

CHAPTER IV

**ETHYLBENZENE REMOVAL BY FROTH FLOTATION UNDER
CONDITIONS OF MIDDLE-PHASE MICROEMULSION FORMATION I:
INTERFACIAL TENSION, FOAMABILITY, AND FOAM STABILITY**

4.1 Abstract

The objective of this study was to investigate the relationship of the froth flotation performance in removal of emulsified ethylbenzene in water with microemulsion formation and with foam formation characteristics. The surfactant used was dihexyl sulfosuccinate (Aerosol MA or AMA) which can form microemulsions with ethylbenzene. The systems studied were designed to form Winsor Type III microemulsions with ethylbenzene which generally correspond to ultra-low interfacial tensions between oil and water phases. By varying the surfactant concentration, NaCl concentration, and oil to water ratio, it was found that the lowest interfacial tension was obtained at 1 wt% AMA and 3 wt% NaCl, while the interfacial tension was not substantially influenced by the oil to water ratio. The highest oil removal was achieved in froth flotation with 0.3 wt% AMA and 3 wt% NaCl. No separation was experienced when the NaCl concentration exceeded 4 wt% due to the poor foamability of the froth formed under these conditions. Therefore, these results demonstrate that both interfacial tension and foam characteristics influence the efficiency of oil removal in the froth flotation process.

4.2 Introduction

Flotation processes are useful for separation of various species such as ore, emulsified oil, and various products that are extracted from an aqueous medium, and can be used for product recovery or for wastewater treatment (1-3). There are two main types of froth flotation: dissolved air flotation; and induced air flotation (4). The dissolved and induced air flotation processes have been used for many years in the separation of suspended solids, oils, greases, and fibers from wastewaters (5). In induced air flotation, air is introduced at the bottom of a froth flotation column. Emulsified oils can co-adsorb at the air-water interface and be carried over the top of the column.

Surfactants play an important role in the froth flotation by inducing foam formation, reducing the coalescence rate of bubbles within the liquid phase, and increasing the retention time of bubbles in the column (6). Two advantages of froth flotation over other conventional separation processes include simplicity of operation and low operational cost. As a result, substantial research has been carried out to develop froth flotation as a waste treatment process (7-16). In this study, the froth flotation technique was implemented in batch mode to remove emulsified ethylbenzene from water; the ethylbenzene concentration is far above its solubility in water.

Ethylbenzene is a colorless organic liquid with a sweet and gasoline-like odor. It is widely used as a solvent and also found in a variety of products in the chemical industry. The USA and Canada limit the amount of ethylbenzene in wastewaters to between 2.4 and 5 $\mu\text{g/L}$ (17). Ethylbenzene may be found in industrial discharges or leakage of underground storage tanks. The relevant routes for ethylbenzene exposure are through oral intake and inhalation, and it can damage the liver of humans.

From our previous work (6, 18, 19), the relationship between the type of microemulsion and the efficiency of oil removal has been investigated, and the maximum oil removal was found to correspond to the formation of a Winsor Type III microemulsion. However, the mechanism of this relationship has not been fully understood. It is known that a Winsor Type III microemulsion has unique characteristics of ultra-low interfacial tension and high solubilization. Chavadej *et al.* (19) reported that most of the oil removed in the froth flotation column came from the excess oil phase rather than from the middle phase microemulsion always present in Type III systems. That work also showed that the relationship between high oil removal and ultra-low interfacial tension in a Winsor Type III microemulsion – the presence of a middle phase microemulsion is coincidental to oil removal.

Depending upon the application, the presence of foam can be either undesirable or preferable. Foam can cause operational problems in many processes such as sewage treatment, coating applications, and crude oil processing (20). However, in some processes, such as fire fighting and froth flotation, foams are critical to successful process performance. Foam is simply produced by introducing air beneath the liquid surface (21). The two-sided aqueous films separating discrete gas bubbles are called lamellae. As liquid drains out of the lamellae, the foam is eventually destroyed. As a result, foam is unstable thermodynamically (20, 21).

Foams can be classified into three classes: unstable foams; metastable foams; and solid foams. Unstable foams remain for a very short time and collapse rapidly due to the thinning of the lamellae by pressure gradient between the lamellae and the plateau triangle regions and water drainage by gravitational forces (20). Metastable foams possess a persistence or degree of stability ranging from a few seconds to months. Added surfactants stabilize foams by retarding the loss or drainage of liquid from the lamellae. In addition, the foams produced can be disrupted by many factors including dust particles, vibration, evaporation, and pressure change (20). The last class of foams is solid foams which are mechanically rigid due to an irreversible chemical process during foam formation (20). Sometimes foams only refer to the situation where the lamellae are liquid, so “solid foams” would be considered as sponges. In this study, we are interested in metastable foams.

Carre *et al.* (22) suggested that one of the key criteria for the success of a flotation operation is to produce a stable froth. Apart from the ultra-low interfacial tension in the Winsor Type III region, it is hypothesized that the following factors influence oil removal in the froth flotation process: the amount of oil attached to the froth, foamability (the rate of foam formation), and foam stability. The objective of this work is to probe the importance of oil/water interfacial tensions, foamability and foam stability in froth flotation of ethylbenzene.

4.3 Experimental Section

4.3.1 Materials

Ethylbenzene (98% purity), obtained from Fluka Company was used as the model oil in this study. The surfactants studied were as follows: di-1, 3-dimethylbutyl sulfosuccinate (Aerosol MA or AMA) with 80% in aqueous solution containing a maximum of 5% isopropanol in the solution supplied by CYTEX corporation, Los Angeles, USA; sodium bis-2-ethylhexylsulfosuccinate (Aerosol-OT or AOT) with 98% purity from Fluka Company; and mono- and di-hexadecyl diphenyloxide disulfonate sodium salt (Dowfax 8390 or DPDS) in 36% solution from Dow Company, Midland, Michigan. Analytical purity grade sodium chloride (NaCl) from Aldrich Chemical Company Inc. was used as electrolyte in this work. All

Chemicals were used as received without further purification. Deionized water was used to prepare all aqueous solutions.

