CHAPTER II
CLEAN-UP OF OILY WASTEWATER BY FROTH FLOTATION: EFFECT OF MICROEMULSION FORMATION II: USE OF ANIONIC/NONIONIC SURFACTANT MIXTURES

2.1 Abstract

Froth flotation can be an effective method to remove emulsified oil from wastewater. In this series of studies, the relationship between surfactant phase behavior (which type of microemulsion is formed between the oil and water) and efficiency of the flotation of ortho-dichlorobenzene is being investigated. The phase behavior is related to surfactant composition and salinity in this work. Use of anionic/nonionic surfactant mixtures here permit examination of a wide range of conditions compared to the use of only anionic surfactants. Optimum oil flotation corresponds to a Winsor Type III microemulsion, which also corresponds to minimum interfacial tensions between oil and water and to maximum solubilization of water and of oil into a surfactant rich microemulsion phase. In this work, high selectivity for oil compared to water in the overhead froth was demonstrated, a necessary criterion for an effective separation.

2.2 Introduction

Chlorobenzene and its derivatives, such as ortho-dichlorobenzene (ODCB), are classified as hazardous substances. However, they are widely used in several applications (1). Ortho-dichlorobenzene is representative of pollutants found in wastewater and ranks high in the US Environmental Protection Agency list of organic priority pollutants (2). Removal of ODCB and similar compounds from wastewaters prior to discharge is a challenging chemical engineering operation. We focus here on removal of ODCB as emulsified droplets in water, rather than as molecularly dissolved pollutants, although froth flotation can be used to remove the latter (3) where it is sometimes called foam flotation or foam fractionation. For a sparingly soluble chemical like ODCB, oil loadings can be many orders of magnitude higher for emulsified oil compared to dissolved oil.

Flotation is a surfactant-based separation process (4 – 6) in which surfactant is added to an aqueous solution and air is sparged through the solution. Air bubbles from sparging air are used as a means of separation (7). Dissolved molecules or ions, solid particles (e.g. ore colligend or ink), or droplets of an emulsified oil can attach to the air bubbles and be carried over to the top of a flotation cell with the foam (called a froth in this case). As a result, the formation of stable bubble-particle aggregation is required in the flotation technique (8) to increase separation efficiency. Flotation operations are suitable for dilute waste treatment because it has several advantages including rapid operation, low space requirement, high efficiency of removal, flexibility of application to various pollutants at various scales, and low cost (9). Consequently, flotation has been used in addressing many waste treatment problems (3, 7, 9 – 16). For example, Feng and Aldrich (13) found that up to 99% of diesel oil could be removed from water by flotation with cationic surfactants.

The presence of surfactant located at the air-water interface in flotation operations promotes the formation of a froth. Moreover, surfactants dramatically increase the dispersion of air in the flotation column or tank, reduce the coalescence of individual bubbles within the liquid, and decrease the rate at which the bubbles rise to the surface. Zouboulis et al. (7) reported that without surfactant, air flotation alone had very little effect on separation. A pure liquid does not foam (17) and so addition of a surfactant is necessary to induce frothing. When a surfactant is added to water, the surface tension of the solution decreases as a result of preferential adsorption of surfactant molecules at the air/water interface. The surfactant molecules are arranged at the interface such that the hydrophilic or polar group (head group) is situated in the water phase and the hydrophobic or hydrocarbon chain (tail group) in the air phase (6). Moreover, a suitable amount of surfactant (9), and a suitable amount of salt (1) are needed to optimize the flotation system.

Under normal conditions, surfactants can reduce the interfacial tension between an oil phase and a water phase from ca. 30 mN/m to ca. 1 mN/m due to the preferrable adsorption of surfactants at the oil/ water interface. In the complex froth flotation process, this interfacial tension reduction is probably partially responsible for the surfactant being important. At an optimal mixing ratio of brine, oil, surfactant, and cosurfactant, a microemulsion is formed (18). Microemulsions are transparent or

translucent dispersions containing two immiscible liquids with droplets of 10 to 100 nm diameter (17) which do not separate (19) because they are themodynamically stable. It is well-known that ultralow oil/water interfacial tension (e.g., 10^{-3} mN/m) corresponds to formation of a third phase, in equilibrium with the oil and water phases, called a middle phase (20); the system under these conditions is known as a Winsor Type III microemulsion. It has been hypothesized by us that synergisms in froth flotation of oily wastewater could correspond to a Type III microemulsion. In the first paper in this series (1), we showed that maximum ODCB removal did occur in an anionic surfactant system when the system was formulated so that a Winsor Type III microemulsion was present. However, limitations on formulation freedom do not permit flotation at the composition at which minimum interfacial tensions are expected in that system (called optimum conditions) because surfactant precipitation and liquid crystals form under these conditions for that system. When microemulsions are used in enhanced oil recovery, surfactant-enhanced subsurface remediation, and consumer products (to name just three applications), this problem is often avoided by adding a hydrotrope like a low-molecular weight alcohol (e.g., isopropanol), often at levels like a few percentage in solution. The hydrotrope reduces the tendency to form ordered surfactant structures like solid crystals or liquid crystals, avoiding phase separation problems (17).

