



CHAPTER III EXPERIMENTAL

3.1 Materials

Alcohol ethoxylates with different numbers of EO groups (three EO groups, five EO groups, seven EO groups, eight EO groups and nine EO groups), nonylphenol ethoxylates with nine EO groups (a nonionic surfactant) and methyl ester sulfonate (MES, anionic surfactant) were obtained from PTT Chemical Public Company Limited. Analytical grade n-octane with 99% purity and analytical grade n-butanol with 99.5% purity were purchased from Italmar (Thailand) Co., Ltd. Palm oil (Palm olein, Morakot) was purchased from TOP supermarket in Thailand in 2005. Analytical grade NaCl was purchased from LabScan Asia Co., Ltd. Motor oil which is commercially available for use in gasoline engines, type SAE 10W-30 (Castrol GTX) was used as a model oily soil. All chemicals were used as received without further purification.

3.2 Methodology

In this work, microemulsion formation of alcohol ethoxylates was studied by obtaining phase diagrams of various surfactant systems with motor oil to form different Winsor Type of I-III-II-IV, the so-called fish diagrams. For all experiments, the surfactant concentration, cosurfactant concentration, and salinity are expressed as percent weight by volume based on aqueous solution.

3.2.1 Microemulsion Formation

In the microemulsion formation study, the experiments were carried out in a series of 20 ml vials. Firstly, 5 ml of aqueous surfactant solutions prepared at different surfactant concentrations, cosurfactant concentrations and salinities were added into vials. After that, 5 ml of motor oil was added to achieve a volumetric ratio of unity to the series of vials with teflon screw caps. In this research, microemulsion studies were carried out using both single nonionic and mixed surfactant systems

with n-butanol as a cosurfactant. For the single nonionic system, alcohol ethoxylates with three EO groups (AE3) was used to form microemulsions with motor oil at different temperatures (20–40 °C). For the mixed system, AE3 was mixed with methyl ester sulfonate (MES) at a weight fraction of MES = 0.02 at different NaCl concentrations (0–7 %wt). After that, each vial was shaken gently by hand for 1 min and then equilibrated in a temperature-controlled incubator (BINDER, KB400/E2) or water bath until the system reached equilibrium for approximately one month. Figure 3.1 shows the procedure for the microemulsion phase study. The equilibrium state was justified by observing that the volume of each phase in the vial remained constant. After equilibration, the height of each liquid phase was measured 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 were calculated in terms of the solubilization parameter, which is the volume of either oil or water dissolved per weight of total surfactants.

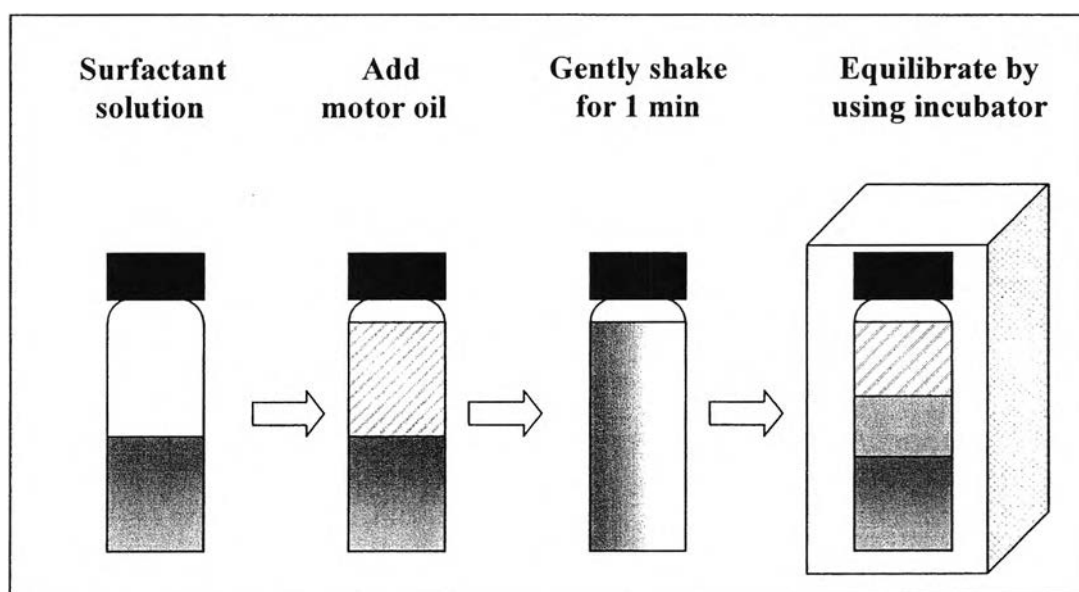


Figure 3.1 Schematic of experiment for microemulsion formation.

The types of microemulsions were justified by the visual observation. To construct fish diagrams, surfactant concentration was plotted with cosurfactant concentration at different temperatures for single nonionic systems and different NaCl concentrations for mixed surfactants systems. These phase plots are called fish

diagrams. The fish diagrams are generally used to determine a minimum surfactant concentration required to form a Winsor Type III microemulsion which is known as the critical microemulsion concentration ($C_{\mu C}$) and form single phase which reveals the solubilization capacity.

3.2.2 Surface Tension Measurements

The surface tension of surfactant solutions having different concentrations was determined by Krüss, K10T. 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).

3.2.3 Interfacial Tension Measurements

The interfacial tension values between) between the surfactant solution and the oil phase which were in equilibrium at different conditions were measured by a spinning drop tensiometer (SITE 04, Kruss GmbH, Hamburg).