CHAPTER III
EXPERIMENTAL

3.1 Materials

Alcohol ethoxylates with different numbers of EO groups (three EO groups, five EO groups, seven EO groups, and nine EO groups called AE3, AE7 and AE9, respectively) with purities of 99.7% in a liquid form are obtained from PTT Chemical Public Company Limited. Sorbitan monooleate (Span 80) are obtained from ICI UniqemaCo. (Wilmington, DE).Sodium methyl 2-sulfohexadecanonate (MES) with purities of 88.6% is obtained from PTT Chemical Public Company Limited. The general properties of the studied surfactant are summarized in Table 3.1. Motor oil which is commercially available for use in gasoline engines, type SAE 10W-30 (Castrol GTX) are used as a model oily soil. All chemicals were used as received without further purification.

Table 3.1 General properties of studied surfactants

Surfactant	Molecular weight	Chemical formula
Dehydol LS 3	318.75	$C_{12-14}(CH_2CH_2O)_3OH$
Dehydol LS 7	496.46	$C_{12-14}(CH_2CH_2O)_7OH$
Dehydol LS 9	578.35	$C_{12-14}(CH_2CH_2O)_9OH$
MES	372	$CH_3(CH_2)_{13,15}CH(SO_3Na)COOCH_3$
Span 80	428.6	$C_{24}H_{44}O_6$

3.2 Equipment

3.2.1 Cathetometer (model TC–II) with digimatic height gauge (model 192–631)

3.2.2 Tensiometer (Kruss, Easy Dyne) with wilhelmy plate metod

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

3.2.4 Water Bath

3.2.5 Refrigerator

3.3 Methodology

In this work, the microemulsion formation of alcohol ethoxylates by obtaining phase diagram of motor oil/surfactant/hydrophilic surfactant/linker/water system to form Winsor's Type I–III–II–IV, called fish diagram, is studied. The various temperatures of microemulsion formation are set up at 20, 30, 40 and 50 °C. For all experiments, the surfactant, the hydrophilic surfactant and the linker concentrations are expressed as percent weight by volume based on aqueous solution.

3.3.1 Microemulsion Formation

In the microemulsion formation study, the experiment is carried out in 10 ml vials. Firstly, 5 ml of aqueous surfactant solutions prepared at different concentrations of surfactant, hydrophilic surfactant and linker are put into vials. After that, 5 ml of motor oil are added to achieve a volumetric ratio of unity to the series of vials and Teflon screw caps are placed on each vial.

In this research, microemulsion study is carried out by using surfactant (alcohol ethoxylate with 3-EO group), hydrophilic surfactant (alcohol ethoxylate with 7- and 9-groups or MES) and liphophilic linker (span80). Mixing between Surfactant, hydrophilic surfactant and liphophilic linker are used to from micriemulsion with motor oil at 20, 30, 40 and 50 °C which the optimum ratio of mixed surfactant between AE3:AE7, AE3:AE9 and AE3:MES are 1:2, 1:5 and 1:10 respectively. After that, each vial is shaken gently by hand for 1 min and then equilibrated in a temperature-controlled incubator (BINDER, KB400/E2), a water bath, or a refrigerator until the system reaches equilibrium for an approximately one month. The experiment is illustrated in Figure 3.1. The equilibrium state is justified by observing that the volume of each phase in the vial remains constant.

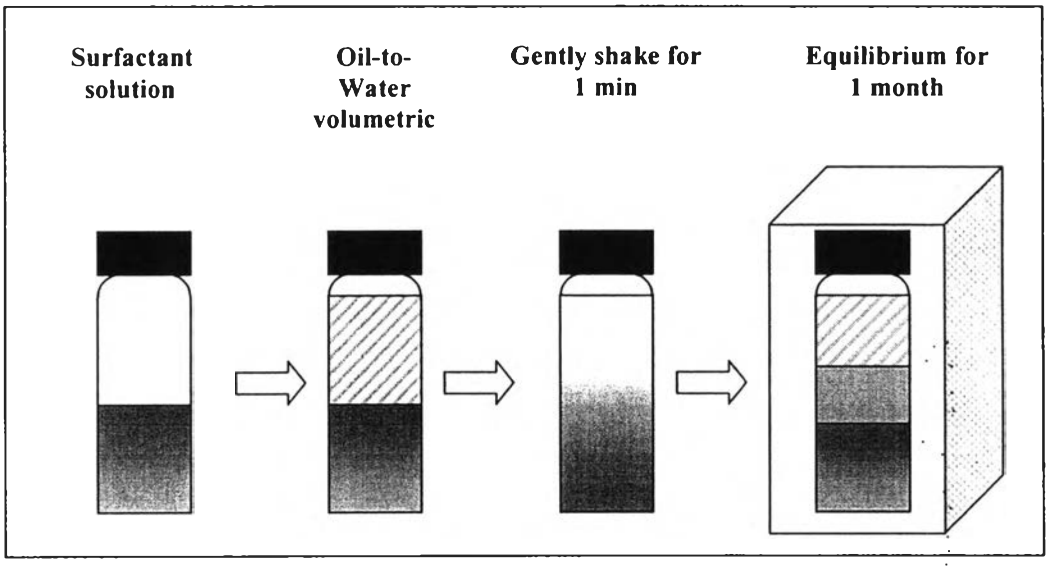

Figure 3.1 Schematic of experiment for microemulsion formation.

3.3.2 Fish Diagram Study

The types of microemulsions of the fish diagram are classified by the visual observation which plotted as linker concentration versus total alcohol ethoxylate concentration at various temperatures. These phase plots are called fish diagrams. The fish diagrams are generally used to determine a minimum surfactant concentration required to form a Winsor's Type III microemulsions which is known as the critical microemulsion concentration (CμC). Moreover, the intersection between three and single-phase regions which reveals the solubilizaton capacity of the system will be observed.

3.3.3 Solubilization Parameter

After equilibrium, the height of each liquid phase will be measured by using a cathetometer, model TC-II from Titan Tool Supply, Inc. attached to a digimatic height gauge, model 192-631, obtained from Mituyo, with an accuracy of 0.001 mm. The solubilization capacities will be calculated in terms of the solubilization parameter (SP), which is interpreted by the following formulation:

Oil-in-Water (O/W) microemulsions (Winsor's Type I):

$$SPw = \frac{0.5*10}{Ms} \quad (3.1)$$

$$SPo = \frac{(0.5 - Vo)*10}{Ms} \quad (3.2)$$

Middle phase microemulsions (Winsor's Type III):

$$SPw = \frac{(0.5 - Vw)*10}{Ms} \quad (3.3)$$

$$SPo = \frac{(0.5 - Vo)*10}{Ms} \quad (3.4)$$

Water-in-Oil (W/O) microemulsions (Winsor's Type II)

$$SPw = \frac{(0.5 - Vw)*10}{Ms} \quad (3.5)$$

$$SPo = \frac{0.5*10}{Ms} \quad (3.6)$$

where

SPw = solubilization parameter of water

SPo = solubilization parameter of oil

Vw = volume of water solubilized in the micellar solution

Vo = volume of oil solubilized in the micellar solution

Ms = total mass of surfactant in the micellar solution

3.3.4 Critical Micelle Concentration (CMC)

The surface tension of surfactant solutions having different concentrations was determined by Krüss, Esay Dywe with the wilhelmy Plate method. The surface tension measurement was conducted at 30 °C. The plot between surface tension vs total surfactant concentration was used to determine the critical micelle concentration (CMC).