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Removal of Diesel from Aqueous Emulsions by Flotation

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ABSTRACT

The induced air flotation of aqueous diesel oil emulsions generated during the remediation of contaminated sand has been studied experimentally in a batch flotation column 35 mM in diameter and 300 mM in height. The stability of these emulsions was characterized and factors such as pH and salinity affecting the stability of the emulsion were investigated. The effects of anionic and cationic surfactants, original diesel content, air-flow rate, surfactant dosage, and the air distributor sinter size were investigated, and it was found that up to 99% of the diesel could be removed. This amounted to a residual diesel concentration of less than 10 ppm.

Key Words. Induced air flotation; Diesel; Emulsions

INTRODUCTION

Oily wastewaters are generated during the production, processing, transportation, storage, and use of petroleum and its derivatives. For instance, in polymer flooding of petroleum reservoirs, the high-molecular-weight polymers and organic surfactants that are used to push oil to a producing well tend to generate highly stable aqueous oil emulsions. Likewise, other types of floods, such as caustic, steam, and firefloods, are notorious for causing problem emulsions.

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Flotation is widely used to treat these effluents. In oil refineries, flotation has been an effective way to treat oily wastewaters before release to a watercourse. In the petroleum industry, it is one of the few reliable methods to treat refractory emulsions produced by secondary and tertiary oil recovery techniques (1).

The flotation process consists of four basic steps: (a) bubble generation in oily water; (b) contact between a gas bubble and an oil droplet suspended in the water; (c) attachment of an oil droplet to a gas bubble; and (d) rising of the bubble-droplet assembly to the surface where the oil can be removed by skimming. There are two main modes of operation for flotation cells, depending on how the air is introduced. In *dissolved air flotation*, air is first dissolved in the effluent at elevated pressure (2–3). Just before the effluent is introduced into the flotation cell the pressure is reduced, resulting in the air leaving the supersaturated solution in the form of small bubbles 50–100 μm in diameter. In *induced air flotation*, the air is either dispersed mechanically in the liquid or sparged into the separation vessel through a distributor (4–5). Under these conditions, the bubble size distribution is typically in the range of 400–2000 μm . In general, induced air flotation machines can be characterized by high liquid flow with low retention times, compared to dissolved air flotation machines with low liquid flows and high retention times.

Although induced air flotation of oil emulsions has been studied for years, it has not found widespread industrial use. This is because most industry research has focused on the implementation of induced air flotation in agitated vessels. Although the equipment is relatively simple and affords a high throughput, it is not very efficient. Mechanical agitation in induced air flotation cells leads to highly turbulent flow, which tends to regenerate very fine droplets that are difficult to treat. This may change with new developments in induced air flotation columns. These columns do not suffer from the same drawback associated with mechanical flotation cells, because a relatively quiescent separation zone can be formed in the column. Also, comparatively small bubbles can be generated through a porous air distributor.

In crude-oil refinery plants, the pretreatment stage before flotation often consists of simple gravity separation by standard API or parallel plate separators. The crude oil–water emulsions leaving these separators normally have oil droplets less than 30 μm in diameter and oil concentrations around 200 mg/L. Dissolved-air flotation is involved for the crude-oil removal, and the average oil-removal efficiency for oil droplets in the range of 0–30 μm is only approximately 20% for industrial size units (4).

Various studies have described the influence of, among others, the height of the foam–liquid interface, the air-flow rate, the bubble diameter, the feed concentration (6–7), as well as the concentration of the added electrolyte (8–9) on the efficiency of oil removal. For example, Hung (10) reported oil removal of

95% by flotation with a polyelectrolyte additive. Van Ham et al. (11) studied the flotation of highly stabilized aqueous oily emulsions and reported that 99.5% of the oil could be removed by using a cationic emulsified surfactant. Angelidou et al. (12) studied the flotation rate of emulsified oil droplets in water using air bubbles averaging approximately 150 μm in diameter and found that high percentages of oil removal were achieved in a short time.

