



## CHAPTER IV

### RESULTS AND DISCUSSION

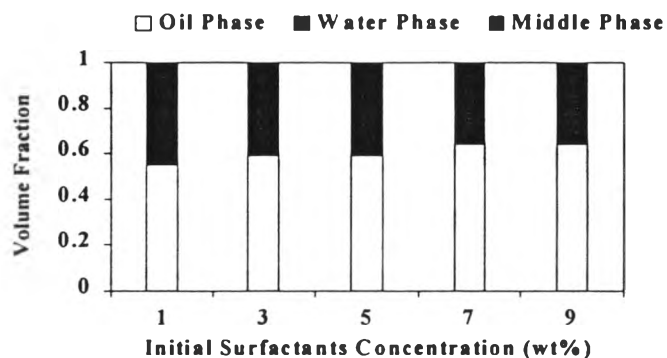
In this study, the term of  $X_{\text{SDS}}$  is the surfactant-only based weight fraction of SDS in the initial solution defining as the weight of SDS divided by total weight of the two surfactants used (SDS and NP(EO)<sub>10</sub>). The calculated value of  $X_{\text{SDS}}$  is based on the total solution, not on any of the two or three individual phases present.

#### 4.1 Microemulsion Formation Experiment

All experimental data of the microemulsion formation study is given in Appendix B. The experimental results are presented clearly below.

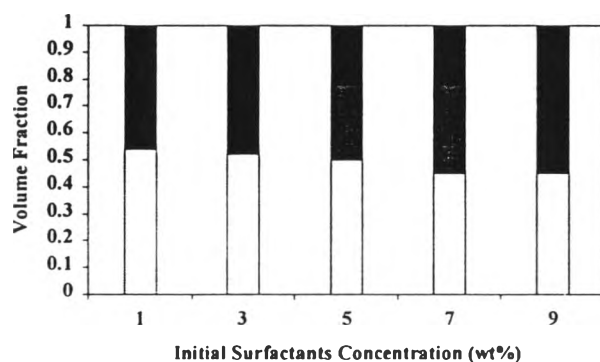
##### 4.1.1 Effect of Initial Surfactant Concentration on Phase Behavior

The effect of initial surfactant concentration on the phase behavior is shown in Figures 4.1. At the  $X_{\text{SDS}}$  value equal 0, the oil phase is a little bit higher in the height when the initial surfactant concentration increases as can be seen in Figure. 4.1(a). Figure 4.1(b) illustrate the phase behavior at  $X_{\text{SDS}}$  equal 0.5. It shows that as the initial surfactant concentration increases the solution changes from two phase system to three phase system. Figure 4.1(c) shows the phase behavior at  $X_{\text{SDS}}$  equal 1. The solution has only two phases: water and oil phases. The height of oil phase slightly decreases as the initial surfactant concentration increases. From the experimental results, it indicates clearly that



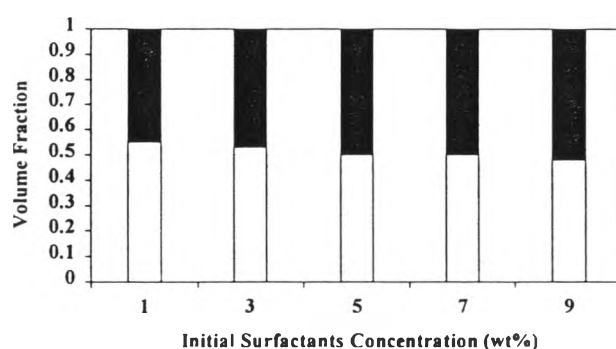
$$X_{SDS} = 0$$

(a)



$$X_{SDS} = 0.5$$

(b)



$$X_{SDS} = 1$$

(c)

Figure 4.1 Phase volume of microemulsion system at different initial surfactants concentrations (ISC) and  $X_{SDS}$  with initial oil/water volume ratio = 1/1, and temperature = 30°C

the presence of the microemulsion phase is dependent upon the percentage of initial surfactant concentration in the system. If the percentage is very small, the surfactant phase or the microemulsion phase cannot exist and the system appears to contain only two phases. If the percentage of surfactant is extremely large, the aqueous and non-polar phases may be completely solubilized in the surfactant phase and the system may contain only single phase (Rosen, 1989). However, in this study the initial surfactant concentration was not high enough to reach this type of phase behavior.

#### 4.1.2 Effect of Weight Fraction of SDS on Phase Behavior

The effect of weight fraction of SDS ( $X_{\text{SDS}}$ ) on the phase behavior is shown in Figures 4.2 which oil/water initial volume ratio is 1/1 and temperature is 30°C. At the initial surfactant concentration equal 1 and 3 wt.% the solution has only two phases of water and oil. As can be seen from Figures 4.2(a) and 4.2(b), the volume of water phase is slightly less than the volume of oil phase and the volume of both water and oil phases do not change much at different SDS weight fractions. At the initial surfactant concentration equal 5 and 7 wt.%, the formation of microemulsion phase was observed (see Fig. 4.2(c) and 4.2(d)). It was very interesting to know that when the initial surfactant concentration increased to 9 wt.%, the system contained only two phases (see Fig. 4.2(e)) and the volume of oil phase decreased as the value of  $X_{\text{SDS}}$  increased. From the results, it indicates clearly that phase behavior depends very much upon the SDS weight fraction.