The option of adding a hydrotrope at these fairly high concentrations is not feasible in the froth flotation process for two reasons. First, the hydrotrope can volatilize if its vapor pressure is too high, leading to air pollution. Second, the hydrotrope will probably remain in unacceptably high concentrations in the water, after the emulsified oil is removed, to permit discharge to the environment. It is interesting to note that in surfactant-enhanced subsurface remediation, if the surfactant is to be reused after flushing the solution through contaminated soil, and the contaminant is volatile (e.g., trichloroethylene), a stripping operation is commonly used to, for example, air strip to remove trichloroethylene from the surfactant solution. The presence of a high concentration of a volatile hydrotrope can dramatically increase the load on the stripper as well as requiring replacement of the hydrotrope upon recycle of the surfactant solution. Salvager's hydrophobic linker (21) and the use of co-surfactants (like long-chain alcohol) by Uchiyama et al. (22) were employed to replace the function of the hydrotrope by (hopefully a much lower concentration of) a higher

molecular weight material. In this study, mixtures of an anionic surfactant (sodium dodecyl sulfate) and a nonionic surfactant (nonylphenol polyethoxylate) were used to adjust phase boundaries without a volatile hydrotrope present.

In our previous study (1) of ODCB removal using an anionic surfactant, while enhancement of oil removal with the froth in the microemulsion region was established, water carry-over with the foam was not measured. Measurement of both water and oil in froth carry-over in this work indicates the selectivity of the separation for the oil.

2.3 Experimental Section

2.3.1 Materials

The oil used was ortho-dichlorobenzene (ODCB) obtained from Fisher Scientific Co., with 99.9% purity. The surfactants were sodium dodecyl sulfate (SDS) from Henkel company with at least 90% purity and nonylphenol ethoxylate ($NP(EO)_{10}$) obtained from ICI Australia Operations Pty Ltd. with a purity of greater than 99.9%. Sodium chloride (NaCl), analytical purity grade, was obtained from Aldrich Chemical Co. Deionized water was used in all of the experiments.

2.3.2 Methodology

In both the microemulsion phase behavior and the froth flotation experiments, the surfactant concentration and salinity were expressed as weight percent of the overall system; namely, water, oil, surfactant, and salt. All experiments were conducted at 30° C.

In investigation of phase behavior, 5 mL of a well-mixed aqueous solution, comprised water, surfactant, and salt, was mixed with 5 mL of ODCB in a vial and sealed with a screw cap and hold in a constant temperature water bath at 30° C. The vials were shaken every 2 hours for 12 hours, and then to stand in the water bath at 30° C for 1 month until equilibrium was achieved. The volume of each phase at equilibrium was determined by measurement of phase height.

A schematic diagram of the froth flotation apparatus used in this study is shown in Figure 2.1. A cylindrical glass column with 5 cm internal diameter and 70 cm height was used as a froth flotation column. One liter of well-mixed solution was immediately transferred into the flotation column. The studied solutions were prepared according to the phase behavior results which will be described in detail later. Filtered air was introduced at the bottom of the froth flotation column at a constant flowrate of 250 mL/min through a sintered glass disk, having pore size diameter about 16 – 40 μm. Foam from the top of the column was collected at 20, 30, and 60 minutes and was then broken for analysis as a liquid. Concentrations of surfactants and ODCB in each phase of the solution were analyzed by a HPLC (Hewlett Packard, Series 1050) with a UV detector.

2.4 Results and Discussion

In this study, X_{SDS} is defined as the weight fraction of SDS which is weight of SDS divided by total weight of mixed surfactants (SDS and $NP(EO)_{10}$). Moreover, the total surfactant concentration is reported in units of weight percentage of total solution (water, oil, salt, and surfactants).

2.4.1 Phase Behavior Results

As reviewed in detail in part I of this series (1), microemulsions can be classified as Winsor Type I, II, or III (20, 23, 24). In a Type I system, the surfactant–containing water phase is in equilibrium with an excess oil phase which contains little surfactant while the surfactant–containing oil phase is in equilibrium with an excess water phase which contains little surfactant in a Type II system. For a Winsor Type III system, a third or middle phase is in equilibrium with an excess oil phase and an excess water phase. The middle phase forms between the excess phases since it has intermediate density between oil and water, and this middle phase contains almost all of the surfactants. In general, as the system is adjusted to make the surfactant more hydrophobic (e.g., increased salt concentration for an anionic surfactant; increased temperature for a nonionic surfactant), the order of transition is Type I to III to II. When the volume of water equals the volume of oil in the middle phase, interfacial

tensions between phases are at a minimum and this is known as the optimum condition. In part I of this series (1), we showed that increasing salinity for a SDS system of ODCB and water caused a Winsor Type I to III transition. However, surfactant solubility problems did not permit the optimal salinity from being reached. The traditional solution to solve these solubility problems, adding low–molecular weight alcohol to the system, is unacceptable in our wastewater application since this alcohol will be present at a substantial concentration in emitted water. So, in this work, a less hydrophilic cosurfactant (NP(EO)$_{10}$) was added to SDS to attain optimum conditions.

2.4.1.1 Effect of single surfactant concentration on microemulsion formation

The effect of either SDS or NP(EO)$_{10}$ concentration on microemulsion formation in single surfactant system without added NaCl is illustrated in Figures 2.2 and 2.3, respectively. When SDS concentration increases from 1% to 9% by weight, the volume of water phase slightly increases and that of oil phase slightly decreases because SDS solubilizes more oil into the water phase. The system is a Winsor Type I microemulsion. In the case of NP(EO)$_{10}$, a Winsor Type III microemulsion is observed with a maximum relative volume of the middle phase microemulsion at 7% by weight of surfactant. At this composition, substantial amounts of both oil and water are solubilized (0.165 mL of oil and 0.335 mL of water). It is well known that the minimum interfacial tension (sum of water / microemulsion and oil / microemulsion interfacial tensions) corresponds to equal volumes of oil and water solubilized in the middle phase microemulsion; furthermore, the higher the volume of either oil or water solubilized at this "optimal condition", the lower the interfacial tension is observed (25, 26). In part I of this series (1), it was found to be impossible to obtain conditions where a significant amount of water was solubilized in the middle phase microemulsion for SDS–only system because liquid crystals were formed at high surfactant concentrations.

