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Diesel Oil Removal by Froth Flotation Under Low Interfacial Tension Conditions I: Foam Characteristics, and Equilibration Time

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Abstract: Froth flotation is a surfactant-based separation process which is suitable for treating dilute wastewaters. To achieve high performance for the froth flotation operation, the combination of an ultra-low interfacial tension (IFT) between excess oil and excess water phases, high foam production rates, and high stability of the foam produced, must be attained. To obtain the ultra-low interfacial tensions, a Winsor Type III or middle phase microemulsion has to be formed. In this study, branched alcohol propoxylate sulfate sodium salt with 14–15 carbon number and 4 PO groups (Alfoterra 145–4PO) was used to form microemulsions with diesel oil. From the results of this work, an increase in surfactant concentration decreased the IFT, and increased foam stability. To obtain the minimum IFT in the region of a Winsor Type III microemulsion, the addition of 5 wt.% NaCl was needed. However, this optimum salinity does not result in effective froth flotation due to poor foam characteristics. The results indicate that both the IFT and the foam characteristics should be optimized to achieve high efficiency of oil removal in a froth flotation operation. Unlike the previously-studied ethylbenzene system, agitation of the solution before introduction into the flotation column yielded the lowest diesel oil

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removal efficiency because of the poor foam characteristics compared to either unagitated systems or systems allowed to equilibrate for one month.

Keywords: Microemulsion, froth flotation, foam formation, foam stability, diesel oil removal

INTRODUCTION

In many countries, including Thailand, diesel oil consumption is much higher than gasoline consumption. This is because diesel oil provides more energy per unit volume than gasoline (1). In the US and Latin America, diesel oil is used primarily for the transportation of goods. Moreover, in Europe, Japan, and elsewhere, diesel oil is a significant source of energy for personal transportation (2). The demand for diesel oil is forecast to grow faster than the demand for other fuels in general. Therefore, the contamination of groundwater by diesel oil leakage from gas stations or underground storage tanks is of increasing concern. Since many components in diesel oil have low water solubility, diesel oil has been found to be present as an oil-in-water emulsion in polluted water.

Froth flotation is the technique investigated in this work to remove the emulsified diesel oil from water. Froth flotation has been widely used for decades to separate a desired ore from unwanted substrates in mineral processing (3). Moreover, nowadays, the froth flotation technique is widely employed in wastewater treatment applications (4–8) and in paper deinking processes (9, 10). There are two main types of froth flotation: dissolved and induced air flotation. In the latter, which was employed in the present study, filtered air is bubbled into the solution through a sintered glass disk. Droplets of emulsified oil can attach to rising bubbles, and then rise to the top of the column to form a foam or froth. However, the stability of these bubble-droplet aggregates is generally low, leading to a lower separation efficiency. To achieve a higher separation efficiency in traditional froth flotation, a proper type of surfactant with an optimum concentration is introduced into the solution to stabilize the foam (11). Chang et al. (12) reported that the surfactant added had to be adjusted at an appropriate concentration to obtain high foam stability. As described in previous work (13–16), our approach is to generate conditions in the flotation column corresponding to ultra-low interfacial tensions between the oil and water phases through microemulsion formation by adding appropriate surfactants while still needing to maintain good foam stability in order to maximize the removal efficiency of several types of oils.

In previous work (13), the maximum oil removal was found to correspond to the formation of a Winsor Type III microemulsion for ortho-dichlorobenzene. Later, Chavadej et al. (14) found that most of the oil removed from the column during the flotation operation came from the excess oil phase rather than the middle phase (phase rich in oil, water, and surfactant) in Winsor Type III microemulsion systems with ortho-dichlorobenzene. After that, Yanatatsaneejit et al. (15) found that the ultra-low oil-water interfacial tension characteristic of a Winsor Type III microemulsion was not the only factor affecting the performance of froth flotation, but foam stability was also crucial to achieving a high flotation efficiency for ethylbenzene removal. In addition, Yanatatsaneejit et al. (16) reported the effect of equilibration time on the performance of froth flotation. For the ethylbenzene system, the highest performance of froth flotation was achieved when the microemulsion system was allowed to equilibrate prior to flotation. Interestingly, pre-mixing of the solution for 40 min (called induced-equilibrium) was found to produce good oil removal. In this present work, the performance of froth flotation to remove diesel oil from water was correlated with interfacial tension and foam characteristics. In addition, the coalescence time between the oil droplets was independently measured and correlated to flotation efficiency in this work.

