CHAPTER III
EXPERIMENTAL

3.1 Chemicals

3.1.1 Surfactants

There were two types of surfactants used in this work which were sodium dodecyl sulphate and nonylphenol polyethoxylate.

Sodium dodecyl sulphate (SDS) was supplied by Henkel company with a manufacture reported purity of at least 90%. SDS is an anionic surfactant, with a negatively charged sulfate head group and alkyl chain length of twelve carbon units. SDS has molecular weight of 288.38 and its chemical formula is $C_{12}H_{25}SO_4Na$.

Nonylphenol ethoxylate ($NP(EO)_{10}$) or TERIC N10 was supplied by ICI Australia Operations Pty Ltd. $NP(EO)_{10}$ is a nonionic surfactant with containing approximately ten moles of ethylene oxide per mole of nonylphenol. Table 3.1 shows chemical properties of TERIC N10

Table 3.1 Chemical properties of TERIC N10

Formula	$(C_2H_4O)_{10}C_{15}H_{24}O$
Molecular weight	660
Appearance at 25°C	Clear viscous liquid
HLB(hydrophilic lipophilic balance)	13.3
Melting point,°C	5
Specific Gravity at 20°C	1.063
Viscosity, cp at 20°C	360

$NP(EO)_{10}$ is readily soluble in water, glycol ethers, simple esters, alcohol and common chlorinated and aromatic solvents and is insoluble in mineral and vegetable oils and in most aliphatic hydrocarbon solvents such as kerosene.

3.1.2 Studied oil

Ortho-dichlorobenzene (ODCB) was chosen to use in this work as a studied oil and purchased from Fisher Scientific Co., Fair lawn, New Jersy with a purity of 99.9 %. The chemical and physical properties of ortho-dichlorobenzene are shown in Table 3.2.

Table 3.2 Chemical and physical properties of ortho-dichlorobenzene

Formula	$C_6H_4C_{l2}$
Molecular weight	147.01
Boiling point	180.5°C at 760 mm. Hg
Melting point	-17.0 °C
Water solubility	156 mg/L at 25°C
Vapor pressure	1.47 mm. Hg at 25°C

Health Hazard: The symptoms are lacremation depression of central nervous system, anesthesia and liver damage.

3.1.3 Water

In this work, distilled water was used for preparing surfactant solutions and cleaning glassware. Distilled water 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 obtained from Aldrich Chemical Company, Inc.

3.2 Experimental procedures

There were two parts of the experiment which were to study the formation of microemulsion and to apply froth flotation for removing orthodichlorobenzene from wastewater.

3.2.1 Study of phase behavior and formation of microemulsion

For investigation of phase behavior, Homogenous surfactant solutions were prepared at 1%, 3%, 5%, 7% and 9% by wt. 5 mL surfactant solutions was mixed with 5 mL ortho-dichlorobenzene in a vial and sealed with a screw cap. In addition, NaCl concentration was also varied in order to determine the effect of NaCl on phase behavior of the studied system. The series of samples were well mixed by shaking and kept in an incubator at 30° C until equilibrium as illustrated in Figure 3.1. The height or the volume of each phase was measured and the appearance of each phase was observed everyday until they did not change with time. Therefore, it showed that it reached an equilibrium.

