obtained from ICI Uniquema Co. (Wilmington, DE, USA). The extended surfactant used in this work which was sodium alkyl polypropylene oxide sulfate (R-(PO)₃-SO₄Na) with the alkyl (R) group consisting of a branched hydrocarbon chain with 14 to 15 carbons (C₁₄₋₁₅) and 3 propylene oxide numbers connecting between the head and tail groups was provided by Sasol North America Inc (Lake Charles, LA) as a 28.6% solution. Secondary alcohol ethoxylate with five EO groups, nonionic surfactant, was purchased from Utica Chemical Co.Ltd, the supplier of Dow Chemical in Thailand.

Palm oil (Palm olein, Morakot) was purchased from TOP supermarket in Thailand in 2005. Analytical grade NaCl was purchased from LabScan Asia Co, Ltd. Motor oil which is commercially available for use in gasoline engines, type SAE 10W-30 (Castrol GTX) was used as a model oily soil. Oil red O (solvent Red 27, CI. No. 26125) was purchased from Aldrich Chemical Company, Inc. All chemicals were used as received without further purification. Standard unsoiled pure polyester, pure cotton and a polyester/cotton blend (65/35) were purchased from Test Fabrics Co. (Middlesex, NJ, USA).

3.2 Methodology

The experiments of this research can be divided into two parts. The first part is to study the phase behavior and microemulsion formation of mixed surfactant systems with motor oil or palm oil and another part is detergency experiments. For

all of the experiments, the concentrations of surfactant and electrolyte are expressed in weight percent of the aqueous solution.

3.2.1 Phase Behavior and Microemulsion Formation

Phase studies were performed by first adding an aqueous surfactant solution to flat-bottom-screw-cap tubes. Then, the studied oil (motor oil or palm oil) was added to the aqueous surfactant solution to achieve a volumetric ratio of unity. The aqueous surfactant solutions added were prepared with different concentrations of surfactants and NaCl. In this study, both surfactant and NaCl concentrations are reported in weight percentage based upon the aqueous phase. All vials containing the mixtures were well shaken for 3 min and placed in an incubator to equilibrate at 30 °C for approximately one month. Figure 3.1 illustrates the steps of the solution preparation of phase studies.

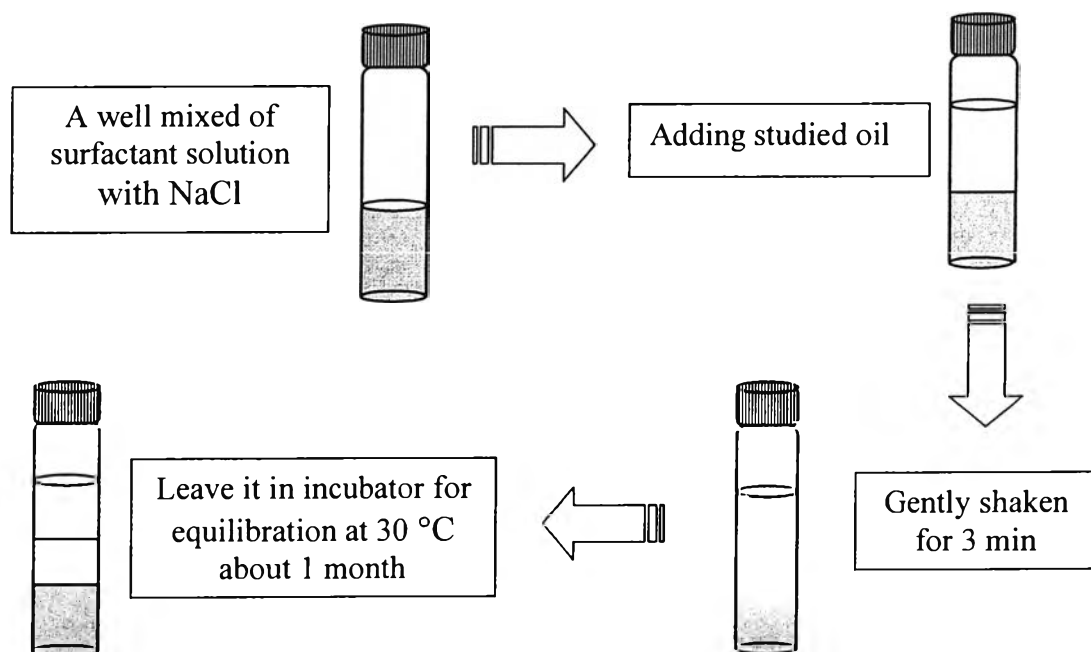


Figure 3.1 Experimental procedure of microemulsion formation.

