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 (Span 80).

Alkyl diphenyl oxide disulfonate (ADPODS) known as Dowfax 8390 in 36% solution used in this research was a commercial grade anionic surfactant supplied by Dow Chemical Co. (Midland, MI, USA).

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

Sorbitan monooleate (Span 80) in 100% solution was obtained from ICI Uniquema Co. (Wilmington, DE, USA). Span80 is a nonionic surfactant. Properties and selected characteristics of the surfactants studied are shown in Table 3.1.

3.1.2 Studied Oil

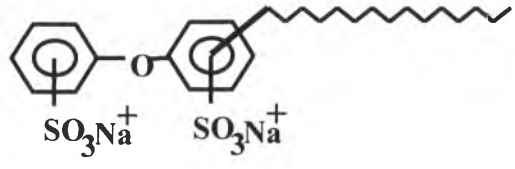
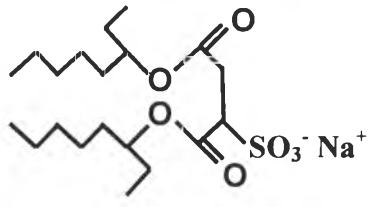
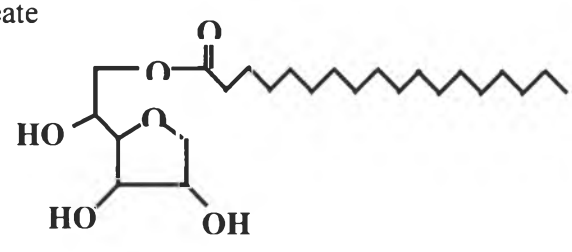
Motor oil which is commercially available for use in gasoline engines, type SAE 10W-30 (Castrol GTX) used as model oily soil. Since the motor oil used in this research is a commercial product and can vary in composition, a single batch of oil was used throughout this research. The oil was kept in a refrigerator at 4 °C until used.

3.1.3 Water

Distilled water was used throughout this research for preparing aqueous surfactant solutions, as rinsing water and cleaning glassware. It was

purchased from Government Pharmaceutical Organization, Bangkok, Thailand.

Table 3.1 Properties of surfactants used in the study (Tongcumpou *et al.*, 2003)

Chemical name	Chemical structure	MW	HLB
Alkyl diphenyl oxide disulfonate (Dowfax 8390)		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 Dyed Oil

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 fabric samples.

3.1.6 Fabrics

Three types of fabric for detergency tests, a standard unsoiled pure polyester, pure cotton and polyester/cotton blend (65/35), were 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 Procedure

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

3.2.1 Phase Behavior and Microemulsion Formation

Phase studies were carried out by first adding an aqueous surfactant solution to flat-bottom-screw cap-tubes. Then the oil was added at a water to oil volumetric ratio of unity. Aqueous surfactant solutions were prepared with different concentrations of three surfactants. Each of mixture (surfactant solution and motor oil) was well shaken for 3 min and left in an incubator for equilibration at 30 °C Figure 3.1 illustrates how to carry out the phase studies.

The equilibrium of the system means the volume of each phase does not change. For the studied system in this research, it took about 1 month to reach equilibrium. The volumes of all phases of microemulsion were measured by using a cathetometer, consisting of a telescope (model TC-II, Titan) and a digimatic height gauge (model 192-631, Mitutoyo) with 0.01 mm accuracy. The solubilization capacities were determined in terms of solubilization parameter as ml of motor oil dissolved per gram of surfactants. The Interfacial tension between equilibrated phases (water excess phase and middle phase or middle phase and oil excess phase) were measured by a Spinning drop tensiometer (SITE 04, Krüss GmbH, Hamberg).