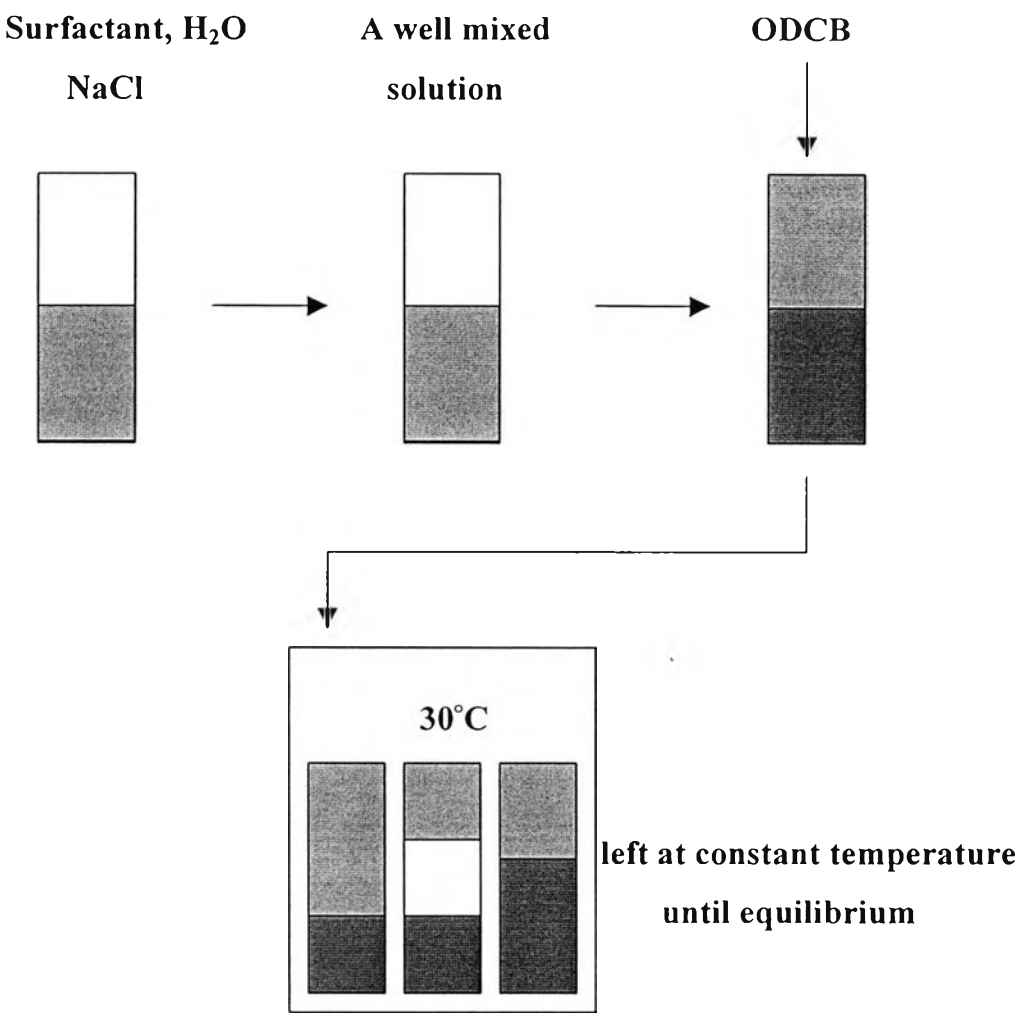

Figure 3.1 Schematic experiment of microemulsion formation

3.2.2 Froth flotation experiment

Approximately six liters of the microemulsion solutions under the desired conditions were prepared and separated into three systems (water and middle phase, water and oil phase, and water, middle and oil phase) as illustrated in Figure 3.2. The volume fraction of each phase depended on the volume fraction in the microemulsion formation part.

A schematic diagram of the froth flotation apparatus used in this study is shown in Figure 3.3. The froth flotation apparatus consisted of a cylindrical glass column with 5 cm internal diameter and 70 cm height. Filtered air was introduced into the bottom of the column at constant flowrate of 250 ml/min through a sintered glass disk with having pore size diameter about 16-40 μm. One liter of well mixed solution (water and middle phases, water and oil phases, and water, middle and oil phases) under different ratios of mixed surfactants was transferred into the column. The air bubble rose through this solution. The foam came out from the column and was collected over a desired period of time at 20, 60, 90, 120, 150 and 180 min. The foam was broken for analysis concentration of ODCB as a liquid by using HPLC or High Performance Liquid Chromatography with a UV detector.