2.4.1.2 Effect of mixed surfactant composition on microemulsion formation

The effect of mixed surfactant composition without added NaCl on phase behavior is illustrated in Figures 2.4 – 2.8. The optimum volume fraction of the middle phase microemulsion is at X_{SDS} of 0.2 to 0.4, indicating the synergism of surfactant mixtures compared to either anionic or nonionic surfactant alone. At total surfactant concentrations of 1 and 3% by weight, an insignificant amount of water is solubilized in the middle phase microemulsion, as was observed with SDS–only systems (1). Therefore, optimal conditions (minimum interfacial tensions) are not achieved. However, at a surfactant concentration of 5% by weight and X_{SDS} at 0.5, approximately equal volume of oil and water are solubilized in the microemulsion phase, due to a proper balance between hydrophobicity and hydrophilicity of the surfactant mixture. When total surfactant concentration is equal to or greater than 7% by weight, liquid crystals appear. Consequently, 5% by weight of total surfactant concentration was chosen to study in froth flotation experiment because it has a high volume fraction of the middle phase without liquid crystals, which corresponds to minimum interfacial tensions.

Phase scans like that in Figures 2.4 – 2.8 have been also carried out for this system at added NaCl concentration up to 2.5% by weight. Since flotations were only performed without added NaCl, these phase volume plots are not reported here but available in a thesis (27).

2.4.1.3 Effect of NaCl on microemulsion formation of mixed surfactant system

For anionic or anionic/nonionic surfactant systems, added electrolyte can dramatically affect microemulsion phase behavior. From Figure 2.9 at X_{SDS} of 0.4 and a surfactant concentration of 1% by weight, an increase in NaCl concentration leads to a Winsor Type III to Winsor Type II transition. This is because the surfactant solubility decreases for the mixed anionic/nonionic system (just like an anionic–only system), so the transition from the Type III to II occurs at lower values of X_{SDS} at higher NaCl concentrations. From the result, the optimum salinity is 1.5% by weight which gives the highest volume fraction of the middle phase and equal volume of both

oil and water solubilized. In wastewater clean–up, the added salt will largely end up in the excess water phase which would be emitted to the environment and could make that water exceed total allowable dissolved solid content. All phase behaviors of the system with added NaCl between 0% and 2.5% by weight are available in a thesis (27). The added salt gives the ability to adjust the phase behavior of a system. Also, the effect of salinity is important to know because the natural salinity of a waste stream can vary widely.

2.4.2 Froth Flotation Results

Froth flotation studies were conducted at 5% by weight of total surfactant concentration since the system has the highest solubilization parameters of both oil and water in the absence of added salt. In this work, the effects of weight fraction of SDS (X_{SDS}) on oil removal, on surfactant removal, and on foam composition are shown in Figures 2.10 – 2.14. As X_{SDS} increases, the tendency for foam production increases, perhaps because the repulsive force between the negatively charged head groups of SDS can electrostatically stabilize the film lamellae of the foam. Therefore, the efficiency of oil and of surfactant removal increases when X_{SDS} increases. At 5% by weight of total surfactant concentration, the optimum X_{SDS} from the phase behavior is 0.5. However, the highest oil and surfactant removal was achieved at $X_{SDS} = 0.8$ which is still in Winsor Type III microemulsion region as shown in Figures 2.10 – 2.12. Therefore, the efficiency of froth flotation depends on not only equilibrium microemulsion phase behavior with the maximum solubilization parameters but also other effects such as air dispersion, froth stability, and nonequilibrium effects. An increase in aeration time directly enhances both surfactant and ODCB removal since more foam is produced with time. At an aeration time of 120 minutes, almost 100% removal of ODCB and $NP(EO)_{10}$ is observed while the removal of SDS is around 80% as shown in Figure 2.13. This is probably because SDS is more hydrophilic than ODCB and $NP(EO)_{10}$, so SDS adsorbs less at the air bubble – water interface than the $NP(EO)_{10}$ does. It is particularly interesting that at higher concentrations of SDS, the additional SDS not only induces increased oil flotation, but also induces increased $NP(EO)_{10}$ and SDS removal. This is probably because foam stability increases when SDS concentration increases, leading to an increase in amount of surfactant associated

with the froth. From Figures 2.10 – 2.12, removal of ODCB, SDS, and NP(EO)$_{10}$ is higher in the Winsor Type III regime than in the Type I regime (as in previous results (1)), although not at the optimum conditions from equilibrium phase diagrams. So, ultra-low oil/water interfacial tensions are important for good oil removal, although not the only factor. We will quantify foam stability and equilibration time effects in future work.

High oil removal efficiency is a necessity for an effective flotation operation, but it is not the sole factor to be considered. If oil and water are present in the froth in the same proportions as in the solution, no selectivity is present and no separation of oil and water is occurring. Figure 2.14 shows the relative fractions of oil and of water in the foam phase as a function of X$_{SDS}$ at a total surfactant concentration of 5% by weight and at 120 minutes aeration time. When in the Winsor Type III microemulsion regime, more oil than water was indeed carried over with the foam. For example, at X$_{SDS}$ = 0.8 (at which composition, fractional oil recovery is the highest observed), the weight ratio of ODCB : water is 2:1 in the collapsed foam. For all these experiments, the initial oil : water volume ratio was 1:1, corresponding to a very high oil loading in a wastewater. Selectivity may be improved further at lower oil loadings. It is very important to note that the separation efficiency of oil is worse when a Winsor Type I microemulsion is formed, and which even shows inverted results with more water than oil being carried overhead when a Type I system is present. These results support further the advantages of formulating the system to be in Winsor Type III microemulsion regime.