These referenced studies were mainly focused on heavy-petroleum products and little appears to have been published on the flotation of light-petroleum products, such as diesel and gasoline. In this paper, the various factors and mechanisms influencing the removal of diesel from stable aqueous emulsions are consequently discussed.

EXPERIMENTAL

Materials

The emulsions were generated in a jet reactor in which sand polluted with diesel fuel was cleaned. A schematic diagram of the jet reactor for the cleaning of diesel-contaminated sand is shown in Fig. 1. The water-jet pressure was approximately 30 MPa, and the flow rate of the sand slurry was 16 kg/min at a slurry concentration of 15% solids. The diesel content in the simulated sand mixture was 5% by mass. The diesel was stripped off the sand surface by the mechanical effect of the high-speed jet. The mixture of the sand and diesel emulsions was subsequently subjected to solid-liquid separation. These emul-

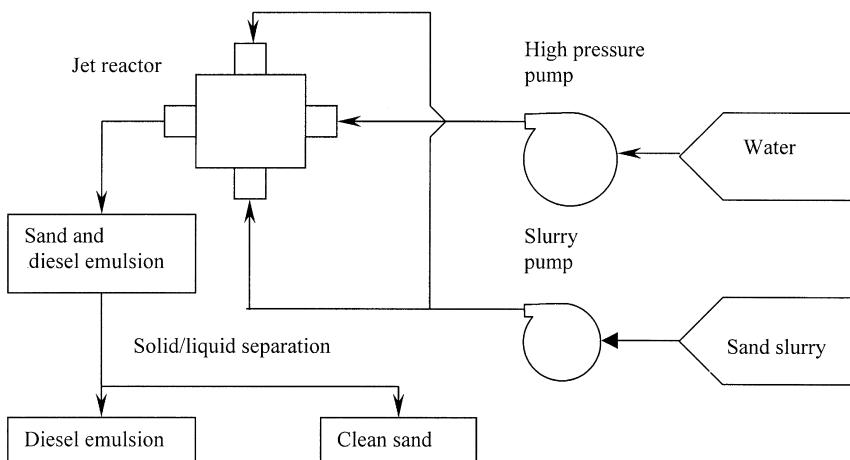


FIG. 1 Schematic diagram of the jet reactor for diesel-contaminated sand cleaning.

sions initially contained diesel concentrations of approximately 10,000 ppm. The emulsions were first allowed to settle for 1 wk, after which the oily surface layers were removed and the resulting emulsions with concentrations of less than 400 ppm were used for the following stability and flotation experiments. An emulsion batch was kept for 6 h with continuous stirring by a mechanical stirrer at a speed of about 60 rpm. Similar emulsions were used for all comparative experiments.

Sodium dodecyl sulphate with a purity of 90–91% was obtained from Saarchem (Pty) Ltd, (South Africa). Analytically pure octadecylamine chloride (ODAC), cetyl trimethyl ammonium chloride (CTMAC), and sodium hydroxide were obtained from Sigma. Analytically pure sodium hydroxide, hydrochloric acid, and methylene chloride were obtained from Merck. Deionized water was used in all the experiments.

Experimental Set-up

Figure 2 shows a schematic diagram of the air flotation equipment used in this study. The test flotation column was made of glass having a diameter of 35 mM and a height of 300 mM. The sparged air was distributed through a sinter glass with pore size 3 (40–60 μm) and pore size 4 (10–15 μm). The experiments were conducted at an ambient temperature of 20°C. Froth overflow occurred automatically and care was taken to clean the column between runs. The pulp level was kept constant by continuously adding rinse water.

Three froth samples were taken at 4, 10, and 20 min during each experimental run. The samples were analyzed for diesel content with the extraction-weighting method described below. All the test runs were duplicated.

