CHAPTER III
METHODOLOGY

3.1 Materials

3.1.1 Surfactants

The Gemini surfactant used in this study was a commercial mixture of mono and di-hexadecyl diphenyloxide disulfonate, sodium salt (Dowfax#8390). It was obtained in a liquid form, 35% active, from Dow Company, Midland, Michigan. Dowfax#8390 is an anionic sulfonated surfactant.

The chemical formula and other general information of Dowfax8390 are shown in Table 3.1.

Table 3.1 General properties of Dowfax8390

Properties	Dowfax8390
Physical Properties	
- chemical formula	$(C_{16}H_3)_nC_{12}H_7O\text{-}(SO_3Na)_2$ n = 1 or 2
- average molecular weight	643
- active ingredient, min	35 %
- density, g/cc @ 25 °C	1.03-1.15
- viscosity, cps @ 25 °C	10
- boiling point	100 °C, 212 °F
Synonym	DADS

3.1.2 Studied Oil

Perchloroetylene (PCE) (tetrachloroethylene, 99% purity) was selected as the studied oil. It was purchased from Aldrich Chemical Co., Milwaukee, WI. The properties of this organic compound are listed in Table 3.2.

3.1.3 Other Compounds

Octanoic acid (99% purity) was purchased from Aldrich Chemical Co., Milwaukee, WI.

Anhydrous calcium chloride (desiccant grade) was purchased from Fisher Scientific.

Magnesium chloride hexa-hydrate ($MgCl_2.6H_2O$) was obtained from J.T. Baker Inc., Phillipsburg, NJ.

Deionized water was used as the aqueous solvent.

Table 3.2 General information of perchloroethylene and octanoic acid

Properties	Perchloroethylene (PCE)	Octanoic acid
Physical Properties		
- chemical formula	C_2Cl_4	$CH_3(CH_2)_6CO_2H$
- molecular weight	165.83	144.22
- melting point, °C	-22	16-16.5
- boiling point, °C	121	237
- Density (g/ml)	1.623	0.910
Health Hazard	Cancer suspect agent. Mutagen harmful vapor. Target organs are nerves, heart, liver, and kidney.	Corrosive

3.2 Experimental Procedures

The occurrence of middle phase microemulsions or single phase microemulsions was verified by visual observation. This method has been proven to be accurate as compared with all other methods (Bourrel and Schechter, 1988).

3.2.1 Study of Phase Behavior of Microemulsion and Solubilization

The microemulsion systems were prepared in 15 ml graduated test tubes (Fisher Scientific Co.) by using 1:1 volume ratio of oil (PCE + octanoic acid) to surfactant solution. Octanoic acid was weighed-in relative to Dowfax#8390 (weight ratio of Dowfax#8390 to octanoic acid, R = 0.75) into the tubes. PCE was added to make the oil volume to 5 ml, and then 5 ml surfactant in aqueous solution was filled in. Solid electrolyte was added to the system until a Type III or Type IV microemulsion was observed, or until a gel formed. The solutions or precipitate were shaken by hand until homogeneous, then allowed to equilibrate at room temperature (i.e., 24 °C).

3.2.2 Co-surfactant Studies

The microemulsion systems were prepared the same way as described in section 3.2.1, except the weight ratio of Dowfax8390 to octanoic acid was 1.0. This experiment determined the impact of octanoic acid on the phase behavior, on tetrachloroethylene solubilization, and on the presence of gel in the Type II region, when the amount of octanoic acid increased.

3.2.3 Mixed Electrolyte Studies

Preparation of section 3.2.1 was repeated except a mixed electrolyte (equal mole ratio of $MgCl_2.6H_2O$ to $CaCl_2$) was used instead of a single electrolyte ($CaCl_2$). The experiments were conducted at ratios of Dowfax#8390 to octanoic acid equal to 0.75 and 1.0. This experiment determined the effect of mixed electrolyte on phase behavior, perchloroethylene solubilization, and the presence of gel in the Type II-region.

3.2.4 Temperature Studies

All sets of the microemulsion systems prepared in sections 3.2.1, 3.2.2, and 3.2.3 were equilibrates at room temperature (24 °C) and in a water bath at 35 °C and 45 °C.