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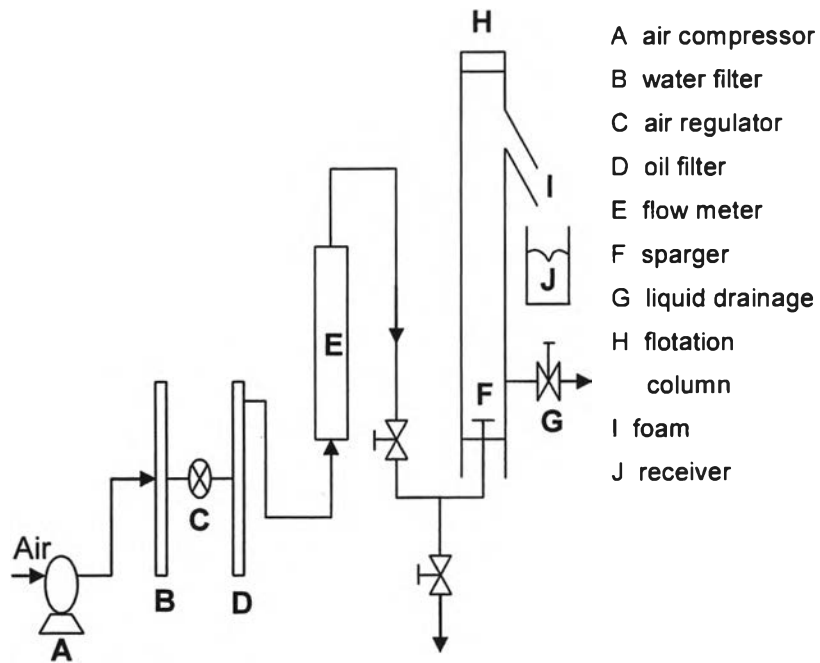
Figure 2.1 Schematic diagram of the froth flotation system
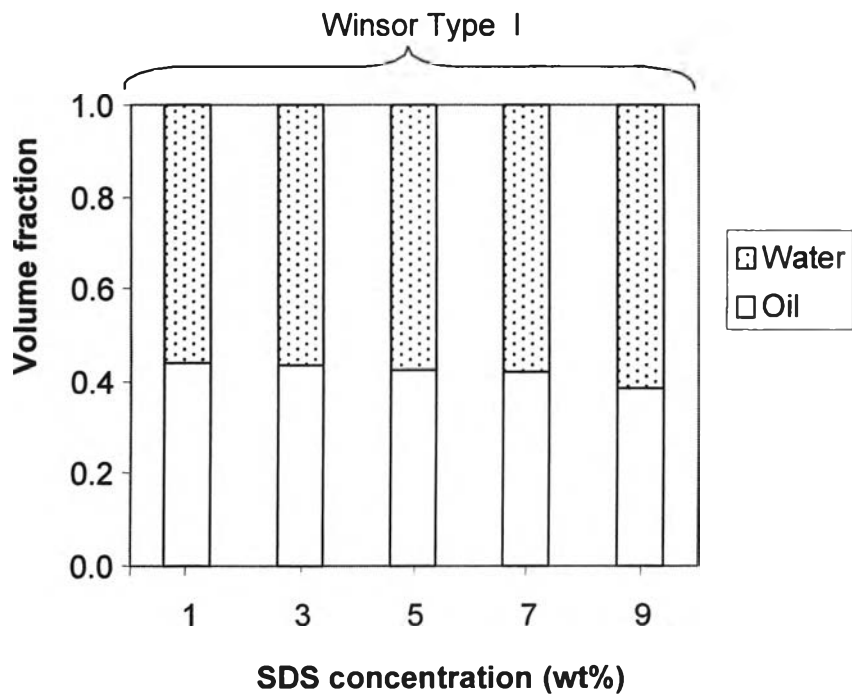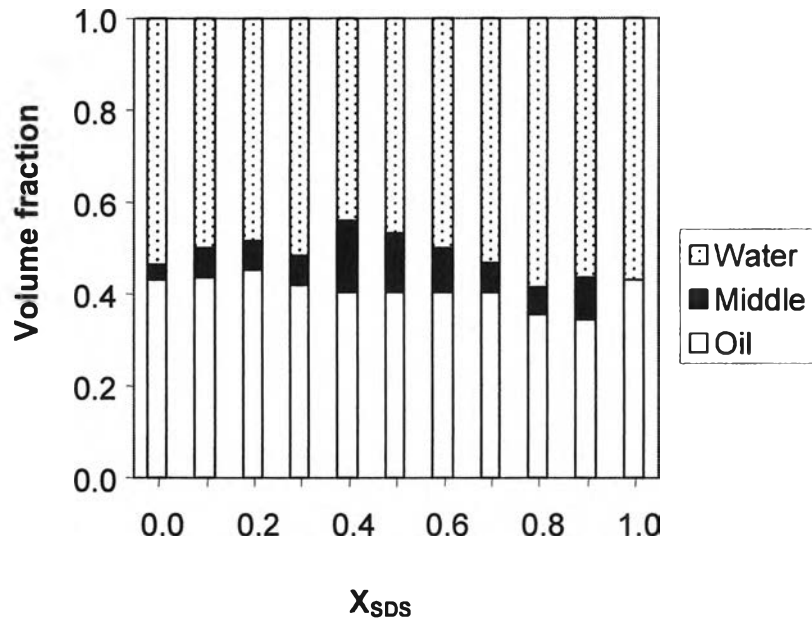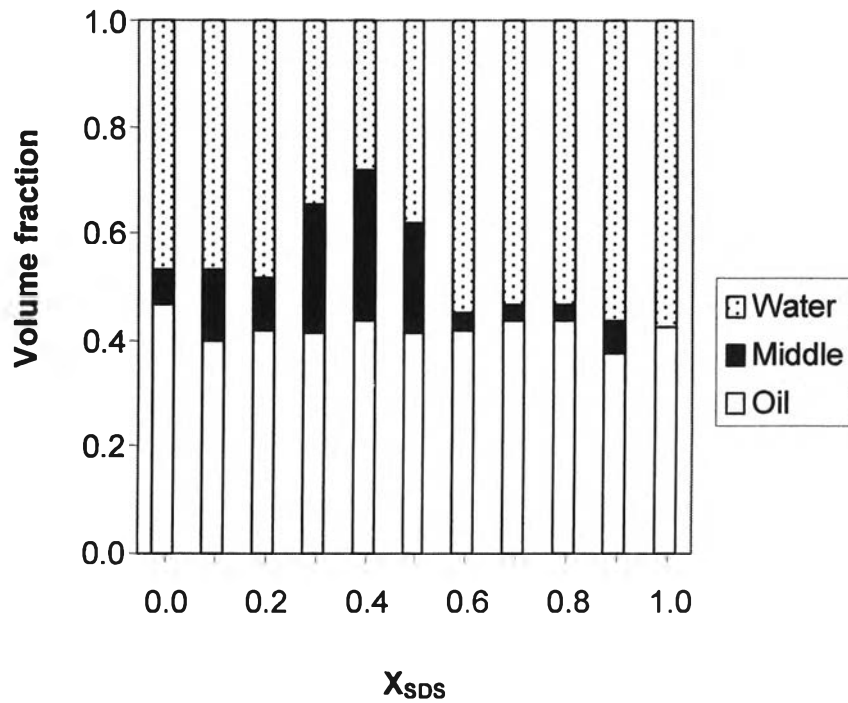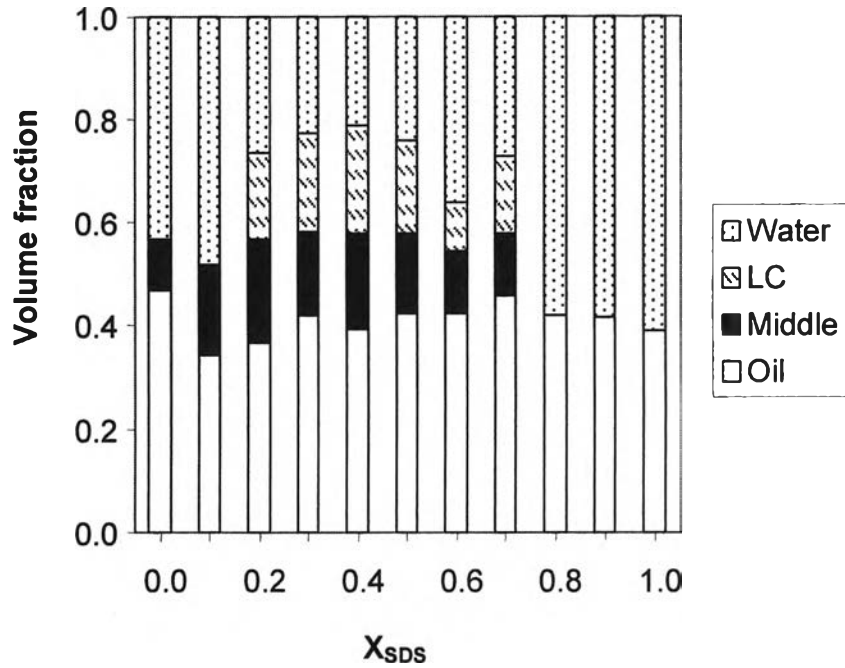