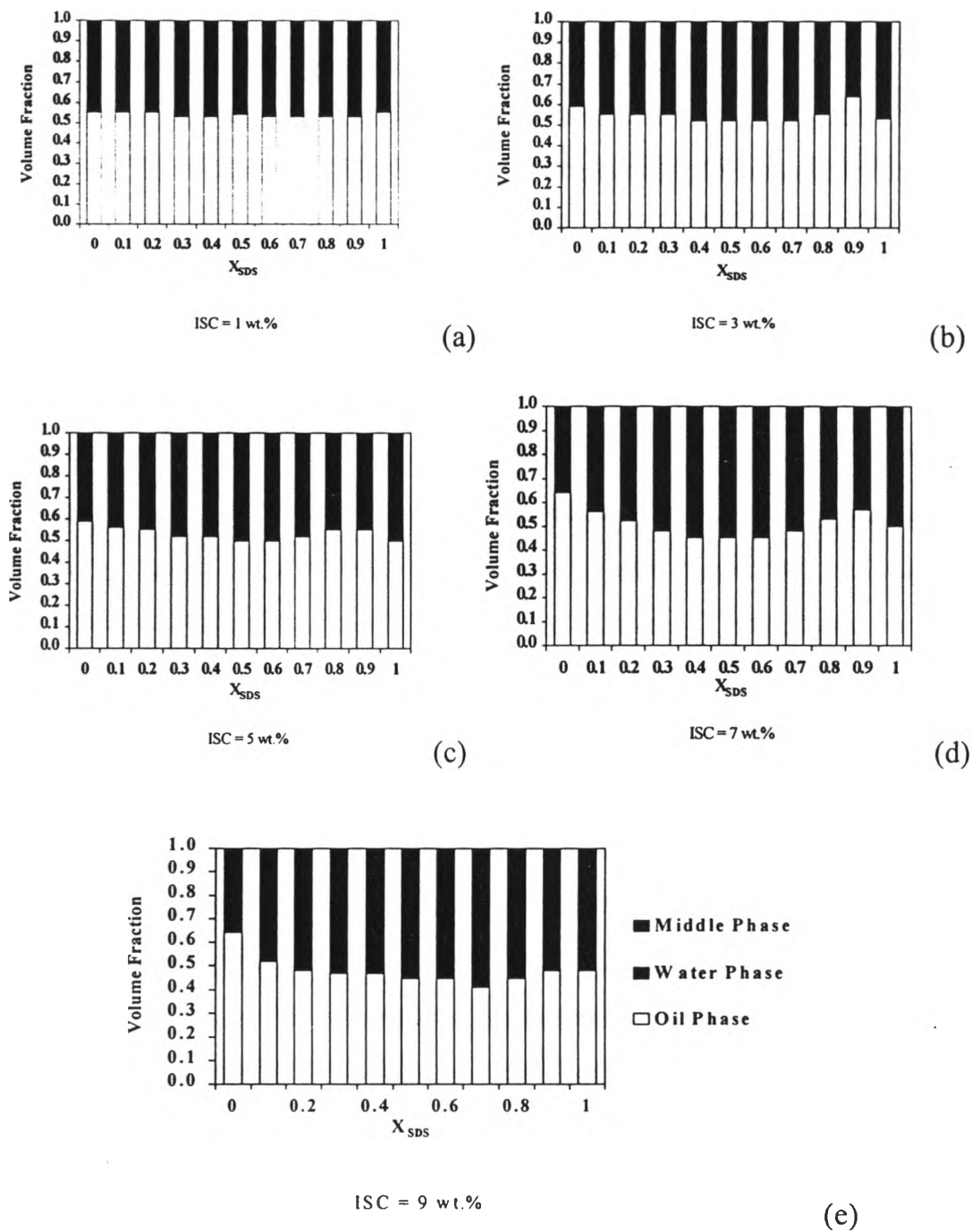


Figure 4.2 Phase volume of microemulsion system at different weight fraction of SDS and initial surfactants concentration (ISC) with initial oil/water volume ratio = 1/1 and temperature = 30°C

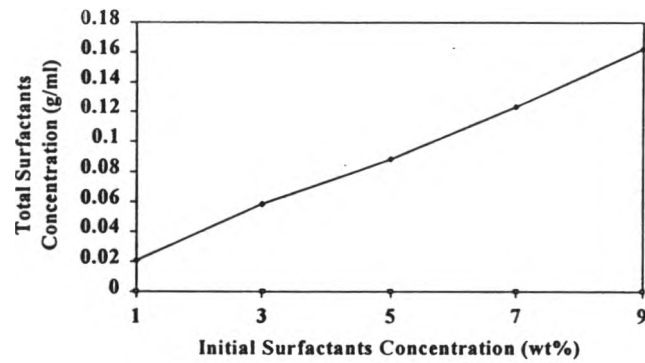
#### 4.1.3 Effect of Total Surfactant Concentration on Winsor-Type Formation

The effect of the initial surfactants concentration on the Winsor-type formation is illustrated in Figures 4.3. At the weight fraction of SDS equal 0, most of surfactants stay in the oil phase creating the Winsor's type II system as can be seen from Figure 4.3(a). An increase in the initial surfactants concentration resulted in the total surfactants concentration in the oil phase increased. Because at  $X_{\text{SDS}}$  equal 0, the system contain only NP(EO) 10 which loves to dissolve in oil. Figure 4.3(b) shows the effect of the initial surfactants concentration on the total surfactants concentrations in the water and oil phases at  $X_{\text{SDS}}$  equal 0.5. It showed that when the initial surfactant concentration increased, the surfactants concentration in the oil phase slightly increased while the surfactants concentration in water phase increased significantly. This indicates that the system could transfer from Winsor's type II to type III and to type I when the initial surfactants concentration increased. At the weight fraction of SDS equal 1, most of the surfactant stay in the water phase, when an increase the initial surfactant results in increasing the total surfactant concentration in water phase as shown in Figure. 4.3(c). Because at  $X$  equal 1 all of surfactant was SDS which love to dissolve in water more than in the oil so the solution appeared to be the Winsor's type I system.

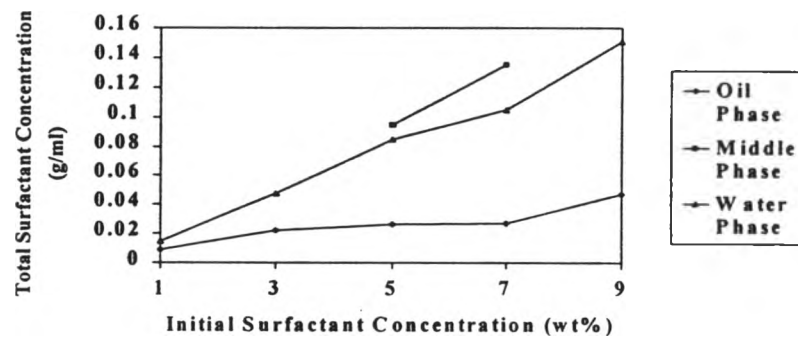
#### 4.1.4 Effect of Weight Fraction of SDS on Winsor-Type Formation

The effect of the weight fraction of SDS on the Winsor-type formation can be seen in Figures 4.4. At the initial surfactant concentration equal 1 wt.%, the system transferred from Winsor's type II to type I when the weight fraction of SDS increased (see Fig. 4.4(a)) since SDS loves to dissolve in water and NP(EO)<sub>10</sub> loves to dissolves in oil. Figure 4.4(b) illustrates the total surfactants concentration in both water and oil phases at the condition of

