# CHAPTER III EXPERIMENTAL

#### 3.1 Materials

## 3.1.1 Surfactants

There are four types of surfactants used in this work which are branched alcohol propoxylate sulfate, sodium dodecyl sulfate, sodium dodecyl bezene sulfonate and sodium bis-2-ethylhexylsulfosuccinate.

Branched alcohol propoxylate sulfate or Alfoterra 145-5PO Sulfate was supplied in the liquid form by Sasol North America Inc., Texas, USA with a manufacture reported purity of 28.7%. Alfoterra 145-5PO Sulfate is an anionic surfactant with a negatively charged sulfate head group.

Sodium dodecyl sulfate (SDS) was purchased from Carlo Erba Reagenti with purity in the range of 94-98 %. SDS is an anionic surfactant with a negatively charged sulfate head group and alkyl chain length of twelve carbon units.

Sodium dodecyl benzene sulfonate (SDBS) was purchased from Carlo Erba Reagenti with purity in the range of 80 %. SDBS is an anionic surfactant with a negatively charged sulfate and benzene head group and alkyl chain length of twelve carbon units.

Sodium bis-2-ethylhexylsulfosuccinate (Aerosol-OT or AOT) was purchased from Fluka company with 98% purity. AOT is an anionic surfactant, with a negatively charged sulfosuccinate head group and alkyl chain length of twenty carbon units. The general properties of the studied surfactants are shown in Table 1.

Table 3.1 General properties of studied surfactants

Surfactant	Molecular weight	Chemical formula
Alfoterra 145-5PO Sulfate	595	C <sub>16</sub> H <sub>32</sub> (C <sub>3</sub> H <sub>6</sub> O) <sub>5</sub> SO <sub>4</sub> Na
SDS	288	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>11</sub> OSO <sub>3</sub> Na
SDBS	348.48	C <sub>12</sub> H <sub>25</sub> C <sub>6</sub> H <sub>4</sub> SO <sub>3</sub> Na
AOT	444.57	C <sub>20</sub> H <sub>37</sub> O <sub>4</sub> SO <sub>3</sub> Na

#### 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<sub>15</sub>-C<sub>24</sub> and having a boiling point higher than 316 °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, 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 are expressed as percent age of weight based on aqueous phase.

### 3.2.1 Study of Microemulsion Formation

In the microemulsion formation, the experiment was carried out in 20 ml vials. Firstly, 5 ml of aqueous surfactant solution prepared at different surfactant concentrations and salinities was added in vials. 5 ml of motor oil was then added in a series of vials with Teflon screw caps. To form microemulsion system, surfactant concentrations, salinity, and oil to water ratio were varied. By fixing Alfoterra concentration at 0.20 wt% and varying AOT concentration at 0.10, 0.50, 0.70, 1 wt%. To determine the salinity effect, sodium chloride concentration was varied from 2 to 4 wt%. Besides, 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 equilibrate in a temperature-controlled incubator (BINDER, KB400/E2) at 30 °C until the system reach equilibrium the procedure of microemulsion illustrate in Figure 3.1. The equilibrium state was justified by observing that the volume of each phase of microemulsion remained constant. The measurement of phase height was conducted by using a cathetometer (model TC-II Titan Tool) attached to a digimatic height gauge (model 192-631, Mituyo) with 0.002 mm in accuracy. The interfacial tension between each phase was measured by a spinning drop tensiometer (SITE 04, Kruss GmbH, Hamburg).

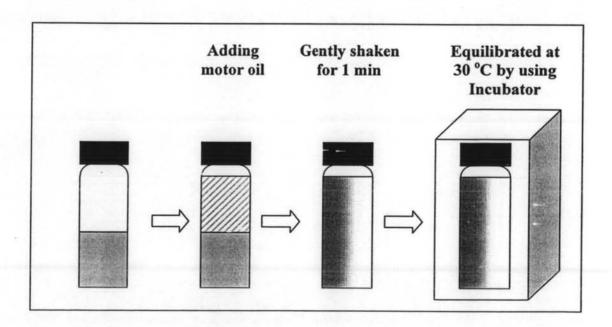


Figure 3.1 Schematic experiment of microemulsion formation.

# 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 it was operated in a continuous mode. Compressed air is first filter 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 of 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 ascend 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 cutting oil concentrations in the froth. In addition, effluent samples was collected at the same period of time as foam collected for analysis of cutting oil 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 was carried out at room temperature of 25-27 °C.

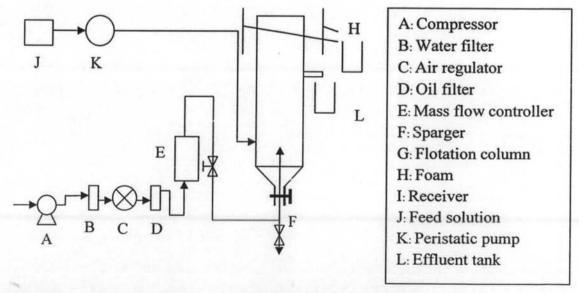


Figure 3.2 Schematic diagram of the froth flotation apparatus.

# 3.2.3 Foamability and Foam Stability Experiments

The investigation of foam ability and foam stability, was carried out using a glass cylindrical column with 5 cm inside diameter and 120 cm height. A quantity of 250 ml of solution containing different surfactant concentration was added into the column. The filtered air was introduced through the sinter glass disk, having a pore size diameters of 16-40 m. The flow rate of air was controlled constant at 100 ml/min by using the mass flow controller. For each run, the maximum foam height was recorded. After that, the filtered air introduce 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<sub>1/2</sub>) is the time required for the foam volume to collapse by half.