Figure 2.2 Volume fractions of water, middle, and oil phases at different SDS
concentrations, with initial oil:water ratio of 1:1, and w/o NaCl

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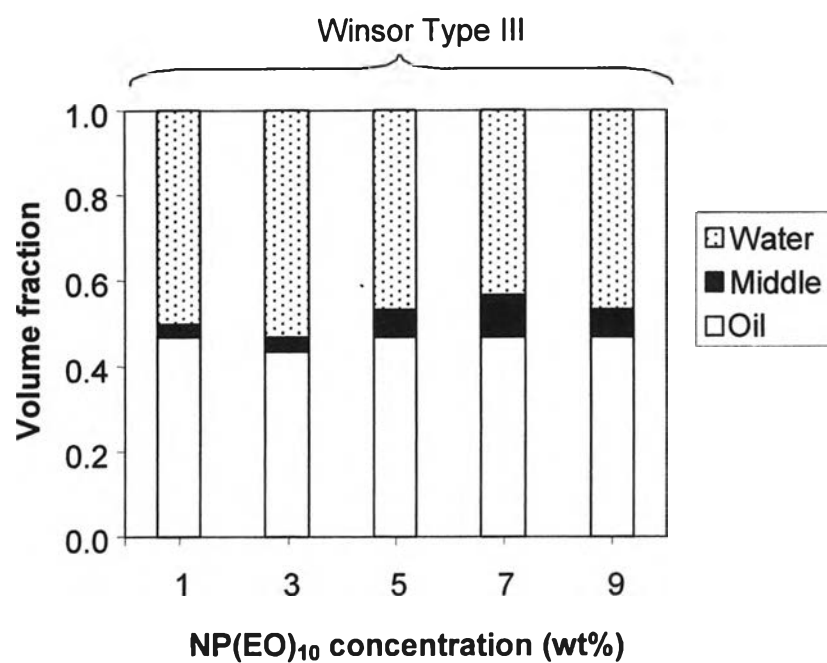
Figure 2.3 Volume fractions of water, middle, and oil phases at different

