CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Surfactants

There were three types of surfactants used in this work which are sodium bis-2-ethylhexylsulfosuccinate (Aeorsol-OT or AOT), mono- and di-hexadecyl diphenyloxide disulfonate sodium salt (Dowfax 8390), and sodium di-1, 3-dimethylbuty sulfosuccinate (Aerosol-MA or AMA).

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.

Mono- and di-hexadecyl diphenyloxide disulfonate sodium salt (Dowfax 8390) in 36% solution was supplied by Dow Company, Midland, Michigan. Dowfax 8390 is an anionic surfactant with two negatively charged sulfonate head groups.

Sodium di-1, 3-dimethylbutyl sulfosuccinate (Aerosol-MA or AMA) with 80% purity and in a mixture of isopropanol and water was supplied by CYTEX Corporation, Los Angeles, USA. AMA is an anionic surfactant, with a negatively charged sulfosuccinate head group and alkyl chain length of sixteen carbon units. 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
AOT	444.57	$C_{20}H_{37}O_4SO_3Na$
Dowfax8390	642	$C_{16}H_3C_{12}H_7O(SO_3Na)_2$
AMA	388	$C_{16}H_{29}O_4SO_3Na$

3.1.2 Studied Oil

Ethylbenzene was selected as a model oil in this work. It was purchased from Fluka Company and has purity of higher than 98%. The chemical and physical properties of ethylbenzene are shown in Table 3.2

Table 3.2 Chemical and Physical Properties of Ethylbenzene

Properties	Ethylbenzene
Chemical formulation	C_8H_{10}
Molecular weight	106.16
Boiling point, °C	136
Melting point, °C	5
Water solubility, g/mL	0.01
Specific gravity at 20 °C, g/mL	0.867

3.1.3 Water

Distilled water was used throughout this work for preparing aqueous surfactant solutions and cleaning glassware. It was purchased from The Government Pharmaceutical Organization, Bangkok, Thailand.

3.1.4 Electrolyte

Sodium chloride (NaCl), analytical purity grade, was used as an electrolyte and purchased from Aldrich Chemical Company, Inc.

3.2 Experimental Procedures

The experiment of this work was divided into two parts. The first part was to study the phase behavior and the formation of microemulsion and the second part was froth flotation experiment. All of the experiments, the concentration of surfactant and electrolyte were expressed in weight percent of the aqueous solution.

3.2.1 Study of Phase Behavior and Formation of Microemulsion

For the phase behavior studies, microemulsion was carried out in 20 mL test tubes. Aqueous surfactant solutions were prepared at desired concentrations of surfactants and NaCl. An equal volume of 5 ml of aqueous surfactant solution and oil were added in a series of test tubes with Teflon screw cap. Each test tube was then shaken gently by hand for 3 min and equilibrated in a temperature-controlled circulating water bath at 30 °C until equilibrium was reached as illustrated in Figure 3.1. The system reached equilibration when the volume of each phase did not change. It was found that the studied system took about 3 weeks to reach equilibrium. The volumes of all phases of microemulsion were measured by using a cathetometer, model TC-II from Titan Tool Supply, Inc. attached to a digimatic height gauge, model 192-631, obtained from Mitutoyo with 0.01 inch accuracy. The surfactant solubulization capacities were calculated in terms of solubilization parameter. The interfacial tension measurements of the two and three phase systems was measured by using a spinning drop tensiometer (SITE 04, KrÜss GmbH, Hamburg).