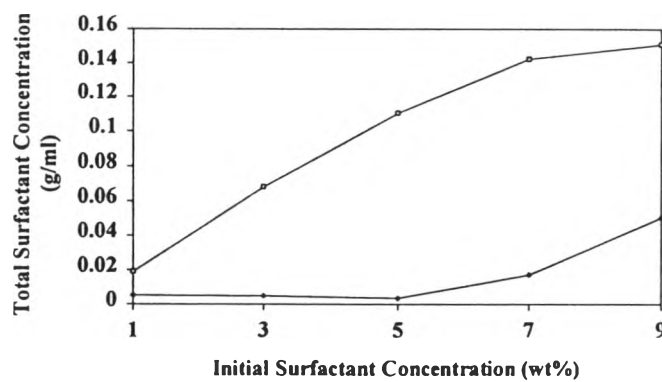
the initial surfactants concentration equal 3 wt.%. The system could move from Winsor's type II to type I system as increasing in  $X_{\text{SDS}}$ . The value of  $X_{\text{SDS}}$  that resulted in the system transferred from Winsor's type II to type I slightly decreased as increasing in the initial surfactants concentrations from 1 wt.% to 3 wt.%. At the initial surfactants concentrations equal 5 and 7 wt.%, the system transfers from Winsor's type II to type III and to type I when the weight fraction of SDS increases as shown in Figures 4.4(c) and 4.4(d). It can be seen clearly that the middle phase had the highest total surfactants concentration when compared with the other two phases. For a low value of the weight fraction of SDS, the system had a high content of NP(EO)<sub>10</sub> and a low content of SDS. As NP(EO)<sub>10</sub> is an oil lover, the system appeared to be the Winsor's type II which the total surfactants concentration in the oil phase was much higher than in the water phase. For a large value of the weight fraction of SDS, the system SDS much higher than NP(EO)<sub>10</sub>. As SDS is a water lover and so the system exhibited the Winsor's type I which the water phase contained the higher total surfactants concentration than the oil phase did. For an intermediate value of the weight fraction of SDS, the microemulsion phase existed. This similar structure of the middle phase in equilibrium with both excess water and oil phases was also be investigated by many researchers (Rosen, 1989). As can be seen clearly in Figure 4.4(e), when the initial surfactants concentration increases up to 9 wt.%, the middle phase (Winsor's type III) can not exist. At this initial surfactants concentration of 9 wt.%, the system would move from the Winsor's type II to type I as increasing in  $X_{\text{SDS}}$ .

 $X_{SDS} = 0$ 

(a)

 $X_{SDS} = 0.5$ 

(b)

 $X_{SDS} = 1$ 

(c)

Figure 4.3 Total surfactants concentrations in oil, water and middle phases at different initial surfactants concentration (ISC) and  $X_{SDS}$  with initial oil/water volume ratio = 1/1 and temperature = 30 °C

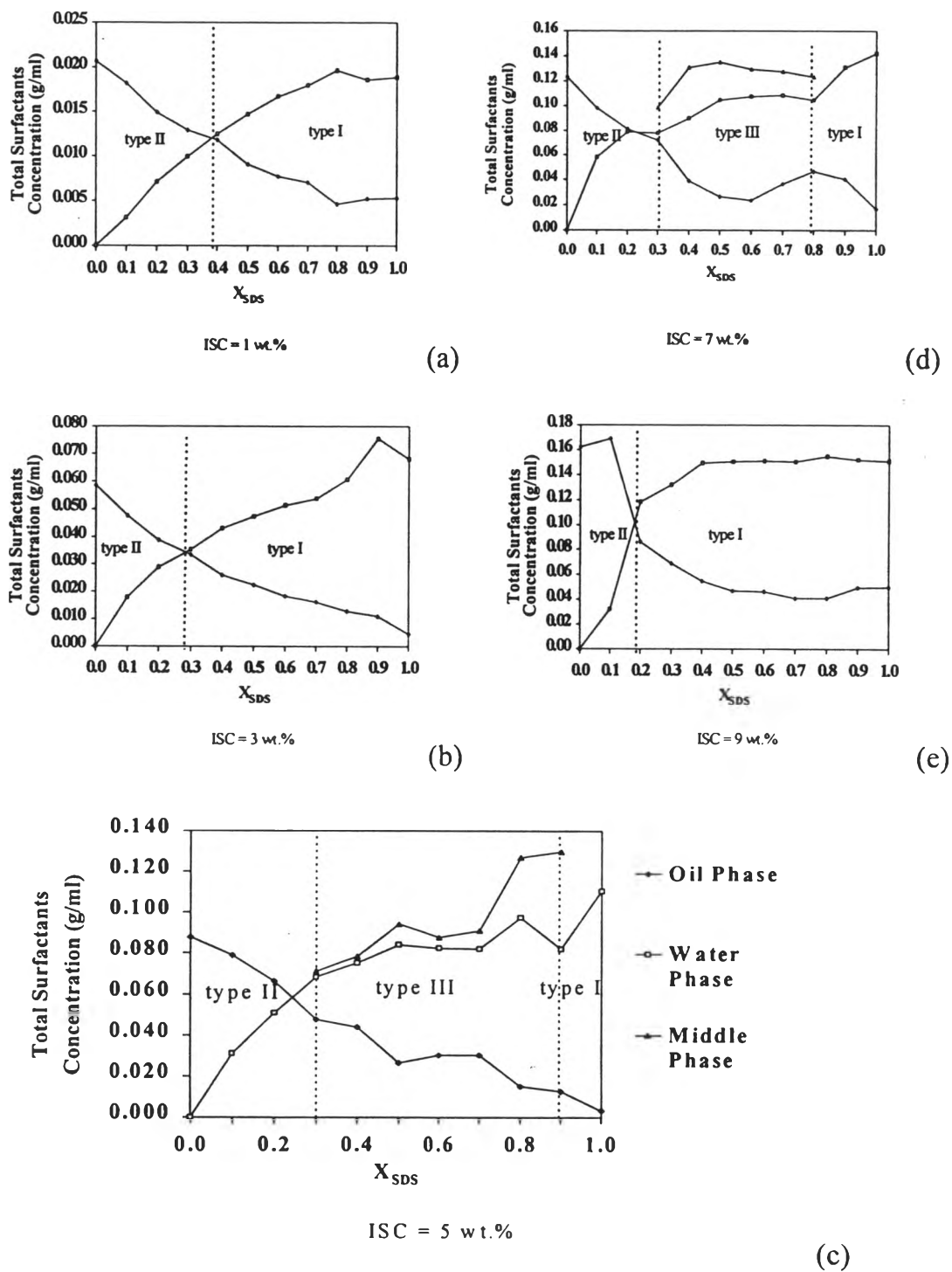


Figure 4.4 Total surfactants concentrations in oil, water and water phases at different  $X_{SDS}$  and initial surfactants concentrations (ISC) with initial oil/water volume ratio = 1/1 and temperature = 30 °C



