

CHAPTER III EXPERIMENTAL

In this research, the experiments of detergency testing and analyzing methods were described below.

3.1 Materials and Equipment

3.1.1 Materials

- Extended surfactant (Alfoterra C_{12,13}-4PO-SO₄Na) supplied by Sasol North America (Lake Charles, LA). This surfactant has a sulfate head group and a hydrophobic tail of methyl-branched alkyl of 12-13 carbons with approximately four propylene oxide (PO) units which having an active content of 29.5 % with 3.8 % free oil (unsulfated alcohol), 0.75 % Na₂SO₄, and 65.95 % water. This sample was used as received and for research purposes.
- Sodium mono- and dimethyl naphthalene sulfonate (SMDNS, 95+%) supplied by CK Witco (Houston, TX).
- 1-dodecanol (98+%) supplied by Sigma-Aldrich.
- Methyl palmitate was purchased from RCI Labscan.
- Sodium chloride (NaCl) > 99 % purity was purchased from RCI Labscan.
- Distilled water
- Oil-soluble dye Oil-red-O was purchased from Sigma-Aldrich.
- All types of fabrics were obtained from Test fabrics, Inc. (Middlesex, NJ, USA).
- Commercial liquid detergent (Breeze excel) from Unilever Company was purchased from a supermarket in Bangkok.

3.1.2 Equipments

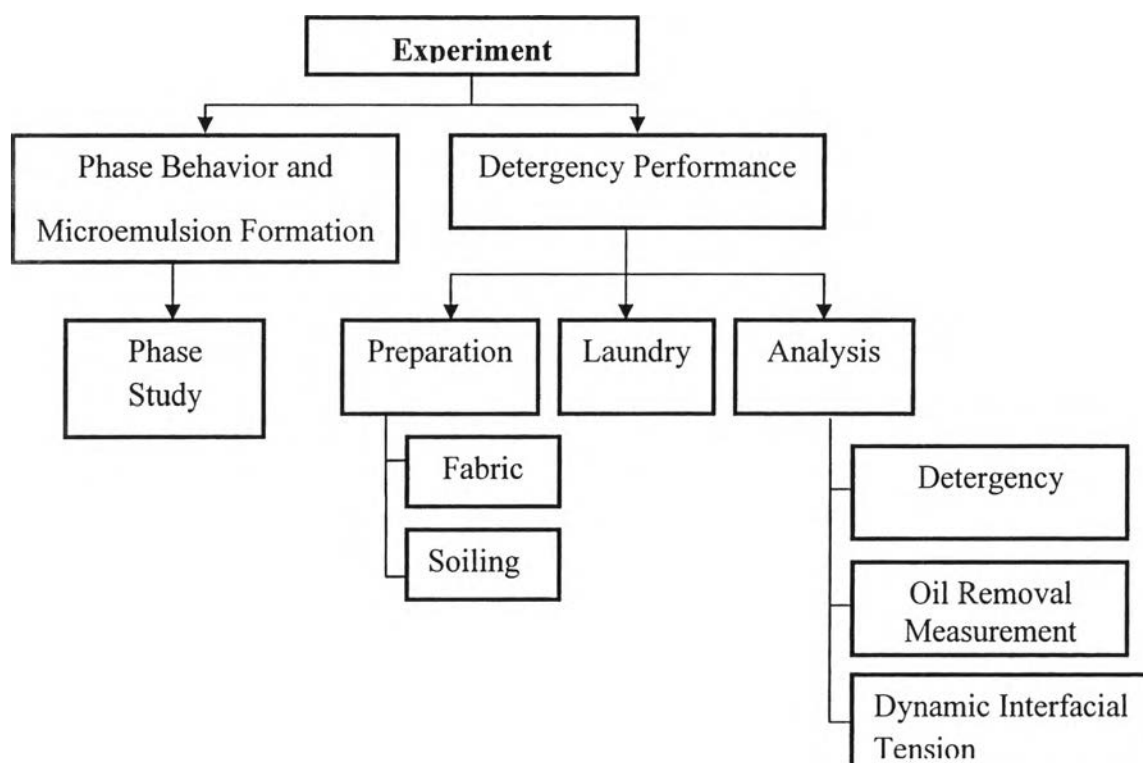
- Temperature-controlled incubator (BINDER, KB400/E2).

