



## 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<sup>®</sup> C<sub>14,15</sub>-4PO-SO<sub>4</sub>Na) supplied by Sasol North America (Lake Charles, LA). This surfactant has a sulfate head group and a hydrophobic tail of methyl-branched alkyl of 14-15 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 % Na<sub>2</sub>SO<sub>4</sub>, and 65.95 % water. This sample was used as received and for research purposes.
- Methyl palmitate was purchased from RCI Labscan.
- Sodium chloride (NaCl) > 99 % purity was purchased from SAFC.
- Distilled water
- Oil-soluble dye Oil-red-O was purchased from Sigma-Aldrich.
- Fabric which was white fabric of polyester/cotton (65:35) blend was obtained from Testfabrics, Inc. (Middlesex, NJ, USA).
- Commercial liquid detergent (Breeze excel) from Uniliver Company was purchased from a supermarket in Bangkok.

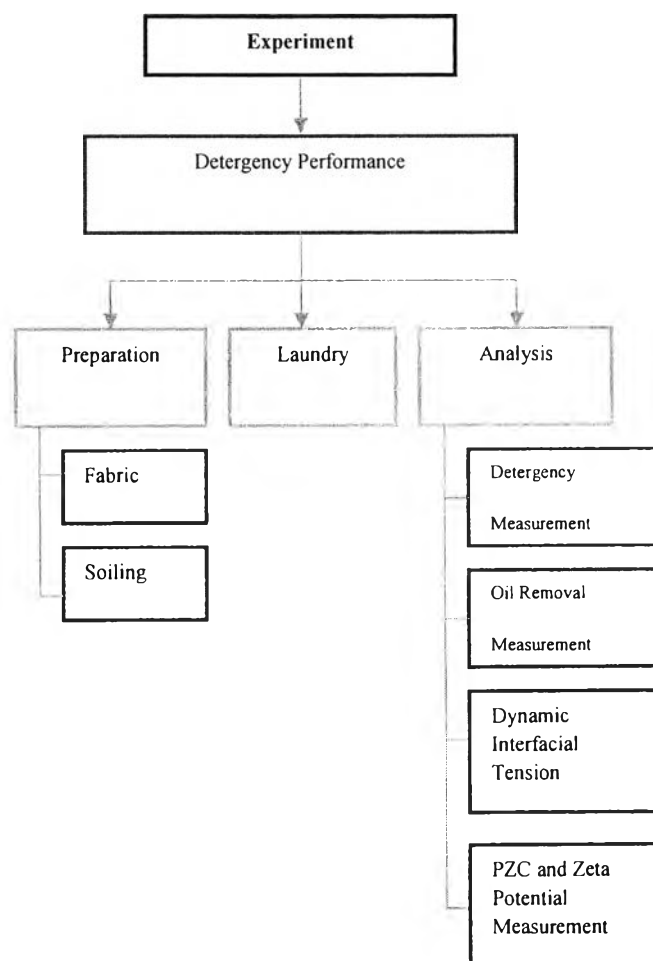
#### 3.1.2 Equipments

- Spinning drop tensiometer (SITE 04, Kruss GmbH, Hamburg).
- Temperature-controlled incubator (BINDER, KB400/E2).
- Water bath
- Terg-O-Tometer
- UV/VIS spectrophotometer

- Colorimetric spectrophotometer, Colorflex (Hunter Lab)
- pH meter
- Zeta meter

### 3.2 Experimental Methodology

Overview of Procedures:



#### Detergency Testing

##### 3.2.1 Fabric Preparation

The swatch (polyester/cotton blend fabric sample) having a size of (3×4) inches was prewashed 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