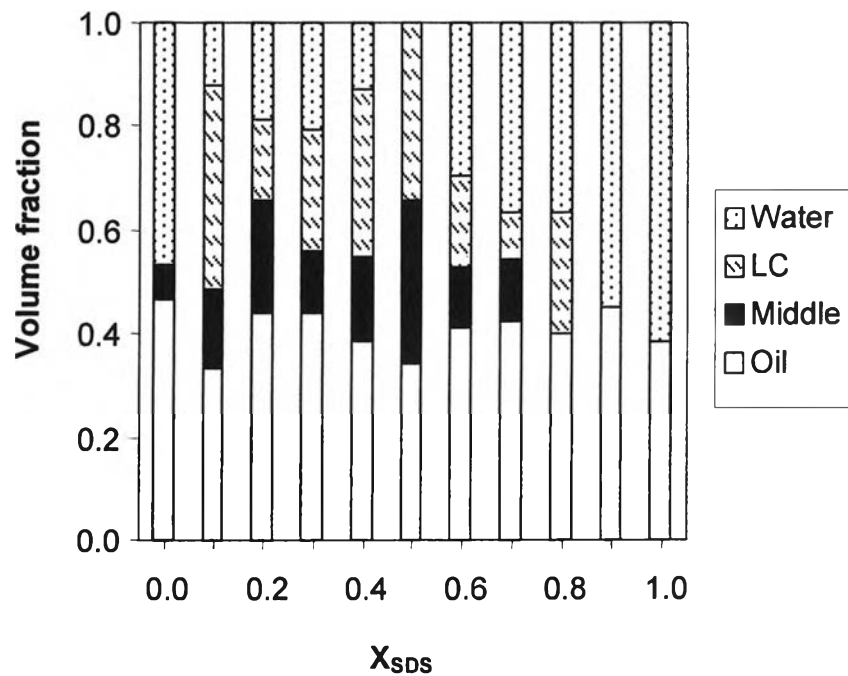NP(EO)$_{10}$ concentrations, with initial oil:water ratio of 1:1, and w/o NaCl
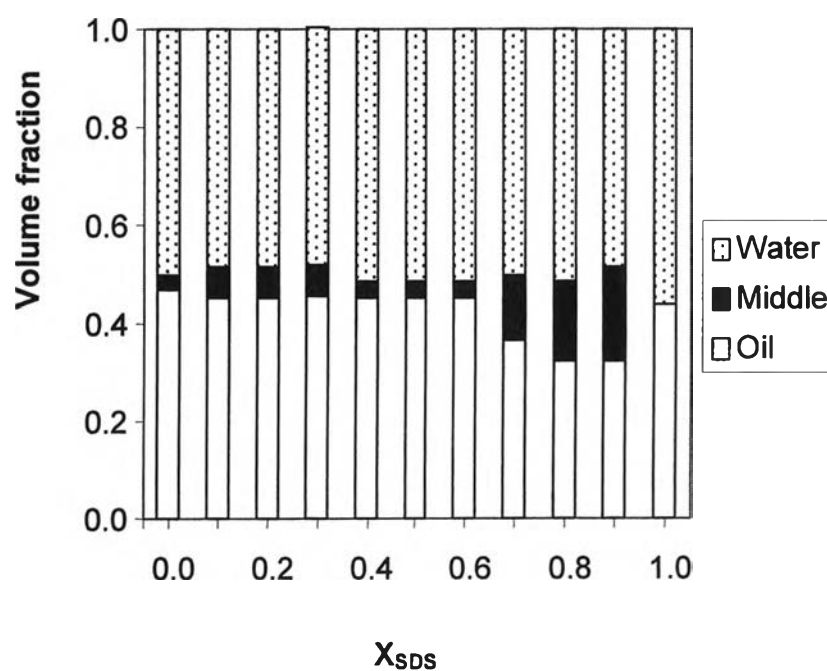
Figure 2.4 Volume fractions of water, middle, and oil phases at 1% wt total

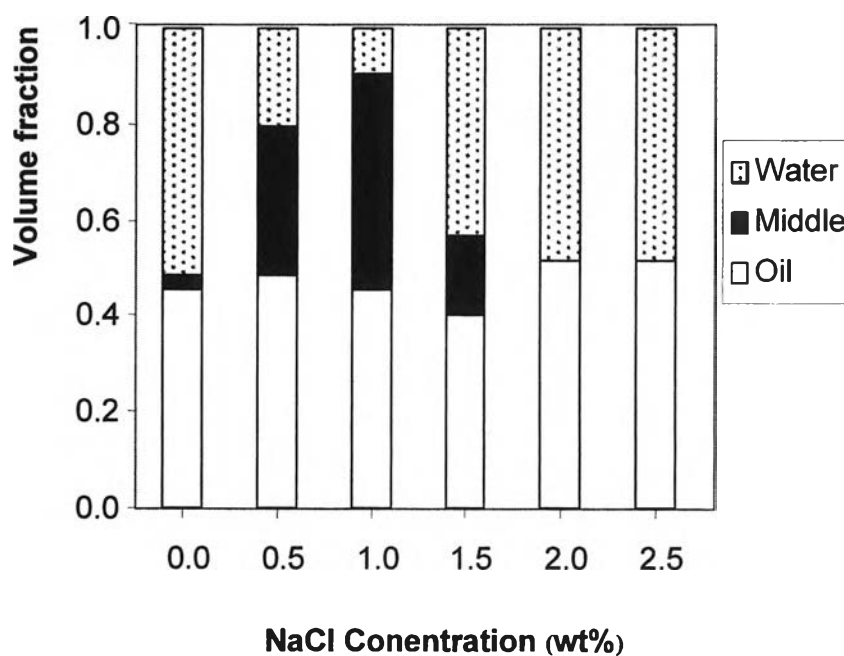surfactant concentration, with initial oil:water ratio of 1:1, and w/o NaCl