EXPERIMENTAL SECTION

Materials

The diesel oil used in this study is a commercial grade diesel oil obtained from the Petroleum Authority of Thailand (PTT) and is a mixture of hydrocarbons having carbon numbers predominantly in the range of C9-C20 with having not more than 65% by volume recovered at 240°C and not less than 85% by volume recovered at 350°C (17). A branched alcohol propoxylate sulfate sodium salt with 14 to 15 carbons in the alkyl chain, and an average of 4 propylene oxides ($C_{14\text{-}15}(PO)_4SO_4$), trade name Alfoterra 145-4PO where PO represents propylene oxide, an anionic surfactant which is an experimental (not yet commercially available) surfactant specially synthesized by Sasol Company (formerly Condea Vista Company), Rosebank, South Africa, was used in this study. Analytical purity grade sodium chloride (NaCl) from Aldrich Chemical Company Inc. was used as the added salt in this work. All chemicals were used as received without further purification. Deionized water was used to prepare all aqueous solutions.

Methodology

This experimental work is divided into four parts. The first part is the equilibrium microemulsion phase behavior and oil/water interfacial tension (IFT) of aqueous solutions containing different $C_{14\text{-}15}(PO)_4SO_4$ concentrations with diesel oil. The second part is the oil separation efficiency in a batch flotation column. The third part is the foamability and foam stability under different conditions. The fourth part is the coalescence rate between oil droplets. In all experiments, the surfactant and electrolyte concentrations are expressed in weight percentage (wt.%) per volume of the aqueous solution consisting of water, surfactant, and electrolyte.

To investigate the phase behavior of microemulsions, 5 mL of a homogeneous aqueous solution, prepared at various surfactant and NaCl concentrations, was mixed with 5 mL of diesel oil in a vial sealed with a screw cap. These vials were shaken every day for 3 days, and then allowed to equilibrate at a constant temperature of 30°C in an incubator for 1 month to reach equilibrium, which was verified by the invariant height of each phase. The height of each phase was measured by using a cathetometer with a precision of ±0.01 mm. The interfacial tension (IFT) between equilibrated excess oil and excess water phases was measured by a spinning drop tensiometer (SITE 04, Krüss GmbH, Hamburg).

A schematic of the froth flotation unit used in this work is shown in Fig. 1. A glass cylindrical column with 5 cm internal diameter and 120 cm height was used as the froth flotation column. A 750 mL sample with different initial oil-to-water ratios and various surfactant and NaCl concentrations which had been equilibrated at 30°C for 1 month in the incubator, was transferred to the froth flotation column. Filtered air at different flow rates using a mass flow controller was introduced at the bottom of the column through a sintered glass disk having pore size diameters of about 16–40 μm. The generated air bubbles rose through the solution to the top of the column. All experiments were terminated when no more foam came overhead from the column, i.e. the stage at which the surfactant concentration in the solution became too low to generate sufficient foam to reach the overhead weir. The total foam collected in the