#### 4.1.5 Effect of Initial Surfactants Concentration and Weight Fraction of SDS on Solubilities of NP(EO)<sub>10</sub> and SDS in water and Oil Phases

The effect of  $X_{\text{SDS}}$  on the solubility of NP(EO)<sub>10</sub> in the water and oil phases are illustrated in Figure 4.5. An increase in  $X_{\text{SDS}}$  could improve the solubility of NP(EO)<sub>10</sub> in water but decreased the solubility of NP(EO)<sub>10</sub> in oil. The solubility of NP(EO)<sub>10</sub> in the water phase and SDS in the water and oil phases is shown in Fig. 4.6. An increases in the value of  $X_{\text{SDS}}$  resulted in the higher concentration of SDS in both water and oil phase. An increase in the initial surfactant concentration lead to increases the solubility of NP(EO)<sub>10</sub> in water phase and increases the solubility of SDS in the oil phase. The solubility of NP(EO)<sub>10</sub> and SDS in water increases as an increases in the value of  $X_{\text{SDS}}$  and initial surfactant concentration which is the one reason for the transferring of the surfactant from Winsor's type II system to the other type of microemulsion.

In the system of initial surfactant concentration equal 5 and 7 wt.% the water, oil and microemulsion phases were analyzed for determine the percentage of water, oil and surfactant as shown in Fig. 4.7 and 4.8. The water phase is composed of ODCB, surfactant and water by the water is the main composition surfactant. Also the water is the main composition in the microemulsion phase except the oil phase which has the ODCB to be the main composition and has very less surfactant.

However, the microemulsion study are interesting. For examples, microemulsion formation with mixed chlorinated hydrocarbon liquids(Baran, 1994), the formation of microemulsion by study the effect of temperature and polyacrylamide (John, and Rakshit, 1993) and the study of phase behavior on microemulsion system by varies type of surfactant (Chhatre and Kulkarni, 1991; Binks, 1993; Kegel and Lekkerkerker, 1993). Because the microemulsion

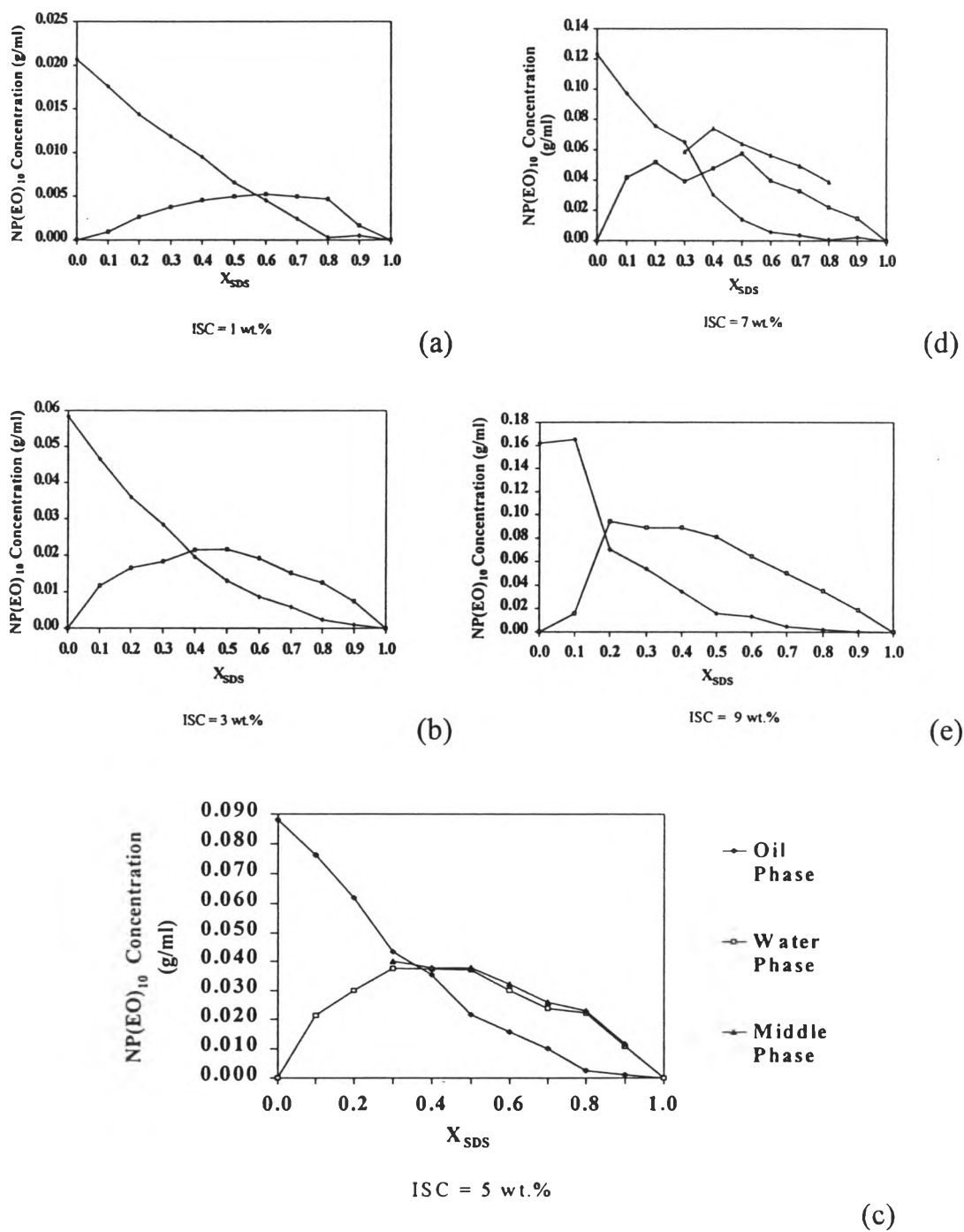


Figure 4.5 NP(EO)<sub>10</sub> concentration in oil, water and middle phases at different  $X_{SDS}$  and initial surfactants concentration (ISC) with initial oil /water volume ratio = 1/1 and temperature = 30<sup>0</sup>C