- Water bath
- Terg-O-Tometer
- UV/VIS spectrophotometer
- Colorimetric spectrophotometer, Colorflex (Hunter Lab)
- Spinning drop tensiometer (SITE 04, Kruss GmbH, Hamburg).
- pH meter

3.2 Experimental Methodology

Overview of Procedures:

Phase behavior and microemulsion formation



In this research, the microemulsion formation of extended surfactants ($C_{12,13}\text{-4PO-SO}_4\text{Na}$) by obtaining phase diagram of methyl palmitate (monoglyceride oily soil)/surfactant/hydrophilic linker/lipophilic linker/water system to form Winsor's Type I-III-II was studied. The temperatures of microemulsion

formation were set up at 40°C. For all experiments, the surfactant, the hydrophilic linker, and the lipophilic linker concentrations were expressed as percent weight by volume based on aqueous solution.

3.2.1 Microemulsion Formation

For microemulsion formation, the experiments were carried out in 10 mL vials. First, 5 mL of aqueous surfactant solutions containing different concentrations of extended surfactant ($C_{12,13}\text{-4PO-SO}_4\text{Na}$), lipophilic linker (1-dodecanol), and hydrophilic linker (SMDNS) were put into vials. After that, 5 mL of methyl palmitate was added to achieve a volumetric ratio of unity (oil-to-water ratio of 1:1) to a series of vials with Teflon screw caps. Each vial was shaken gently by hand for 1 min and then equilibrated in a temperature-controlled incubator (BINDER, KB400/E2) for several weeks. The equilibrium state was justified by observing that the volume of each phase in the vial remained unchanged. Schematic of the experiment for microemulsion formation shows in figure 3.1.

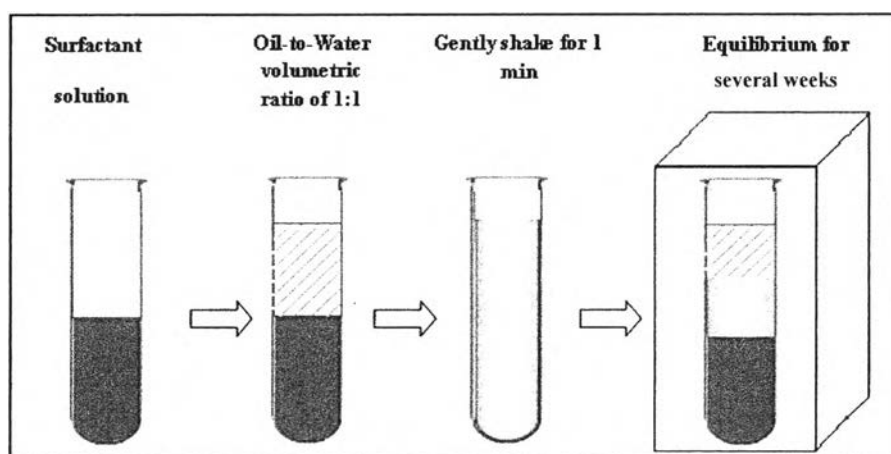


Figure 3.1 Schematic of the experiment for microemulsion formation.

Detergency Testing

3.2.2 Fabric Preparation

The swatch (fabrics sample) having a size of (3×4) inches was pre-washed before soiling to get rid of the residues of mill finishing agents. The prewashing was performed with 1000 mL of distilled water, a 10-min wash cycle with an agitation speed of 120 rpm. After being finished the prewashing process, the swatch was

left to dry overnight at room temperature. This method was followed according to the ASTM standard guide D4265-98.

3.2.3 Soiling Experiment

This method was followed according to Goel (1998)'s method. First, the oil (methyl palmitate) was dyed by the oil soluble Oil-red-O dye; approximately 0.1 g of oil soluble Oil-red-O dye was added to 100 mL of the oil, before applying it to the fabric. Second, the dyed oil was diluted by using dichloromethane (dimethyl chloride) at appropriate ratio of dyed oil to dichloromethane. After that the pre-washed swatch was immersed into a container which containing the dyed oil solution for a specific time. Finally, the prewash soiled swatch was left to dry overnight at room temperature and continued to laundry experiment.

3.2.4 Laundry Experiment

The washing experiments, were conducted by using a Terg-O-Tometer (Copley, DIS8000). Three soiled swatches were washed for 20 min with 1 L of washing solution having different surfactant concentrations (0 - 0.5 %w/v of $C_{12,13}$ -4PO-SO₄Na) and different salinities (0 - 7 %w/v of sodium chloride), and followed by two rinse cycles of 3 min and 2 min, respectively, with 1 L of distilled water of each rinse. All of the experiments were conducted with three soiled swatches as replicates at various washing temperatures (20 - 50 °C). The postwash swatches were allowed to dry overnight at room temperature before determining the detergency performance.