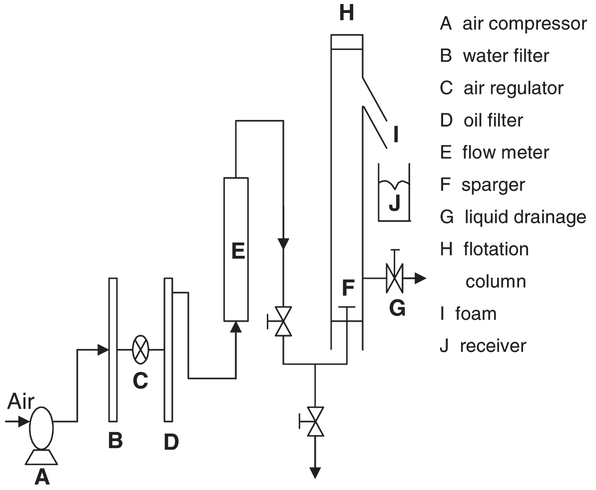

Figure 1. Schematic of the froth flotation apparatus.

receiver was then broken by freezing for diesel oil and surfactant concentration analyses by using the methylene chloride extraction method (18) and the titration method with methylene blue chloride (19), respectively. Since the froth flotation was operated in a batch mode, total cumulative removal of surfactant and diesel oil was used to assess the process performance. Moreover, the solution in the column was sampled at the same time interval as the foam collected for analysis of residual diesel oil and surfactant concentrations.

In order to obtain a better understanding about the phenomena important in the froth flotation process, foamability and foam stability experiments were conducted independently in the same flotation column. A 250 mL sample containing a given surfactant and NaCl 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 desired flow rate until the maximum foam height in the column was achieved. This maximum foam height was used to indicate foamability. Then, the air injection was ceased and the time required for the foam volume to collapse to half of the maximum height was recorded to quantify foam stability. All experiments of froth flotation operation, foamability, and foam stability were conducted at room temperature of 25–27°C. The ratio of maximum foam height to initial solution height is considered as foamability while foam stability ($t_{1/2}$) is defined as the time required for the foam to collapse to half the maximum foam height.

To investigate the coalescence time between oil droplets, an aqueous solution having different surfactant and NaCl concentrations was mixed with diesel oil in a vial at various oil-to-water ratios. After that, this mixture was gently shaken for 1 minute. The intensity of light generated from a Light Emitting Diode (LED) having a wavelength of 568 nm passing through the solution in the vial was measured at various times by a photo sensitive detector until reaching a constant value. In this research, the time when the light intensity begins to reach a plateau is defined as the coalescence time between oil droplets. The plateau light intensity indicates that the coalescence between oil droplets is completed. The measurement of the coalescence time was also conducted at room temperature of 25–27°C.

RESULTS AND DISCUSSION

To gain a better understanding of the mechanism of the froth flotation process, all results obtained from the four experimental parts are presented in the same graph in order to determine the correlation of all process parameters. Figure 2 shows the proposed mechanism of oil separation in the froth flotation operation which consists of four sequential steps. Firstly, air bubbles are generated within the liquid solution by introducing air into the bottom of the flotation column. In this step, oil droplets adhere to the surface of the air bubbles. In the second step, as the air bubbles rise in the solution, the

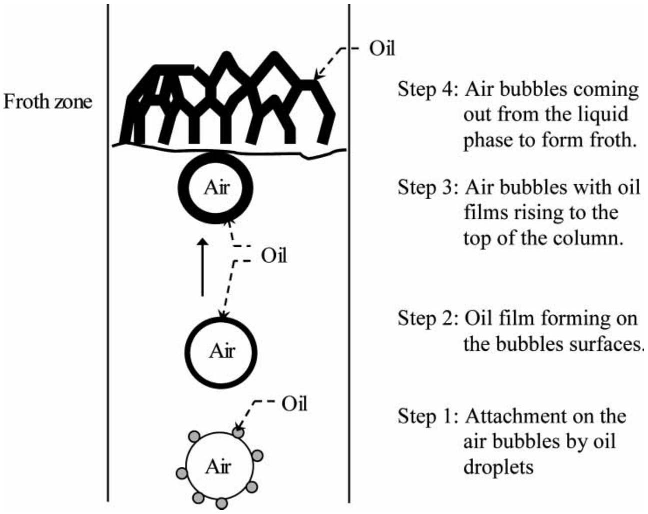

Figure 2. Proposed mechanism in froth flotation operation.