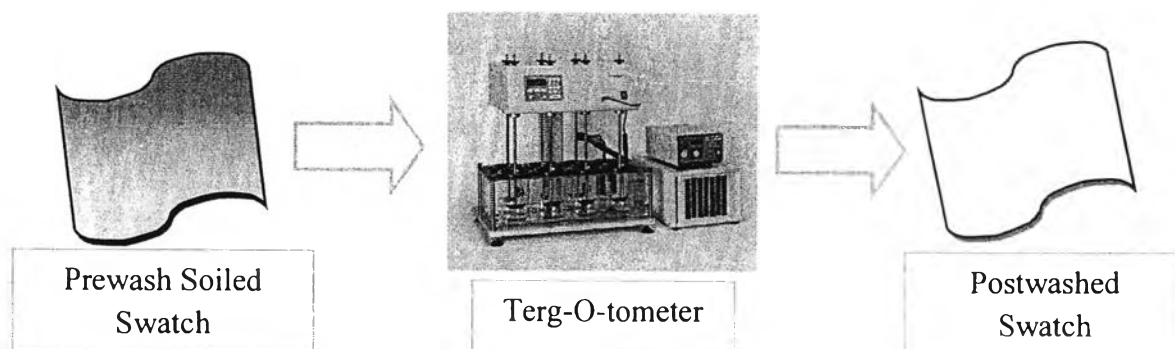
process, the swatch was left to dry overnight at room temperature. This method was followed according to the ASTM standard guide D4265-98.

### 3.2.2 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 prewashed 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. This research the difference ratios of dyed oil to dichloromethane which were 1 to 3, 1 to 5, 1 to 7, and 1 to 8 were carried out at difference soiling time (2 min, 5 min, and 10 min) in order to find the appropriate ratio and soiling time.

### 3.2.3 Laundry Experiment

For laundry experiment, it was conducted by using a Terg-O-Tometer (Copley, DIS8000). Three prewash soiled swatches were washed for 20 min with 1 L of a washing solution having different surfactant concentrations (0 - 0.5 %w/v of Alfoterra<sup>®</sup> C<sub>14,15</sub>-4PO-SO<sub>4</sub>Na) and different salinities (1 - 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 prewash soiled swatches as replicates at various washing temperatures (10 - 50 °C). The postwash swatches were allowed to dry overnight at room temperature before determining the detergency performance.



**Figure 3.1** Overview of laundry experiment.

### 3.3 Measurement and Analysis Methods

#### 3.3.1 PZC Measurement

The samples were added into distilled water which having different solution pH. The solution pH was adjusted at different values by using a 0.1 M HCl or 0.1 M NaOH solution. After that, the solution was then kept at 20 °C for 24 h. The initial pH value of this solution before adding a sample and the final pH after 24 h were measured with a pH meter (Ultra basic DENVER Instrument).

#### 3.3.2 Zeta Potential

An amount of 3.5 mg of semi-solid oil was added into a surfactant solution that having different surfactant concentrations and salinities. Then, the mixture was stirred and kept at 20 °C for 24 hours. After that, the solution was transferred to an electrophoretic cell of a zeta meter (Zeta Meter 3.0+ unit) equipped with a microscope module. After applying a suitable voltage, the charged particles move towards to the electrode until attaining a steady state (the particles move with constant velocity). Their velocities were measured and referred as their electrophoretic mobility which automatically calculated the zeta potential in millivolt units by using the Helmholtz-Smoluchowski equation. For a given data set, at least 20 particles were monitored and the average zeta potential value was reported.

### 3.3.3 Detergency Efficiency Determination

The detergency performance of methyl palmitate (semi-solid 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.

### 3.3.4 Semi-Solid Oil 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 semi-solid oil removal was quantified in terms of the percentage of semi-solid oil removal by using the equation as follow.

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

where  $C_0$  is the average semi-solid oil concentration of the soiled swatches before washing and  $C_1$  is the average semi-solid oil concentration remains of the soiled swatch after washing.

### 3.3.5 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 3 %w/v of sodium chloride and different surfactant concentrations, and the light phase was the semi-solid oil (methyl palmitate). Additionally, the Dynamic IFT of our studied formulation (0.1 %w/v extended surfactant concentration and 3 %w/v sodium chloride) was also determined at different temperatures. The diameter of the oil drop was measured after the sample was circulated for 20 minutes.