4.3.2 Methodology

There were three experimental parts in this research work. The first part was to study microemulsion phase behavior of the aqueous solutions with ethylbenzene. The second part was to investigate foamability and foam stability. The third part of this work was to study the efficiency of froth flotation. In all experiments, the surfactant and electrolyte concentrations are expressed in weight percentage (wt%) per volume of the aqueous solution comprising water, surfactant, and salt (not including oil).

To investigate the phase behavior of microemulsions, 5 mL of homogeneous aqueous solution, prepared at various surfactant and NaCl concentrations, was mixed with 5 mL of ethylbenzene in a vial and sealed with a screw cap. The vials were shaken every day for 3 days, and then allowed to equilibrate at a constant temperature of 30°C in a water bath for 1 month to reach equilibrium, which was verified by the invariant height of each phase. The interfacial tensions between equilibrated excess oil and excess water phases were measured by a spinning drop tensiometer (SITE 04, Krüss GmbH, Hamburg).

A schematic diagram of the froth flotation unit used in this work is shown in Figure 4.1. A glass cylindrical column with 5 cm internal diameter and 120 cm height was used as the froth flotation column. A 250 mL sample containing a given surfactant concentration and an oil to water ratio of 1:1 was transferred to the column. Filtered air was introduced at the bottom of the column through the solution at a constant flowrate of 100 mL/min until the foam height in the column was constant. The maximum foam height was then measured. Then the filtered air was stopped, and the time required for the foam volume to collapse to one half of the maximum height was recorded to quantify foam stability.

In order to better understand the froth flotation process, froth flotation experiment was conducted in the same column. A 750 mL sample with an initial oil:water ratio of 1:1 and various surfactant and NaCl concentrations was prepared, equilibrated at 30°C for 1 month in the incubator, and finally transferred to the froth flotation column. Filtered air was introduced at the bottom of the column at a constant flow rate of 300 mL/min through a sintered glass disk having pore size diameters of

about 16 – 40 μm . The foam collected in the receiver (J in Figure 4.1) over a period of time was broken by freezing with subsequent ethylbenzene analysis. Moreover, the solution in the column was sampled at the same time interval as foam collected for analysis of ethylbenzene and surfactant concentrations. All experiments were stopped when solution surfactant concentrations became low enough that no more foam came overhead from the column. All experiments of froth flotation operation, foamability, and foam stability were conducted at a room temperature of about 25 – 27° C.

4.4 Results and Discussion

In this study, wt% is based upon the aqueous system consisting of water, salt, and surfactant. Foamability is defined as the ratio of maximum foam height to initial solution height whereas foam stability ($t_{1/2}$) is the time required for the foam to collapse to 50% of the maximum height.

4.4.1 Phase Behavior

To form Winsor Type III microemulsions with ethylbenzene, multiple surfactant systems (e.g. AMA, AOT, and the mixture of AOT and DPDS) were tested systematically. For conditions used in this study, only AMA or mixtures of AOT and DPDS could form middle phase microemulsions (Winsor Type III) with ethylbenzene. In operating froth flotation with the mixture of AOT and DPDS, the foam generated was not able to reach the overflow outlet of the column, indicating poor foamability and foam stability. Consequently, only results from the AMA system are reported here. The phase behavior of ethylbenzene with the mixture of AOT and DPDS are not shown here but are available elsewhere (23).

The effect of AMA surfactant concentration on interfacial tension (IFT) is shown in Figure 4.2. Winsor Type III microemulsions are formed at AMA concentrations above 0.3 wt% as indicated by the presence of 3 phases at equilibrium: excess water phase; excess oil phase; and a middle phase. The shape of the IFT curves in Figure 4.2 is typical of systems that transition from micellar solutions to optimum microemulsions (24). The surfactant concentration where the first microemulsion “droplet” is formed and ultra-low IFT is reached is called a critical microemulsion concentration ($C_{\mu C}$) (24); in this case, the $C_{\mu C}$ is 0.3 wt% AMA. The $C_{\mu C}$ also

corresponds to the Type I to Type III transition concentration. At an AMA concentration of 1 wt%, the minimum observed value of IFT was obtained. At this IFT minimum, the surfactant is balanced between water and oil solubility and can form a bicontinuous structure in the middle phase, leading to high oil and water solubilization in this phase corresponding to the minimum IFT value (25). However, as shown in Figure 4.2, the IFT increases slightly when the AMA concentration is greater than 1 wt%, a trend that has been reported before and seems to be linked to strong surfactant – surfactant interactions (26).

The lowest AMA concentration which can form a Winsor Type III microemulsion ($C_{\mu C} = 0.3$ wt%) was selected to determine the effect of salinity on IFT. The effect of NaCl concentration on IFT and other process parameters are shown in Figure 4.3. The minimum IFT is observed at approximately 3 wt% NaCl: this is known as the optimum salinity. The changes of IFT versus electrolyte concentration in Figure 4.3 are typical of Winsor Type I – III – II transition. Systems with 2 wt% or less NaCl show Type I microemulsion systems (oil – swollen micelles) and systems with 4 wt% NaCl or more show Type II systems (reverse micelles); at intermediate salinity values, a Type III system is observed.