formation of an oil film on the surface of the air bubbles visually appears. The thickness of the oil film should be great enough to achieve a high oil removal; thus the coalescence between the oil droplets has to be maximized. The third step is the rising of bubbles (with attached oil droplets) to the top of the column. High stability of the air bubbles covered by an oil film is required in this step. The fourth step is the air bubbles emerging from the liquid phase to form froth. To operate froth flotation successfully, high stability of the froth is needed to obtain a dry foam with a high oil content. In this work, all important parameters including interfacial tension obtained from the phase behavior study, foam stability and foamability obtained from the foam characteristic study, and oil droplet coalescence time are simultaneously analyzed to correlate to the efficiency of the froth flotation separation.

Phase Behavior

According to previous work, interfacial tension (IFT) is one of the major factors affecting the performance of the froth flotation operation (12, 13). As shown in Fig. 3, at 3 wt.% NaCl concentration, increasing surfactant concentration decreases the IFT monotonically between excess oil and excess water phases, presumably because of the increasing adsorption of surfactant at the oil/water interface (20). However, if the surfactant concentration exceeded 0.15 wt.%, stable macroemulsions were found to form, causing the oil/water separation to be too slow for equilibration of the phases to be practically obtainable.

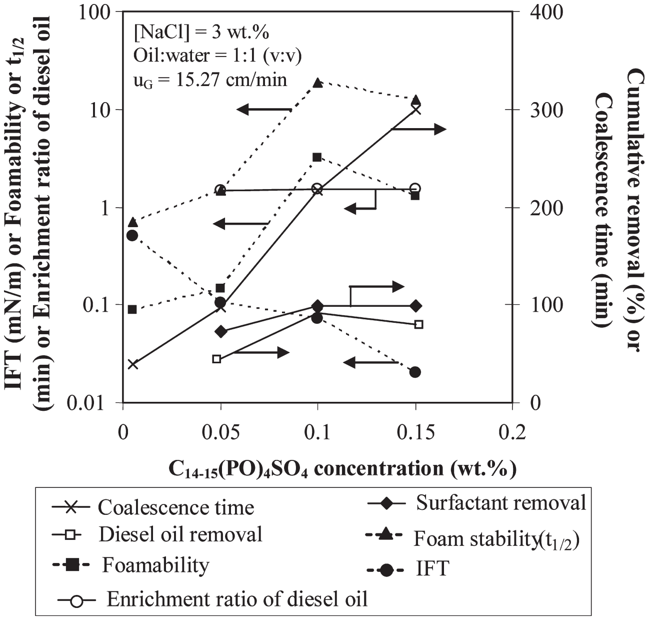

Figure 3. Effect of surfactant concentration on process parameters.

As shown in Fig. 4, at a fixed $C_{14\text{-}15}(PO)_4SO_4$ concentration of 0.10 wt.%, a minimum IFT is observed at 5 wt.% NaCl. For anionic surfactants, a minimum in IFT with varying salinity is commonly observed and this salinity is known as the optimum salinity; this effect has been discussed in our previous study of the relationship between the microemulsion phase behavior and froth flotation performance of ethylbenzene (15).

Figure 5 shows the insignificant effect of oil-to-water ratio on IFT at 0.1 wt.% $C_{14\text{-}15}(PO)_4SO_4$ concentration and 3 wt.% NaCl concentration, which is consistent with the ethylbenzene (with different surfactant systems) results (16). Since oil-to-water ratios in practical froth flotation operations can be quite different from those used in traditional phase studies (of 1:1 v/v), this lack of sensitivity on oil-to-water ratio justifies the use of phase studies to guide the design and operation of flotation for real wastewater, which generally has very low ratios of oil-to-water.

