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Electrical Aspects of Adsorbing Colloid Flotation. XVIII. Flotation with Mixed Surfactant Systems

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Electrical Aspects of Adsorbing Colloid Flotation. XVIII. Flotation with Mixed Surfactant Systems

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Abstract

The effect of a number of alcohols in reducing the critical micelle concentration (cmc) of sodium dodecylsulfate (SDS) was measured conductimetrically; it was found that alcohols quite substantially reduce the cmc. This suggested that these alcohols might be used as relatively low-cost surfactant extenders in precipitate and adsorbing colloid flotation separations. It was found that *n*-butanol, *n*-hexanol, *n*-octanol, and octanoic acid quite markedly reduced the quantity of SDS needed for the flotation of ferric hydroxide flocs and of ferric hydroxide flocs in which Cu(II) had been coprecipitated/adsorbed. Octanoic acid was found to be the most effective cosurfactant for the adsorbing colloid flotation of Cu(II) with ferric hydroxide.

INTRODUCTION

Adsorptive bubble separation methods have been used for the concentration of minerals from ore pulps for many years (*1*, *2*). More recently they have been studied for use in concentrating trace elements for analysis, treating industrial wastewaters, and for recovering metals from ore leaching solutions; a number of reviews describe this work (*3*–*11*). We recently published a foam flotation procedure for the removal of antimony from aqueous systems (*12*). McIntyre et al. (*13*) showed the feasibility of this method at the pilot-plant scale for waste treatment, and have made a cost analysis of the technique. One of the major recurring costs in these flotation methods is the cost of the surfactants needed to make the particles hydrophobic, so that they will attach to bubbles and be floated from the solution. One might hope that the use of mixed surfactant systems could reduced the cost of surfactants; the use of such mixed surfactant systems in ore flotation has been common practice for many years (*1, 2*) but apparently this has not been done in adsorbing colloid and precipitate flotation. The adsorption of binary surfactant mixtures on surfaces was investigated theoretically earlier by us (*14, 15*), and those results led to the present experimental study.

In this paper we investigate the use of alcohols or an intermediate chain-length fatty acid as cosurfactants with sodium dodecyl sulfate (SDS). Many of the factors which are involved in the formation of condensed monolayers of surfactant on precipitate surfaces are also involved in surfactant micelle formation—van der Waals forces, hydrophobic interactions, etc. In view of this, some conductimetric titrations were carried out to determine the effects of alcohol concentration and chain length on the critical micelle concentration (cmc) of SDS. The existence of aggregates in detergent solutions above the cmc has been recognized for many years; plots of a number of properties of solutions of amphipathic electrolytes such as SDS show abrupt changes in the concentration dependences of these properties at the cmc (*16*). Shinoda showed that alcohols such as *i*-pentanol, *n*-hexanol, and *n*-heptanol reduce the cmc's of alkanoate soaps (*17*). Recently Shah and Mahmood reported on an extensive study of the effects of short-chain alcohols (ethanol, *n*-propanol, and *n*-butanol) in aqueous solution of the cmc's of sodium and lithium dodecyl sulfates (*18*). They used surface tension, refractive index, electrical conductivity, viscosity, and UV spectroscopic measurements. Electrical conductivity measurements are easily carried out and lead to relatively accurate determinations of the cmc if the solution does not contain high concentrations of other electrolytes. These results indicated that alcohols very markedly enhanced micelle formation, and encouraged us to carry out flotation experiments with mixtures of SDS and an alcohol (or with octanoic acid) as surfactant and with ferric hydroxide as the floc to be removed. These results in turn encouraged us to test the removal of Cu(II) by flotation with mixed surfactant systems and ferric hydroxide; Chatman et al. (*19*) previously showed that Cu(II) is readily removed from aqueous systems by adsorbing colloid flotation with ferric hydroxide and sodium dodecyl sulfate.

EXPERIMENTAL

The SDS used in this work was obtained from Sigma Chemical Co. and Fluka AG; both samples were reportedly more than 99% pure. Both gave cmc's by conductimetric titration of approximately 8.32 mmol/L at room temperature, in good agreement with the reported value (*20*) of 8.08 mmol/L at 25°C. Fisher certified grade 1-butanol, *t*-butanol, 1-pentanol, 1-octanol, 2-octanol, 1-decanol, 1-dodecanol, ferric chloride, and sodium chloride were used. The 1-hexanol and octanoic acid were from Eastman (practical grade) and the cupric sulfate was from Baker and Adamson. All solutions were made with deionized water.

Conductivity measurements were made with a YSI Model 31 conductivity bridge. A stock solution containing 20.000 g/L SDS was prepared and used as the titrant. Stock solutions of the various alcohols in water were made up by volume (butanols) or by preparing saturated solutions of the alcohols in water at 25°C and calculating the concentrations of these solutions from the data compiled in Ref. *21*. These solubility data are given in Table 1.