Figure 2.5 Volume fractions of water, middle, and oil phases at 3% wt total
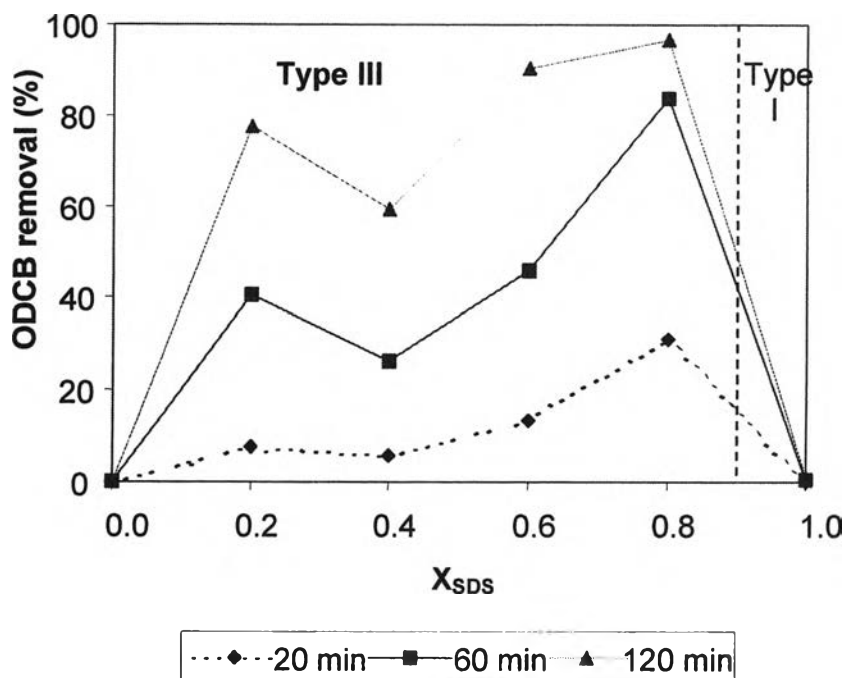surfactant concentration, with initial oil:water ratio of 1:1, and w/o NaCl

Figure 2.6 Volume fractions of water, middle, and oil phases at 5% wt total surfactant
concentration, with initial oil:water ratio of 1:1, and w/o NaCl

Figure 2.7 Volume fractions of water, middle, and oil phases at 7% wt total
surfactant concentration, with initial oil:water ratio of 1:1, and w/o NaCl

Figure 2.8 Volume fractions of water, middle, and oil phases at 9% wt total
surfactant concentration, with initial oil:water ratio of 1:1, and w/o NaCl

Figure 2.9 Volume fractions of water, middle, and oil phases at 1% wt total
surfactant concentration, X_{SDS} of 0.4, and initial oil:water ratio of 1:1

Figure 2.10 Dynamic removal efficiency of ODCB as a function of X_{SDS} at 5% wt total
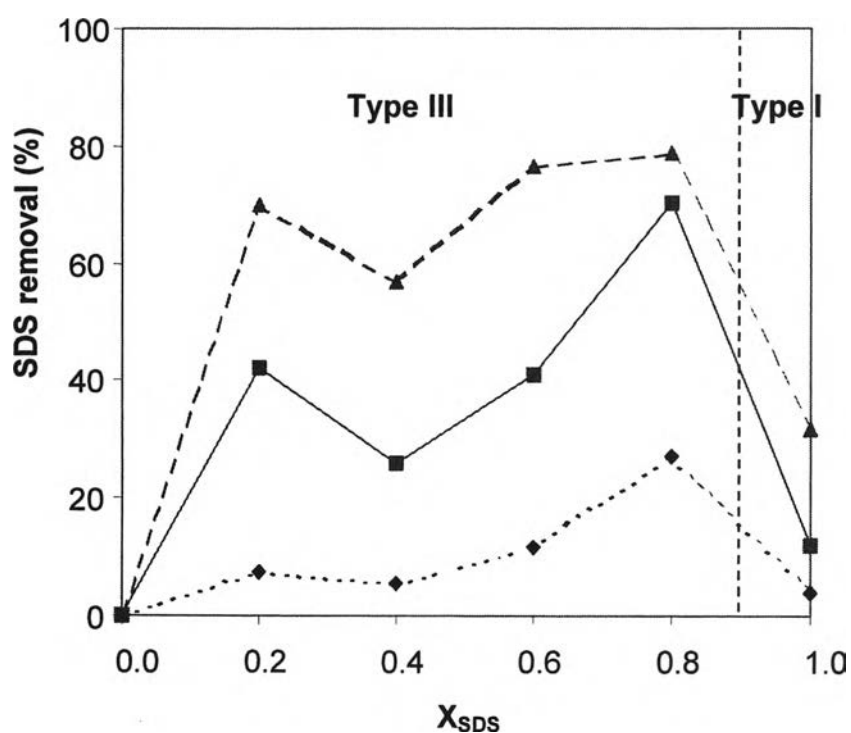surfactant concentration with initial oil:water ratio of 1:1, and w/o NaCl