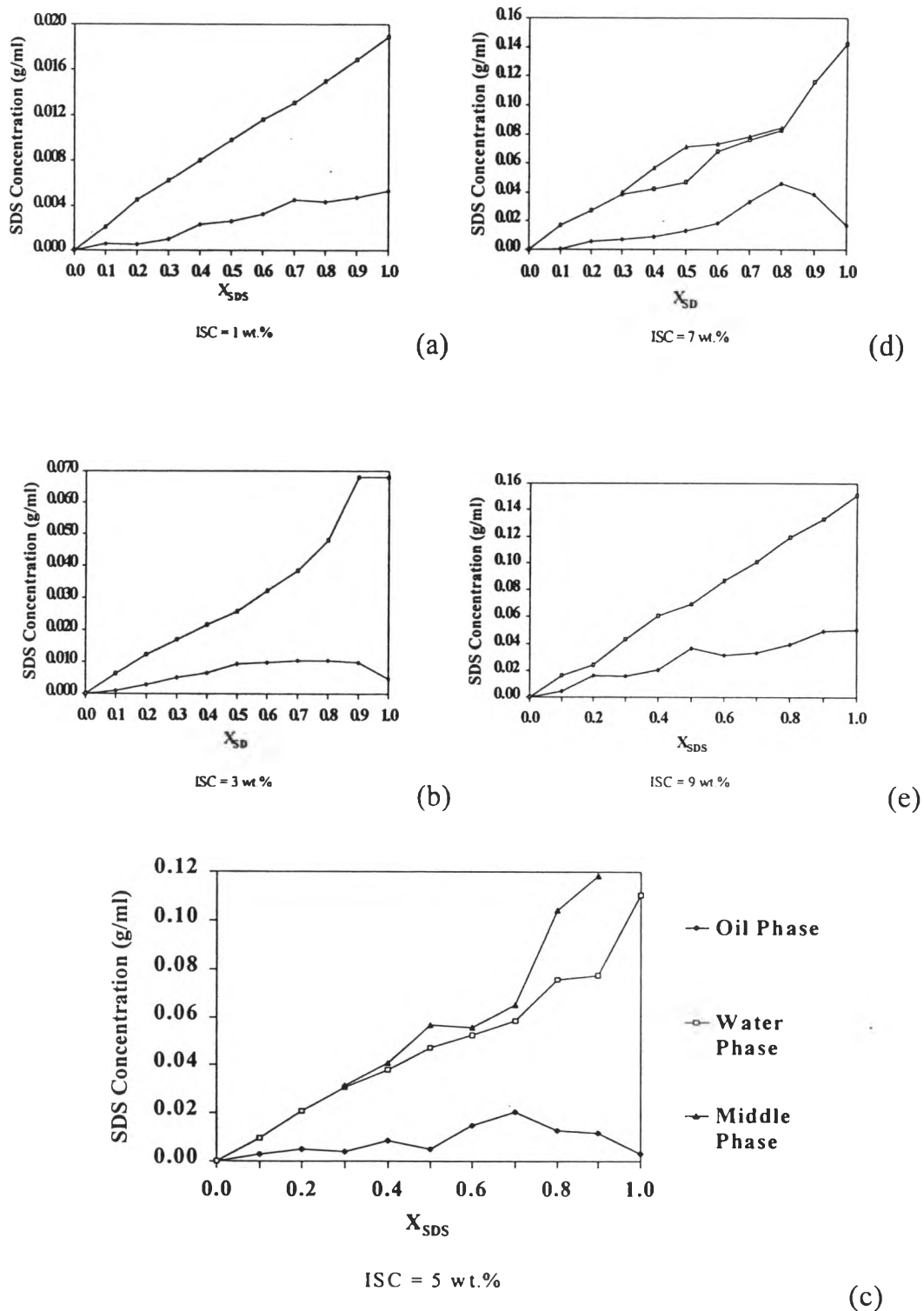
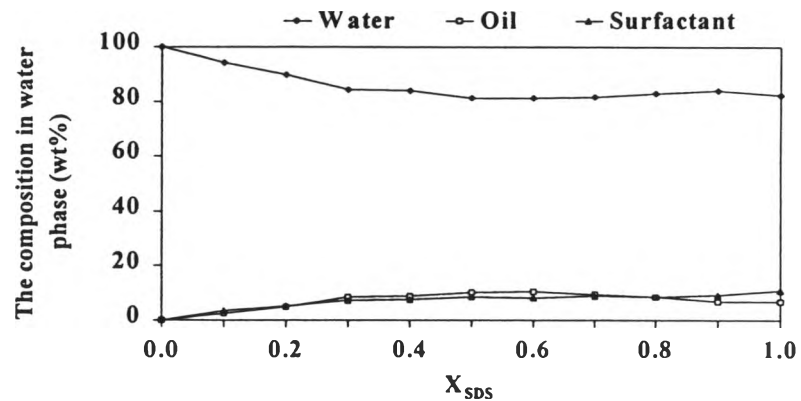
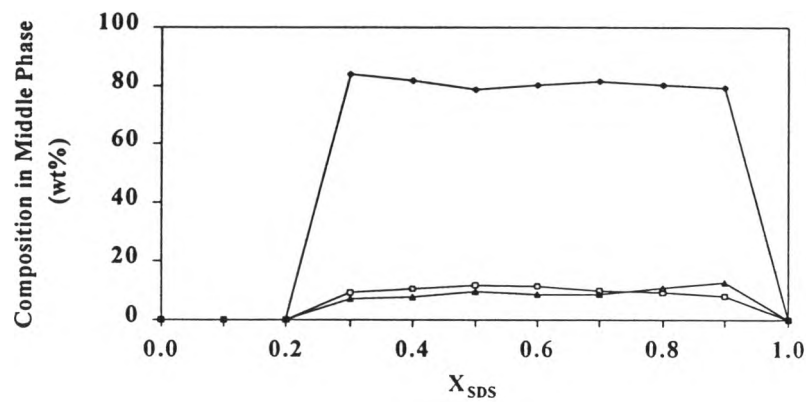


Figure 4.6 SDS concentration in oil, water and middle phases at different  $X_{SDS}$  and initial surfactants concentration (ISC) with initial oil/water volume ratio = 1/1 and temperature = 30°C



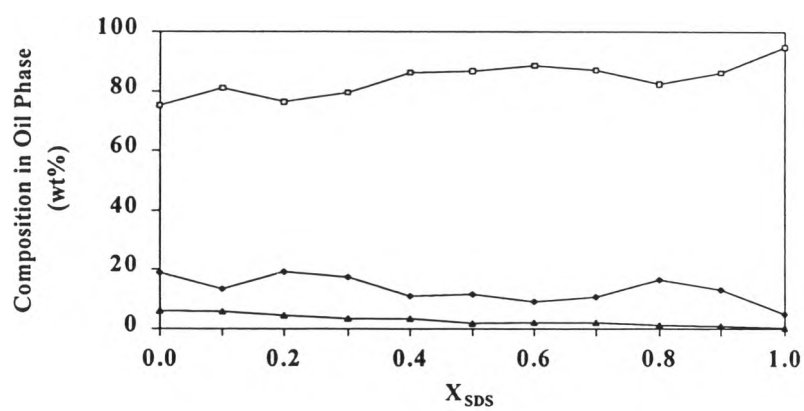
Water phase

(a)