An aliquot of the desired alcohol solution was placed in a 500-mL beaker and diluted to 200 mL for titration. The solution was stirred magnetically, and small volumes of the SDS titrant solution were added from a 50-mL buret. The volumes of added titrant and the corresponding conductivities were recorded. Titrant was added until well beyond the point at which the changes in conductivity per milliliter of added titrant had become relatively small and the known cmc of SDS had been exceeded. A typical plot of specific conductivity versus SDS concentration is shown in Fig. 1(a). The break at the cmc is quite clearly seen. The data (volumes of added SDS titrant and specific conductivities) were entered into a Zenith Model 150 microcomputer. The computer program used calculated SDS concentrations in the solution being titrated and displayed the following plots: (a) specific conductance versus concentration of SDS (mmol/L), (b) specific conductance versus the square root of the SDS concentration, and (c) equivalent conductance versus the square root of the SDS concentration. The computer program calculated the cmc's by first fitting straight lines by least squares to the roughly linear segments of the plots on either side of the breaks in the curves (see Figs. 1a and 1c) and then calculating the point of intersection of these two lines. (As seen in Fig. 1b, the plots of specific conductance versus the square root of the SDS concentration were not sufficiently linear to be suitable for calculating cmc's.) The abscissa of the point of intersection of

TABLE 1
Solubilities of Alcohols in Water

Compound	g/100 mL	mol/L	Mole fraction
1-Pentanol	2.20	0.2496	0.00458
1-Hexanol	0.60	0.0587	0.00106
1-Octanol	0.054	0.00415	0.0000747
2-Octanol	0.40	0.0307	0.000555
1-Decanol	0.0037	0.00023	4.2×10^{-6}
1-Dodecanol	0.0004	0.00002	3.9×10^{-7}

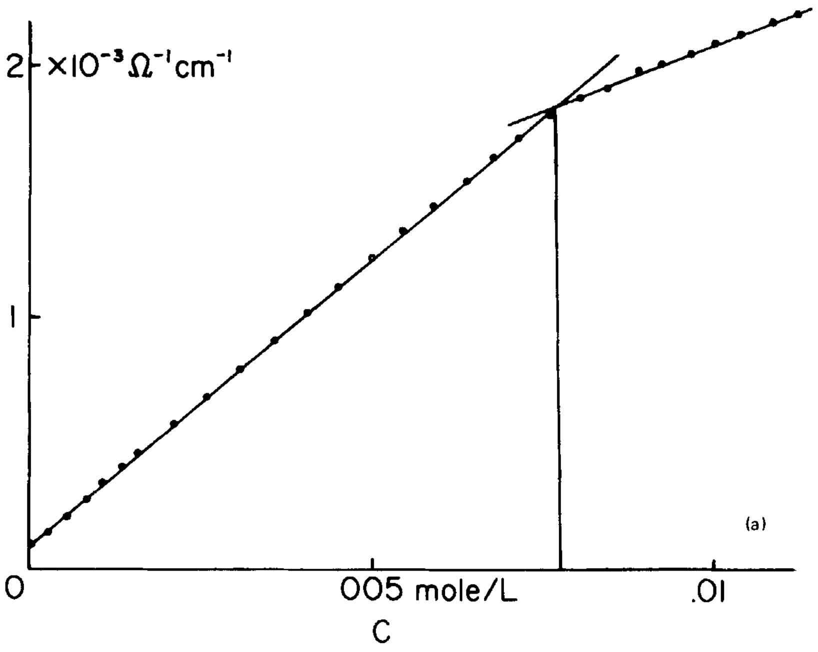
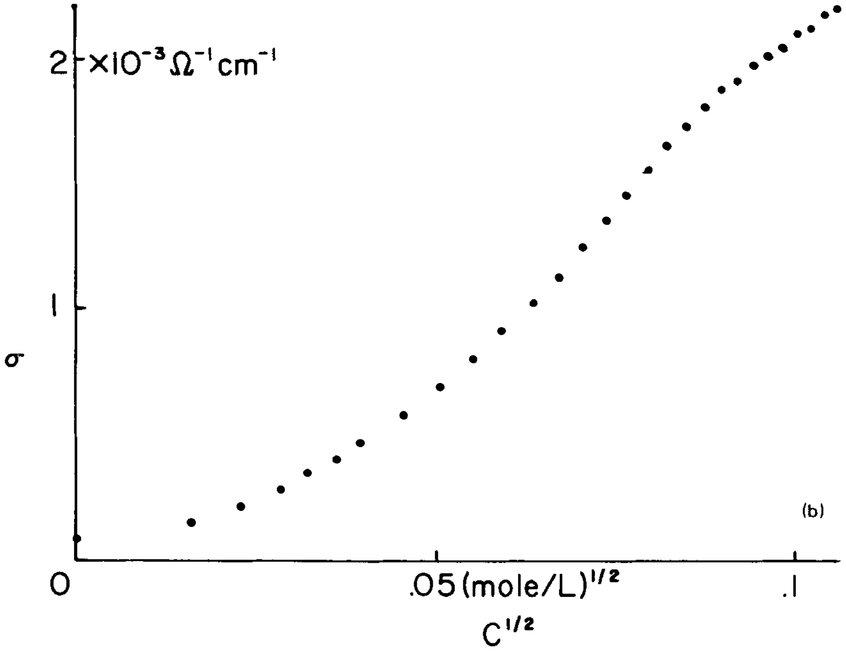
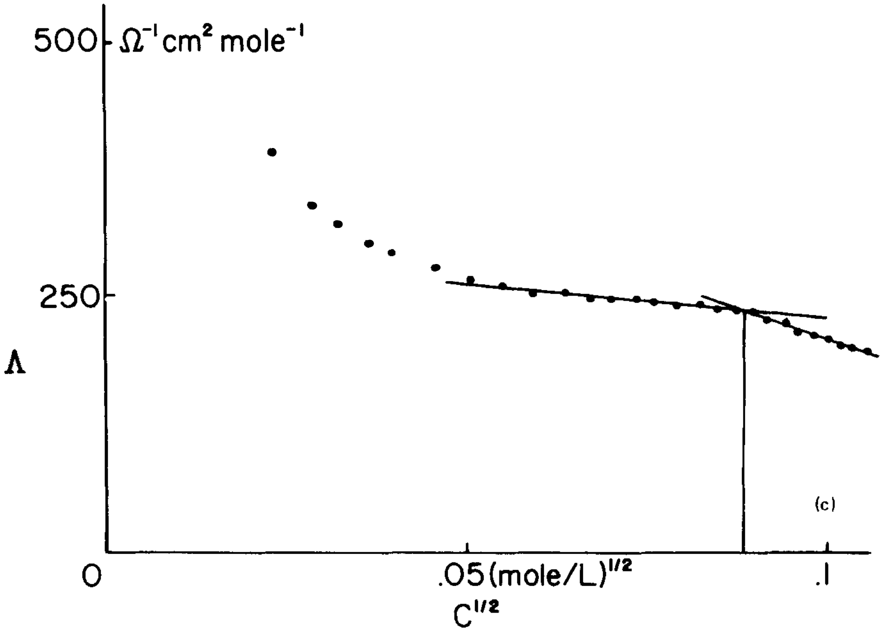