Foam Characteristics

As described in previous work (15, 16), the removal efficiency of oil in a froth flotation operation is improved by adjusting the system to have both higher foamability and foam stability. The effects of $C_{14\text{-}15}(PO)_4SO_4$ concentration on both foam stability and foamability are illustrated in Fig. 3. Both foam

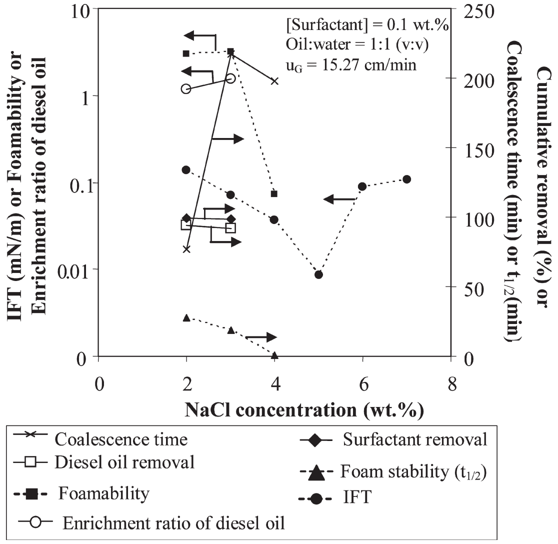

Figure 4. Effect of NaCl concentration on process parameters.

stability and foamability reached a maximum at 0.1 wt.% surfactant concentration. From Fig. 4, at 0.1 wt.% $C_{14\text{-}15}(PO)_4SO_4$, foamability reaches a maximum at 3 wt.% NaCl while foam stability decreases with increasing NaCl concentration over the range studied. As shown in Fig. 5, both

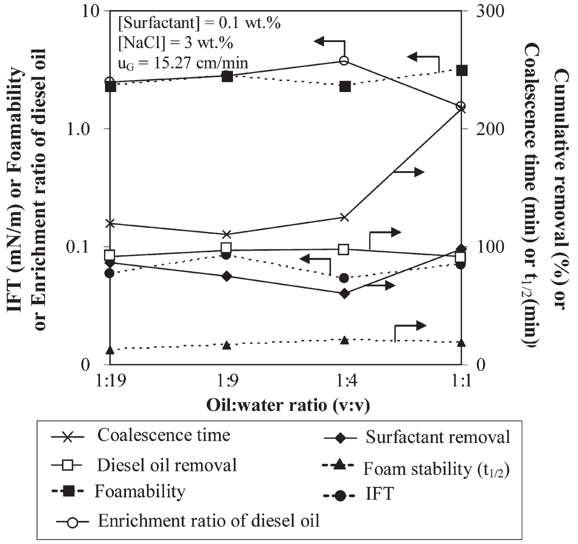

Figure 5. Effect of oil-to-water ratio on process parameters.

foamability and foam stability tend to increase slightly with increasing oil-to-water ratio, consistent with the results of the ethylbenzene system (16).

In systems with anionic surfactant, salt, and oil, there are a number of factors affecting foamability and foam stability, including Gibbs-Marangoni effects, surface viscosity, viscosity in film lamellae, packing of the surfactant monolayer at the air-water interface, and bridging of foam lamellae by oil droplets, to name a few. Therefore, explanations of these effects of surfactant and salt concentrations and oil-to-water ratio are largely speculative. A possible explanation for the maximum foamability at 3% NaCl is the tendency to increase foamability/foam stability due to reduced repulsion between charged head groups in a monolayer, leading to increased surface viscosity, while there is a tendency to decrease foamability due to reduced electrostatic repulsion between the two surfactant monolayers. The maximum foamability and foam stability occur at a surfactant concentration slightly lower than the critical micelle concentration, CMC (11). The CMC of $C_{14\text{-}15}(PO)_4SO_4$ is approximately 0.01 wt.%. Since the froth flotation unit used in this study was operated in batch mode and so the surfactant concentration varied from 0.1 wt.% at the initial time to a very low concentration at the end of operation. The average surfactant concentration throughout the operation period might be close to the CMC.

