CHAPTER III EXPERIMENTAL

3.1 Materials

3.1.1 Surfactants

Alfoterra 145-5PO Sulfate, branched alcohol propoxylate sulfate, sodium sulfate, was used in this work. It is an anionic surfactant with a negatively charged sulfate head group supplied in the liquid form by Sasol North America Inc., Texas, USA, with a manufacture reported purity of 28.7%. The general properties of the studied surfactant are shown in Table 3.1.

Table 3.1 General properties of studied surfactants

Surfactant	Molecular weight	Chemical formula
Alfoterra 145-5PO Sulfate	595	$C_{14-15}H_{32}(C_{3}H_{6}O)_{5}SO_{4}Na$

3.1.2 Studied Oil Contaminant

Motor oil was selected as a model oil contaminant in this research which is commercially available for use in gasoline engines, type SAE 10W-30 with synthetic guard (Castrol GTX). Motor oil is complex in composition and has high hydrophobicity. It generally consists of at least five main components: n-paraffins, isoparaffins, cycloparaffins, aromatic hydrocarbons, and mixed aliphatic and aromatic compounds.

3.1.3 <u>Water</u>

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 <u>Electrolyte</u>

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 four main parts. The first part was a study of microemulsion formation (phase behavior), the second part was foamability and foam stability experiments, the third part was froth flotation experiments and the fourth part was a study of bubble mechanism. 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 motor oil 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 and salinity were investigated by varying Alfoterra concentration at 0.1-10 wt.% and sodium chloride at 1-20 wt.%. 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 values of interfacial tension and dynamic interfacial tension 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 Foamability and Foam Stability Experiments

In the investigation of foamability 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 μ m. The flow rate of air was maintained 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.

3.2.3 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 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 μ m. 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 peristaltic 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 motor oil concentrations in the froth. In addition, effluent samples were collected at the same period of time as foam collected for analysis of motor oil and surfactant concentrations by using the extraction with methylene chloride method and titration with methylene blue chloride method, respectively. All experiments of the froth flotation were carried out at room temperature of 25-27 °C.



Figure 3.2 Schematic diagram of the froth flotation apparatus.

3.2.4 Bubble Size Distribution

The bubble size distribution of the systems was obtained by photographic method. The photographs were taken by capturing the image with a digital camera, and then processed and enhanced using the image processing software (Image-Pro Plus 5.1) that enabled to distinguish the bubble boundaries clearly. They were taken at three axial positions for different operating conditions. The bubble diameters were automatically computed by the software that is based on the assumption that the bubbles are symmetric as shown in Figure 3.3.

In order to obtain the distribution curves about 500 bubbles for each distribution were analyzed, since this is the number of bubbles to be measured in order to establish a statistically stable distribution (Colella *et al.*, 1999).

Because of the limitation of photographic method for measuring the bubble size, only the bubbles close to the wall can be completely seen. However, as the column wall was relatively thin (2 mm), the optical effects caused by the distance differences between bubbles and the digital camera were neglected.



Figure 3.3 Schematic diagram of photographic method.