FIG. 1. Determination of critical micelle concentrations by conductivity titration. (a) Plot of specific conductance against surfactant concentration, C. (b) Plot of specific conductance versus $C^{1/2}$. (c) Plot of equivalent conductance versus $C^{1/2}$. The initial concentration of 1-butanol in this run is 5.0×10^{-4} mol fraction.


2
×10^{-3} Ω^{-1}cm^{-1}
1
σ
(b)
0
.05 (mole/L)$^{1/2}$
.1
C$^{1/2}$


500
Ω^{-1}cm^{2} mole^{-1}
250
Λ
(c)
0
.05 (mole/L)$^{1/2}$
.1
C$^{1/2}$

the two line segments is the cmc (for plots of specific conductivity versus SDS concentration) or the square root of the cmc (for plots of equivalent conductivity versus square root of the concentration). It was found that plots of specific conductivity versus concentration yielded the most reproducible cmc's, and the cmc's reported here were all calculated by that method. Good agreement between cmc's obtained in that way and cmc's obtained graphically was found. We observed that the breaks in the plots of equivalent conductivity versus the square root of the SDS concentration were often hard to see at higher alcohol concentrations.

The flotation apparatus used was built in our laboratory, and is diagrammed in Fig. 2. House air was filtered and humidified, then blown through the column at a rate of about 100 mL/min. Air flow rates were measured with a soap film flow meter and stopwatch with liquid in the column. A fritted glass disk of medium pore size was sealed into the bottom of the column to disperse the air as fine bubbles. It was possible to place a magnetic stirrer immediately beneath the column to disperse the bubbles still further; this did not seem to yield any significant improvement in these results (in which the separations, if they work at all, are extremely rapid), and was not done for most of the runs. Stock solutions of ferric chloride [10.000 g/L Fe(III)], SDS (20.000, 10.000, and 1.000 g/L), and cupric sulfate [12.5 g/L Cu(II)] were prepared. Solutions of cosurfactants used were 1-octanol (saturated, 540 mg/L, and 200 mg/L), 1-butanol (200 mg/L), 1-decanol (saturated, 37 mg/L), and 1-dodecanol (saturated, 4 mg/L).

