

CHAPTER III EXPERIMENTAL

3.1 Materials

3.1.1 Surfactant

Alfoterra 145-3PO, branched alcohol propoxylate sulfate, used in this work is an anionic surfactant with a negatively sulfate head group which was supplied in the liquid form by Sasol North America Inc., Texas, USA, with a manufacture reported purity of 28.6%. The general properties of the studied surfactant are shown in Table 3.1.

Table 3.1 General properties of studied surfactant

Surfactant	Molecular weight	Chemical formula
Alfoterra 145-3PO	483	$C_{14-15}H_{32}(C_3H_6O)_3SO_4Na$

3.1.2 Studied Oil Contaminant

Cutting oil was selected as a model oil contaminant in this research work. Cutting oil 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 C_{15} - C_{24} and having a boiling point higher than $316^{\circ}C$.

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 sodium chloride (NaCl) was used as an electrolyte and obtained from Labscan Asia Co., Ltd. with a 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 foamability 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 Microemulsion Formation

In the microemulsion formation studied, the experiment was carried out in 20 ml vials. Firstly, 5 ml of aqueous surfactant solutions prepared at different surfactant concentrations and salinities was put into vials. After that, 5 ml of cutting oil was added to the series of vials and Teflon screw caps were placed on each vial. Surfactant concentration, salinity, and oil to water ratio were varied. In determining the salinity effect, sodium chloride was varied from 1-10 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. The microemulsion experiments are illustrated in Figure 3.1. The equilibrium state was justified by observing that the volume of each phase in the vial remained constant. After equilibration, the measurement of each 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 an accuracy of 0.002 mm. The interfacial tension values between two phases were measured by a spinning drop tensiometer (SITE 04, Kruss GmbH, Hamburg).

Secondly, after the optimum condition of surfactant concentration and salinity was known, the effects of oil loading was studied by varying the cutting oil at 1:1, 1:4, 1:9, and 1:19 volumetric ratio. The volume of the system was controlled at 10 ml. The experimental procedure was performed as in the first part.

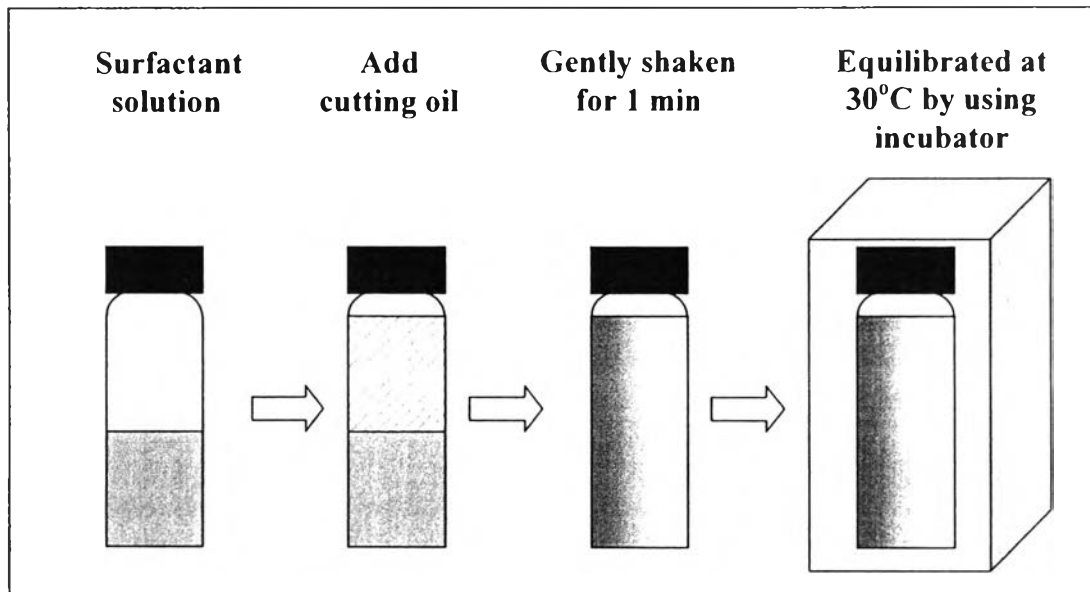


Figure 3.1 Schematic of experiment for microemulsion formation.

3.2.2 Froth Flotation Experiments

Figure 3.2 shows a schematic 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 μm . A well-mixed solution, which comprised surfactant, water, and oil under different microemulsion conditions 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 overflowed from the column was collected over different time intervals. After that, the froth was collapsed to analyze cutting oil and surfactant concentrations in the froth. In addition, effluent samples were collected at the same period of time as foam collected for analysis of cutting oil and surfactant concentrations by using the methylene chloride extraction

method (Feng *et al.*,2000) and the titration with methylene blue chloride method (ASTM D1681-92), respectively. All experiments of the froth flotation were carried out at room temperature of 25-27 °C.