Figure 2.11 Dynamic removal efficiency of SDS as a function of X$_{SDS}$ at 5% wt total
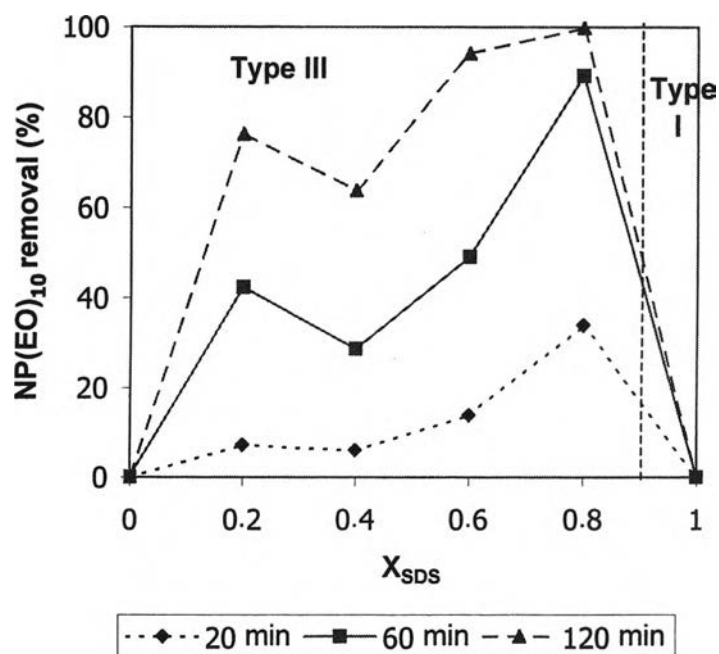surfactant concentration with initial oil:water ratio of 1:1, and w/o NaCl

Figure 2.12 Dynamic removal efficiency of NP(EO)$_{10}$ as a function of X$_{SDS}$ at 5% wt
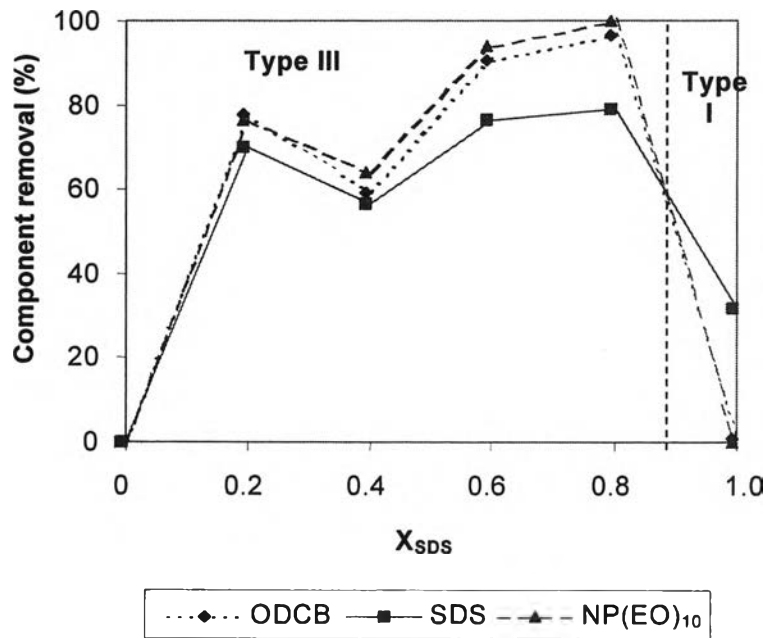total surfactant concentration with initial oil:water ratio of 1:1, and w/o NaCl

Figure 2.13 Comparison between removal efficiency of ODCB, SDS, and NP(EO)$_{10}$ at 5% wt total surfactant concentration with initial oil:water volume ratio of 1:1, w/o NaCl, and 120 min aeration time
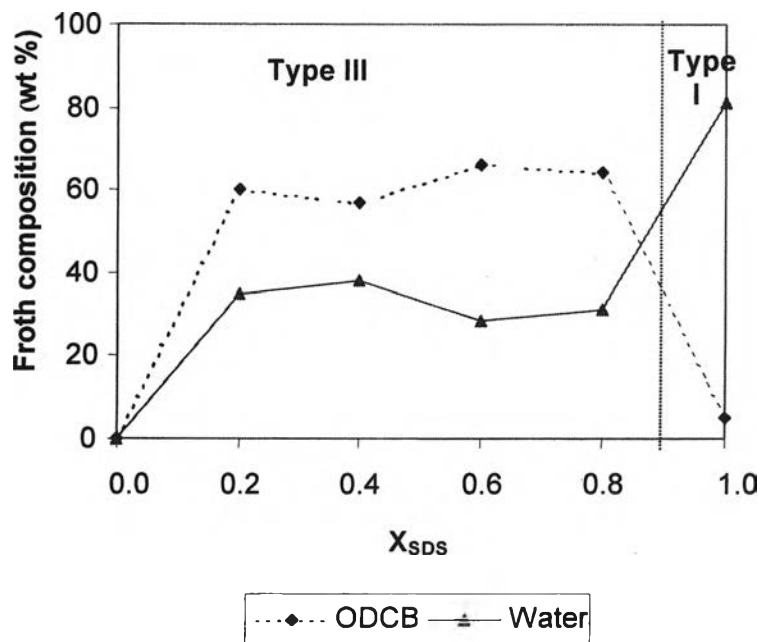

Figure 2.14 ODCB and water content in collapsed foam fraction as a function of X$_{SDS}$ at 5% wt total surfactant concentration with initial oil:water volume ratio of 1:1, w/o NaCl, and 120 min aeration time