Flotation runs were carried out as follows. Approximately 100–250 mL deionized water was placed in a 1-L beaker and the desired quantities of Fe(III) solution and alcohol (or octanoic acid) solution were added. The test solution was then diluted up to a volume of about 480 mL, placed on a magnetic stirrer, and the pH adjusted with 1 *M* NaOH solution to a value roughly 0.5 above the desired final value. Stirring was continued for approximately 8–12 min, after which the desired quantity of SDS solution was added, the volume of the solution was brought to 500 mL, and the pH of the solution was adjusted to the desired value by dropwise addition of 0.1 *M* NaOH or nitric acid. This dilute slurry was then stirred slowly for 2–5 min, after which it was poured into the column, through which air was already flowing. A stopwatch was immediately started, and the changes in color of the solution and the presence or absence of suspended solids were noted visually several times during the course of the run. The column was brightly illuminated and a piece of white paper was held behind the solution in the column to facilitate detecting color and any suspended particles. When copper removal was studied and the effects of added salt on this separation tested, the Cu(II) and sodium

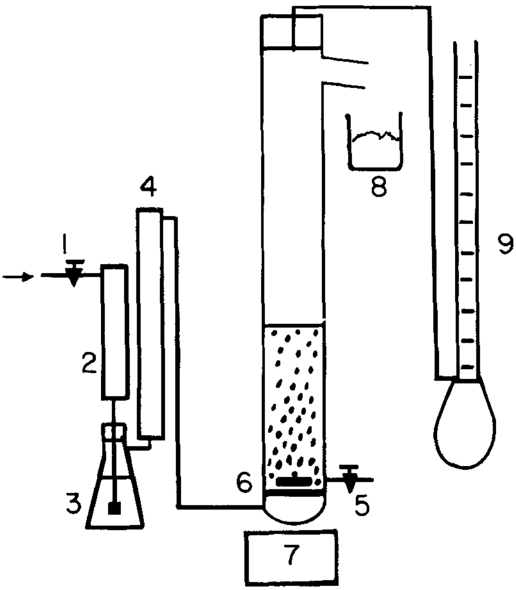


FIG. 2. The flotation apparatus. (1) Air inlet, (2) ascarite tube, (3) humidifier, (4) glass wool filter, (5) drain tap, (6) fritted glass disk, (7) magnetic stirrer, (8) foam catcher, (9) soap film flow meter.

nitrate were added to the slurry just after the addition of the ferric chloride solution. The initial concentration of Cu(II) used was always 25 mg/L. After removal by flotation of ferric hydroxide [together with coprecipitated and adsorbed Cu(II)], samples were collected from the sample tap at the bottom of the column for residual copper analysis. The sample tap was purged before each sample was collected by wasting approximately 5 mL of solution from the column. The collected samples were analyzed for Cu by atomic adsorption at 324.7 nm; a Perkin-Elmer Model 305B instrument was used.

RESULTS

The results of a typical conductivity for determining cmc's in solutions containing both SDS and alcohols have already been shown in Fig. 1. The values of the cmc's obtained by this procedure are given Table 2, and are shown graphically in Fig. 3. The data indicate that most of the alcohols lower the cmc of SDS very markedly. The effects of 1-decanol

TABLE 2
Effects of Alcohols on the cmc of Sodium Dodecyl Sulfate

Alcohol	Mole fraction alcohol	cmc of SDS (mol/L $\times 10^{-3}$)
1-Butanol	0.00	8.32
	5.0×10^{-4}	7.74
	7.5×10^{-4}	7.32
	1.0×10^{-3}	7.29
	2.0×10^{-3}	6.21
	3.0×10^{-3}	5.19
	4.0×10^{-3}	4.28
	5.0×10^{-3}	3.68
t-Butanol	1.0×10^{-3}	7.78
	2.0×10^{-3}	7.24
	3.0×10^{-3}	6.63
	4.0×10^{-3}	6.21
	5.0×10^{-3}	5.55
1-Pentanol	1.2×10^{-3}	6.44
	2.3×10^{-3}	4.56
	3.5×10^{-3}	3.16
	4.6×10^{-3}	3.28
1-Hexanol, 1st set	1.33×10^{-4}	6.52
	2.65×10^{-4}	4.69
	3.98×10^{-4}	5.27
	5.30×10^{-4}	4.31
	7.95×10^{-4}	4.40
	1.06×10^{-4}	3.69
2nd set	2.7×10^{-4}	6.08
	5.3×10^{-4}	4.74
	8.0×10^{-4}	4.26
	1.06×10^{-3}	3.10
1-Octanol	1.87×10^{-5}	7.29
	3.74×10^{-5}	5.50
	5.61×10^{-5}	4.59
	7.47×10^{-5}	2.55
2-Octanol	1.4×10^{-4}	7.03
	2.8×10^{-4}	5.94
	4.2×10^{-4}	4.97
	5.6×10^{-4}	2.55
1-Decanol	4.2×10^{-6}	7.82
1-Dodecanol	3.9×10^{-7}	8.18