4.4.2 Foam Formation and Foam Stability

In froth flotation, both the rate of foam formation and the stability of the generated foam are extremely crucial for good separation. Figure 4.2 shows that increasing the AMA concentration decreases foamability. This may be due to enhanced viscosity of the continuous phase which is an important factor in coalescence of bubbles (27). At a low surfactant concentration, the solution viscosity is low. Therefore, the diffusion rate of surfactant to the newly created interface, which occurs during the bubble generation, is fast enough to be adsorbed at the bubble interface. With the absence of further diffusion of surfactant from bulk solution to the interface, the action of the interface elasticity can restore the bubble size by the Gibbs-Marangoni effect (28). This result is consistent with the explanation of Marinhardt (29) that foam of low viscosity is claimed to propagate more easily than foam of high viscosity.

In typical foam situations, a surfactant accumulates at the oil/water interface, and as the water drains, it tends to form a metastable lamellae phase which imposes a near zero curvature to the surfactant molecules. The equilibrium state is a

highly curved micellar solution. When the surfactant is formulated to form a Type III microemulsion system, the equilibrium net curvature of the surfactant film approaches zero (30). It is reasonable to imagine that when foam is produced in these microemulsion systems, instead of forming a lamellae phase, a film of near zero curvature is formed. The hypothesis that “microemulsion films” are formed instead of “lamellae films” is corroborated by the fact that not only the surfactant, but also the oil is removed during the froth flotation process, as indicated in Figures 4.2 and 4.3.

While microemulsion films can satisfy the zero curvature conditions for the surfactant molecules, they are still under the influence of “drainage” effects imposed by the Laplace pressure, and because of that they are still metastable phases, but with longer half time than curved microemulsion systems (Type I & II).

4.4.3 Froth Flotation

To achieve good separation as well as an acceptable degree of treatment in running a froth flotation process, treated wastewater has to contain very low concentrations of both oil and surfactant. Hence, the overhead froth should have high concentrations of both oil and surfactant. In this study, parameters used to indicate the performance of froth flotation are oil removal, surfactant removal, and enrichment ratio of oil. An enrichment ratio of oil is defined as a ratio of concentration of oil in the collapsed froth to a concentration of oil in the initial solution. The enrichment ratio of oil must be greater than unity if separation has been achieved.

4.4.3.1 Maximum removal of ethylbenzene and surfactant

Figure 4.2 shows the effects of surfactant concentration on total cumulative ethylbenzene removal, total cumulative AMA removal, initial IFT, and foamability. Since the surfactant concentration progressively decreases with time during the froth flotation and foamability experiments, the “initial” AMA concentration is used to characterize those experiments. The total cumulative AMA removal and total cumulative ethylbenzene removal are calculated based on the end of the flotation operation when the froth no longer flows overhead. The AMA removal is highest (nearly 100%) at the $C_{\mu C}$ level of AMA. However, the ethylbenzene removal decreases monotonically as the AMA concentration further increases, a trend that can be attributed to foamability.

Figure 4.3 shows the effects of initial NaCl concentration on total cumulative ethylbenzene removal, total cumulative AMA removal, initial IFT, foamability, and foam stability. All flotation experiments were carried out at initial conditions in the Winsor Type III region. In this case, unlike Figure 4.2, the highest total cumulative ethylbenzene removal corresponded to not only the minimum IFT, but also to maxima in both foamability and foam stability. At 4% NaCl and above, no recovery of ethylbenzene or AMA occurred since the froth never reached the overhead outlet. This salinity also corresponds to a sharp decrease in foamability and foam stability even though the IFT was still quite low. Unlike ethylbenzene, total cumulative AMA removal is nearly constant with increasing salinity until the 4 wt% NaCl concentration is reached where froth stability is catastrophically low.

4.4.3.2 Enrichment ratios and dynamics of flotation

Figure 4.4 shows the ethylbenzene enrichment ratio as a function of AMA concentration at 2 wt% and 3 wt% salinities. At an AMA concentration above 2 wt%, an ethylbenzene enrichment ratio of less than unity is observed – the oil is depleted instead of enriched in the froth. The separation is worthless in this region despite total cumulative oil removal of nearly 50% at the AMA concentration of 3 wt%. The highest enrichment ratio occurs at the AMA concentration corresponding to the minimum IFT (i.e. at lower AMA concentrations). The salinity with lower IFT and higher foamability and foam stability has slightly higher ethylbenzene enrichment ratio (1.76 at 3 wt% NaCl versus 1.34 at 2 wt% NaCl) before froth collapse prevented a separation at still higher NaCl concentrations. As the initial AMA concentration increases, the foam wetness and foam production rate reach a minimum (Figure 4.5) while ethylbenzene enrichment ratio tends to decrease, as shown in Figure 4.4.

Dynamic froth flotation results (effect of operating time in the batch experiment) are shown in Figures 4.6 – 4.10. Figures 4.6 and 4.7 show the effect of initial AMA concentration on dynamic ethylbenzene removal and dynamic AMA removal, respectively. The system having 0.3 wt% AMA concentration seems to be a very good condition for ethylbenzene removal because nearly 100% of ethylbenzene is removed within the shortest time due to the highest foam production rate. Even though at the beginning of operation, the ethylbenzene removal from a 2 wt% AMA concentration solution is higher than that from a 1 wt% AMA concentration solution, the

total cumulative ethylbenzene removal from the latter is higher than that from the former because the rate of ethylbenzene removal of the latter increases with time (Figure 4.8). The AMA removal shows this same trend except at 2 wt% initial AMA where removal is lowest. This is because the rate of AMA removal at 2 wt% AMA, as shown in Figure 4.9, is not high enough in the first 3 hours. This may be due to the fact that the foam production rate is lowest for this system. From Figures 4.10 – 4.12, the ethylbenzene removal, the AMA removal, and ethylbenzene enrichment ratio at 3 wt% NaCl is higher than that at 2 wt% NaCl. This is because the rates of both ethylbenzene and AMA removal at 3 wt% NaCl correspond to the foam production rate (Figure 4.5) which is much higher than those rates for 2 wt% salt as shown in Figure 4.13 – 4.14.