After equilibration, the height of each liquid phase was measured using a cathetometer having a high precision of ± 0.01 mm. The solubilization capacities were calculated in terms of the solubilization parameter, which is the volume of either oil or water dissolved per weight of total surfactants. The IFT values between the two equilibrated phases were measured by using a spinning drop tensiometer (Krüss, SITE 04).

3.2.2 Surface Tension Measurements

The surface tension of surfactant solutions having different concentrations with and without NaCl was determined by the DuNouy ring technique using a tensiometer (Krüss, K10T). The surface tension measurement was conducted at 30 °C. The plot between surface tension vs total surfactant concentration was used to determine the critical micelle concentration (CMC).

3.2.3 Fish Diagram Study

The types of microemulsion of the fish diagram were classified by the visual observation, conductivity and IFT measurements. For each condition, the electrolytic conductivity was measured, under gentle stirring with a magnetic stirrer, by using a conductivity meter (Cyberscan, con110). The conductivity and the IFT results were used to plot the phase diagram or conditions where Winsor Type I, II and III microemulsions exist. When plotted as surfactant concentration vs salinity (or vice versa), these phase plots are called fish diagrams.

3.2.4 Detergency Experiment

3.2.4.1 *Fabric Pretreatment and Soiling Procedure*

Pre-washing of test fabrics was done to eliminate the residues of mill-finishing agents. The pre-washing method followed the ASTM standard guide D4265-98 (ASTM D 4265-98, 2000). The studied oil was stained by the oil soluble Oil-Red-O dye using the standard method (Goel, 1998b) before being applied on the fabrics. Approximately 0.1 g of the oil-soluble dye was added to 100 mL of the oil. The colored oil was filtered until clear of solids. The soiling procedure was done by diluting 10 mL of the clear dyed oil with dichloromethane to 100 mL. The

fabric was folded and completely submerged in the dyed oil solution 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. Then the dried soiled fabric was cut into 3×4 inch swatches in wrap and weft directions. All swatches were freshly prepared for each batch of laundry experiments.

3.2.4.2 Laundry Procedure

Detergency experiments were carried out by using a tergotometer (Copley, DIS 8000). The tergotometer simulates home washing-machine action in a bench scale unit. The washing experiments were performed in 1000 mL washing solution with 20 min washing time. Distilled water was used for rinsing. To investigate the effect of rinsing more thoroughly, the total volume of rinsing water used was kept constant at 2000 mL while the volume of rinsing water for each rinse step was varied for three sets of experiments at 1000, 500 and 333.33 mL which corresponded to 2, 4 and 6 rinses, respectively. The first rinse took 3 min, while each subsequent rinse step took 2 min. All experiments were carried out at a constant temperature of 30 °C. Three swatches were washed in each bucket and 3 replication experiments were performed for each experimental condition. In order to determine the correlation between phase behavior and detergency performance, NaCl was added to the washing solutions to obtain the same salinity as that in the Winsor type III microemulsion region in the phase studies.

3.2.4.3 Detergency Measurement

Detergency performance is determined by reflectance measurement of pre-wash and post-wash swatches. Reflectance measurements of the unsoiled swatches, the pre-wash soiled swatches and post-wash soiled swatches are conducted by a colormetric spectrophotometer (Hunter Lab, ColorFlex). The percentage of detergency is calculated by the following equation:

$$\% \text{ Detergency} = [(A-B)/(C_0-B)] \times 100 \quad (3.1)$$

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

3.2.4.4 Oil Removal Measurement

Oil removal is characterized by portion of residual oil on the swatches which is washed out during the detergency process. The attached oil was extracted from the fabric sample by submerging a swatch in 2-propanol overnight at room temperature and the amount of extracted oil in the solution was measured by reflectance using a UV/VIS Spectrophotometer (Hewlett Packard, 8452A), a procedure discussed by Goel (Goel, 1998b).

3.2.4.5 Dynamic IFT Measurement

Dynamic IFT measurements were carried out using a spinning drop tensiometer and a contact angle measurement analyzer by the drop shape analysis method (Krüss, DSA10 Mk2). The heavy phase was the aqueous washing solution or rinsing solution and the light phase was the dyed oil. A volumetric ratio of the aqueous solution to the oil of 100:1 was used to measure IFT values. The diameters of the oil drop were measured as a function of time, while the rotational velocity was held constant.

3.2.4.6 Concentration of Surfactant Measurement

The ADPODS concentration, in both washing and rinsing solutions, was measured by the UV/VIS spectrophotometer at a wavelength of 235 nm, which was shown to be the highest absorbance for ADPODS. The concentration of AOT and Span 80 were assumed to be in the same proportion as that of ADPODS. The surfactant residue on the fabric's surface of each step was calculated by the concentration difference method, assuming that the surfactant adsorption on the glass container in the detergency experiments was negligible.