Middle phase

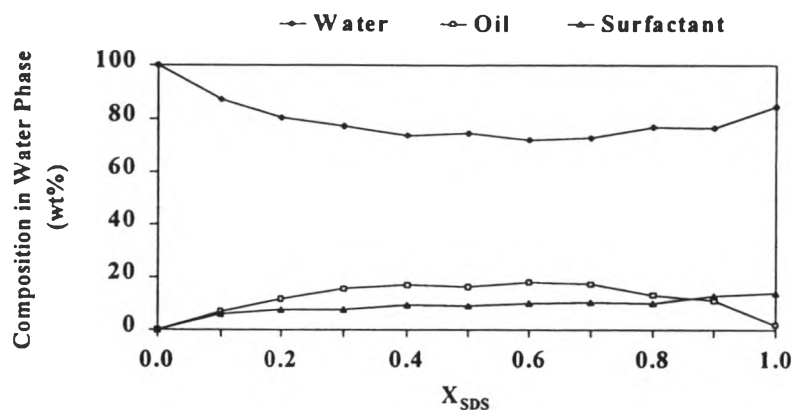
(b)



Oil phase

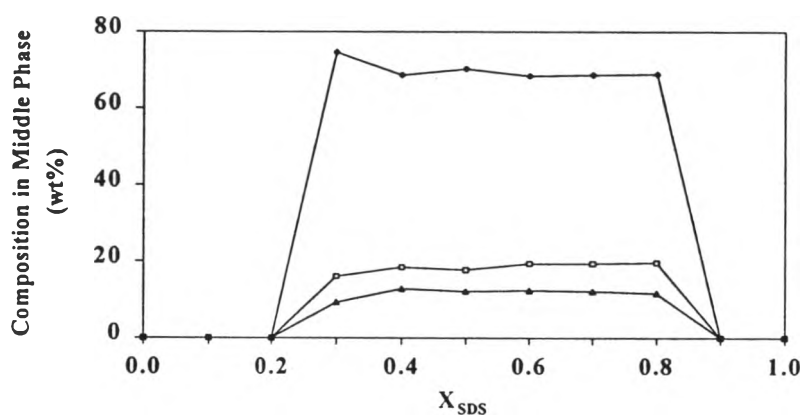
(c)

Figure 4.7 Compositions of three phases at different  $X_{SDS}$  with initial surfactants concentration = 5 wt.%, initial oil/water volume ratio = 1/1 and temperature = 30<sup>0</sup>C



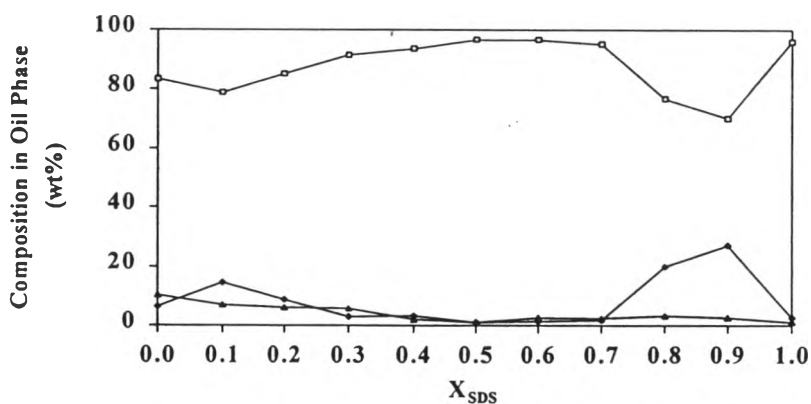
Water phase

(a)



Middle phase

(b)



Oil phase

(c)

Figure 4.8 Compositions of three phases at different  $X_{SDS}$  with initial surfactants concentration = 7 wt.%, initial oil/water volume ratio = 1/1 and temperature = 30<sup>0</sup>C

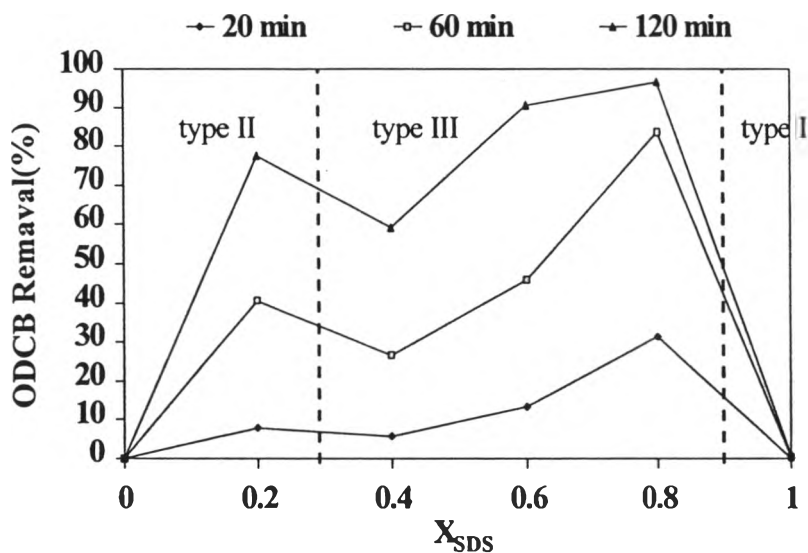
can be used as effective media for interfacial reactions and interfacial synthesis. They are also used as efficient fluid systems in novel applications such as tertiary oil recovery, and organ preservation fluids.

## 4.2 Froth Flotation Study

Appendix C shows the experimental data of the study of froth flotation process under the microemulsion condition.