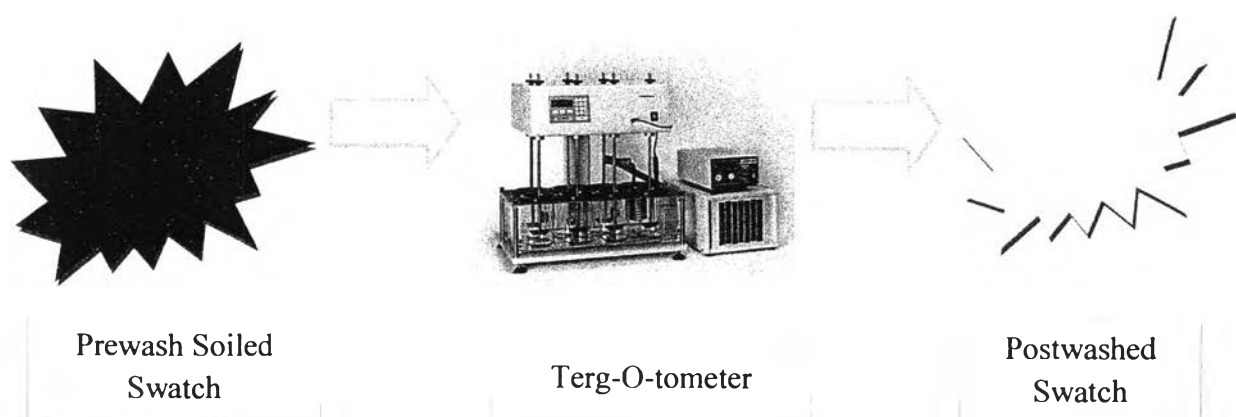


Figure 3.2 Overview of laundry experiment.

3.3 Measurement and Analysis Methods

3.3.1 pH Measurement

The solution was adjusted at different concentration. The pH measurement was conducted by using a pH meter (Ultra basic DENVER Instrument). The pH values were measured the pre-washed solution and the post-washed solution.

3.3.2 Detergency Efficiency Determination

The detergency performance of methyl palmitate (monoglyceride oily soil) was determined by the reflection method. The reflectance measurement was conducted by using a calorimetric spectrophotometer (Hunter Lab, Color Flex). The color change of pre-washed and post-washed swatches was quantified by the lightness parameter (L^*) which reported in the range of 0 (completely black) to 100 (completely white). The calorimeter was calibrated against standard black and white plates before each actual measurement and measured at a minimum of 3 different positions for each sample. The detergency performance was quantified in terms of the percentage of detergency (%D) by using the following equation:

$$\text{Detergency (\%)} = [(A-B) / (C_0-B)] \times 100 \quad [1]$$

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.

3.3.3 Monoglyceride Removal Measurement

This measurement was followed according to Goel (1998)'s method. The residue oil on the test fabric sample was extracted by using 2-propanol and left it overnight at room temperature. The absorbance of extracted solution was measured using an Ultraviolet/Visible spectrophotometer (Shimadzu, UV-1800). In addition, an indirect measurement for oil removal was carried out by using a calorimetric spectrophotometer (Hunter Lab, Color Flex). The monoglyceride removal was quan-

tified in terms of the percentage of monoglyceride removal by using the equation as follow.

$$\text{Monoglyceride removal (\%)} = [(C_0 - C_1) / C_0] (100) \quad [2]$$

where C_0 is the average monoglyceride concentration of the soiled swatches before washing and C_1 is the average monoglyceride concentration of the soiled swatch after washing.

3.3.4 Dynamic Interfacial Tension Measurement

A spinning drop tensiometer (SITE 04, Kruss GmbH, and Hamburg) was used to measure Dynamic Interfacial Tension (IFT). The dense phase was the aqueous washing solution at 0.3 %w/v of total surfactant concentration and different sodium chloride concentrations, and the light phase was the monoglyceride oily soil (methyl palmitate). Additionally, the Dynamic IFT of our studied formulation (0.3 %w/v total surfactant concentration and 3 %w/v sodium chloride) was also determined at different temperatures above melting point. The diameter of the oil drop was measured after the sample was circulated for 20 minutes.