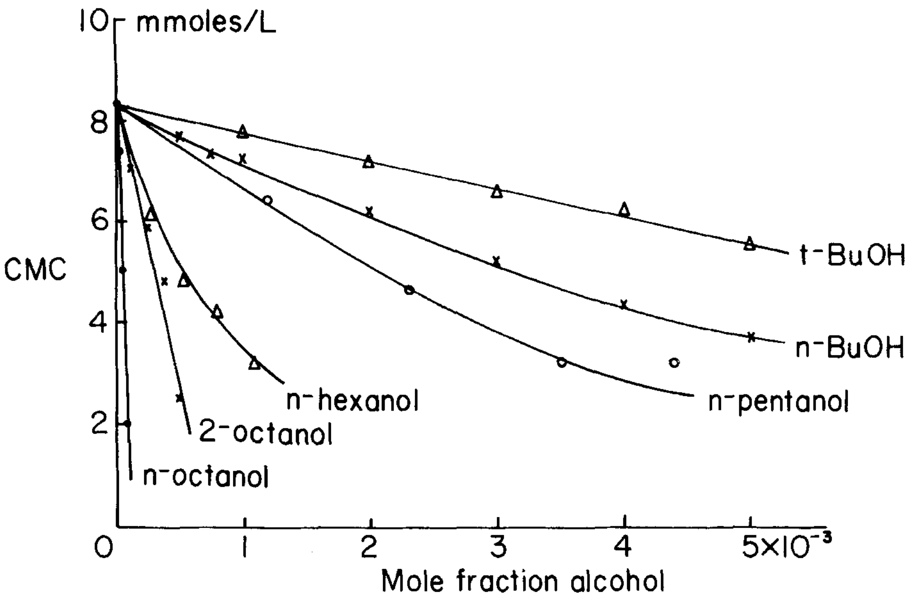


FIG. 3. Critical micelle concentrations of sodium dodecyl sulfate (SDS) as a function of added alcohol concentration.

and 1-dodecanol are slight, probably because of their very low solubilities in water. We can draw a number of conclusions from Fig. 3, where the cmc's are plotted as functions of the initial concentrations of the alcohols. First, as long as the solubility of the alcohol is sufficient, the effectiveness of an alcohol in decreasing the cmc increases with increasing chain length. Second, as indicated by the 1- and 2-octanol results and the 1- and *t*-butanol results, normal alcohols are more effective than branched or secondary alcohols of comparable size. Both of these conclusions are quite consistent with a picture of mixed micelle formation, in which alcohol molecules and dodecylsulfate ions are present in the micellar structure. Increased branching interferes with efficient packing and decreases the extent of the van der Waals interactions; increased alkyl chain length increases the van der Waals and hydrophobic interactions. Our third conclusion is that the magnitude of this effect is quite large for *n*-hexanol and the octanols, and that these alcohols might be suitable surfactant extenders for SDS in flotation processes.

Flotation of freshly prepared ferric hydroxide flocs was carried out with SDS and 1-butanol, 1-hexanol, 1-octanol, and octanoic acid. All four of these substances are substantially cheaper than SDS, and it was felt that the effectiveness of 1-octanol in enhancing micelle formation with SDS justified the investigation of both it and octanoic acid as surfactant extenders in flotation separations using SDS. In these runs the flotation was followed visually, and in all cases the pH was 5.5. In Figs. 4–8 the circles indicate complete removal of the floc within 3 min, and the crosses indicate failure of the flotation run to remove the floc completely within 3 min.

In Fig. 4 we see that 100 mg/L Fe(III) is successfully removed by 20 mg/L SDS alone, but not by 15 mg/L. The addition of relatively large quantities of 1-octanol is seen to permit the removal of the floc at SDS concentrations as low as 5 mg/L, and it is apparent that there is a wide range of concentrations of both SDS and 1-octanol which allow successful flotation. Figure 5 shows essentially similar results for the flotation of 200 mg/L Fe(III); flotation with SDS alone is effective down to a concentration of 60 mg/L but not at 40 mg/L, while added 1-octanol extends the concentration range over which SDS is effective down to 5 mg/L. The concentrations of 1-octanol used in both of these sets of runs include values which are higher than would be desirable for industrial use for the separation; they demonstrate that the separation is not sensitive to overloading with 1-octanol. It is evident that the total quantity of surfactant can be very markedly reduced by using SDS–1-octanol mixtures; since 1-octanol costs slightly over half as much as SDS ($8.53/kg as compared to $15.83/kg in the Aldrich catalog), sustantial savings in surfactant costs could be achieved by using mixed surfactants.

The effectiveness of 1-hexanol–SDS mixed surfactant systems in the flotation of 100 mg/L Fe(III) is shown in Fig. 6. We see here that, as with 1-octanol, on a weight basis the mixed surfactants are quite a bit more effective in making the ferric hydroxide flocs floatable. The cost of *n*-hexanol is $5.67/kg (Aldrich), so that quite substantial savings in surfactant costs could be realized. Flotation results for the removal of 100 mg/L Fe(III) with SDS and 1-butanol are shown in Fig. 7. Despite the relatively low efficiency of *n*-butanol in reducing the cmc of SDS, we see that it results in quite substantial reductions in the amount of SDS required to float the floc. Since *n*-butanol costs $5.20/kg, roughly a third as much as SDS, its use as a surfactant extender could result in significant savings.