4.5 Conclusions

In our previous froth flotation work with ortho-dichlorobenzene (18), we concluded that oil removal increases with decreasing IFT. For this work with ethylbenzene, we have studied foam characteristics in more detail and have found that foam formation and stability can also have profound effects on oil removal. There are conditions where the frothing is so unfavorable that no froth comes overhead and no separation occurs even though IFT values can be ultralow. An important conclusion of this work is that both low IFT and good frothing are necessary for efficient separation. In part II of this series, we examine the effect of oil to water ratio, air flow rate, and equilibration time on the froth flotation performance.

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4.7 References

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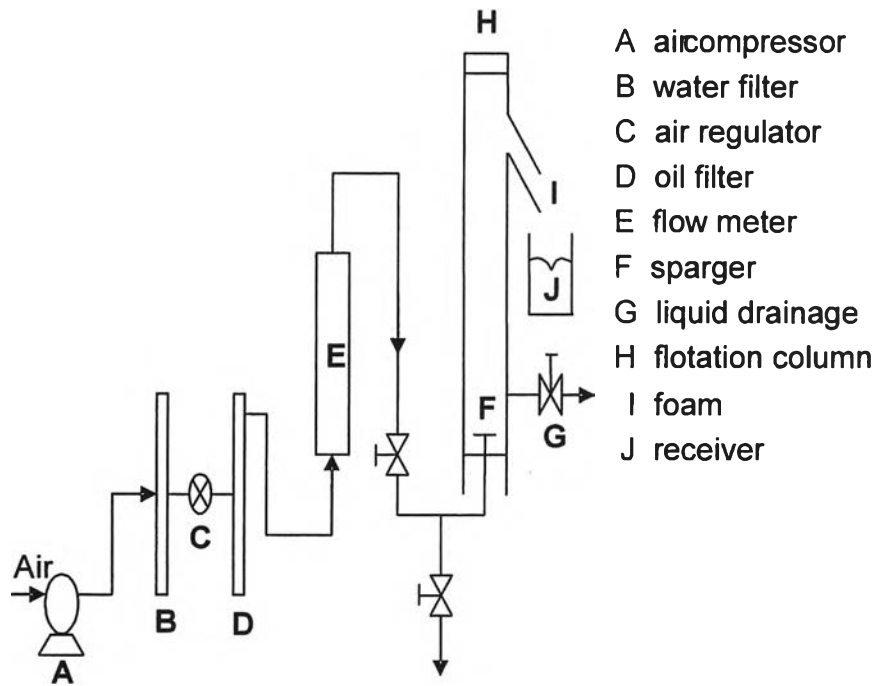


