

CHAPTER III

EXPERIMENTAL PROCEDURES

3.1 Materials

3.1.1 Gemini Surfactants

Gemini surfactants named “FE-1020E” and “FE-1030E” were supported by Miyochi Oil & Fat Co., Ltd. Structure and properties of these surfactants are shown in figure 3.1 and table 3.1 respectively.

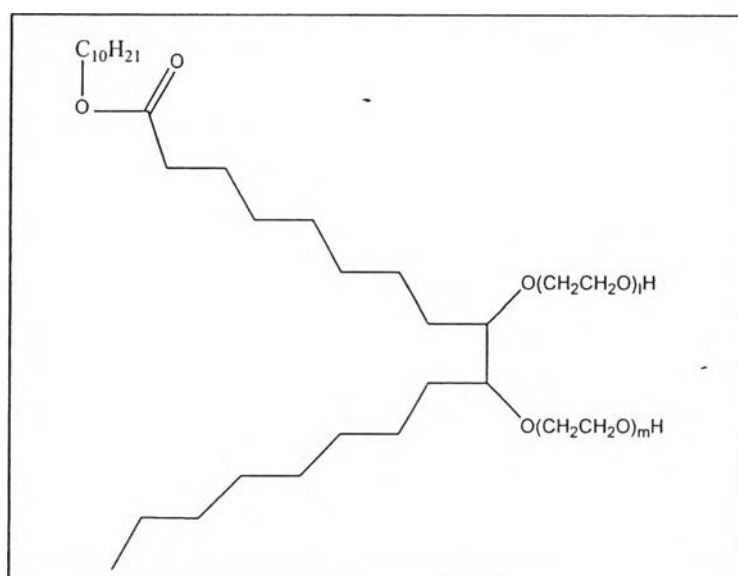


Figure 3.1 Structure of FE-(I+m)E ; (I+m = 20, 30).

Table 3.1 Properties of FE-(I+m)E

	FE-1020E	FE-1030E
OHV	96.8	64.6
pH(1%)	6.2	5.3
Cloud point (degree)	45	72
HLB	12.7	15.1
state (25 degree)	Light yellow liquid	White liquid
cmc (M)	3.6×10^{-6}	2.0×10^{-6}
γ cmc (mN/m)	35	37

3.1.2 Methyl Ester Sulfonate (MES)

Methyl Ester Sulfonate (MES) were supported by Lion Co., Ltd. Structure and properties of these surfactants are shown in Figure 3.2 and Table 3.2 respectively.

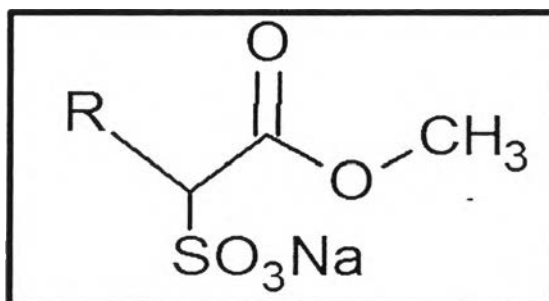


Figure 3.2 Structure of MES.

Table 3.2 Properties of MES

	MES-14C	MES-16C	MES-18C
Weight, g	400.0	500.0	500.0
Active ingredient, wt%	99.8	99.0	99.5
Water content, wt%	0.2	1.0	0.5
Total, wt%	100.0	100.0	100.0

3.1.3 Methyl Ester Ethoxylate (MEE)

Methyl Ester Ethoxylate (MEE) were supported by Lion Co., Ltd. Structure and properties of these surfactants are shown in Figure 3.3 and Table 3.3 respectively.

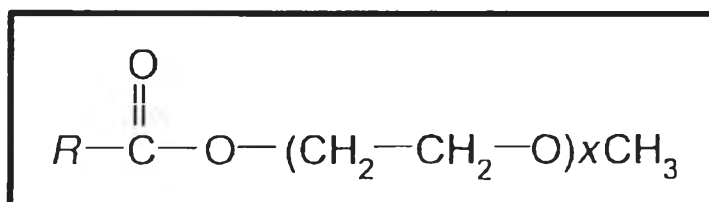


Figure 3.3 Structure of MEE.

Table 3.3 Properties of MEE

	C12MEE-8EO	C12MEE-14EO	C12MEE-18EO
Average EO number	8.3	13.6	17.6
Weight, g	1000	1000	1000
Active ingredient, wt%	91.1	91.8	90.2
Water content, wt%	8.9	8.2	9.8
Total, wt%	100	100	100

3.1.4 Palm Oil

Palm oil (Palm olein, Morakot) was purchased from Tesco Lotus in Thailand in 2013.

3.1.5 Distilled Water

Distilled water is used to prepare all solutions.

3.1.6 Oil-Soluble Dye Oil-Red-O

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

3.1.7 Fabric

Pure cotton and pure polyester are obtained from Testfabrics, Inc. (Middlesex, NJ, USA)

3.1.8 Commercial Grade Liquid Detergent

Commercial grade liquid detergent was purchased from a local store and was tested as a representative commercial liquid detergent.