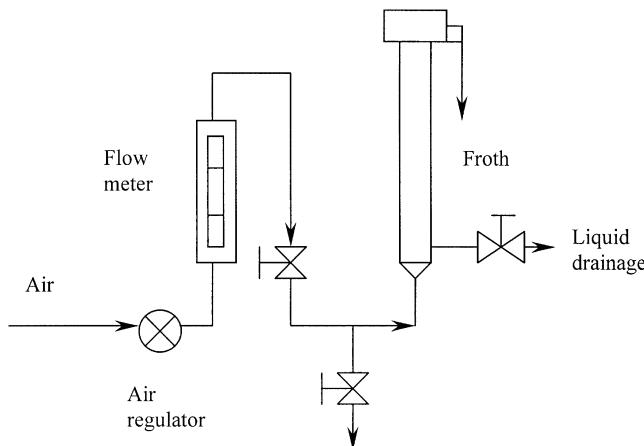


FIG. 2 Experimental set-up for emulsion flotation.

Analytical Method

The diesel was extracted from the emulsions by methylene chloride. Volumetrically equal parts of methylene chloride and emulsion were put into a separating funnel and the mixture was subject to 20 min of intense vibration. After clear stratification was manifest, the diesel-bearing methylene chloride at the bottom was separated from the emulsion. Two extractions were conducted for each sample.

The amount of oil extracted was determined by a gravimetric method (13). The methylene-chloride extract was transferred to a weighing beaker tared to ± 0.0001 g. Two washing cycles requiring about 2–3 mL of methylene chloride each were needed to wash out the beaker. About 30 mL of methylene-chloride extract was obtained for each sample. The methylene chloride in the top-covered beaker was allowed to evaporate slowly in a fume hood at 20°C by covering the beaker. After complete evaporation within approximately 10 h, the dish was reweighed and the amount of oil was determined. The evaporation was considered to be complete after no more weight loss could be observed over 2 h. The analytical method was verified by the evaporation of a known amount of diesel content of methylene-chloride extract. The analytical results were reproducible with an experimental error of less than 1%.

Experimental Methods

The emulsion-stability tests were conducted according to the method developed by Van Ham et al. (11) and Behie et al. (14). This method entailed sampling approximately 2 L of static emulsion in a beaker and following the diesel concentration at the given location of the beaker bottom. Because the rate of change of the concentration was proportional to the concentration at the sampling point, the emulsion stability could be described by a single value, i.e., a first-order stability constant (K_S). K_S is the relative variation in diesel concentration per unit of time. A desired amount of reagent was subsequently added to the emulsions and stirred for 6 min by a magnetic stirrer. The emulsions were then transferred to the flotation column.

RESULTS AND DISCUSSION

Emulsion Stability

Because free diesel oil is naturally floatable, the diesel–water emulsion stability greatly affected diesel flotation. Figure 3 shows the stability of the emulsions. The initial diesel content c_0 was 360 ppm, the diesel content at a given time is denoted by c , and the normalized diesel content is the ratio of c to c_0 . As can be seen from Fig. 3, the diesel content decreased to about 180 ppm (50% of its original value) after 15 h of settling, after which the settling rate

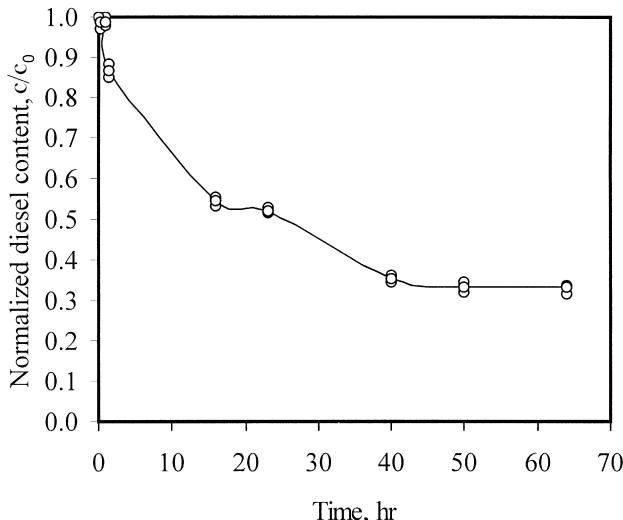


FIG. 3 Typical results from the stability test for a diesel/water emulsion.

decreased. When the diesel content was around 100 ppm, settling almost ceased. As a consequence, a simple settling method could not separate the diesel oil from the emulsions.