Figure 4.1 Schematic diagram of the froth flotation apparatus

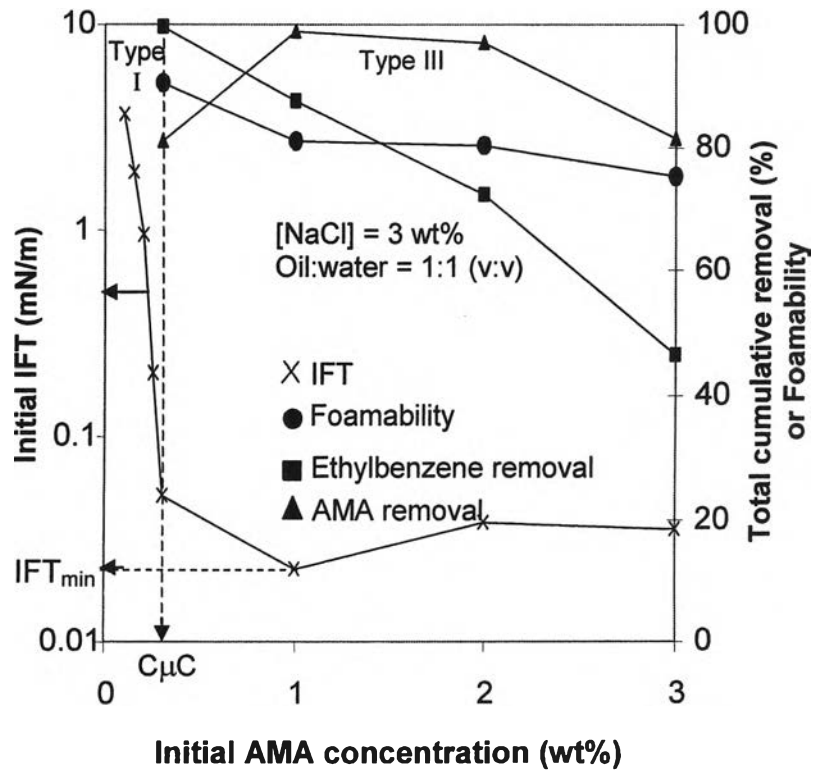


Figure 4.2 Effect of initial AMA concentration on process parameters

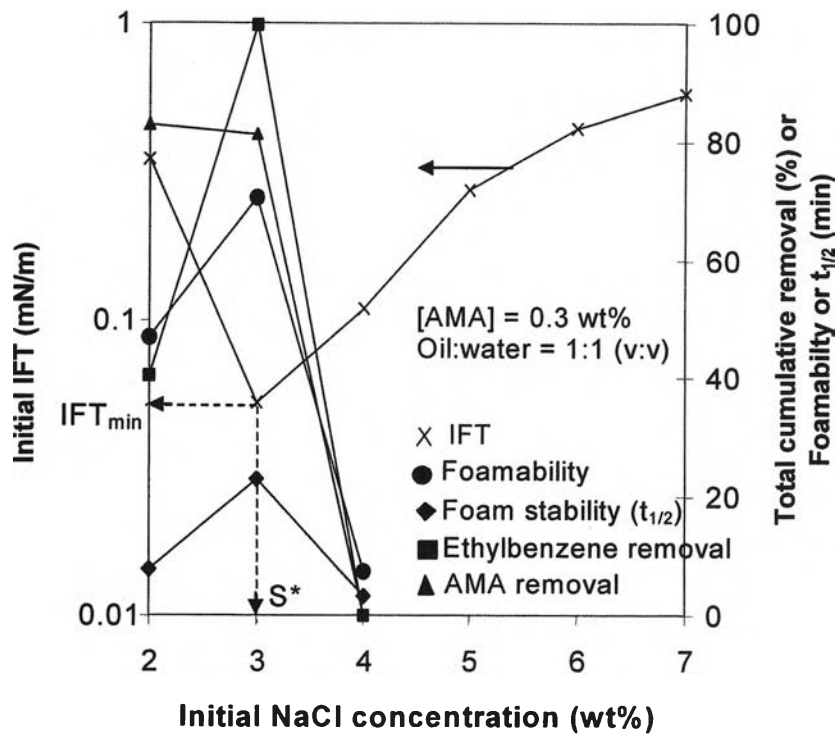


Figure 4.3 Effect of initial NaCl concentration on process parameters (S^* = optimal salinity)

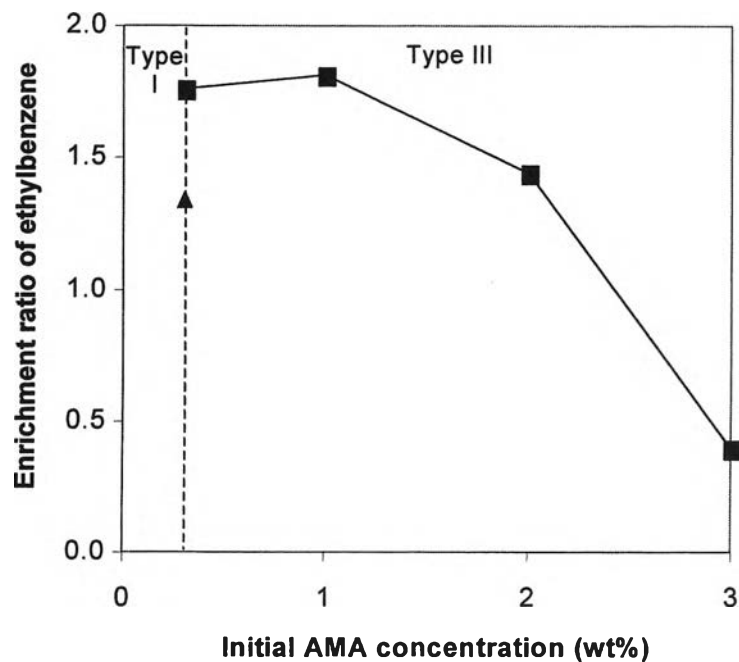


Figure 4.4 Effect of initial AMA concentration on enrichment ratio of ethylbenzene at 2 wt% NaCl (▲) and 3 wt% NaCl (■)

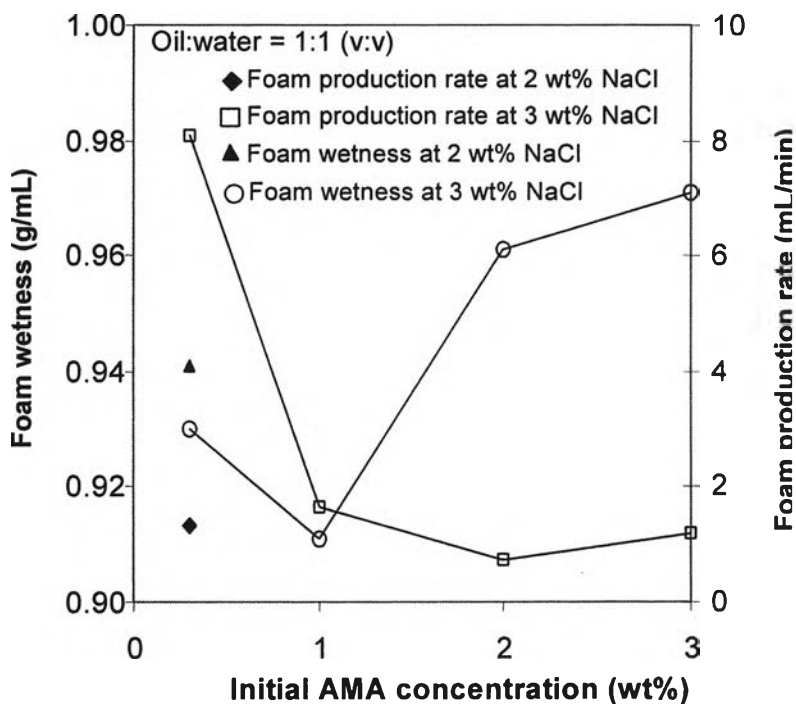


Figure 4.5 Effect of initial AMA concentration on foam wetness and foam production rate