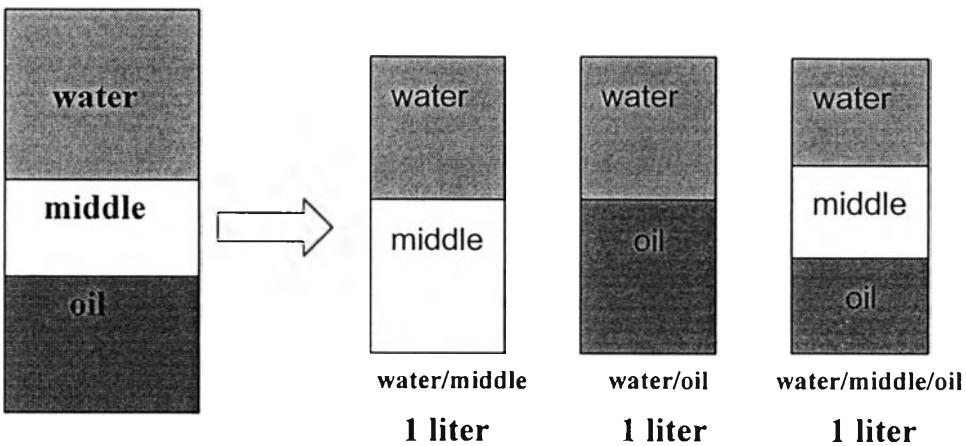

Figure 3.2 Schematic diagram of microemulsion solution in froth flotation experiment

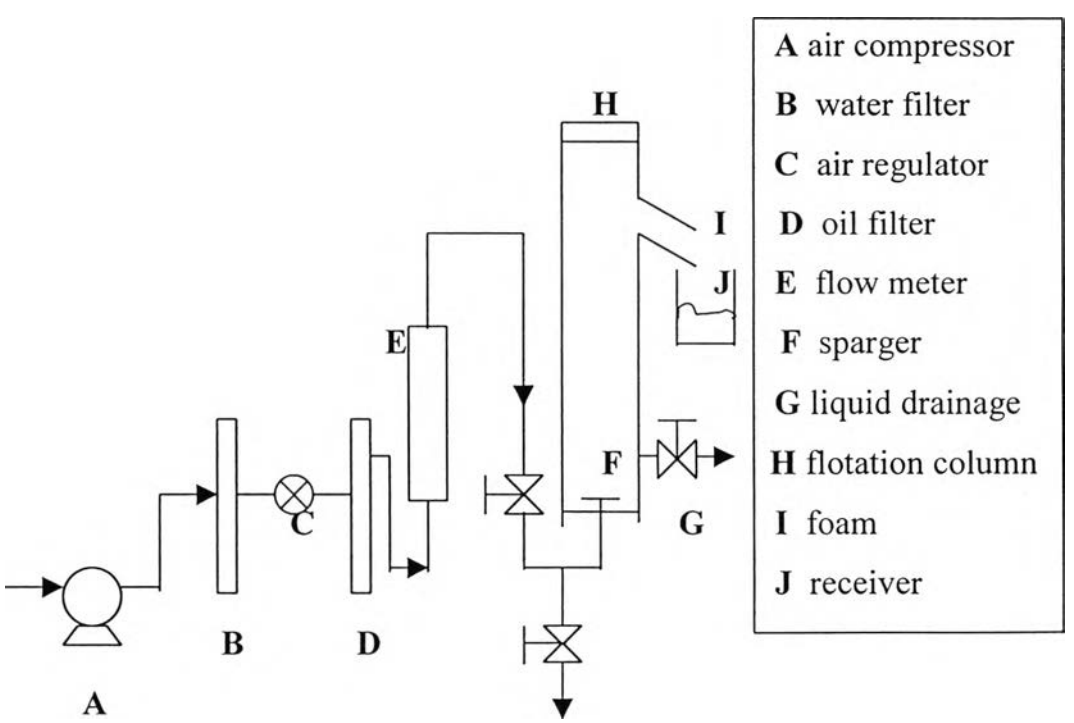

Figure 3.3 Schematic diagram of the froth flotation apparatus

3.3 Analytical methods

HPLC or High Performance Liquid Chromatography which is one of the Chromatography techniques was used to determine the concentrations of ODCB and surfactants. (Hewlett Packard,HP 1050).

The conditions used for analysis by HPLC are as follows:

ODCB: Flowrate 1.0 ml/min

Wave length 254 nm

Detector UV

Column ODS Hypersil Hewlett Packard

Mobile phase 85/15 HPLC grade methanol/distilled and filtered water