Figure 8 demonstrates the effectiveness of SDS–octanoic acid mixtures in the flotation of 100 mg/L Fe(III). It is seen that octanoic acid is a very effective cosurfactant with SDS; despite the fact that it is somewhat more expensive than the alcohols ($12.52/kg from Aldrich), it might well be competitive with them. We note that salts of fatty acids have long been used as flotation agents in their own right (*1, 2*).

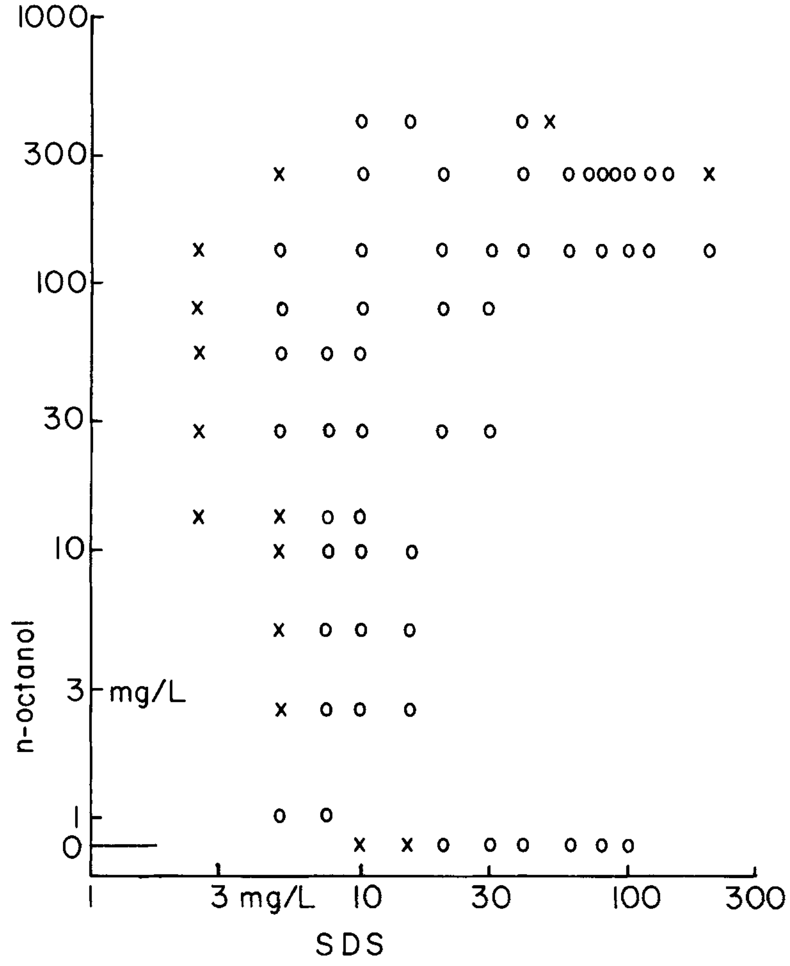


FIG. 4. Flotability of $Fe(OH)_3$ in the presence of various concentrations of SDS and 1-octanol. (O) Floated; (X) failed to float or flotation incomplete after 3 min. 100 mg/L Fe(III) initially, pH = 5.5, air flow rate = 100 mL/min. This is a log-log plot.

The above data indicate the ability of these mixed surfactant systems to remove flocs. Two of these systems were then used to remove Cu(II) (concentration 25 mg/L) from aqueous solutions. Octanoic acid-SDS and *n*-butanol-SDS were selected for this work. The results are presented in Table 3. They indicate that Cu(II) is not effectively removed at or below a pH of 6.0, nor is it effectively removed at or above pH of 7.0. At around a

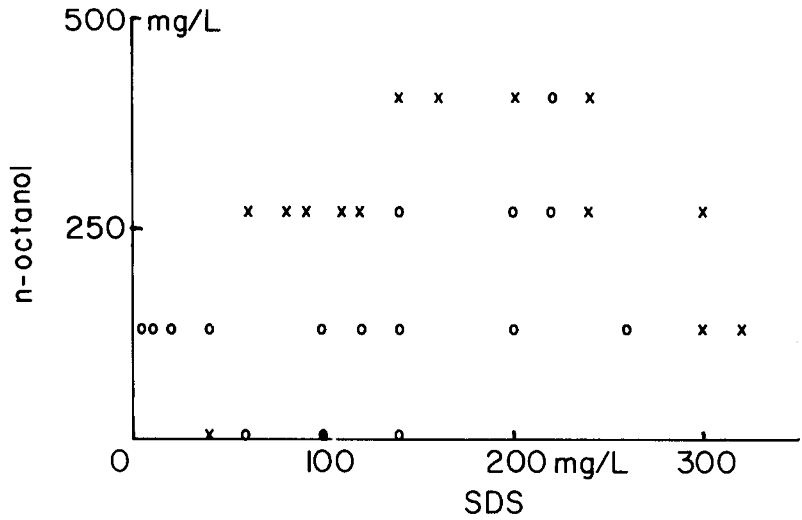


FIG. 5. Same as Fig. 4, but with 200 mg/L Fe(III) initially. A linear plot.