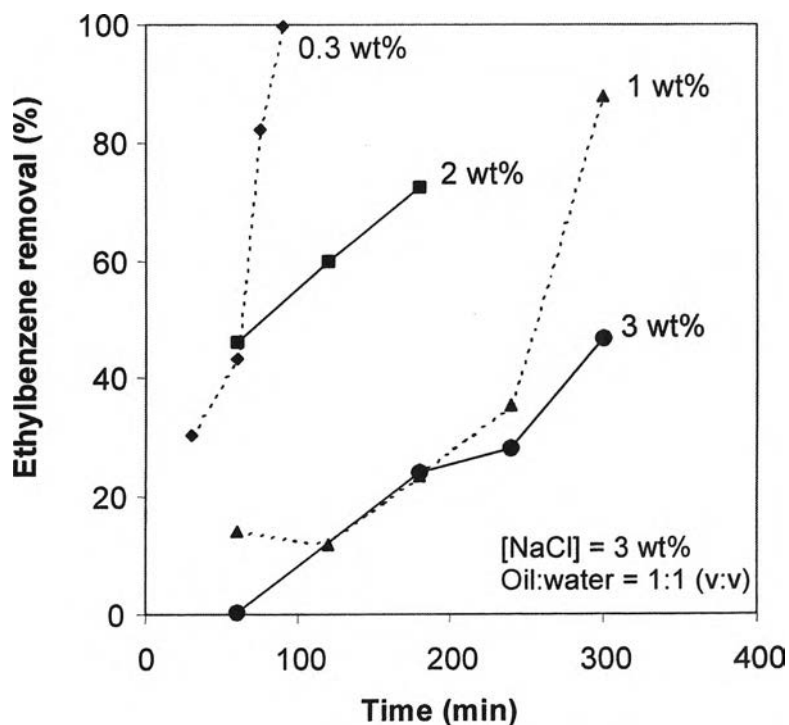


Figure 4.6 Dynamic removal efficiency of ethylbenzene at different initial AMA concentrations

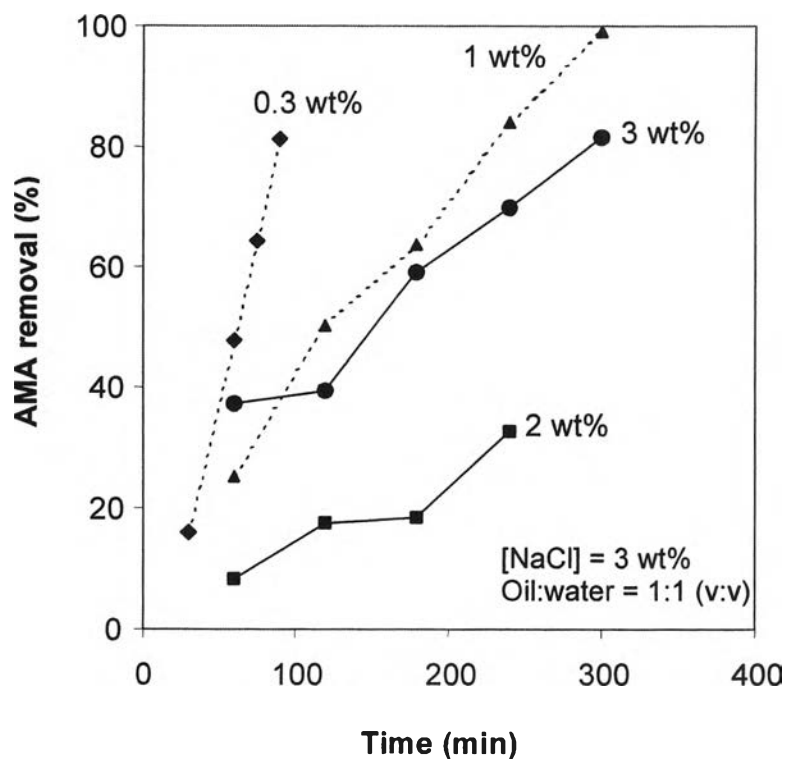


Figure 4.7 Dynamic removal efficiency of AMA at different initial AMA concentrations

