

## CHAPTER IV

### RESULTS AND DISCUSSION

#### **4.1 Water/aerosol OT/n-decane/NaCl microemulsion phase behavior**

The volumes of n-decane and water are dissolved within the aerosol OT micellar solution to form a single thermodynamically stable phase. Since aerosol OT which is double tail anionic surfactant and highly hydrophobic, forms microemulsion type II at room temperature, microemulsion type I can be obtained by increasing temperature to 45°C. The water/aerosol OT/n-decane/NaCl microemulsion phase behavior is observed to acquire a range of microemulsion type I preparation for the adsorption and adsolubilization experiment. The results are shown in Figure 4.1. At low concentration of aerosol OT, the microemulsion phase behavior type changes from I to III and II respectively when percent of salinity is scanned. At aerosol OT concentration 8-10%, the microemulsion phase behavior changes from I to IV and II respectively.

Amount of water and n-decane in aerosol OT micellar solution is termed solubilization. At low salinity the volume of n-decane dissolved in the aqueous micellar phase is very small. An increase in the electrolyte concentration results in an increase in the solubilization of n-decane. For type I systems, small amount of water is in oil phase and virtually all the water is in the micellar continuous phase. When the microemulsion type III regime is reached, water is expelled from the micellar phase. At the same time, the oil uptake in this phase continues to increase up to the point where all the oil present in the system is solubilized. Microemulsion type II system is obtained when further increase in the salinity. For ionic surfactant, the cohesive energy most profoundly changed upon the addition of electrolyte is the interaction between surfactant molecules. Increasing the electrolyte concentration tends to

decrease both the interactions between surfactant molecule and water and between surfactant molecules. The counterion binding associates strongly with charged hydrophilic part. The net result is a reduction in the hydrophilic effect of surfactant. The surfactant grouping is considered simultaneously when dealing with the effect of electrolyte in ionic surfactant. A typical variation of the amount of solubilization is the increasing electrolyte concentration, which in this case promotes the classical microemulsion type I-III-II transformation. Figure 4.1 also showed the expected I-III-II transition with increasing NaCl concentration.

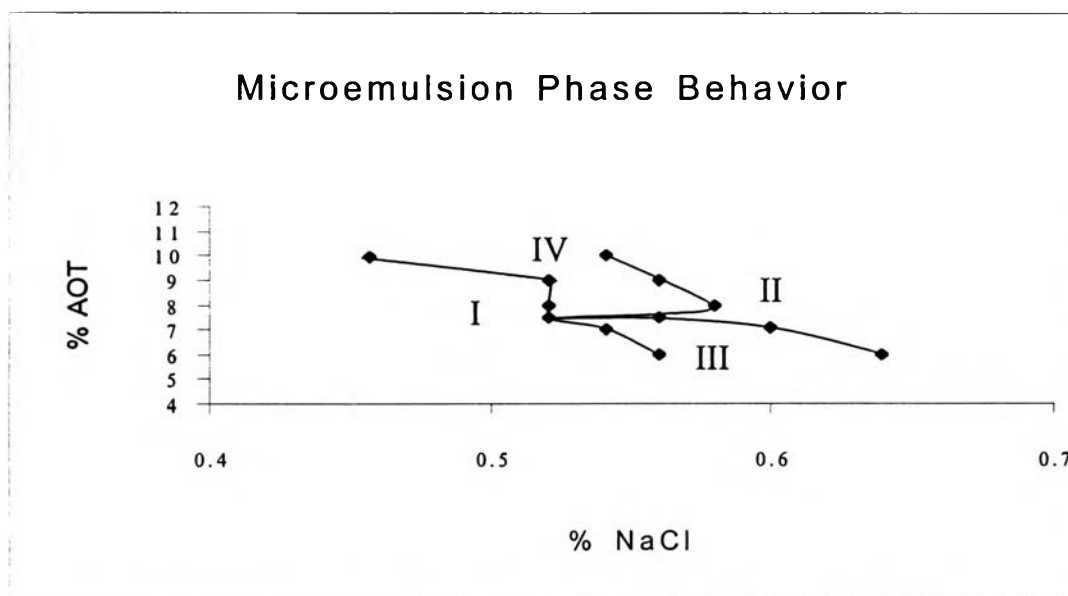


Figure 4.1 The water/aerosol OT/n-decane/NaCl microemulsion phase behavior at 45 °C

