

CHAPTER III EXPERIMENTS

3.1 Materials

All chemicals were obtained commercially. Sodium bis (2-ethylhexyl) sulfosuccinate (Aerosol OT), at purity of 98%, n-decane (99%) were obtained from Fluka Chemical Company and were used without further purification. Alumina (AL5900), which has average pore size greater than 300 angstroms and the specific surface area more than 100 square meter per gram of alumina, was used as the substrate and was obtained from Alltech Associates, Inc. Sodium chloride was obtained from Ajax Chemicals Company.

3.2 Experimental methods

3.2.1 Phase behavior diagram

The experimental part involves measuring phase behavior of aerosol OT/n-decane/water/NaCl microemulsion at 45 °C. The mass fractions of aerosol OT (γ), oil (α), and salt (ε) in microemulsion system are defined as follows,

$$\alpha = \frac{W_{dec}}{W_b + W_{dec}} \quad \text{----- (3.1)}$$

$$\gamma = \frac{W_{AOT}}{W_b + W_{dec} + W_{AOT}} \quad \text{----- (3.2)}$$

$$\varepsilon = \frac{W_{NaCl}}{W_{NaCl} + W_{water}} \quad \text{----- (3.3)}$$

where W_{AOT} , W_{dec} , W_{NaCl} , W_{water} , W_b were weights of aerosol OT, n-decane, NaCl, water, and brine solution, respectively. The mass fraction of oil (α) was kept constant at 50 % weight, while the mass fraction of aerosol OT (γ) was varied 6-10% and the mass fraction of salt (ϵ) was varied 0.3-0.7%. Preparation of microemulsion as shown in Appendix A was done by respectively adding aerosol OT, deionized water, and NaCl salt in n-decane. The mixture solution was mixed by using VORTEX mixer and left in a temperature controlled bath at 45 °C for 2 days until it reached equilibrium state. After that, the system was observed for the type of microemulsion and volume of continuous phase was measured.

3.2.2 Adsorption of aerosol OT and adsolubilization of n-decane

Adsorption experiment was done by separating continuous phase of microemulsion type I into a tube containing alumina. Since the microemulsion system was sensitive to temperature change, transfer was carefully done in a controlled environment at 45 °C. Alumina, tubes and pipette tips were also preheated at 45 °C. Adsorption experiment was done in 2 ways by equilibrating alumina with the continuous phase without excess n-decane and with excess n-decane covering. The mixing solution before obtaining microemulsion type I was divided into 2 portions and separately equilibrated in tubes to get microemulsion type I. Then the continuous phase of each tube was brought to adsorption experiment.

3.2.2.1 Microemulsion type I without excess oil phase. The continuous phase of microemulsion type I was transferred into the test tube containing alumina. A ratio of the volume of continuous phase to mass of alumina was 1 ml. to 5 mg. Then the system was quickly put back to equilibrate for adsorption experiment. The adsorption and adsolubilization were allowed to proceed for 2 days. After the system reached the equilibrium state, the aerosol OT and n-decane remaining in the microemulsion solution were measured by HPLC and GC respectively. The amount of aerosol OT

adsorption and n-decane adsolubilization were determined from the concentration difference before and after adsorption.

3.2.2.2 Microemulsion type I with excess oil phase. The first step of preparation was the same as that described for the system without excess oil phase as mentioned in section 3.2.2.1. In the second step, a portion of the excess n-decane phase was carefully pipetted and slowly added to cover the microemulsion phase with the least disturbance of the system equilibrium. It was noted that the volume ratio of microemulsion phase to excess n-decane phase was 1 to 0.5.

3.3 Analysis methods

3.3.1 Analysis of aerosol OT and n-decane in microemulsion type I phase

The concentration of aerosol OT in the microemulsion phase was diluted 100 times by deionized water and analyzed by the Hewlett Packard HPLC Series 1050 with Alltech Alltima C18 column LC 5 μm ., 150 mm. x 4.6 mm., and Alltech 350 conductivity detector. The mobile phase composition was 80 % methanol (HPLC grade) and 20% water. The mobile phase flow rate was 1.0 ml/min. The total amount of aerosol OT in continuous phase was calculated from the aerosol OT concentration and the volume of microemulsion phase.

The concentration of n-decane was determined by using the Perkin Elmer AutoSystem ARNEL gas chromatography with Supelco pack column 10 % carbowax x 20 m, 80/100 supelcoport and flame ionization detector. The GC conditions setting was 20 ml./min. of nitrogen as a carrier gas, 150 °C of oven temperature, 220 °C of injector temperature, and 260 °C of detector temperature. The 2 ml. of diluted solution in a glass vial capped with PTFE-faced silicone septa was equilibrated at 100 °C using Perkin Elmer

HS 40 headspace for 15 minutes, pressurized for 0.1 min., withdrawn 0.2 min., and transferred at 100 °C of needle temperature to the GC injector.