pH of 6.5, however, where about 20 mg/L SDS alone is required to bring about successful removal, a mixed surfactant system containing 5 mg/L SDS and 5 mg/L octanoic acid allows one to achieve removals of copper down to well below 1 mg/L (the drinking water standard for copper). The presence of Cu(II) appears to interfere with *n*-butanol's ability to act as a cosurfactant, since many of the Cu-containing flocs were not floated by SDS-*n*-butanol mixtures under conditions which had resulted in the flotation of pure ferric hydroxide. At the lower pH's the solubility of $Cu(OH)_2$ is sufficient large that good separations are not obtained even when the ferric hydroxide floc is floated. At high pH's, presumably the floc is negatively charged due to adsorption of excess hydroxide and/or desorption of protons, so that it is unable to attract the negative dodecyl sulfate surfactant ions; the floc therefore remains hydrophilic and is unable to float. The effects of pH on copper removal are illustrated in Fig. 9; these runs were all made with 5 mg/L octanoic acid, 5 mg/L SDS, 25 mg/L Cu(II), and 100 mg/L Fe(III). The effects of added salt (sodium nitrate) are shown in Fig. 10. In these runs the pH was 6.5, and the surfactant system was 5 mg/L each of octanoic acid and SDS. We see that the removal of Cu(II) decreases in efficiency with increasing ionic strength for this system, just as it does when SDS is used alone as the

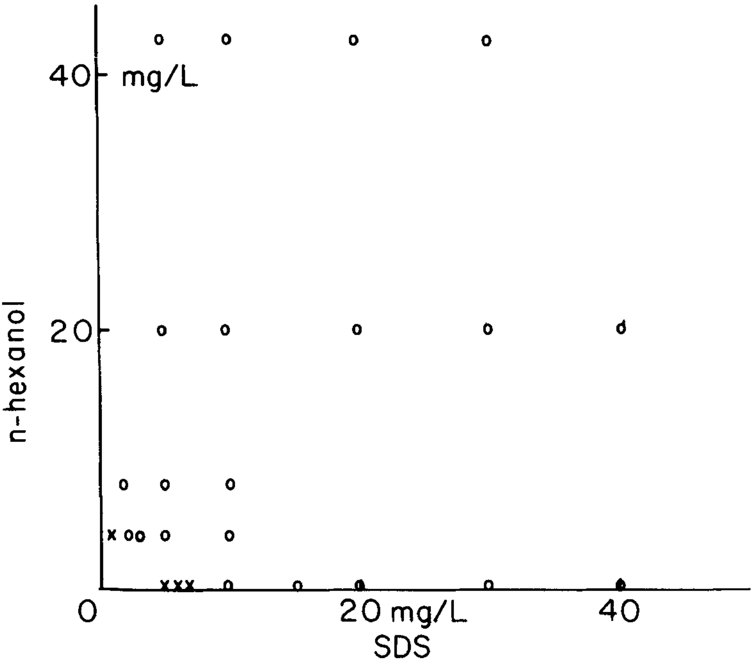


FIG. 6. Flotability of $Fe(OH)_3$ in the presence of SDS and 1-hexanol. 100 mg/L Fe(III) initially, pH = 5.5, air flow rate = 100 mL/min.

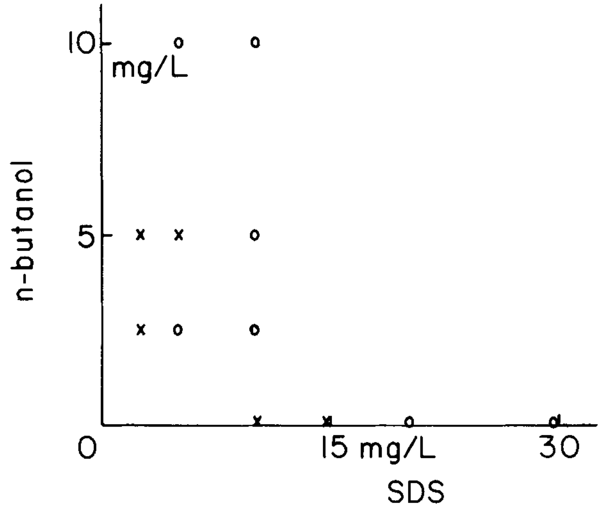


FIG. 7. Flotability of $Fe(OH)_3$ in the presence of SDS and 1-butanol. Conditions as in Fig. 6.