#### **4.2 The solubilization of aerosol OT and n-decane in microemulsion type I**

Before the adsorption and adsolubilization experiments were carried out, the initial amounts of aerosol OT and n-decane were first investigated by varying percent weight of NaCl and aerosol OT to obtain solubilization amount of aerosol OT and n-decane in the microemulsion phase. Figure 4.2 shows that volume of microemulsion phase increases with increasing percent weight of NaCl and further increases with increasing percent weight of aerosol OT. When the percent weight of aerosol OT in the microemulsion phase is very high (8-10%), formation of gel component occurs. Especially, when the weight fraction of salt gets close to the transition phase line, the volume of the microemulsion phase cannot be precisely measured. Furthermore at very high aerosol OT the microemulsion system is very sensitive to the temperature change. Therefore, it requires more attention to control the temperature of working environment.

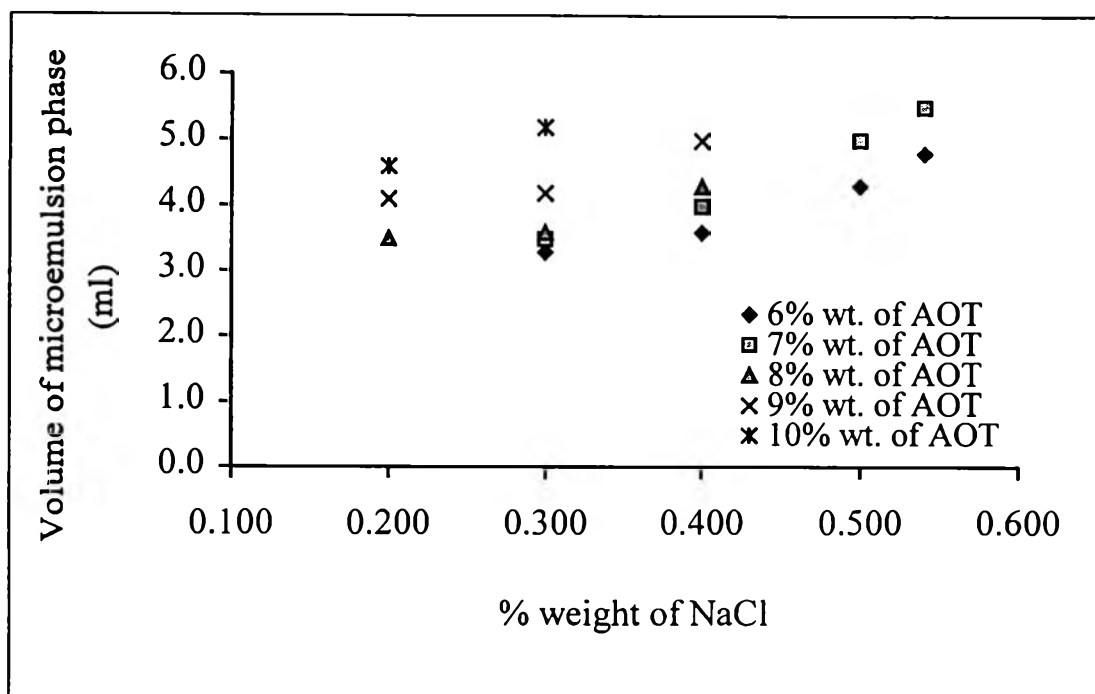


Figure 4.2 Increase of the volume of microemulsion phase in microemulsion type I system at 45 °C