Because normal coalescence in the emulsion is prevented by the electric double layer surrounding the droplets, the potential difference between the dispersed droplets and the dispersed medium has a direct impact on the stability of the emulsion. When the contact potential at the oil–water interface decreases, the stability decreases commensurately. Factors such as pH and the presence of electrolytes in the emulsion can therefore play an important role in the stability of the emulsion and the ultimate floatability of the oil.

The effect of solution pH on the emulsion stability was studied with an initial diesel concentration of 128 ppm. The initial pH of the emulsion was 7.0. The pH was adjusted with hydrochloric acid and sodium hydroxide. Each pH value was changed from the initial emulsion pH of 7. The emulsion stability constant (K_S) increased with an increase in pH, as indicated in Fig. 4. In acidic environments, the emulsion stability constant was low and the diesel–water emulsions were stable. Conversely, in basic environments, the emulsion constant was high and the diesel–water emulsions were unstable.

Destabilization with an increase in pH did not occur gradually, because the emulsion stability decreased rapidly when the pH exceeded 11, leading to very

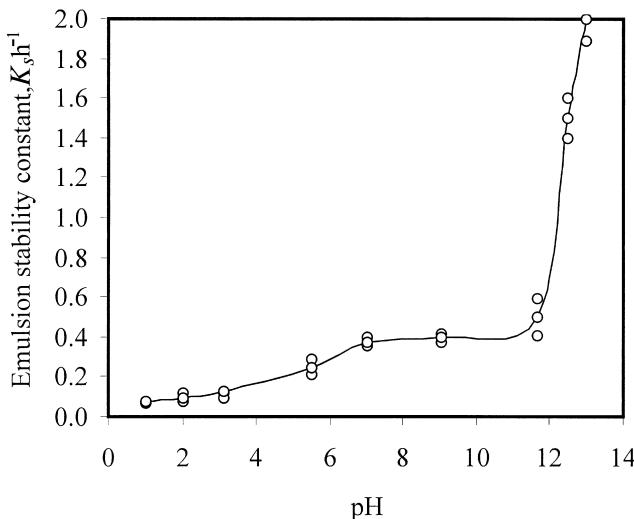


FIG. 4 Variation of emulsion stability constant with pH.

rapid destruction of the emulsions. At these high pH values, some small diesel flocs appeared and floated to the solution surface, and the emulsions became clear. This could be attributed to the uncommitted electron pairs of OH^- groups in basic environments that provided ligands for the hydrocarbons. The ionic strength decreased from 0.005 at pH 2 to 0.0005 at pH 11, and was too low to affect the emulsion stability.

The effect of the NaCl concentration on the emulsion stability was conducted at pH 7 and an initial diesel concentration of 128 ppm. The results are shown in Fig. 5, where it can be seen that the emulsion was gradually destabilized by an increase in the percentage of NaCl. The presence of the electrolyte affected the emulsion stability through its influence on the electric double layer of the droplets, as was also observed by Pondstabodee et al. (15), for example, and is an indication of the importance of collectors and other reagents in the flotation system.

The flotation recovery of diesel at different pH values is shown in Fig. 6 without surfactants. The initial diesel content was 128 ppm, the air-distributor size was 3, and the air-flow rate was 100 mL/min. As can be seen from Fig. 6, the diesel removal with pH was closely related to the change in the emulsion stability, and the more stable the emulsions, the lower the oil removal. The diesel could not be removed from the initial emulsion with a pH of 7 in the absence of surfactants.