3.1.9 Dichloromethane

Analytical grade dichloromethane (99.8% purity) was purchased from LabScan Asia Co.,Ltd.

3.1.10 Propan-2-ol

Analytical grade propan-2-ol (99.8% purity) was purchased from LabScan Asia Co.,Ltd.

All the materials were used as received without further purification and for research purposes.

3.2 Equipment

- Spinning drop tensiometer (Operating manual Dataphysics SVT 20)
- Temperature-controlled incubator (BINDER, KB400/E2)
- Water Bath
- Terg-O-Tometer
- UV/VIS Spectrophotometer (Hewlett Packard, 8452A)
- Colorimetric spectrophotometer ,Colorflex (Hunter Lab)
- Total Organic Carbon Analyzer (TOC)
- Tensiometer (Wilhelmy Plate method)

3.3 Experimental Procedures

The experiment part of this research was divided into three parts. The first part was to determine basic properties of surfactants. The second part was to study the phase behavior and microemulsion formation and the last part was detergency experiment.

3.3.1 Basic Property Studies

3.3.1.1 *Solubility*

➤ Anionic Surfactant

Precipitation point, defined as the point at which an aqueous surfactant solution separates to two phases, liquid phase and solid phase. 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.

➤ Nonionic Surfactant

Water solubility was examined by measuring the point at which an aqueous surfactant solution separates to two phases. Each surfactant was varied concentration and was left to separates to two phases at room temperature.

Water solubility was examined by measuring the inverse cloud point by heating 1% aqueous solutions of nonionic surfactants and determining the temperature at which the solution clouds (the “cloud point”).

3.3.1.2 *Critical Micelle Concentration (CMC) and Surface Tension*

Critical Micelle Concentration (CMC) and Surface Tension were measured by using a contact angle measurement instrument (DSA10 Mk2, Kruss GmbH, Hamberg)

3.3.2 Phase Behavior and Microemulsion Formation

The dynamic interfacial tension (IFT) between aqueous phase and the oil phase which were in equilibrium at different conditions was measured using a spinning drop tensiometer (Operating manual Dataphysics SVT 20). The IFT measurements were made using 1-2 μL of oil injected into 300 μL of aqueous phase in capillary tube. Reported IFT values were recorded at twenty minutes after oil injection.

3.3.3 Detergency Experiments

3.3.3.1 *Fabric Preparation*

➤ **Fabric:** Pre-washing of test fabrics was done before soiling in order to get rid of contaminants. This method was followed the ASTM standard guide D4265-98.

➤ **Soiling:** The studied oil was stained by the oil soluble Oil-Red-O dye using Goel (1998)'s method (Goel, 1998) before being applied on the fabrics.

a) The fabric was cut into 3x4 inch swatches in wrap and weft directions.

b) Approximately 0.1 g of the oil-soluble dye was dissolved to 100 mL of the palm oil.

c) The oil soluble Oil-Red-O dye was filtered until clear of solids.

d) Dilute 8 mL of clear dyed oil with dichloromethane to 80 mL.

e) The swatches were soaked in a dyed oil solution for a minute.

f) The soiled swatches were then laid on a flat plate in a ventilated hood at room temperature overnight so as to dry the soiled swatches.

g) Keep them in a sealed glass container in order to prevent evaporation.

Note; All swatches were freshly prepared for each batch of laundry experiments.

3.3.3.2 Laundry Procedure

Detergency experiments were accomplished by using a Terg-O-Tometer (Copley, DIS 8000) simulating home washing-machine action in a bench scale unit. The soiled swatches were put in the bucket of a Terg-O-Tometer. The conditions were set at a liter of washing solution, twenty minutes of washing step, three minutes of first rinse step, and two minutes of second rinse step. All washing and rinse steps were conducted at an agitation speed of 120 rpm and carried out at proper washing temperatures.

3.4 Analysis

3.4.1 Detergency Measurement

Detergency performance is determined by reflectance measurement of pre-wash and post-wash swatches. Reflectance measurements of the unsoiled swatches, the pre-wash soiled swatches and the post-wash soiled swatches were conducted by a colorimetric spectrophotometer (Hunter Lab, ColorFlex). The percentage of detergency is calculated by the following equation:

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

Where ; “A” is the average reflectance of the soiled swatches after washing.

“B” is the average reflectance of the soiled swatches before washing.

“C₀” is the average reflectance of the unsoiled swatches before washing.

3.4.2 Oil Removal Measurement

This measurement was followed Goel (1998)'s method (Goel, 1998). Oil removal was characterized by portion of residual oil on the swatches that is washed out during the detergency process. The attached oil was extracted from the pre-wash soiled swatches by submerging the fabric in propan-2-ol overnight at room temperature. After washing, submerge the post-wash soiled swatches in propan-2-ol overnight at room temperature. Then, measure the amount of extracted oil in the solution via UV/VIS Spectrophotometer (Hewlett Packard, 8452A). Finally, calculate extracted oil concentration from the calibration curve.