CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Surfactants

Two types of surfactants were used in this work, branched alcohol propoxylate sulfate, sodium salt (Alfoterra 145-5PO Sulfate), and sodium dodecylsulfate (SDS). Alfoterra 145-5PO Sulfate with purity of 28.7% was supplied in the liquid form by Sasol North America Inc., Texas, USA. Alfoterra 145-5PO Sulfate is an anionic surfactant with a negatively charged sulfate head group. SDS is an anionic surfactant with a negatively charged sulfate head group and alkyl chain length of twelve carbon units. SDS was purchased from Carlo Erba Reagenti with purity in the range of 94-98 %. The general properties of the studied surfactants are shown in Table 3.1.

Table 3.1 General properties of studied surfactants

Surfactant	Molecular weight	Chemical Formula
Alfoterra 145-5PO Sulfate	595	$C_{16}H_{32}(C_3H_6O)_5SO_4Na$
SDS	288	$CH_3(CH_2)_{11}OSO_3Na$

3.1.2 Studied Oil Contaminant

Diesel was selected as a model oil contaminant in this research work. It was purchased from PTT Public Co., Ltd. Diesel is a complex combination of hydrocarbons produced by the distillation of crude oil. It consists of hydrocarbons having carbon number predominantly in the range of C9-C20 and having a boiling point in the range of approximately 163-357 ^{o}C.

3.1.3 Water

Distilled water was used in all experiments for preparing aqueous surfactant solutions and rinsing glassware. It was supplied by The Government Pharmaceutical Organization, Bangkok, Thailand.

3.1.4 Electrolyte

Analytical grade of sodium chloride (NaCl) was used as an electrolyte and obtained from Labscan Asia Co., Ltd. with purity of 99%.

All chemicals were used as received without further purification.

3.2 Experimental Procedures

In this work, experiments were divided into three main parts. The first part was a study of microemulsion formation (phase behavior), the second part was froth flotation experiments, and the third part was foam ability and foam stability experiments. For all experiments, the surfactant concentration and salinity were expressed as percent by weight based on aqueous solution. All experiments were conducted at 30 °C.

3.2.1 Phase Behavior Experiment

In the microemulsion formation studied, the experiment was carried out in 20 ml vials. Firstly, aqueous surfactant solution prepared at different surfactant concentrations and salinities was added in vials. 5 ml of diesel was added in a series of vials with Teflon screw caps which already had 5 ml of aqueous solution contained surfactant, salt, and water. Surfactant concentrations, salinity, and oil to water ratio were varied. By fixing Alfoterra concentration at 0.10 wt% and varying SDS concentration at 0.10, 0.50, 0.70, 1 wt%. In determine the salinity effect, sodium chloride concentration was varied at 2, 3, and 4 wt%. The oil to water ratio was varied at 1:1, 1:4, 1:9, 1:19. After that, each vial was shaken gently by hand for 1 min and then equilibrated in a temperature-controlled incubator (BINDER, KB400/E2) at 30 °C until the system reached equilibrium as illustrated in Figure 3.1. The equilibrium state was justified by observing that the volume of each phase in the

vial remained constant. The measurement of phase height was conducted 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 0.002 mm in accuracy. The interfacial tension values between two phases were measured by a spinning drop tensiometer (SITE 04, Kruss GmbH, Hamburg).