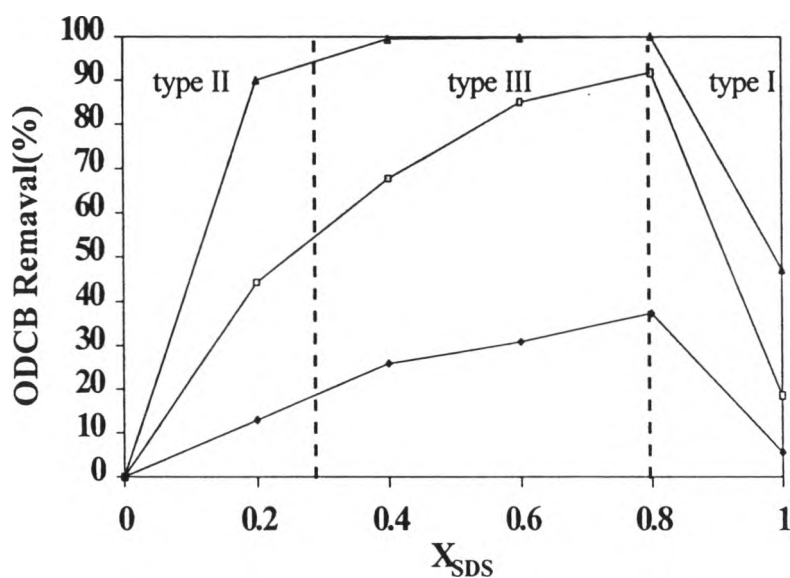
### 4.2.1 Effect of Weight Fraction of SDS on ODCB and Surfactant Removal

The effect of surfactant composition on ODCB, and surfactants removals can be seen in Figures 4.9 to 4.11. The removal of ODCB increases as the value of  $X_{\text{SDS}}$  increases when the system transferred from Winsor's type II to type III. On the other hand, the removal of ODCB decreases as the value of  $X_{\text{SDS}}$  increases when the system transfer from Winsor's type III to type I system. The results reported here would imply that when the solution is in Winsor's type III system, the flotation efficiency is greater than when the solution is the Winsor's type II or type I system. From the previous works, adding NaCl in the froth flotation process where microemulsion was present can improve the ODCB removal (Pongstabodee, et al.). In contrast, adding NaCl in the froth flotation process where no microemulsion was present and it resulted in lower oil removal (Peng and Di, 1994). Therefore, occur of microemulsion solution was one reason for improve the flotation efficiency. Also, the effect of surfactant composition on the NP(EO)<sub>10</sub> and SDS removal is the same as the ODCB removal. By the removal of NP(EO)<sub>10</sub> and SDS increases with increasing the value of  $X_{\text{SDS}}$ , when the system transfer from



Initial surfactants concentration = 5 wt.%

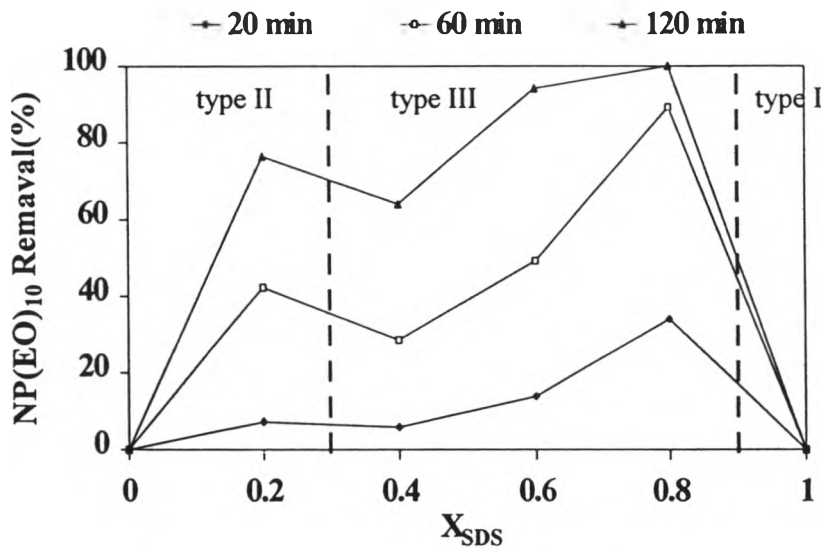
(a)



Initial surfactants concentration = 7 wt.%

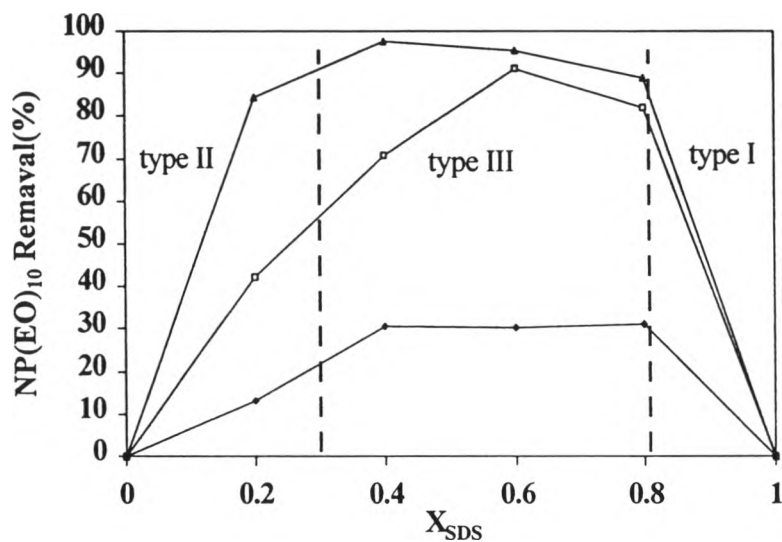
(b)

Figure 4.9 Removal efficiency of ODCB at different types of microemulsion, and two different initial surfactants concentration with initial oil/water volume ratio = 1/1 and temperature = 30°C



Initial surfactants concentration = 5 wt.%

(a)

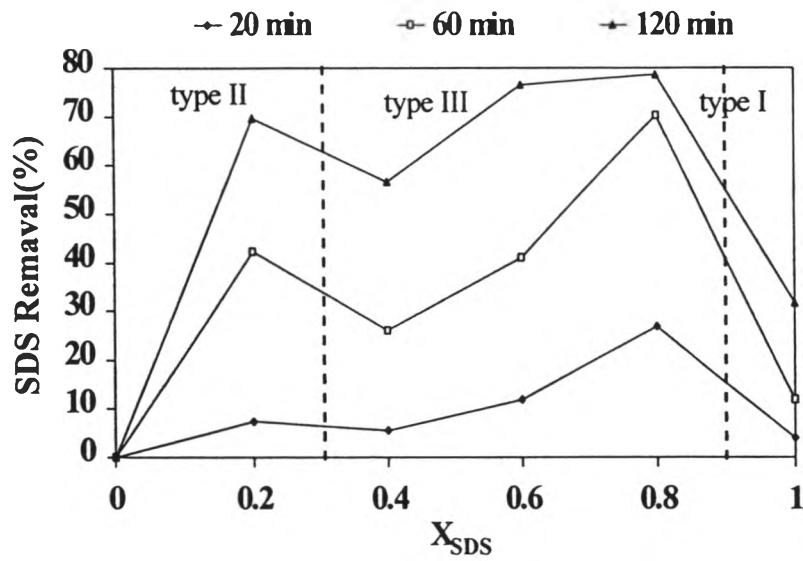


Initial surfactants concentration = 7 wt.%

(b)