Figure 6 shows the effect of air superficial velocity (u_G), which is calculated from the applied air flow rate divided by the cross-sectional area of the column, on foam characteristics of the system, having an $C_{14\text{-}15}(PO_4)SO_4$

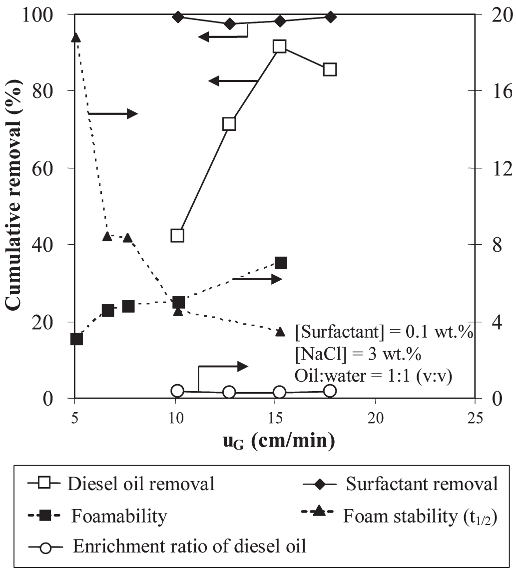

Figure 6. Effect of superficial air velocity on process parameters.

concentration of 0.10 wt.%, a NaCl concentration of 3 wt.%, and an oil-to-water ratio of 1:1. As shown in Fig. 6, the foam stability is strongly affected by u_G; the foam stability decreases substantially with increasing u_G. This is because at a higher u_G, the velocity of the bubble swarm rising through the column is higher, leading to increasing coalescence between air bubbles. However, in the case of foamability, the foam formation is proportional to u_G because the number of bubbles increases when u_G increases. In this studied system, above a u_G of 15 cm/min, measurement of foam characteristics was not possible because foam did overflow from the flotation column.

Coalescence of Oil Droplets

The coalescence time between oil droplets is expected to be one of the major parameters affecting the performance of froth flotation since there should be a difference between floating many small droplets vs. a few large drops. The coalescence time increases with increasing $C_{14\text{-}15}(PO)_4SO_4$ concentration, as shown in Fig. 3. The coalescence time first generally increases and then decreases with increasing salinity, and reaches a maximum at 3 wt.% NaCl, as seen in Fig. 4. Figure 5 shows the effect of oil-to-water ratio on the coalescence time. There are several important factors affecting coalescence rates and their complex interaction makes the prediction of the effects of operation variables difficult. Increasing viscosity tends to increase coalescence times (21) (decrease coalescence rates), probably explaining the effects of surfactant concentration and oil-to-water ratio. Decreasing IFT reduces the decrease in system free energy upon the loss of interfacial area upon coalescence, tending to decrease coalescence times. Interfacial tension gradients also tend to decrease as the surfactant concentration decreases, reducing Gibbs-Marangoni effects which help stabilize the thin aqueous film between oil droplets, which tends to decrease coalescence times also. Increasing salinity compresses the electrical double layer, reducing electrical repulsion between the oil droplets (which have induced charge due to the adsorbed anionic surfactant), also tending to reduce coalescence time. Increasing salinity could also cause denser packing of the surfactant molecules, increasing interfacial viscosity, tending to increase coalescence rates. The complex interaction between these effects results in the maximum in coalescence time with salinity, as seen in Fig. 4. The effects of salinity on the coalescence rates for emulsions have been discussed in the literature (22).

Evaluation and Correlation of Froth Flotation Performance

As in previous work (15), the three parameters of oil removal, surfactant removal, and the enrichment ratio of oil are used to characterize the performance parameters of the froth flotation operation. In this work, the enrichment ratio of diesel oil is defined as the diesel oil concentration in the collapsed froth to the diesel oil concentration in the initial feed as an indication of selectivity of diesel oil removal compared to water. Therefore, to achieve any separation, the enrichment ratio of diesel oil has to be higher than unity.

