CHAPTER III
EXPERIMENTAL

3.1 Material

3.1.1 Surfactants

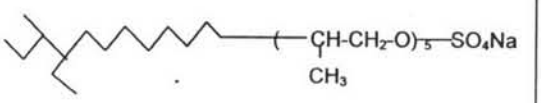
There were two types of surfactants used in this research work which are Branched alcohol propoxylate sulfate sodium salt with 14 – 15 carbons and 3 propylene oxides (Alflotera 145-3PO) and secondary alcohol exthoxylate nonionic surfactant (Tergitol 15S5).

Branched alcohol propoxylate sulfate sodium salt with 14 – 15 carbons 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

Secondary alcohol exthoxylate nonionic surfactant (Tergitol 15S5) was purchased from Union Carbide Co., Ltd.

The properties and characteristics of the surfactants studied are shown in Table 3.1

Table 3.1 Properties of surfactants used in the study

Chemical name	Chemical structure	MB	HLB
Alflotera 145-3PO		595	-
Tergotol 15S5	$CH_3(CH2)_{15}$-O($CH_2CHO)_5H$	415	10.5

3.1.2 Oil

In this work, motor oil which is commercially available for use in gasoline engines, Castrol GTX type SAE 10W-30, is used as the model of oily soil. A single batch of oil was used throughout this research since the motor oil can vary in composition. The oil was kept in refrigerator at 4 degree celsius under close system.

3.1.3 Water

Distilled water was used throughout this research for preparing aquous surfactant solutions, as rinsing water.

3.1.4 Electrolyte

An electrolyte, Sodium chloride (NaCl), was used in this research and perchased 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 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 Commercial Detergent

Brand a commercial liquid detergent available in Thai market was also used in order to compare the detergency performance with the selected formulation. The composition was contained; Sodium Linear Alkybenzene Sulphonate 4% (W/W); Ethoxylate Alcohol 2% (W/W); and, Sodium Lauryl Ether Sulphate 8.5% (W/W).

3.1.8 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 part was to study the phase behavior and microemulsion 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

Firstly, an aqueous surfactant solution was added to a series of flat-bottom-screw cap-tubes. Then the oil was added at a 1:1 water to oil volumetric ratio. Aqueous surfactant solutions were prepared with different concentrations of the two surfactants and NaCl. Each mixture was shaken well for 3 min and left in an incubator for equilibration at 30°Cas 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 which the solubilization parameter of water equals 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). .

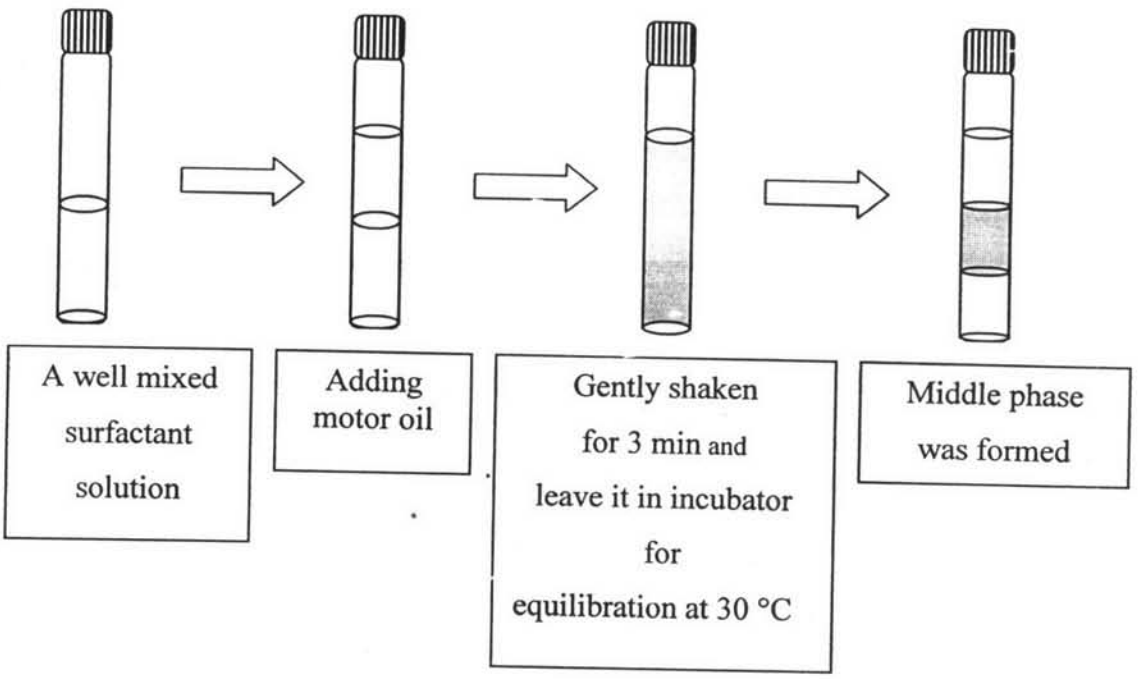


Figure 3.1 Schematic experiment of microemulsion formation.

3.2.2 Detergency Experiments

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 di-methyl 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-OTometer(Copley, Model DIS 8000). The Terg-O-Tometer 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. Rinsing in 1000 ml of distilled water was performed for 3 min in the first rinse and 2 min in the second rinse. All experiments were carried out at a constant temperature of 30° C. Three swatches were washed in each bucket for one 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 the washing solutions so that the salinity corresponding to the microemulsion composition was simulated.

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 was determined from the quantity of residual oil on the swatches. The quantity of residual oil was extracted from the fabric sample by submerging a swatch in 2- Propanol overnight at room temperature and the extracted solution was measured for absorbance at 520 nm by a UV/VIS Spectrophotometer (Hewlett Packard, 8452A). 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 solutions (Goel, 1998).

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).