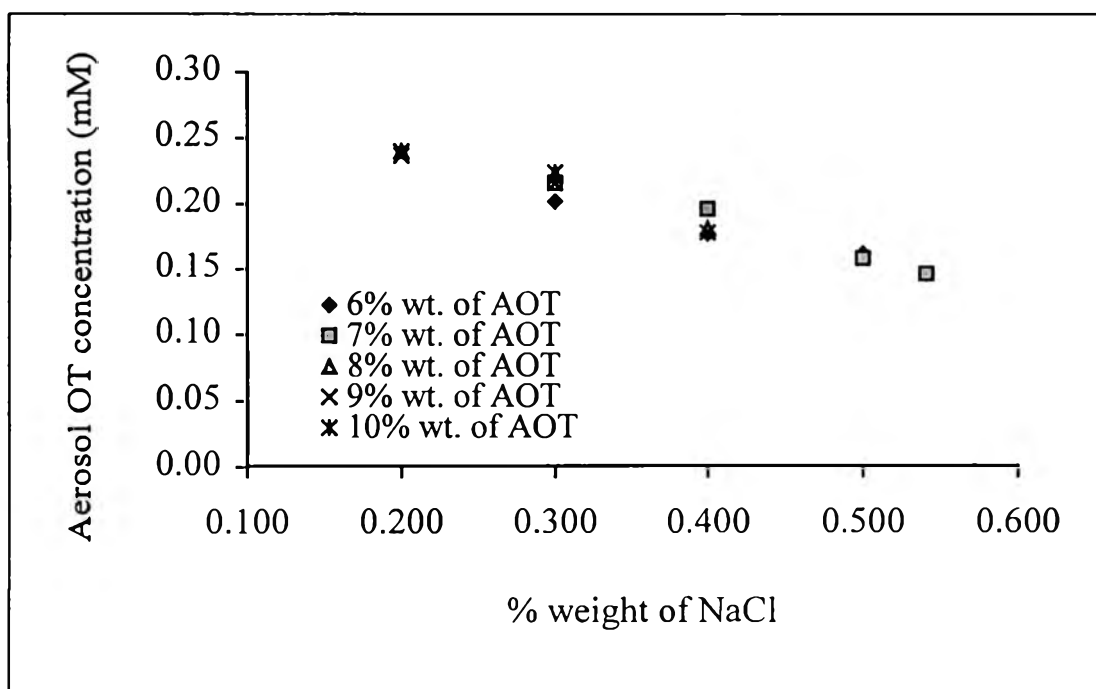


Figure 4.3 The aerosol OT concentration in microemulsion type I phase

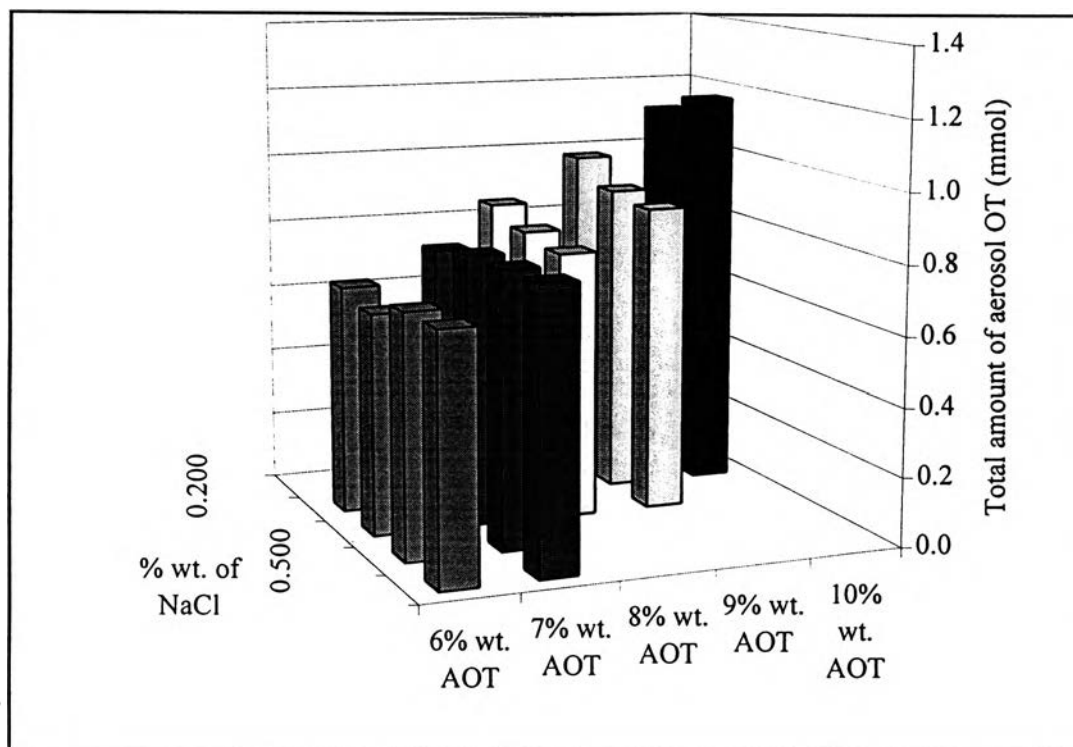


Figure 4.4 The total amount of aerosol OT in microemulsion type I phase

The results shown in Figures 4.3-4.4 are plots of aerosol OT present in microemulsion type I as the weight fraction of NaCl. As the percent weight of NaCl is increased, the concentration of aerosol OT in the microemulsion phase is decreased, however total mole of aerosol OT is rather constant as shown in Figure 4.4. The result of determination of n-decane solubilization in the microemulsion phase is shown in Figure 4.5.

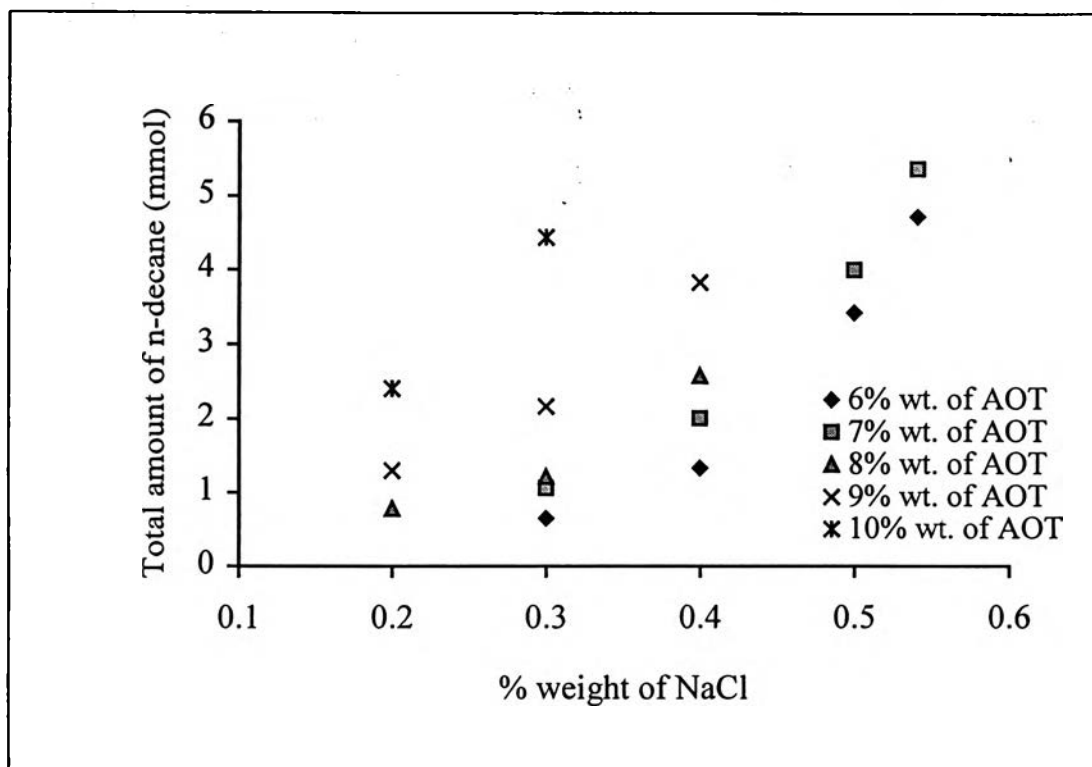


Figure 4.5 The total amount of n-decane in microemulsion type I phase

It is indicated that the amount of n-decane increases as the percent weight of NaCl is increased and further increases as the percent weight of aerosol OT is increased. It is noticed that large volume of n-decane occurs near the transition phase line. The change in the volume of microemulsion phase leads to the conclusion that the addition of electrolyte to the system helps to stabilize ionic head group of aerosol OT, thus increases the extent of solubilization of n-decane in the inner core of aerosol OT micelle (called swollen micelle). Furthermore, the addition of electrolyte reduces the repulsion between the similarly charged aerosol OT head groups and increases hydrophobicity, thus the aerosol OT micelles can form easily.

