CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Surfactants

There were three types of surfactants used in this research work which are alkyl diphenyl oxide disulfonate (ADPODS or Dowfax 8390), dioctyl sodium sulfosuccinate (Aerosol-OT or AOT) and sorbitan monooleate known as Span80.

Alkyl diphenyl oxide disulfonate (ADPODS) in 36% solution used in this research was a commercial grade anionic surfactant supplied by Dow Chemical Co. known as Dowfax8390 (Midland, MI, USA).

Dioctyl sodium sulfosuccinate (Aerosol-OT or AOT) was purchased from Fluka Company with 98% purity. AOT is a hydrophobic anionic surfactant with a negatively charged sulfosuccinate head group and alkyl chain length of twenty carbon units.

Sorbitan monooleate (Span 80) in 100% solution was obtained from Sigma-Aldrich (Steinheim, Germany). Span80 is a nonionic surfactant. Selected characteristics and properties of the studied surfactants are shown in Table 3.1.

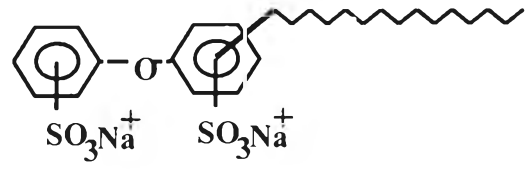
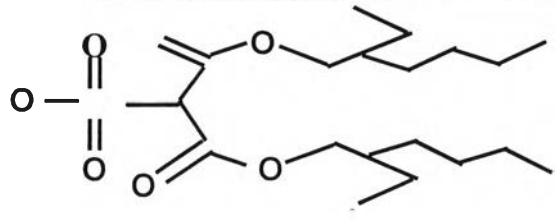
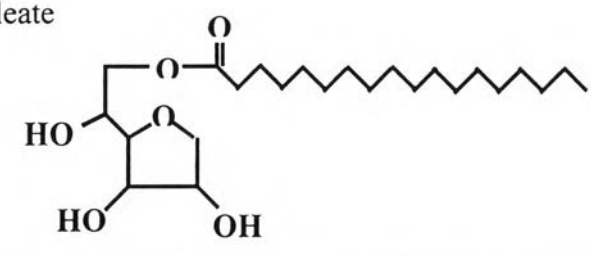
3.1.2 Studied Oil

Motor oil used in the study is commercially available for use in gasoline engines, type SAE 10W-30 (Castrol GTX). As the motor oil used in this research is a commercial product and vary in composition, the same batch of oil was used throughout this research.

3.1.3 Water

Distilled water was used throughout this research for preparing aqueous surfactant solutions, washing step, rinse step and cleaning glassware. It was purchased from Government Pharmaceutical Organization, Bangkok, Thailand.

Table 3.1 Properties of surfactants used in the study

Chemical name	Chemical structure	MW	HLB
Alkyl diphenyl oxide disulfonate (ADPODS)		642	+40
Bis (2-etylhexyl) sulfosuccinate acid Sodium salt (AOT)		444	10.2
Sorbitan monooleate (Span80)		428.6	4.3

3.1.4 Electrolyte

Sodium chloride (NaCl), analytical purity grade, was used as an electrolyte and purchased from LabScan Asia CO, Ltd.

3.1.5 Oil Dye

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

3.1.6 Fabric

Fabric for detergency tests, a standard unsoiled polyester/cotton blend (65/35), was purchased from Test Fabrics Co. (Middlesex, NJ, USA)

3.1.7 Other Chemicals

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

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

3.2 Experimental Procedures

The experiment part of this research was divided into two parts. The first one was to study the phase behavior and microemulaion formation with mixed surfactant systems and another part was detergency experiment. All of the experiments, the concentrations of surfactant and electrolyte were expressed in weight percent of the aqueous solution.

3.2.1 Phase Behavior and Microemulsion Formation

Phase studies were prepared by first adding an aqueous surfactant solution to flat-bottom-screw cap-tubes. Then the oil was added at water to oil volumetric ratio of unity. Aqueous surfactant solutions were prepared at different concentrations. The resultant mixture (surfactant solution mixed with the oil) was gentle shaken and left in an incubator for equilibration at 30 °C as illustrated in Figure 3.1.

The equilibrium of microemulsions was found to take a few weeks. After equilibration, each of phase volume was used to determine the solubilization parameters of both oil and water. The S^* value was determined at the point where the solubilization parameter of water equal the solubilization parameter of oil. The volumes of all phases of microemulsion were measured by using a cathetometer, model TC-II from Titan Tool Supply, Inc. attached to a digimatic height gauge, model 192-631, obtained from Mitutoyo with 0.01 inch accuracy. The Interfacial tension between equilibrated phases was measured by a spinning drop tensiometer (SITE 04, Krüss GmbH, Hamberg).