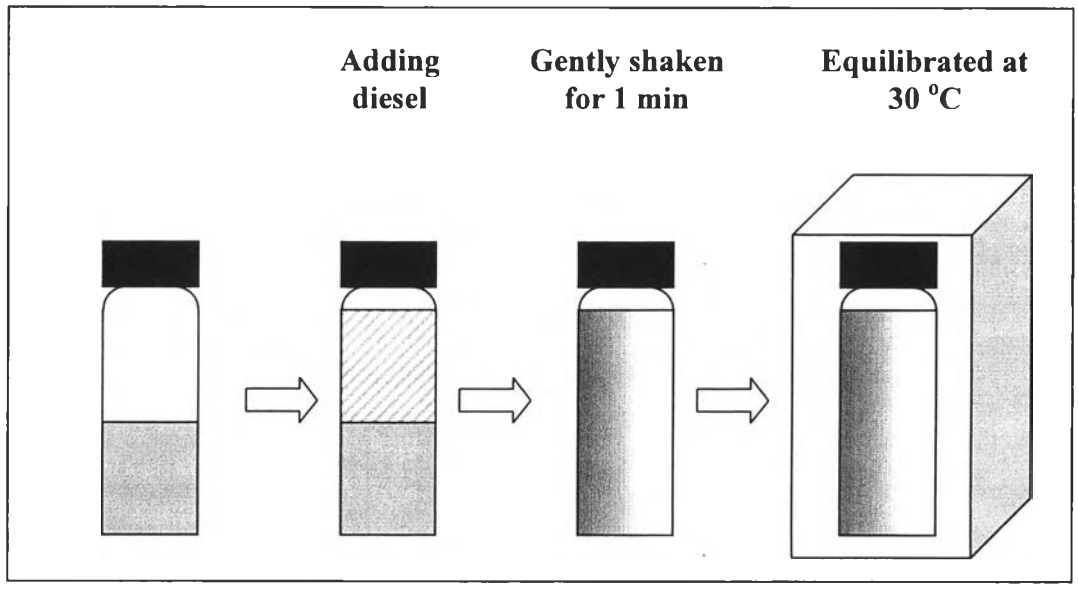

Figure 3.1 Schematic experiment of microemulsion formation.

3.2.2 Froth Flotation Experiment

Figure 3.2 shows the schematic diagram of froth flotation apparatus used in this study. The froth flotation apparatus consisted of a glass cylindrical column with 5 cm inside diameter and 120 cm height and operated in a continuous mode. Compressed air was first filtered to remove all particles and oil and then passed through a water filter. The flow rate of the filtered air was regulated by a mass flow controller (AALBORG, GFC171S) before it was introduced into the bottom of the column through a sinter glass disk with pore size diameters about 16-40 μm. A well-mixed solution, which composed surfactant, water, and oil under microemulsion condition was fed continuously with a desired flow rate into the froth flotation column by using a peristatic pump (Masterflex, easy-load II). The level of solution in the column was adjusted by a three-way flexible tube in order to vary the foam

height. The air bubbles ascended through the solution generated foam. The foam overflown from the column was collected over different time intervals. After that, the froth was collapsed to analyze diesel concentrations in the froth. In addition, effluent samples were collected at the same period of time as foam collected for analysis of diesel and surfactant concentrations by using the extraction with methylene chloride method and titration with methylene blue chloride method, respectively. All experiments of the forth flotation were carried out at room temperature of 25-27 °C.

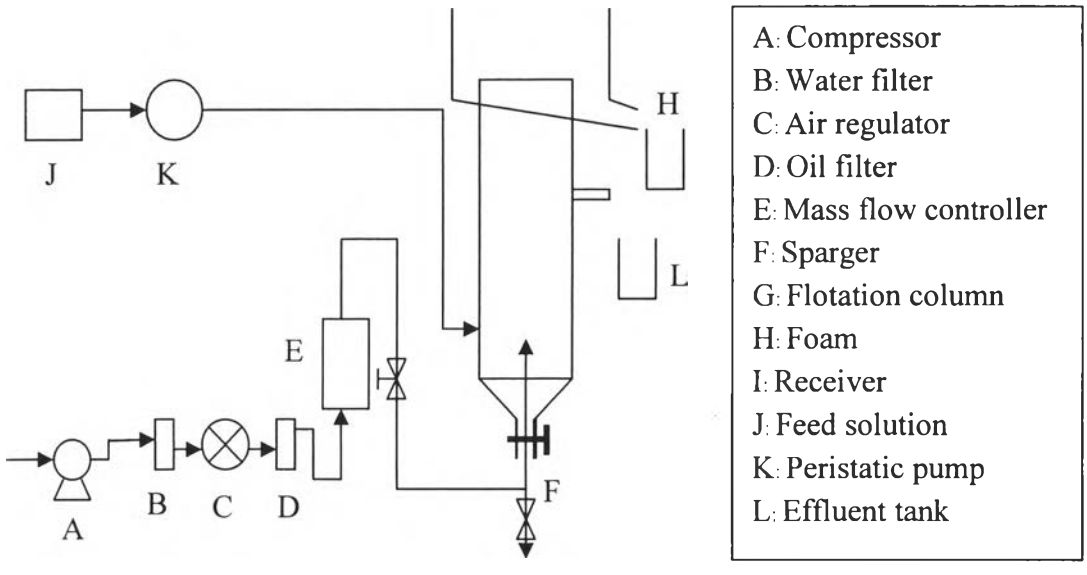

Figure 3.2 Schematic diagram of the froth flotation apparatus.

3.2.3 Foamability and Foam Stability Experiments

The investigation of foam ability and foam stability, 250 ml of the sample was transferred to a glass cylindrical column with 5 cm inside diameter and 120 cm height. The filtered air was introduced through the sinter glass disk, having pore size diameters of 16-40 μm. The flow rate of air was controlled constant at 100 ml/min by using the mass flow controller. The solution in the column was aerated continuously until the foam height in the column was constant. Thus, the maximum foam height was recorded. After that, the filtered air to the column was stopped. The time required for the foam volume to collapse by half was recorded. Foamability is defined as the ratio of maximum foam height to initial solution height whereas foam stability ($t_{1/2}$) is the time required for the foam volume to collapse by half.