### 4.3 Aerosol OT adsorption and n-decane adsolubilization

#### 4.3.1 Microemulsion without excess oil phase

The microemulsion phase was brought in contact with alumina and left to reach equilibrium in the controlled at 45 °C. When the microemulsion system does not have excess n-decane covering, the results of adsorption of aerosol OT on alumina is shown in Figure 4.6. The adsorption of aerosol OT per gram of alumina decreases as the percent weight of NaCl is increased (i.e. approaching to the transition phase line). It is noted that the microemulsion system containing 10% weight of aerosol OT shows that the aerosol OT adsorption drops dramatically because the gel formation on alumina prevents the contact of aerosol OT on alumina.

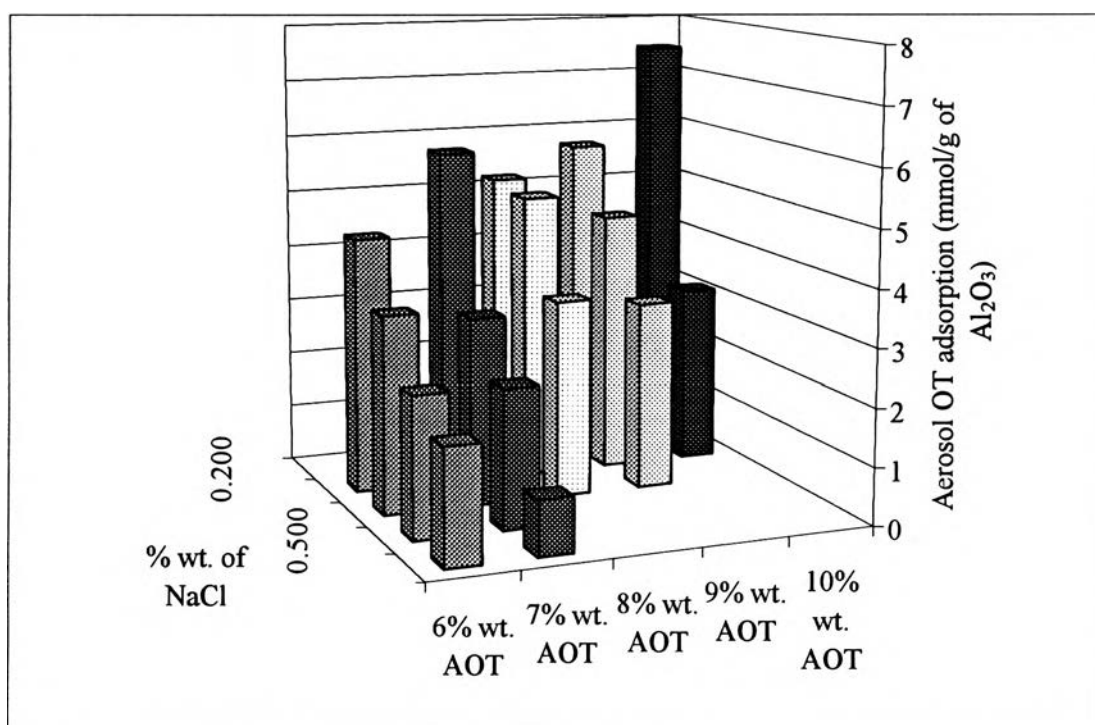
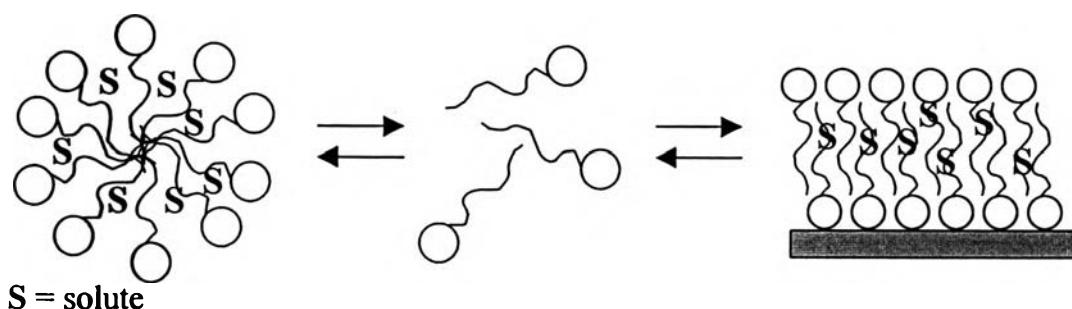