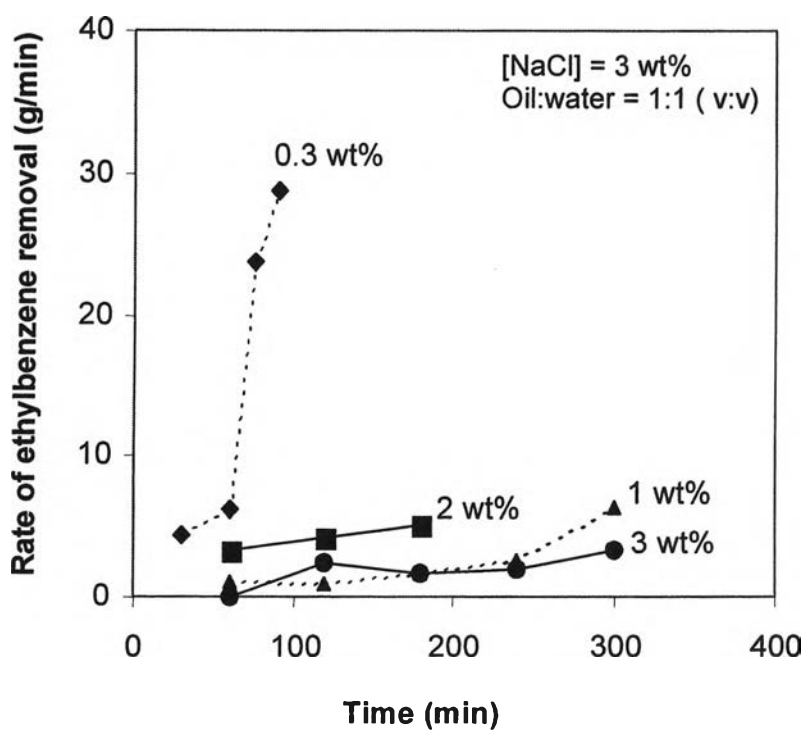


Figure 4.8 Rate of ethylbenzene removal at different initial AMA concentrations

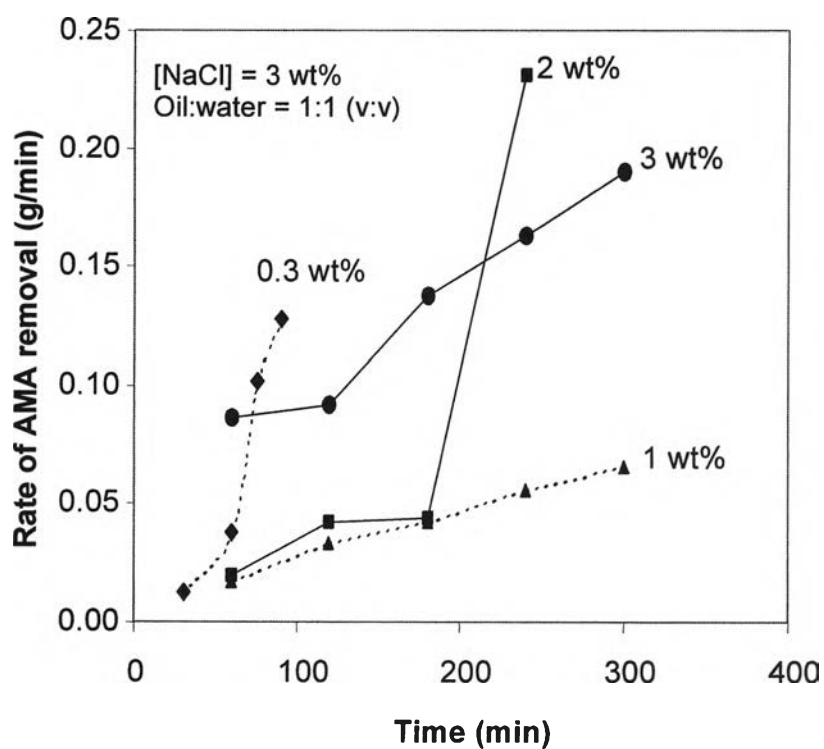


Figure 4.9 Rate of AMA removal at different initial AMA concentrations

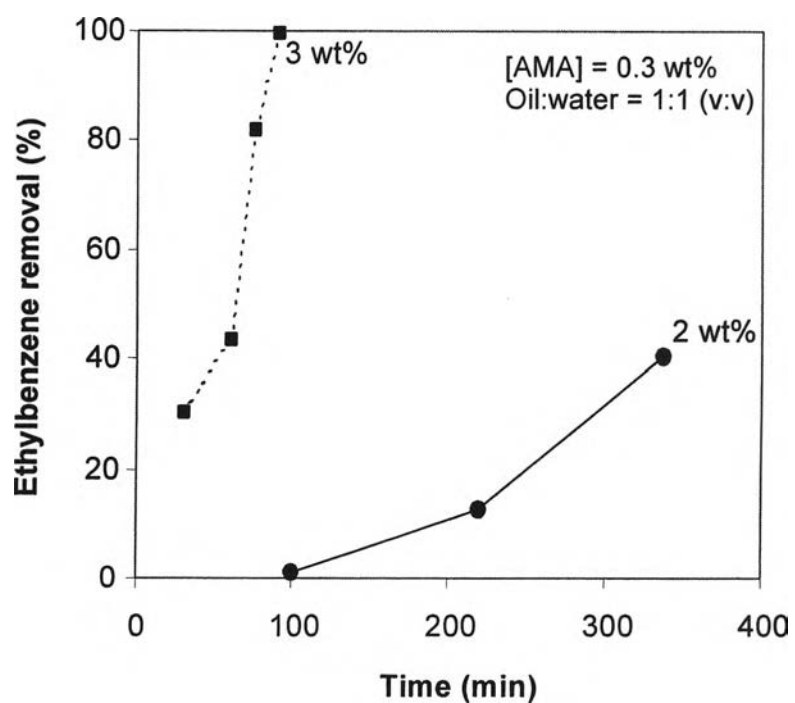


Figure 4.10 Dynamic removal efficiency of ethylbenzene at two different NaCl concentrations

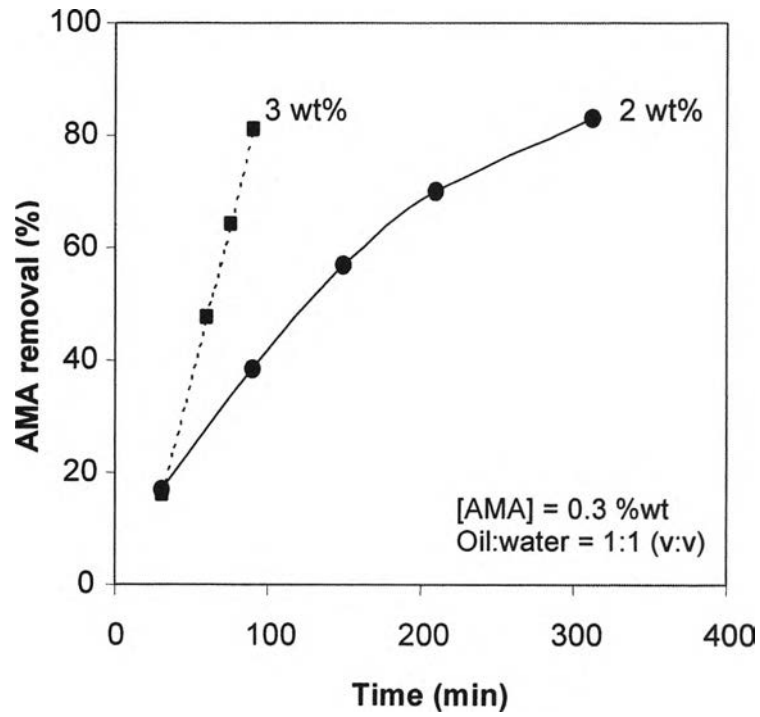


Figure 4.11 Dynamic removal efficiency of surfactant at two different NaCl concentrations