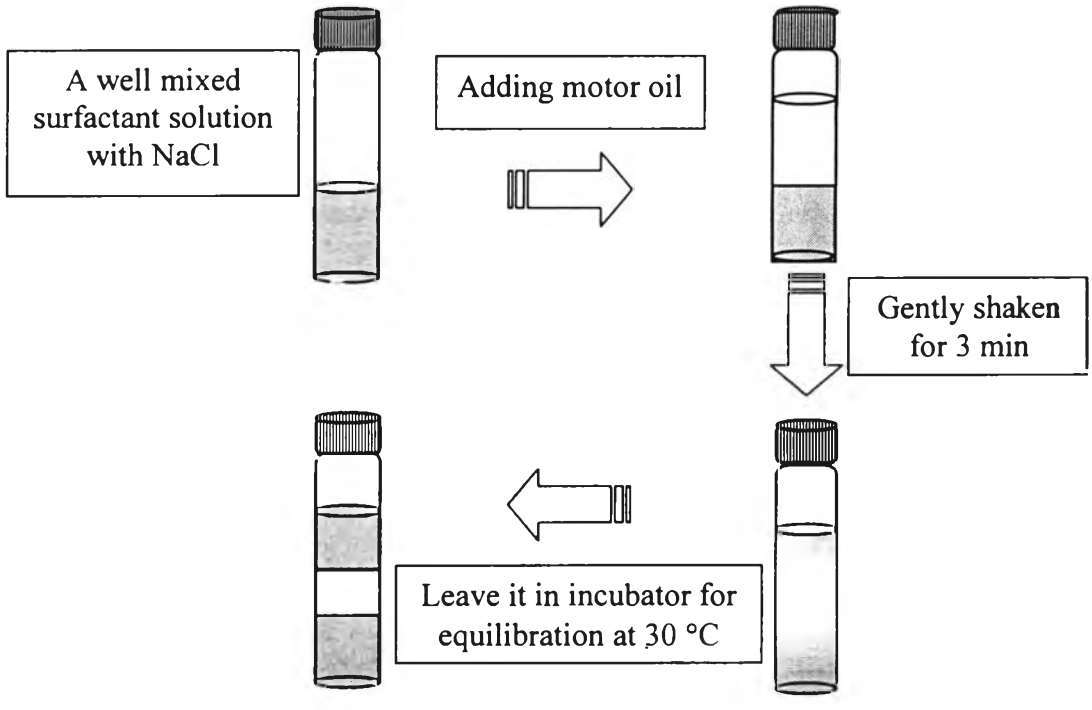


Figure 3.1 Schematic experiment of microemulsion formation.

3.2.2 Detergency Experiment

3.2.2.1 Fabric Preparation

Before soiling, the fabric was pre-washed to eliminate residues of mill finishing agents, which might influence oil removal results. The pre-washing method followed ASTM standard guide D4265-98 (Annual Book of ASTM Standards, 2000).

3.2.2.2 Soiling Procedure

A tested oil was dyed by an oil soluble Oil-Red-O dye using the standard method (Goel, 1998), before being applied on the fabric. Approximately 0.1 g of oil-soluble dye having λ_{max} around 520 nm was added to 100 mL of the oil was prepared for use as color soil for detergency experiments. The colored oil was then filtered until clear. The soiling procedure was done by diluting 10 mL of the clear dye oil with dimethyl chloride to 100 mL. The fabric was folded and put in a grass container, and then the dyed oil solution was poured until the fabric was completely submerged. It was left for 1 min before it was taken and rinsed to remove the adhered solution. The soiled fabric was then unfolded and lay on the flat plate in ventilated hood to dry at room temperature overnight. After that the fabric

was cut into 3×4 inch swatches in a wrap and weft directions. All soiled swatches were kept in a sealed glass container before use. All swatches were freshly prepared for each batch of laundry experiment. By this soiling method, the average weight ratio of oil to fabric was approximately 0.15.

3.2.2.3 Laundry Procedure

Detergency experiments were carried out by using a Terg-O-Tometer (Copley, Model DIS 8000). The Terg-O-Tomcter simulates home washing-machine action in a bench scale unit. The washing experiments were performed in 1000 ml washing solution and 20 min washing time. Rinsing water of 1000 ml, 500 ml and 333.33 ml were performed for studying the effect of rinse steps, 3 min first rinse and 2 min after first rinse with distilled water. All experiments were carried out at a constant temperature of 30° C. Three swatches were washed in each bucket for on cycle as replication. From the phase behavior results, the surfactant composition which offered the lowest IFT and the lowest salinity was selected as formulation for this detergency experiment. First, the selected formulation was diluted to obtain different active surfactant concentration. In order to examine the correlation between phase behavior and detergency performance, salt was added to washing solutions so that the salinity corresponding to the microemulsion composition were simulated. Brand a commercial detergent available in Thai market was also used in order to compare the detergency performance with the selected formulation.

3 2.2.4 Detergency Measurements

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

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

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.

3.2.2.5 Oil Removal Measurement

Oil removal percentage was determined from the quantity of residual oil on the swatches. The quantity of residual oil was extracted from the fabric with 2-propanol by submerging a swatch in 2- propanol overnight at the room temperature and the extracted solution was measured the absorbance at 520 nm by UV/VIS Spectrophotometer. UV/VIS spectrophotometer (Shimadzu 2550) was used to quantify the dye content in the solution at 520 nm. The residual concentration of oil was calculated from the calibration curve of control oil solution. The %oil removal was obtained from the value of oil levels on the swatch before and after wash. This method showed that the dye and the oil were removed by the surfactant solution in the same proportional which they were loaded on the fabric (Goel, 1998).

3.2.2.6 Dynamic Interfacial Tension Measurement

Dynamic interfacial tension was measured by using the spinning drop tensiometer. The heavy phase was the aqueous washing solution at 0.119 % of the studied formulation (0.5% ADPODS, 5% AOT, 3% Span 80) at different salinity and the light phase was the dyed oil.

3.2.2.7 Concentration of Surfactant Measurement

Concentration of each surfactant was assumed as a proportion to Dowfax 8390 that was measured by UV/VIS-Spectrometer (Shimadzu, 2550) at a wavelength of 235 nm.