Figure 4.6 The adsorption of aerosol OT at 45 °C in the type I microemulsion phase without excess n-decane phase covering

The adsorption of monomeric aerosol OT on  $\text{Al}_2\text{O}_3$  occurs by the electrostatic attraction force between the charge of the anionic head group of aerosol OT and the charge on the  $\text{Al}_2\text{O}_3$  surface. The adsorption increases as the number of monomeric aerosol OT is increased. However, the monomeric aerosol OT is in equilibrium with the adsorption on  $\text{Al}_2\text{O}_3$  and the micellization as shown in the schematic below.



The adsorption of aerosol OT on alumina decreases micellization of aerosol OT. Increase number of micellization by increasing the percent weight of aerosol OT in the microemulsion phase also increases probability of monomeric aerosol OT to adsorb on the surface (as shown in Figure 4.6). As the percent weight of NaCl increases, chemical potential of aerosol OT in oil-swollen micelles is increased which favors decrease in chemical potential of monomeric aerosol OT for adsorption and solubilization of n-decane in micellar core is increased as well. As the chemical potential of monomeric aerosol OT for adsorption is increased, the admicellization is also increased. However, increase the percent weight of NaCl reduces hydrophilicity of aerosol OT and also helps to stabilize ionic repulsion between head groups, thus reduces the electrostatic attraction between the aerosol OT head groups and the alumina surface. The results still show that the aerosol OT adsorption decreases as the percent weight of NaCl is increased indicating that reduction of hydrophilicity at micellization has greater effect than adsorption of aerosol



OT on alumina. The adsorption decreases, as ionic strength of the microemulsion phase is very high.

Adsolubilization of n-decane in the admicelles of the microemulsion phase is shown in Figure 4.7. The results show that the adsolubilization of n-decane increases as the percent weight of NaCl is increased and significantly increases when the percent weight of NaCl is varied closely to the transition phase line even though the adsorption of aerosol OT is decreased in this region. It can be explained that the large increase in adsolubilization of n-decane is due to increase hydrophobicity of aerosol OT in admicelles and also desorption of n-decane from micellar phase and the admicellar region provides larger area for n-decane to adsolubilize than micellar core of the micellar phase. Furthermore, n-decane has more adsolubilization when increase the percent weight of aerosol OT in the microemulsion phase because more aerosol OT adsorbs on  $\text{Al}_2\text{O}_3$  surface.