Removal of Diesel Oil and Surfactant

Figure 3 shows the effects of $C_{14\text{-}15}(PO)_4SO_4$ concentration on IFT, total cumulative diesel oil removal, total cumulative $C_{14\text{-}15}(PO)_4SO_4$ removal, foamability, foam stability, and coalescence time between oil droplets. Similar to the ethylbenzene system (15), the total cumulative diesel oil removal is the highest at the $C_{14\text{-}15}(PO)_4SO_4$ concentration corresponding to the maximum foamability and the maximum foam stability, but not the minimum IFT. The same trend is observed for the total cumulative $C_{14\text{-}15}(PO)_4SO_4$ removal. Both diesel oil and $C_{14\text{-}15}(PO)_4SO_4$ removal was found experimentally not to correlate to the coalescence time between oil droplets.

The interfacial tension (IFT), total cumulative diesel oil removal, total cumulative $C_{14\text{-}15}(PO)_4SO_4$ removal, foamability, foam stability, and coalescence time between oil droplets as a function of NaCl concentration are shown in Fig. 4. Enough foam could not be generated to overflow the flotation column when the NaCl concentration was greater than 3 wt.%, resulting in no diesel oil or surfactant removal above 3 wt.% NaCl. Between 2 and 3 wt.% NaCl, the removal of diesel oil and surfactant was found to be almost constant.

Figure 5 shows the IFT, total cumulative diesel oil removal, total cumulative $C_{14\text{-}15}(PO)_4SO_4$ removal, foamability, foam stability, and coalescence time as a function of oil-to-water ratio. Similar to our previous work (16), the removal efficiency of oil was not significantly affected by the oil-to-water ratio, whereas the surfactant removal reached a minimum value at an 1:4 oil-to-water ratio for the diesel oil system, but 1:9 ratio for the ethylbenzene system (16). The variables most affected by oil-to-water ratio are the coalescence time, which increases dramatically as the oil-to-water ratio approaches 1:1, and the enrichment ratio of diesel oil, which approaches unity (no selectivity for oil removal vs. water) as the oil-to-water ratio approaches 1:1. At extremely high oil loadings in the wastewater, the froth flotation technique is not effective, which is expected. It is important to note that the IFT is almost independent of oil-to-water ratio. Since microemulsion phase studies used to equilibrate oil/water/microemulsion phases prior to measurement of the IFT are typically performed at an initial oil-to-water ratio of unity, whereas oily wastewater typically has an oil-to-water ratio much lower, the robustness of IFT with respect to oil-to-water ratio means that IFT values in this study are relevant to real wastewater having a low oil-to-water ratio.

The effects of superficial velocity (u_G) on the total cumulative diesel oil removal, total cumulative $C_{14\text{-}15}(PO)_4SO_4$ removal, foamability, and foam

Table 1. Effect of equilibration condition on process parameters at a fixed surfactant concentration of 0.10 wt.%, a NaCl concentration of 3 wt.%, and an oil-to-water ratio of 1:1

System	IFT (mN/m)	Total cumulative removal (%)		Enrichment ratio of diesel oil	Foamability	$t_{1/2}$ (min)
		Diesel oil	Surfactant			
Non-equilibrium (no premixing)	0.109	91.54	98.30	1.53	3.13	18.82
Induced-equilibrium (40 min mixing)	0.159	54.46	46.89	1.62	0.11	0.87
Equilibrium (1 month storage)	0.036	96.86	74.06	1.53	3.21	20.03

stability are shown in Fig. 6. The highest removal efficiency of diesel oil corresponded to the u_G which had the highest foamability but relatively low foam stability. This is because the number of bubbles passing through the solution increases rapidly with increasing u_G, leading to a higher foam production rate. The results indicate that under the studied conditions, the effect of foamability is more significant than that of foam stability on the diesel oil removal. In contrast, the total cumulative $C_{14\text{-}15}(PO)_4SO_4$ removal was not significantly affected by u_G, even though the volume of foam generated increased. This is due to some of the surfactant as well as diesel oil being entrained back into the solution since the circulation velocity of the bubble swarms is higher at a higher u_G.