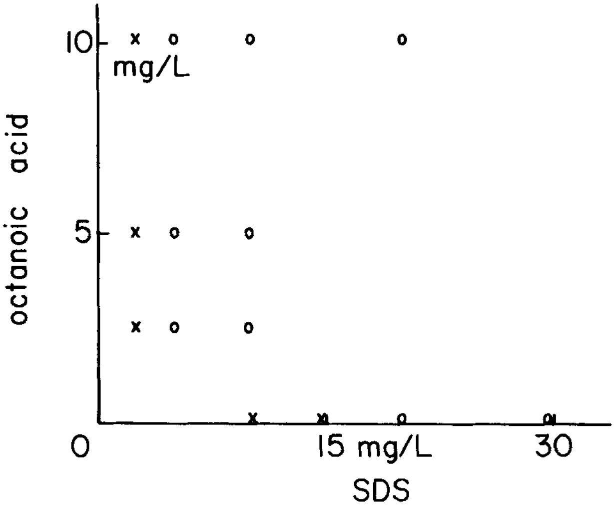


FIG. 8. Flotability of $Fe(OH)_3$ in the presence of SDS and octanoic acid. Conditions as in Fig. 6.

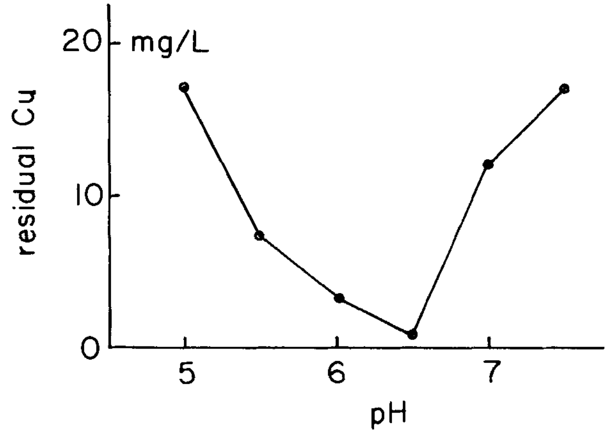


FIG. 9. Effect on pH on Cu(II) removal by adsorbing colloid flotation with Fe(III) (100 mg/L), 5 mg/L SDS, 5 mg/L octanoic acid. The initial Cu(II) concentration was 25 mg/L. Air flow rate = 100 mL/min.

TABLE 3
Compiled Results for the Removal of Cu(II)[a]

pH	Concentration of surfactants (mg/L) SDS	1-Butanol	Octanoic acid	Time sample collection (min)	Floc removed, yes/no	Residual Cu(II) (mg/L)
5.0	5	2.5	5	10	n	17.0
5.0	5	2.5		10	y	4.5
	5	2.5		10	y	6.8
	5		2.5	10	y	8.0
	5		5.0	10	y	7.4
6.0	5		5.0	10	y	2.6
6.5	20			10	y	0.2
	20			10	y	0.3
	5	2.5		10	n	12.0
	5	2.5		15	n	22.5
	5	2.5		15	n	23.5
	5		2.5	15	n	5.5
	5		2.5	15	n	2.5
	5		2.5	15	n	14.0
	5	5		18	n	15.0
	5	5		12	n	8.3
	5		5	10	y	0.4
	5		5	7	y	0.6
	5		5	10	y	0.7
	5		5	10	y	0.7
	5		5	10	y	0.6
7.0	5		5	15	n	12.3
7.5	5		5	12	n	17.0

[a] Initial Cu(II) concentration, 25 mg/L; Fe(III) concentration, 100 mg/L. y = yes, successful run, i.e., the final solution became clear within 3 min. n = not a successful run, i.e., the solution did not clear up. Air flow rate was 100 mL/min in all experiments.

surfactant (*19*). Further work on this needs to be done using more strongly binding surfactants such as dodecyl phosphate (*22*) or dodecyl iminodiacetate (*23*), which would be expected to compete more aggressively with nonamphipathic anions for binding sites on the floc.

We conclude that the utility of mixed surfactant systems in floc and precipitate flotation has been demonstrated, and that use of mixed surfactant systems could result in substantial surfactant cost reductions in the industrial use of these adsorptive bubble separation techniques.

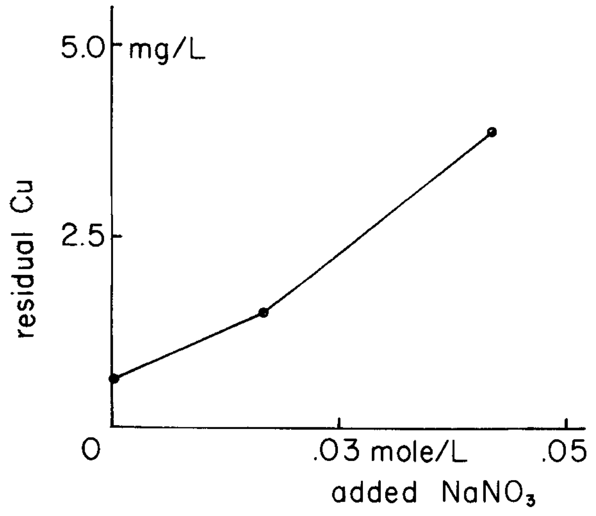


FIG. 10. Effect of added $NaNO_3$ on Cu(II) removal. pH = 6.5, initial Cu(II) concentration = 100 mg/L, 5 mg/L SDS, 5 mg/L octanoic acid, air flow rate = 100 mL/min.

Acknowledgment

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