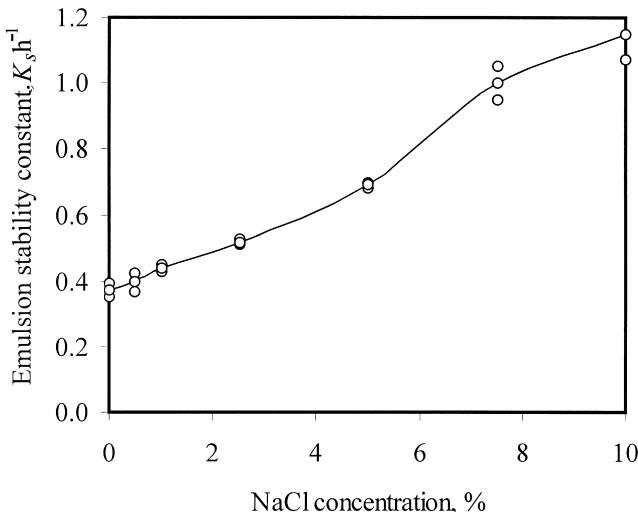


FIG. 5 Variation of emulsion stability constant with NaCl concentration.

Selection of Flotation Collectors

Flotation experiments were conducted with an anionic surfactant, sodium dodecyl sulphate (SDS, CMC 0.0082 M), and two cationic surfactants, octadecyl amine chloride (ODAC, CMC 0.0017 M) and cetyl trimethyl ammonium bromide (CTAB, CMC 0.0017 M).

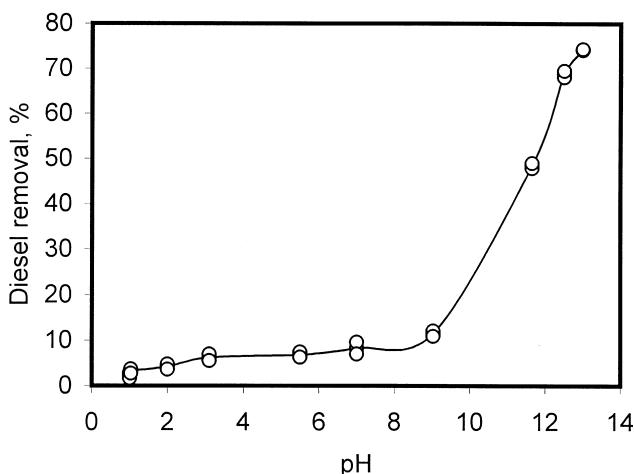


FIG. 6 Effect of emulsion pH on the diesel removal.

nium chloride (CTMAC, CMC 0.016M). An air-flow rate of 100 mL/min, a diesel-feed content of approximately 100 ppm, and an air distributor of sinter size 3 were used throughout. The results are shown in Fig. 7.

The oil could be removed effectively with the cationic surfactants, but less so with the anionic surfactant. The surfaces of most petroleum oil droplets (including diesel) are strongly negatively charged (11, 16) and as a result the negatively charged droplets could not adhere to the anionic surfactant. Instead, the diesel droplets might be solubilized in the micelles of the SDS. At low SDS concentrations, little oil was removed, but removal increased as the SDS concentration increased to the critical micelle concentration of SDS (about 100 ppm). Maximal oil removal could be obtained with the critical micelle concentration of the surfactant SDS, as was also observed by other investigators, such as Wungrattanasopon et al. (9).

The diesel droplets were strongly attracted to the cationic surfactants such as ODAC and CTMAC, and numerous flocs were observed after the cationic surfactants were mixed with the emulsions. The flocs, composed of oil and cationic surfactant, adhered to and floated with the bubbles, even at relatively low surfactant concentrations. The difference in performance between the two cationic frothers can be attributed to their frothing characteristics. Octadecyl amine chloride is a poor frother, and its froth lifetime is too short for the foam to establish itself in the froth zone. In contrast, CTMAC is a good frother, and

