CHAPTER III
EXPERIMENTAL

3.1 Materials and Equipment

3.1.1 Equipment:

1. Cathetometer (model TC–II) with digimatic height gauge (model 192–631)
2. Temperature-controlled incubator (BINDER, KB400/E2)
3. Water Bath

3.1.2 Chemicals:

1. Alcohol ethoxylates ($C_{12\text{-}14}EO_3$ (AE3), $C_{12\text{-}14}EO_5$ (AE5), $C_{12\text{-}14}EO_7$ (AE7), and $C_{12\text{-}14}EO_9$ (AE9)) with 99%purity in liquid form were supplied by Thai Ethoxylate Co.,Ltd.
2. Motor oil
3. *n*-Butanol
4. *n*-Hexanol
5. *n*-Octanol
6. Distilled water

3.2 Experimental Procedures

In this work, the microemulsion formation of alcohol ethoxylates (AEs) by obtaining phase diagram of AE/alcohol/motor oil/water system to form Winsor Types (I, II, III, IV), called fish diagram, was studied. The various temperatures of microemulsion formation were set up from 20 °C, 30 °C, 40 °C to 50 °C. For all experiments, the surfactant and the cosurfactant concentrations were expressed as percent weight by volume based on aqueous solution.

3.2.1 Microemulsion Formation Expèriment

In the microemulsion formation study, the experiment was carried out in 20 ml vials. Firstly, 5 ml of aqueous solutions prepared at different types and concentrations of AE, and different type and concentrations of cosurfactants were put into the 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 microemulsion study, *n*-butanol, *n*-hexanol, and *n*-octanol were used as cosurfactants. After that, each vial was shaken gently by hand for 1 min and then equilibrated in a temperature-controlled incubator (BINDER, KB400/E2) or a water bath at different temperatures until the system reached equilibrium for approximately one month. The equilibrium state was justified by observing that the volume of each phase in the vial remained unchanged. Schematic of experiment for microemulsion formation shows in Figure 3.1.

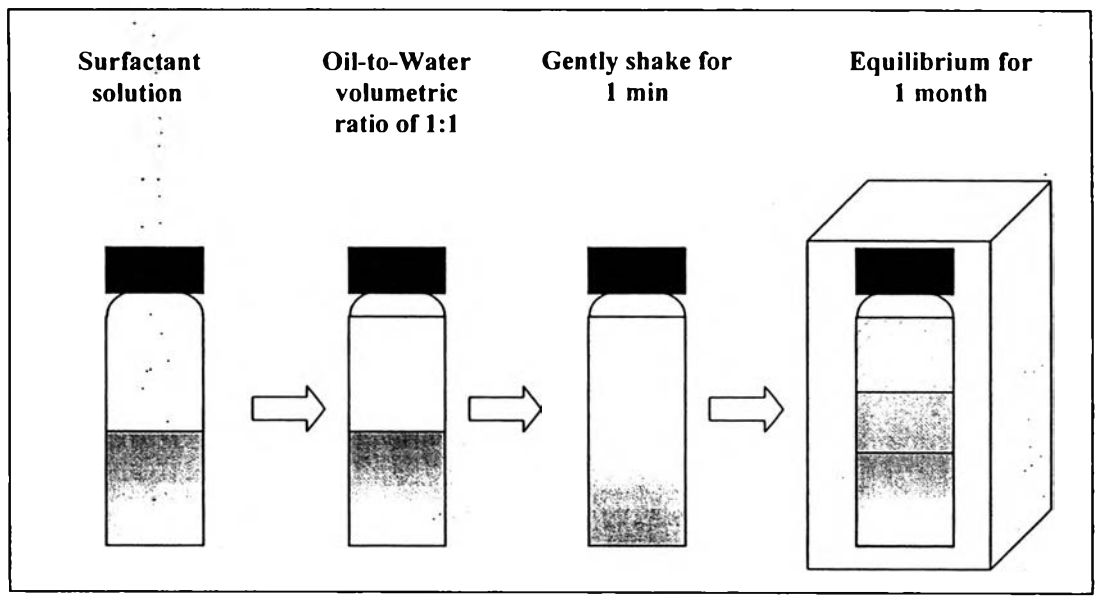

Figure 3.1 Schematic of experiment for microemulsion formation experiment.

3.2.2 Fish Diagram Study

The types of microemulsions were classified by the visual observation. The fish diagrams were plotted as cosurfactant concentration versus surfactant concentration at various temperatures. 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 (CμC). Moreover, the intersection between the three microemulsion regions (Winsor Types I, II, and III) reveals the solubilizaton capacity of the system to obtain a single phase of microemulsion.

3.2.3 Solubilization Parameters (SP)

After equilibrium, the height of each liquid phase was 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 were calculated in terms of the solubilization parameters (SP).

3.2.4 Cloud Point Measurement

The cloud point measurement was modified from the standard test method D 2024-65 [1]. The cloud point of aqueous surfactant solutions were determined visually by noting the turbid temperature and separated-phase temperature while heating 5 ml of such surfactant solution in the screw-capped vial. Therefore, cloud point was taken as the average temperature between the surfactant solutions turned into turbidity and separated into 2 phases upon being heated. The determination was repeated 3 times for each solution and the heating rate was kept at 0.5 °C for 2 hours.