Table 1 shows the effects of equilibration conditions on the IFT, total cumulative diesel oil removal, total cumulative $C_{14\text{-}15}(PO)_4SO_4$ removal, foamability, and foam stability. For the equilibrium system, the total cumulative diesel oil removal, foamability, and foam stability, were the highest, as expected. It is interesting to note that the total cumulative diesel oil removal from the non-equilibrium system was very close to that of the equilibrium system. Unlike the ethylbenzene system (16), the diesel oil removal of the induced-equilibrium system was found to have the lowest oil removal because both foamability and foam stability were extremely low. The induced-equilibrium system also gave the lowest $C_{14\text{-}15}(PO)_4SO_4$ removal, again not consistent with the previous work on ethylbenzene (16). In contrast with the results of the diesel oil removal, the maximum $C_{14\text{-}15}(PO)_4SO_4$ removal was found for the non-equilibrium system.

From a practical viewpoint, these results lead to the important and counterintuitive conclusion that no agitation may be preferable to agitation for a short time for the diesel oil/water mixture. Of course, real wastewater will have emulsified oil in contact with the water for some time before treatment, so optimum contact time will need to be determined for the specific stream of interest. Since different trends have been observed for the two oils studied (diesel oil and ethylbenzene), results may be highly dependent on oil structure.

Mechanistically, a hypothesis to explain the results is that when mixing for 40 minutes, the emulsion droplet size may be reduced, leading to a greater oil/water interface area and more surfactant adsorbed at this interface. The resulting lower surfactant concentration in the bulk aqueous phase leads to higher oil/water interfacial tension, lower foamability, and lower stability, leading to lower diesel oil and surfactant removal. When the system equilibrates, the surfactant primarily incorporates itself into the microemulsion phase and ultralow IFT values are observed. The equilibrium system gives a high diesel oil removal, but low surfactant removal as compared to the non-equilibrium system. Most of the oil being removed comes from the excess oil phase and not from the microemulsion phase, as has been deduced in previous work for dichlorobenzene under equilibrium conditions (14).

Enrichment Ratio of Diesel Oil

The enrichment ratio of diesel oil as a function of $C_{14\text{-}15}(PO)_4SO_4$ concentration is shown in Fig. 3. As the $C_{14\text{-}15}(PO)_4SO_4$ concentration increased, the enrichment ratio of diesel oil was almost constant. Actually, increasing surfactant concentration might be expected to decrease the enrichment ratio of oil because of several aspects such as thicker foam lamellae causing higher water content in foam lamellae, and higher viscosity of the solution leading to a lower drainage rate of water from the foam lamellae. However, the amount of oil in the froth also increases with increasing surfactant concentration due to more hydrophobic regions in the froth.

From Fig. 4, the diesel oil enrichment ratio increases as the NaCl concentration increases from 2 to 3 wt.%. A possible explanation is that the repulsive force between the head groups of the anionic surfactant at the opposite site of the foam lamellae decreases with increasing NaCl concentration, resulting in a thinner foam lamellae, resulting in a lower amount of water carried over with the foam, leading to a higher enrichment ratio of diesel oil. As shown in Fig. 5, the enrichment ratio of diesel oil reaches a maximum at a 1:4 oil-to-water ratio. It is expected that the diesel oil enrichment ratio would increase with increasing oil-to-water ratio, but the mild maximum observed here is due to increasing solution viscosity at high oil-to-water ratio, causing slower water drainage from the foam lamellae. From Fig. 6, we can see that the superficial air velocity has no significant effect on the enrichment ratio of diesel oil while Table 1 shows that equilibration conditions have little effect.

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