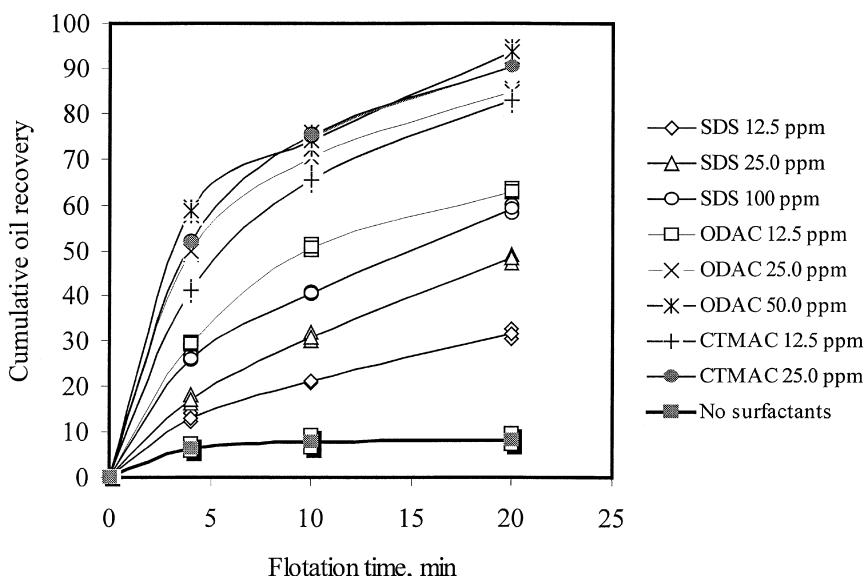


FIG. 7 Effect of surfactants on the diesel removal.

TABLE 1
Factors and Levels Investigated in the Oil-Removal and Kinetics Experiments

Run	Diesel content (ppm)	Air-flow rate (mL/min)	CTMAC dosage (ppm)	Sinter size (no.)
1	200	100	12.5	3
2	200	300	25	3
3	200	300	12.5	4
4	100	300	25	4
5	100	100	25	3
6	200	100	25	4
7	100	100	12.5	4
8	100	300	12.5	3

the loaded froth rapidly established itself in the froth zone. The bubbles in these froths were smaller and more stable than those in the froths associated with ODAC.

Based on the flotation kinetic data in Fig. 7, the first-order kinetics could be expressed as $\ln(1 - R) = kt$, in which R is the oil recovery, t flotation time, and k the slope of the line of $\ln(1 - R)$ vs t . This is consistent with the experimental results of several other investigators (11, 17). The first-order flotation rates shown in Table 1 were based on the removal rate during the first 4 min of flotation.

Effect of Operation Parameters on Oil-Flotation Performance

Four operating parameters were investigated in the experiments, air-flow rate, diesel content, CTMAC dosage, and air-distributor size. Two-level

TABLE 2
Results of Flotation Experiments with Factors Detailed in Table 1

Run	Diesel removal (%)				Flotation rate(%)				Diesel residual (ppm)			
	1	2	Mean	SD	1	2	Mean	SD	1	2	Mean	SD
1	80.07	78.63	79.35	1.02	9.90	10.22	10.06	0.23	35.0	37.0	36.0	1.41
2	91.81	93.17	92.49	0.96	16.38	16.14	16.26	0.17	14.5	13.5	14.0	0.71
3	80.85	78.35	79.60	1.77	14.18	13.94	14.06	0.17	32.5	33.5	33.0	0.71
4	98.98	97.52	98.24	1.05	16.19	15.83	16.01	0.25	2.7	3.3	3.0	0.42
5	91.36	90.00	90.68	0.96	12.28	11.92	12.10	0.25	6.6	7.4	7.0	0.57
6	99.90	98.12	99.01	1.26	14.64	14.42	14.53	0.16	1.8	2.2	2.0	0.28
7	83.91	82.39	83.15	1.07	10.81	10.61	10.71	0.14	11.2	11.8	11.5	0.42
8	87.25	86.25	86.75	0.71	11.48	11.08	11.28	0.28	15.0	16.0	15.5	0.71

fractional factorial experiments were conducted, the results of which are shown in Table 1. All the flotation runs were duplicated and the results are shown in Table 2. The SD of the results was within 2%, as shown in Table 2. The final diesel concentration was measured for all these experiments, and this index was the control target for the final effluent discharge after flotation.

As can be seen from Table 1, almost all the diesel could be removed, while the residual diesel levels could be reduced to less than 10 ppm at optimal conditions (run 6 in Table 1). Flotation can therefore be considered a highly effective method for the treatment of emulsions with cationic surfactants.