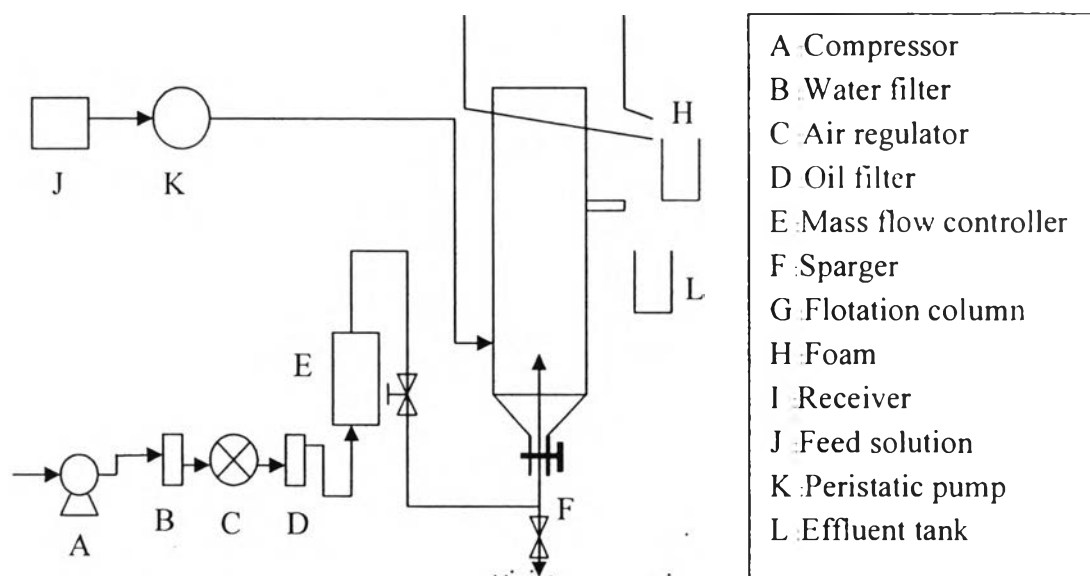


Figure 3.2 Schematic of the froth flotation apparatus.

3.2.3 Foamability and Foam Stability Experiments

For 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 μm . The flow rate of air was kept 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.

3.2.4 Bubble Size Distribution Measurement

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 by using the image processing software (Image-Pro Plus 5.1) that enables 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.

Because of the limitation of photographic method for measuring the bubble size, only the bubbles close to the wall can be seen completely. 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 negligible.

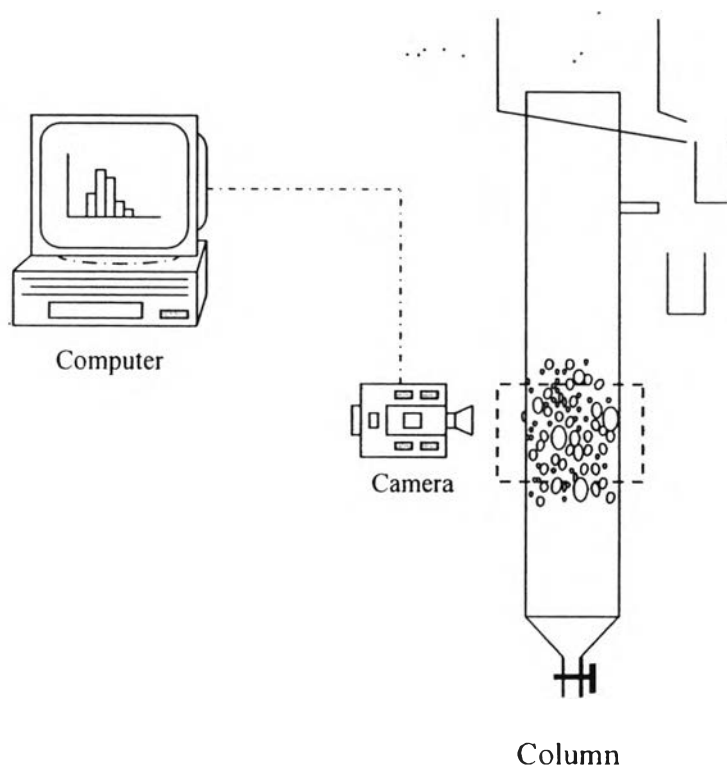


Figure 3.3 Schematic of photographic method.