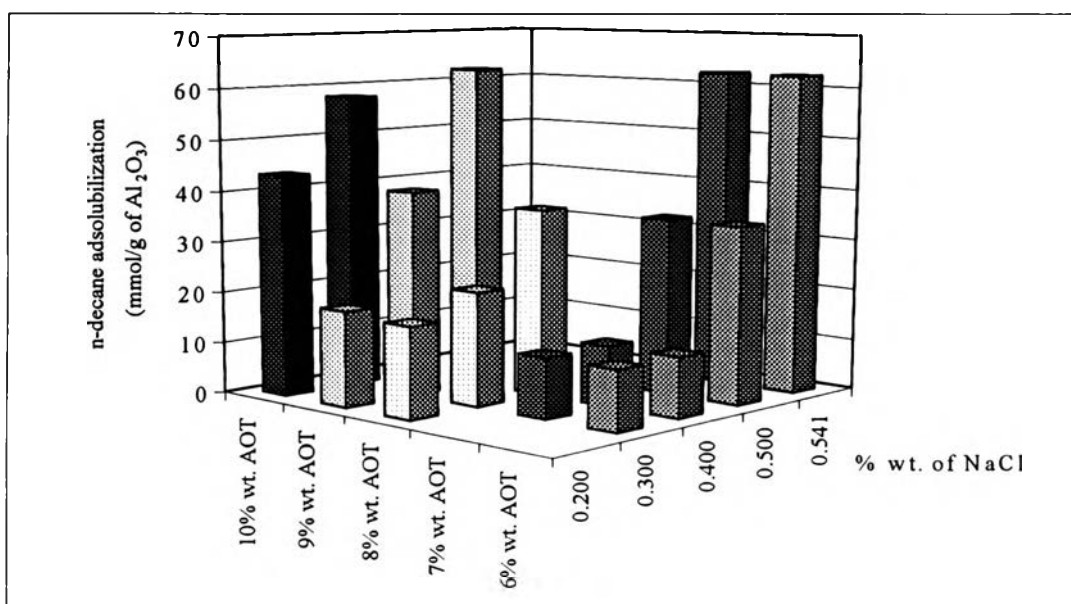


Figure 4.7 The adsolubilization of n-decane at 45 °C in the type I microemulsion phase without excess n-decane phase covering

#### 4.3.2 Microemulsion with excess oil phase

Adsorption of aerosol OT, when the microemulsion system containing excess n-decane was in contact with alumina, is shown Figure 4.8. The results show that as the percent weight of NaCl is increased, the adsorption of aerosol OT per gram of alumina is decreased. The results showed similar adsorption trend for the microemulsion system in the absence of n-decane covering, however the percent adsorption of aerosol OT in the presence of excess n-decane is higher due to a shift of equilibrium of aerosol OT in the excess oil phase to the microemulsion phase. The transfer of the excess was shown by analysis result of aerosol OT remaining in the microemulsion phase after adsorption equilibrium which is higher than before adsorption with excess n-decane phase covering.