3.3.2 Determination of aerosol OT adsorption and n-decane adsolubilization

3.3.2.1 *Microemulsion without excess oil phase.* The quantity of adsorbed aerosol OT on Al_2O_3 and the quantity of n-decane adsolubilization were calculated by the indirect method (Nunn, 1981). The basic procedure for the indirect method is illustrated in Figure 3.1.

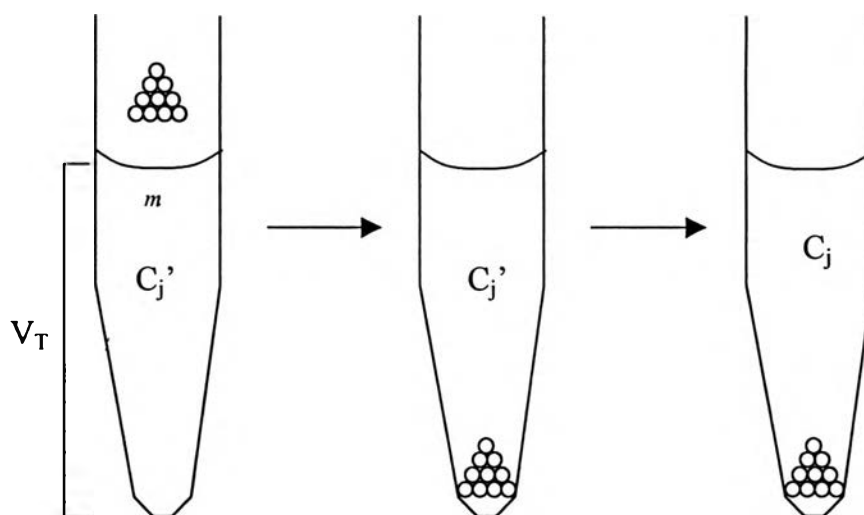


Figure 3.1 Schematic representation of the indirect method experiment for microemulsion without excess oil phase

The first step, the initial concentration of the j th component in the solution (C_j') was measured. After that, m grams of Al_2O_3 were contacted with V_T volume of the solution. After allowing the system to equilibrate, the final concentration of j th component (C_j) was analyzed. The adsorption, Γ_j , in mole per weight of Al_2O_3 was calculated from

$$\Gamma_j = \frac{V_T(C_j' - C_j)}{m} \quad \text{---- (3.4)}$$

3.3.2.2 *Microemulsion with excess oil phase.* Figure 3.2 indicated the procedure to measure aerosol OT adsorption and n-decane adsolubilization in microemulsion with excess oil phase.

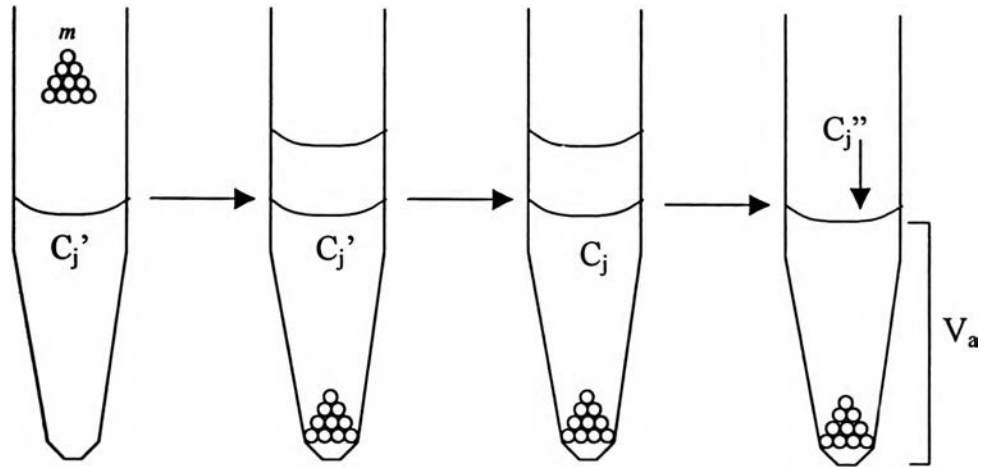


Figure 3.2 Schematic representation of the adsorption and adsolubilization experiment for microemulsion with excess oil phase

The initial concentration of the j th component in the solution (C_j') was measured. After that, m grams of Al_2O_3 were contacted with the microemulsion then excess n-decane was refilled to cover microemulsion phase. After allowing the system to equilibrate, the concentration of j th component (C_j) was analyzed and excess oil phase was removed. The final concentration of j th component (C_j'') was also analyzed from j th component in both of the remained microemulsion phase and alumina. Because the volume of alumina was very small when compare with the whole system, the volume of alumina was neglected. From the final concentration and volume of remained microemulsion phase (V_a), the total mole of j th component was determined. The adsorption, Γ_j , in mole per weight of Al_2O_3 was calculated by j th component mass balance as follow

$$\Gamma_j = \frac{V_a (C_j'' - C_j)}{m} \quad \text{---- (3.5)}$$