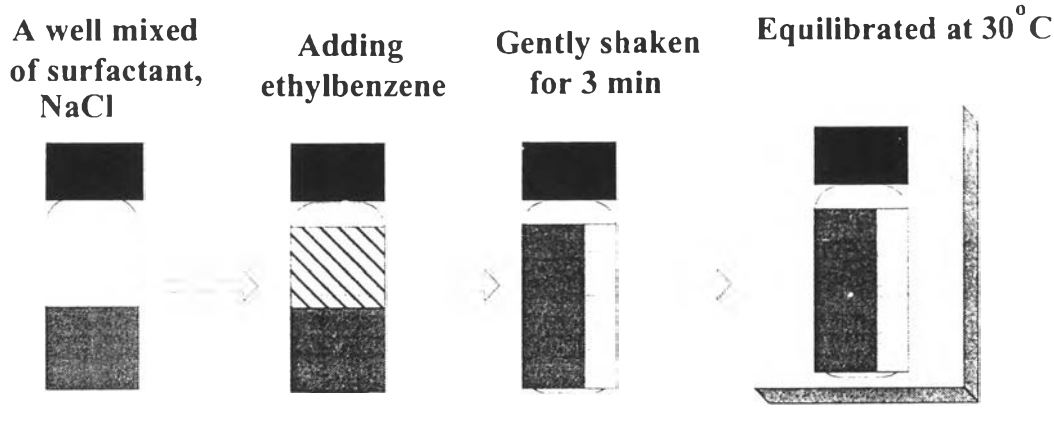

Figure 3.1 Schematic experiment of microemulsion formation.

3.2.2 Froth Flotation Experiment

A schematic diagram of the froth flotation apparatus used in this study is shown in Figure 3.2. The flotation column was made of glass having an internal

diameter of 5 cm and a height of 70 cm. The sparged air was distributed into the bottom of the column through a sinter glass with pore size about 40-60 μm. The experiments were conducted at an ambient temperature of 30 °C. The well mixed solution of 750 ml which composed of surfactants, water and oil was transferred into the column. The solution was prepared at specific concentrations of surfactants according to the results obtained from the phase study. The air bubble rose through this solution and generated foam. The foam came out from the column and was collected over a desired period of time. For analysis, the broken foam and treated solution were determined the concentrations of ethylbenzene by using a GC-Headspace, the amount of surfactant by using titration method (ASTM D1681-92, 1997).

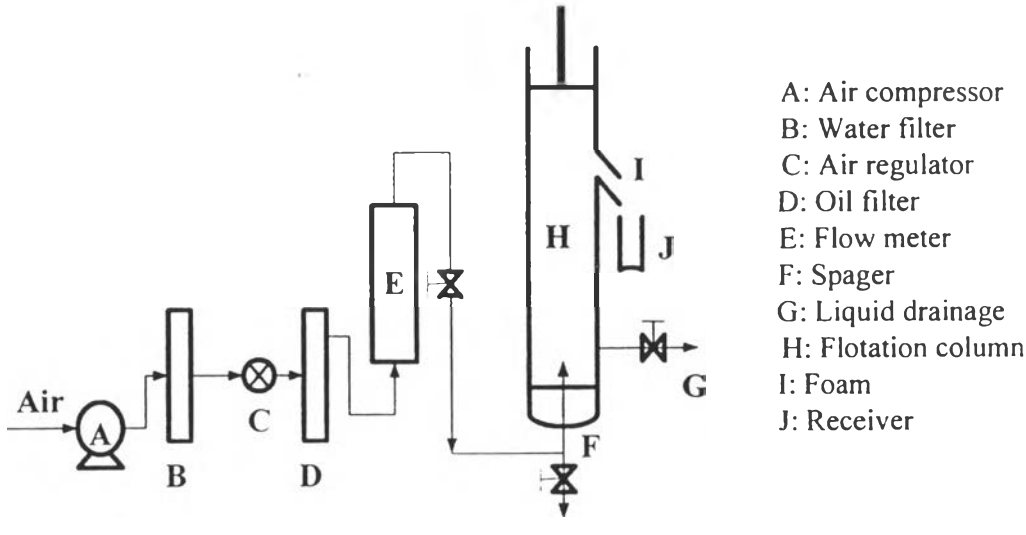

Figure 3.2 Schematic diagram of the froth flotation apparatus.