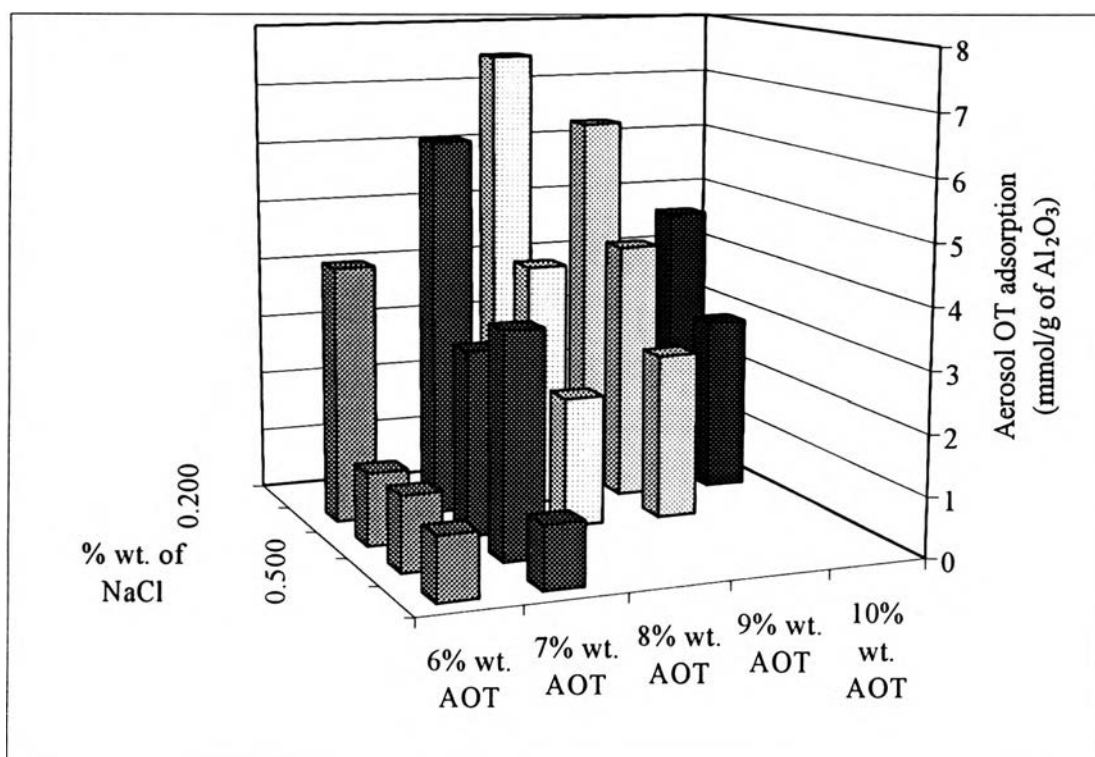


Figure 4.8 The adsorption of aerosol OT at 45 °C in the type I microemulsion phase with excess n-decane phase covering

Adsolubilization of n-decane in the microemulsion system with excess n-decane phase is shown in Figure 4.9. The results show that n-decane adsolubilization increases as increase the percent weight of NaCl and further increases as increase the percent weight of aerosol OT in the system. In all cases, the n-decane adsolubilization in the presence of excess oil is higher than those obtained for the system in the absence of excess oil phase. Again, the adsolubilization in the presence of excess n-decane shows the shift of equilibrium which n-decane transfers from the excess phase to the microemulsion phase and causes very high adsolubilization.

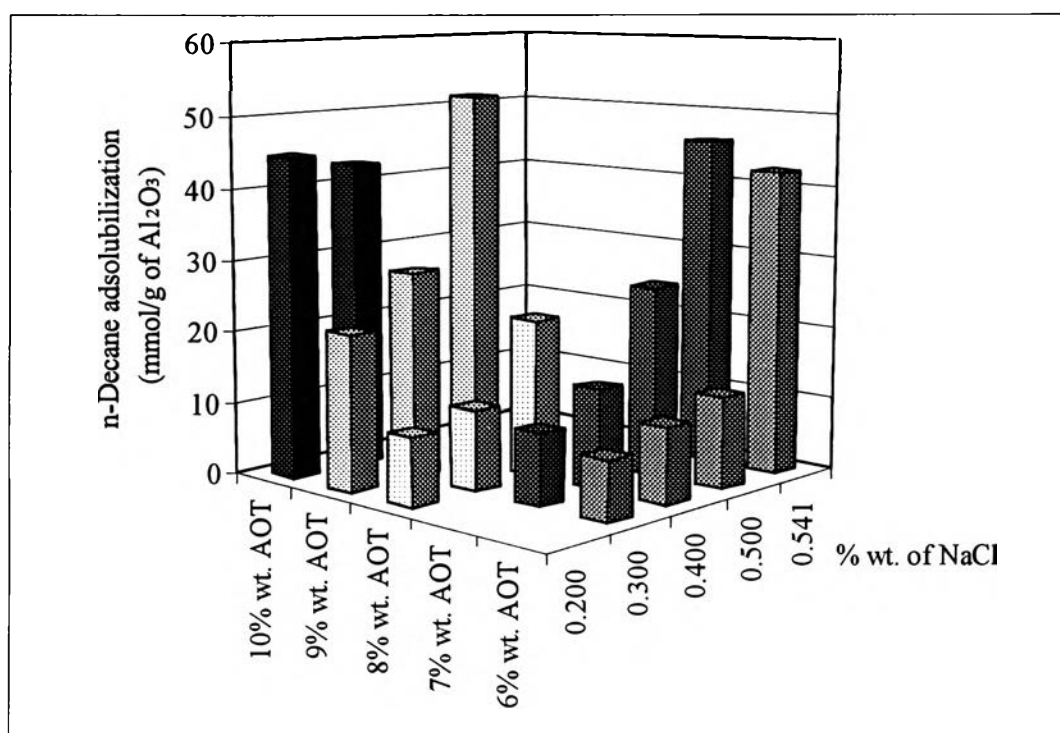


Figure 4.9 The adsolubilization of n-decane at 45 °C in the type I microemulsion phase with excess n-decane phase covering