CHAPTER III
METHODOLOGY

3.1 Chemicals

Anionic surfactant, used in this study was sodium dodecyl sulfate (SDS, 99%) obtained from Sigma (St. Louis, Missouri). The chemical formula is $CH_3(CH_2)_{10}CH_2 O SO_3Na$ (MW = 288.38) and the specific gravity at 20 ^{O}C is $\geq$ 1.1.

n-Hexane (AR grade) was obtained from Mallinckrodt (Paris, Kentucky). The chemical formula is $CH_3(CH_2)_4CH_3$ (MW = 86.18) and the density at 20 ^{O}C is 0.659 g/mL.

Octanoic acid (purum > 98%) was purchased from Fluka (St. Louis, Missouri). Sodium chloride (Unilab grade) was purchased from Ajex Co.Ltd (Auburn, New South Wales). Distilled water was used as the aqueous solvent.

3.2 Experimental Procedures

3.2.1 Study of Phase Behavior of Microemulsion and Solubilization

The microemulsion systems were prepared in 10 ml glass centrifugal tubes by using 1:1 volume ratio of oil (octanoic acid + hexane) to surfactant in aqueous solution. Octanoic acid was weighed in relative to SDS (weight ratio of SDS to octanoic acid, R = 40:60) into the tubes.

The stock solutions of surfactant and sodium chloride were prepared separately. Then the salt solution and the surfactant solution were pipetted into the centrifuge tubes and followed by water to make the aqueous solution volume to 5 mL. The volume of octanoic acid was pipetted in relative to the weight ratio of SDS to octanoic acid into the tubes and then

hexane was added to make up the solution volume to another 5 mL. The tubes were capped with septa and sealed with aluminium cap to prevent volatilization. The solutions were shaken by vortex (Genie-2) until homogenized and allowed to equilibrate at constant temperature (25 ± 0.1 ^{O}C, 35 ± 0.1 ^{o}C) for 2 weeks or 2 months. The occurrence of microemulsion phase were visually observed and the phase volumes were measured. The criterion for phase equilibrium and solubilization was verified by visual observation of unchanged phase volume readings over several days. Solubilization of hexane or water in microemulsion phases was calculated based on the concept of solubilization parameter (SP).

3.2.2 Density Measurements

Densities of microemulsion were determined by pipetting the volume of 1 mL microemulsion phase or excess phases into a vial tube and quickly weighed. The density was reported as an average value at 25 ^{o}C.

3.2.3 Interfacial Tension Measurements

The equilibrium interfacial tension (ITF) of microemulsion solutions at different surfactant concentrations and at 25 ^{O}C were determined. A spinning drop interfacial tension model Kruss SITE 04 was utilized for measuring the IFT. A drop of light phase was injected by a microliter syringe into a rotating capillary tube containing the heavy phase. The glass tube is sealed and spun around its axis at 2000 to 4000 rpm. The shape of this drop is the result of an equilibrium between rotational forces acting to elongate the drop and contraction by interfacial tension acting to minimize surface area. Providing that the drop is quite long relative to its width, interfacial tension can be calculated with Vonnegut's formular

$$\sigma = [r^3 \Delta \rho \omega^2]/4 \qquad\qquad (1)$$

where σ is interfacial tension (mN/m), r is half of drop width, $\Delta\rho$ is the density difference between two phase, and ω is the angular velocity.

In this study the interfacial tension was calculated between two liquid phases by

$$\sigma = e(vd)^3 n^2 \Delta\rho \tag{2}$$

where d is drop diameter, n is number of revolutions which was shown on digital dial, e unit factor is given by e = 3.427E-7 (mN cm^3 min^2)/(m gr mm^3), and v is enlargement factor of lenses, for this study v = 0.31 mm sdv.

3.2.4 Conductivity Measurements

Since a regular conductivity probe is too large to immerse into a microemulsion phase for conductivity measurement, a conductivity detector (Alltech 550) of HPLC (Hewlett Packard Series 1050) was adapted. It was operated at 25 $^{\circ}$C. The standard potassium chloride aqueous solutions (0.5 mM to 2.0 M) were prepared and measured the conductivity values by using the conductivity meter (Orion Model 124) and conductivity cell model 012210. The solution volume of 0.1 ml were injected into an injection loop of HPLC which was connected directly to the conductivity detector. The mobile phase was methanol 100% and the flow rate of 1.0 ml/min. The reading taken from the conductivity detector was corrected for the true value obtained from the conductivity meter. A calibration curve was made from the plot of the corrected conductivity values against the corresponding peak areas. When the microemulsion phase, excess oil and excess water phases were analyzed, the sample volumes of 0.1 mL were performed in a similar manner to the standard solution. The peak areas obtained were converted to the conductivity values of the samples by comparing with the calibration curve.