CHAPTER III
EXPERIMENTAL

3.1 Materials

The five anionic surfactants used in this research were sodium dodecyl sulfate (SDS, 99%+), sodiumhexadecyl diphynyloxide disulfonate or Dowfax8390 (36%), sodium di(2-ethylhexyl) sulfosuccinate or aerosol OT (AOT, 100%), sodium dihexyl sulfosuccinate or aerosol MA (AMA, 80% wt solution in water), and sodium diamyl sulfosuccinate or aerosol AY (AAY, 100%). The five cationic surfactants used in this study were dodecylpyridinium chloride hydrate (DPCl, 98%), dodecyltriammonium bromide (DTAB, 99%), hexadecyl triammonium bromide (CTAB, 97%), didodecyltriammonium bromide (DDAB, 98%), and ditallowdimethylammonium chloride or Arquad2T-70 (70%). SDS is a single tail C_{12} anionic surfactant. Dowfax 8390, which is a mixture of single-tailed and double-tailed diphenyloxide disulfonates (about 3 to 1 mixture), was supplied by Dow Chemical. AOT, AMA, and AAY were obtained from CYTEC. DPCl and DTAB are a single tail C_{12} and CTAB is a single tail C_{16} cationic surfactant. SDS, DPCl, DTAB, and CTAB were purchased from Aldrich Chemical Company. DDAB, a twin tail C_{12} surfactant, was obtained from Fluka. Arquad2T-70 or DTDACl, a twin tail C_{18} surfactant, was obtained in a form of aqueous solution from AkzoNobel. All surfactants were used without further purification. The chemical structure and molecular weight of all surfactants are shown in Table 1. Hexane (99%) was chosen as the main oil in this work. Trichloroethylene (99%) and hexadecane (98%) were also used for studying the effect of hydrophobicity of oil. Table 2 shows the molecular structure, density (ρ_o), molecular weight, and equivalent (EACN) of the oils considered in this work. The salt used was sodium chloride (99%+). Distilled water was used in all experiments.

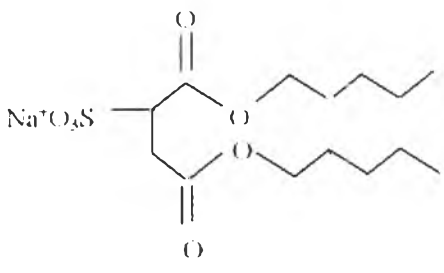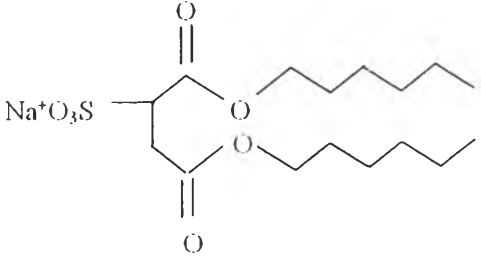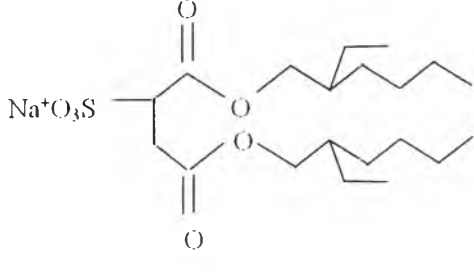**Table 1** Chemical structure and molecular weight of the studied surfactants

Surfactant	Chemical structure	Molecular weight (g/g-mole)
SDS	$CH_3(CH_2)_{11}SO_4^-Na^-$	288.38
Dowfax8390	$C_6H_4(SO_3^-Na^+)O\ C_6H_4(SO_3^-Na^+)C_{16}H_{33}$	642.00
AAY		360.00
AMA		388.46
AOT		444.00
DPCl	$CH_3(CH_2)_{11}$⊕$^+ NCl^-$	283.90
DTAB	$CH_3(CH_2)_{11}N^+(CH_3)_3Br^-$	308.35
CTAB	$CH_3(CH_2)_{15}\ N^+(CH_3)_3Br^-$	364.46
DDAB	$(CH_3(CH_2)_{11})_2N^+\ CH\ (CH_3)Br^-$	462.65
DTDACl	$(CH_3(CH_2)_{17})_2\ N^+\ CH\ (CH_3)Cl^-$	571.50

Table 2 Properties and chemical structure of the studied oils.

Oil	Chemical structure	Density (g/ml)	EACN	Molecular weight (g/g-mole)
Trichloroethylene (TCE)	Cl_2CHCl	1.464	-3.81	131.39
Hexane	$CH_3(CH_2)_4CH_3$	0.659	6	86.18
Hexadecane	$CH_3(CH_2)_{14}CH_3$	0.770	16	226.44

3.2 Experimental

3.2.1 Precipitation boundary

A series of surfactant solutions were prepared by varying anionic surfactant concentration while concentrations of cationic surfactants were kept constant. For studying the effect of added salt, 0.15 M NaCl was added into the solutions. All solutions were cooled down to 1 or $2^{\circ}C$ for 2 days in order to force the precipitation to occur. The solutions were then placed in the $25^{\circ}C$ water bath, shaken periodically, and allowed to equilibrate for at least 4 days before being analyzed. If a solution was outside of the precipitation region, crystals would redissolve so that the solution becomes clear. If crystals remained in the solution after equilibration, the initial solution composition was considered to be inside the precipitation region. Using this procedure, the boundary of the precipitation region could be determined. Surfactant solutions were kept for further study with the oil.

3.2.2 Microemulsion formation

Investigation of phase behavior, the microemulsion systems were prepared by using equal volumes of oil and surfactant solution, the temperature constant in a bath at $25^{\circ}C$. The phase volumes were determined by measuring the levels of each phase in test tube. The phase height and surfactant concentration were used to calculate the solubilization parameter.

3.2.3 Measurement of Microemulsion Interfacial Tension

Interfacial tension (IFT) between a microemulsion phase and excess oil and/or excess water phases at 25°C was determined by utilizing a spinning drop tensiometer (SITE 04; Kruss, Hamburg, Germany). The drop size and the number of revolutions per minute were read at an equilibrium state where there was no variation of drop size with time. The interfacial tension was calculated between two liquid phases by using the following formula:

$$\sigma = e(vd)^3 n^2 \Delta\rho \tag{3.1}$$

where σ is the interfacial tension (mN/m), e is a unit factor given by e = 3.427E-7 (mN cm^3 min^2)/(m gr mm^3), v is enlargement factor of lenses (for this study v = 0.31 mm sdv.), d is the drop diameter, n is the number of revolutions per min, and $\Delta\rho$ is the density difference between two liquid phases. Densities of microemulsions, excess oil and excess water phases were determined by weighing an exact volume of the solutions at 25°C.