

CHAPTER III EXPERIMENTAL

3.1 Materials

3.1.1 Studied Surfactants

In this research work, methyl ester sulfonate sodium salt with 16-18 carbons and 95% purity was supplied by Malaysia Palm Oil Board (MPOB). Second, branched alcohol propoxylate sulfate sodium salt with 14-15 carbon and 3 propylene oxides (Alfotera 145-3PO) was provided by Sasol. Third, alcohol ethoxylate nonionic surfactant (AE) with 99% purity was supplied by Cognis (Thailand). Fourth, linear alkylbenzene sulfonate (LAS) sodium salt with 10-13 carbons and 80% purity was purchased from The East Asiatic (Thailand) Public Co., Ltd.

The chemical structure and molecular weights of the studied surfactants are shown in Table 3.1

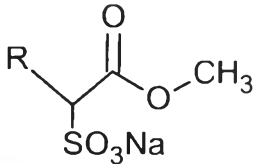
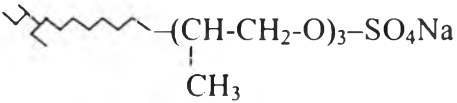
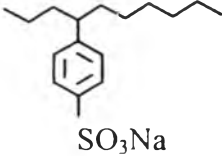
Name of Surfactants	Chemical Structure	Molecular Weight
Methyl Ester Sulfonate (MES)		382.71
Alfotera 145-3PO		483
Linear Alkylbenzene Sulfonate (LAS)		344
Alcohol Ethoxylate (AE)	$C_{12}H_{25}O(CH_2CH_2O)_xH$	318 - 582

Table 3.1 Properties of used surfactants in this work

3.1.2 Oil

In this work, palm oil which is commercially available for use in cooking, Morakot, was used as the model of oily soil.

3.1.3 Dyed Oil

Oil red O (solvent Red 27, No. 26125) was purchased from Aldrich Chemical Company, Inc.

3.1.4 Fabric

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

3.1.5 Electrolyte

An electrolyte, sodium chloride (NaCl) with a purity of 99 %; was purchased from LabScan Asia Co.,Ltd.

3.1.6 Commercial Detergent

A commercial liquid detergent “Breeze” available in Thai market was also used to compare the detergency performance with various studied surfactant systems. The compositions of the commercial detergent are sodium linear alkylbenzene sulfonate of %w/w, sodium lauryl ether sulphate of %w/w, and ethoxylate alcohol of %w/w

3.1.7 Other Chemicals

Dichloromethane, analytical reagent grade, was purchased from Italmar (Thailand) Co.,Ltd. 2-Propanol, analytical reagent grade, was purchased from LabScan Asia Co.,Ltd.

3.2 Experimental Procedures

The experimental work of this research was divided into three parts. The first part was to determine basic properties of MES and the other studied surfactants. The second part was to study the phase behavior and microemulsion formation with mixed surfactant systems and the last part was detergency experiment.

3.2.1 Basic Property Studies

3.2.1.1 *Determination of Tension*

The measurement of surface tension of solutions having different surfactant concentrations was done by using a contact angle measurement instrument (DSA10 Mk2, Kruss GmbH, Hamburg). The plots of surface tension versus initial MES concentration were used to obtain the critical micelle concentration (CMC) of all studied surfactants.

3.2.1.2 *Solubility Determination*

Precipitation point, defined as the point at which an aqueous surfactant solution separates to two phases, liquid phase and solid phase was used to determine the maximum solubility of MES. Each surfactant was varied concentration and was left to precipitate at room temperature. The precipitation point was detected as precipitation concentration (%wt/v) when surfactant solution start to precipitate. In comparison, the higher precipitation concentration the higher solubility in water, since the surfactant remains to be soluble in water.

3.2.1.3 *Foaming Power Determination*

The Ross-Miles test was conducted at 30°C and 0.1 % wt/v of Surfactant concentration. First, 50 ml of surfactant solution was used to rinse the receiver which was controlled at 30°C. Second, the test surfactant solution was filled into a 200 ml pipet which was placed at the top of the receiver. Next, the pipet was opened to allow the test surfactant solution flowed into the receiver. After flowing out completely, the foam height was measured. After 5 min, the remaining foam height was then measured. The first read foam height was used to represent foamability and the second foam height was used to indicate foam stability.

3.2.2 Phase Behavior and Microemulsion Formation Experiment

Phase studies were performed by preparing different aqueous surfactant concentrations. The aqueous surfactant solution and oil were added to a series of flat-bottomed screw-capped tube at a volumetric ratio of unity. The samples were shaken well for 3 and min left in a water bath for attainment of equilibrium at 30°C and a month. After equilibration, the interfacial tension values between the two equilibrated phases were measured by a spinning drop tension meter (SITE 04, Kruss GmbH, Hamberg). Figure 3.1 shows schematic experiment of microemulsion formation.