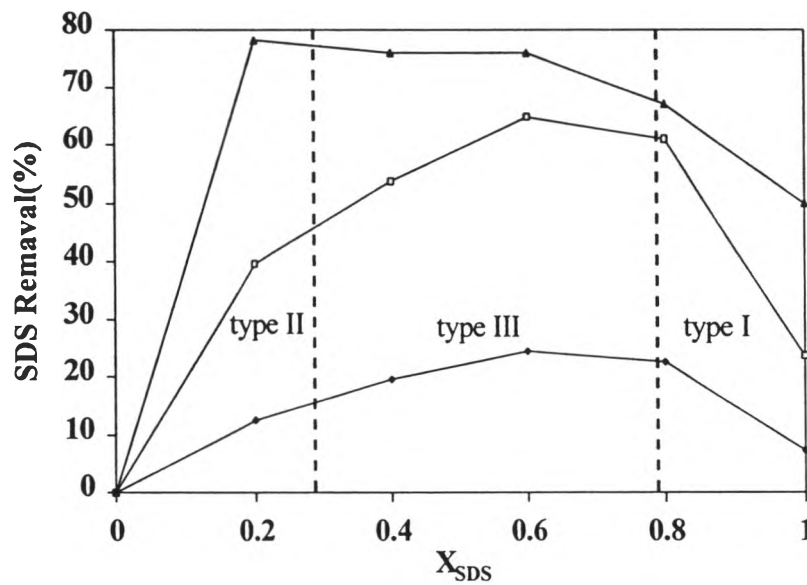
Figure 4.10 Removal efficiency of NP(EO)<sub>10</sub> at different types of microemulsion, and two different initial surfactants concentration with initial oil/water volume ratio = 1/1 and temperature = 30°C





Initial surfactants concentration = 5 wt.%

(a)



Initial surfactants concentration = 7 wt.%

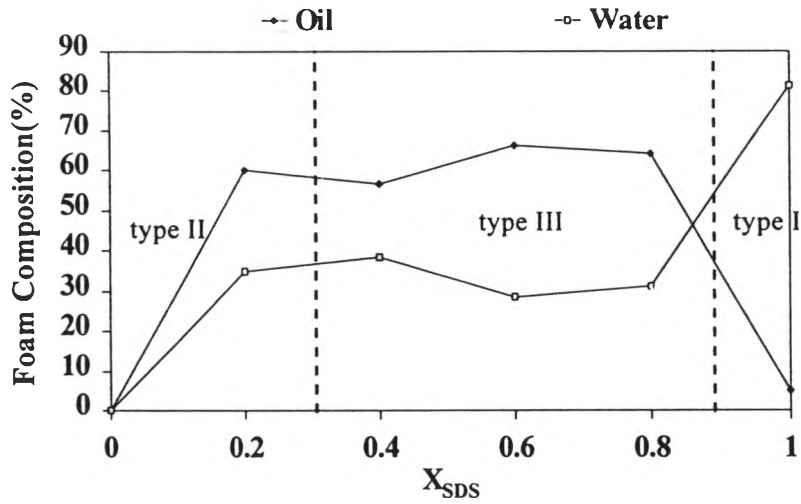
(b)

Figure 4.11 Removal efficiency of SDS at different types of microemulsion, and two different initial surfactants concentration with initial oil/water volume ratio = 1/1 and temperature = 30°C

Winsor's type II to type III system. But the removal of NP(EO)<sub>10</sub> and SDS decreases as the value of  $X_{\text{SDS}}$  increases, when the system transfer from Winsor's type III to type I system. However, the increase of the bubbling time from 20 min to 60 min can improve the flotation efficiency more than the bubbling time change from 60 min to 120 min.

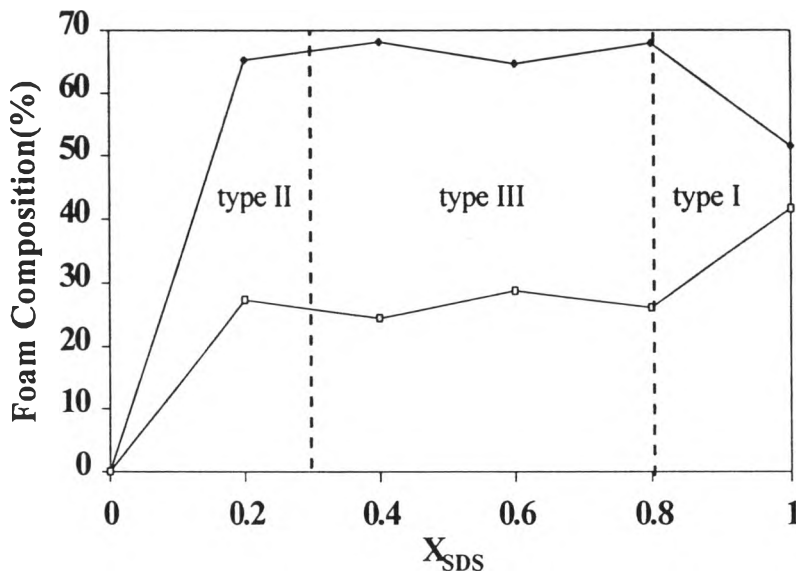
#### 4.2.2 The Effect of Weight fraction of SDS on The Percentage of ODCB and Water in Foam

If consider on the composition in the foam, the efficiency of the froth flotation process can be seen in Figure 4.12. This figure shown the percentage of ODCB and water in foam. Figure 4.12(a) is the result of the froth flotation process at the initial surfactant concentration equal 5 wt.%. The Winsor's type II system give the percentage of ODCB in foam a little higher than the percentage of ODCB in the initial solution and the Winsor's type III system give the percentage of ODCB in foam higher than the percentage of ODCB in the initial solution but the Winsor's type I system give the percentage of ODCB in foam lower than the percentage of ODCB in the initial solution. Figure 4.12(b) is the results of the froth flotation process at the initial surfactant concentration equal 7 wt.% which give the same results as Figure 4.12(a). From these results can be implied that the Winsor's type III system is good for the froth flotation process to increases the percentage of ODCB in foam or decreases the percentage of water in foam.



Initial surfactants concentration = 5 wt.%

(a)



Initial surfactants concentration = 7 wt.%

(b)

Figure 4.12 ODCB and water contents in foam fraction at different types of microemulsion and two different initial surfactants concentration with initial oil/water volume ratio = 1/1, temperature = 30°C and operating time = 120 min