Only the main effects of the factors were considered in the ANOVA. As indicated by the results of the ANOVA in Table 3, only CTMAC had a significant influence on the percentage of removal of the diesel.

Although the residual diesel was influenced most significantly by CTMAC, it was only the air-flow rate that did not have a significant influence on the residual diesel (Table 4). The rate of removal was significantly influenced by all the factors. As in the case of the other two response variables, CTMAC again had the most pronounced influence on the flotation rate (Table 5). The significance of the sinter size of the distributor, especially by the air-flow rate and the surfactant concentration, was in accordance with the observation by Van Ham et al. (11).

TABLE 3
ANOVA of the Main Effects of the Factors on Diesel Removal

ANOVA ^{a, b, c}							
Unique method							
			Sum of squares	df	Mean square	F	Sig.
DIESREM	Main effects	(Combined)	717.520	4	179.380	14.724	.000
		Diesel	17.556	1	17.556	1.441	.255
		Air flow	6.002	1	6.002	.493	.497
		Sinter	28.837	1	28.837	2.367	.152
		CTMAC	665.124	1	665.124	54.595	.000
	Model		717.520	4	179.380	14.724	.000
	Residual		134.013	11	12.183		
	Total		851.532	15	56.769		

^a DIESREM by Diesel, Air flow, Sinter, CTMAC

^b All effects entered simultaneously

^c Because of empty cells or a singular matrix, higher-order interactions have been suppressed.

TABLE 4
ANOVA of the Main Effects of the Factors on Residual Diesel

		ANOVA ^{a, b, c}				
		Unique method				
		Sum of squares	df	Mean square	F	Sig.
DIESRES	Main effects	(Combined)	1953.500	4	488.375	14.891 .000
		Diesel	576.000	1	576.000	17.563 .002
		Air flow	20.250	1	20.250	.617 .449
		Sinter	132.250	1	132.250	4.032 .070
		CTMAC	1225.000	1	1225.000	37.352 .000
	Model		1953.500	4	488.375	14.891 .000
	Residual		360.760	11	32.796	
	Total		2314.260	15	154.284	

^a DIESRES by Diesel, Air flow, Sinter, CTMAC

^b All effects entered simultaneously

^c Because of empty cells or a singular matrix, higher-order interactions have been suppressed.

TABLE 5
ANOVA of the Main Effects of the Factors on Flotation Rate

		ANOVA ^{a, b, c}				
		Unique method				
		Sum of squares	df	Mean square	F	Sig.
FLOTRATE	Main effects	(Combined)	80.609	4	20.152	198.798 .000
		Diesel	5.784	1	5.784	57.058 .000
		Air flow	26.061	1	26.061	257.087 .000
		Sinter	7.868	1	7.868	77.617 .000
		CTMAC	40.896	1	40.896	403.431 .000
	Model		80.609	4	20.152	198.798 .000
	Residual		1.115	11	.101	
	Total		81.724	15	5.448	

^a FLOTRATE by Diesel, Air flow, Sinter, CTMAC

^b All effects entered simultaneously

^c Because of empty cells or a singular matrix, higher-order interactions have been suppressed.

Discussion and Conclusions

On the basis of this work, the following conclusions can be drawn.

- Emulsion pH had an important influence on emulsion stability. The emulsion stability decreased rapidly when the pH exceeded 11, leading to very rapid destruction of the emulsions. Accordingly, removal of diesel by flotation increased rapidly when the pH exceeded 11. The flotation performance was closely related to the emulsion stability; the electrolyte NaCl had some influence on diesel emulsion stability as well.
- Cationic surfactants such as ODAC and CTMAC were effective collectors for diesel emulsion flotation. In contrast, anionic surfactants such as SDS were less effective.
- Under optimal conditions, approximately 99% of the diesel could be removed, while the residual diesel could be reduced to less than 10 ppm. Optimal conditions consisted of high levels of cationic surfactant (CTMAC), low air-flow rates, and a large distributor size.

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