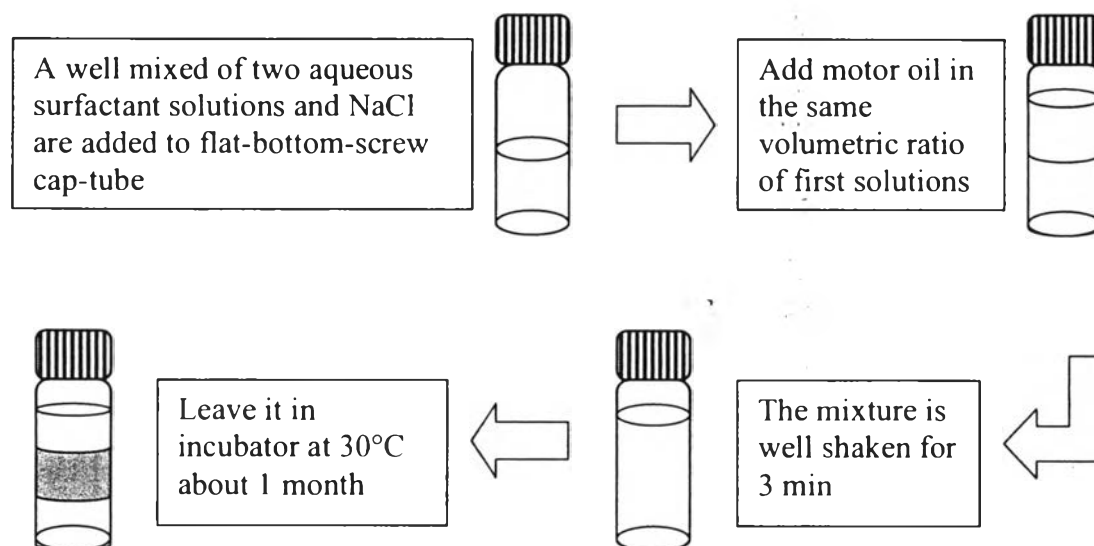


Figure 3.1 Schematic experiment of microemulsion formation

3.2.3 Detergency Experiments

3.2.3.1 *Fabric Preparation*

The test fabric were pre-washed 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).

3.2.3.2 Soiling Procedure

The test oil was dyed by an oil soluble Oil-Red-O dye using standard method (Goel, 1998). Approximately 0.1 g of the oil-soluble dye, having λ_{\max} around 520 nm in 100 mL of the oil, was prepared for use as colored oily 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 test fabric was folded and put in a glass container into which the dyed oil solution was poured until the fabric was 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 was marked to assure that all specimens in the same test series are stained and graded with the same fabric orientation. All soiled swatches were kept in a sealed glass container before use. By using this soiling method, the average weight ratio of oil-to-fabric was approximately 0.15. All soiled swatches were freshly prepared for each set of laundry experiments since a different aging of the soil on the textile has an effect on detergency results.

3.2.3.3 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 had sequential three steps: 20 min for washing, 3 min for a first rinse and 2 min for a second rinse. Deionized water was used as rinse water. The agitation speed was kept constantly at 100 rpm or both steps of washing and rinsing.

3.2.3.4 Detergency Measurements

Detergency performance was measured by reflectance of pre-wash and post-wash swatches to determine the percentage of detergency (%D). The reflectance measurements of the unsoiled swatches, the pre-wash soiled swatches and post-wash soiled swatches were obtained by using colourmetric spectrophotometer (Color Flex, Hunter Lab). The percentage of detergency is calculated by the following equation (ASTM Standards, 2000):

$$\%Detergency = [(A - B)/(C_0 - B)] * (100),$$

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.

3.2.3.5 Oil Removal Measurement

Oil removal is quantified by the 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 was plotted between colored oil concentration and absorbance. The oil removal (%) was obtained from the values of oil levels on the swatch before and after washing, the commercial liquid detergent product (CP) was also selected for the detergency experiments under the same laundering conditions.

3.2.3.6 Residual Surfactant Adsorption on the fabric Determination

For each laundering experiment, a washing and two rinsing solutions were determined or MES concentration by using the titration method according to ASTM standard guide D 1681-92 (Annual Book of ASTM Standards, 2000). The MES concentration was calculated by the following equation

$$M_1V_1 = M_2V_2 \quad (3.2)$$

where M_1 and M_2 are the morality of cationic surfactant and MES concentration in sample respectively, V_1 and V_2 are the used volume of cationic surfactant and sample respectively.

To calculate residual surfactant on the fabric, the principle of mass balance was used. Figure 4.1 shows the calculation method of residual surfactant on the fabric using mass balance principle.

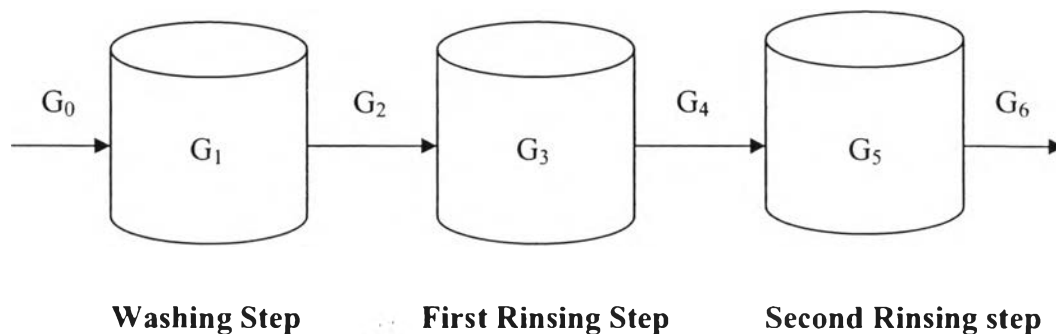


Figure 3.2 Schematic mass balance for calculation of residual surfactant adsorption

G_0 is the initial weight of studied surfactant which is the product of the surfactant concentration and the volume of washing solution. G_1 is the weight of surfactant after washing, obtained from titration method. G_2 , $(G_0 - G_1)$, is the weight of residual surfactant on the fabric after washing. G_3 is the weight of studied surfactant after first rinsing, obtained from titration method which is the product of the surfactant concentration in the first rinse and the volume of the first rinsing water. G_4 , $(G_2 - G_3)$, is the weight of residual surfactant on the fabric after first rinsing. G_5 is the weight of surfactant after second rinsing, obtained from titration method. G_6 , $(G_4 - G_5)$, is the weight of residual surfactant on the fabric after second or last rinsing