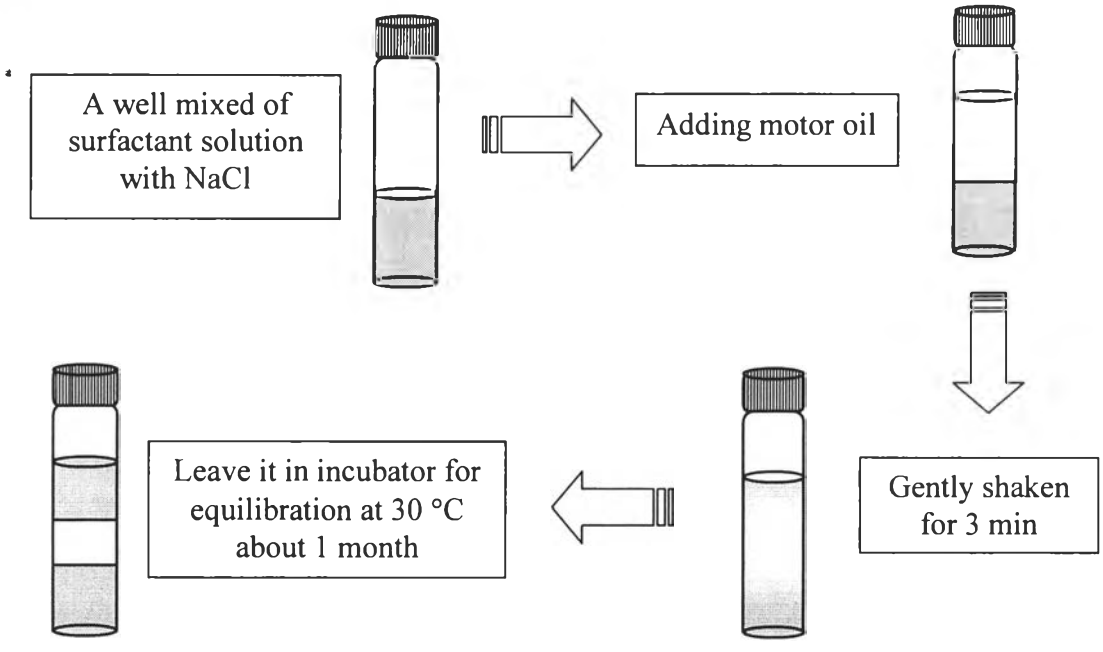


Figure 3.1 Schematic experiment of microemulsion formation.

3.2.2 Detergency Experiment

In detergency test, there were several steps as followings:

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 was followed according to the ASTM standard guide D4265-98 (Annual Book of ASTM Standards, 2000).

3.2.2.2 Soiling Procedure

The tested oil, motor 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 the oil-soluble dye was added to 100 mL of the oil. The colored oil was filtered until clear. The soiling procedure was done by diluting 10 mL of the clear dyed oil with dichloromethane to 100 mL. The fabric was folded and put in a glass container, and then the dyed oil solution was poured until the fabric

was completely submerged. It was left for 1 min and then rinsed to remove the adhered solution. The soiled fabric was then unfolded and lay on a flat plate in a ventilated hood to dry at room temperature over night. 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 until 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 0.21.

3.2.2.3 Laundry Procedure

Washing experiments were carried out by using a Terg-O-Tometer (Copley, model DIS 8000). The Terg-O-Tometer simulates home washing-machine action in a bench scale unit. It consists of 8 vessels with individual paddles for using. These vessels are arranged in water bath for controlling the washing temperature. It has an external thermo-circulator water heater with overtemperature cut-out. The temperature measurement by the thermocouple (Pt100 probe) mounted on the water bath connected to a temperature controller and there also has a cooling coil for cooling down water. The agitator of each bucket is similar to a top-loading home washing machine.

The washing experiments were performed in 1000 ml, 500 ml and 333.33 ml washing solution for the same washing and rinsing times, of 20 min wash, 3 min first rinse and 2 min second rinse with distilled water. Temperatures of both washing solution and rinse water were kept constant at 30°C. Three swatches were washed in each bucket for on cycle as replication. 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.

3.2.2.4 Detergency Measurement

Detergency performance was 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 were conducted by a colormetric spectrophotometer (Hunter Lab, ColorFlex). 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 was determined from the quantity of strained oil on the swatches to be washed out during the detergency process. The quantities of the strained oil before washing and the residual oil after washing were extracted from the fabric with 2-propanol by submerging a swatch in 2- propanol overnight at room temperature and the extracted solution was measured the absorbance at 520 nm by a UV/VIS Spectrophotometer (Hewlett Packard, 8452A). The residual concentration of oil was calculated from the calibration curve of the prepared oil solutions. This method was represented that the dye and the oil were removed by the surfactant solution in the same proportion which they were loaded on the fabric (Goel, 1998).

3.2.2.6 Dynamic Interfacial Tension Measurement

Dynamic interfacial tension was measured by using a spinning drop tensiometer (SITE 04, Krüss GmbH, Hamberg) and a contact angle measurement instrument (DSA10 Mk2, Krüss GmbH, Hamberg). The heavy phase of surfactant mixture was the washing solution and the light oil phase was the dyed oil. A ratio of surfactant solution to oil of 100:1 was used to measure interfacial tension.