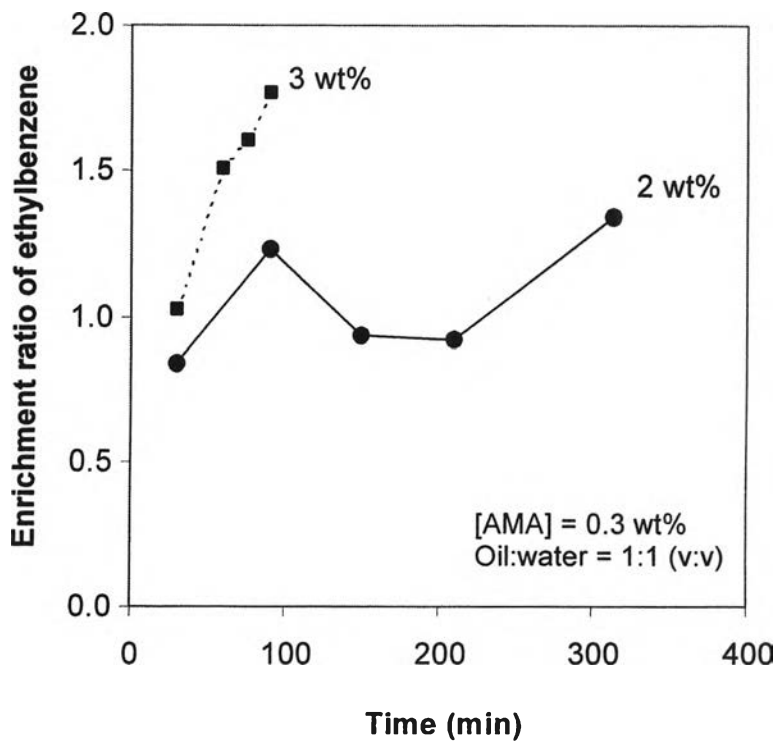


Figure 4.12 Enrichment ratio of ethylbenzene as a function of time at two different NaCl concentrations

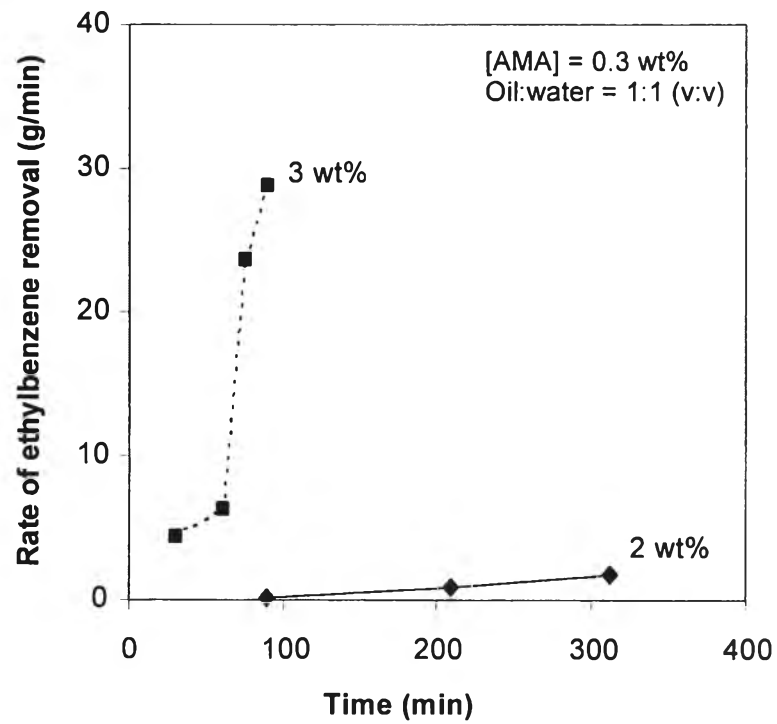


Figure 4.13 Rate of ethylbenzene removal at two different NaCl concentrations

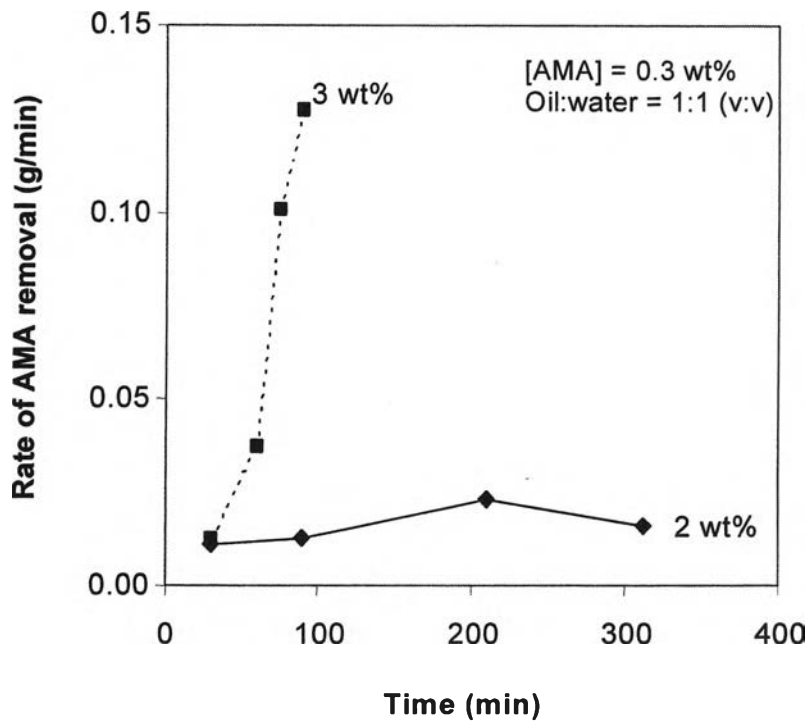


Figure 4.14 Rate of AMA removal at two different NaCl concentrations