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Electrical Aspects of Adsorbing Colloid Flotation. XXI. Flotation with Dodecylphosphate/*n*-Hexanol Mixtures

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Abstract

The adsorbing colloid flotation of Pb(II), Cd(II), and Cu(II) with ferric hydroxide floc and the mixed surfactant system sodium dodecylphosphate (SDP)/*n*-hexanol was investigated. Good removals of Pb and Cu were obtained; removal of Cd was less satisfactory. The effects of interfering anions (sulfate, oxalate, silicate, phosphate) were studied; higher concentrations of these ions could be tolerated with SDP/*n*-hexanol than with sodium dodecylsulfate. Measurements of the cmc of SDP were made; low solubility prevented determinations in the pH range 4–8.5.

INTRODUCTION

Flotation methods have been used for the concentration of minerals from ores for about 70 years, and a number of excellent monographs exist (*1*, *2*). The extension of this approach to the flotation of precipitates, ions, ion pairs, etc. for possible application in the concentration of trace elements for analysis, the treatment of industrial wastewaters, and the recovery of metals from ore leaching solutions is substantially more recent (*3*, *4*), dating from Sebba's pioneering work (*5*).

The use of surfactant mixtures in the flotation of minerals is common practice, but this technique has not been employed to any extent in precipitate or adsorbing colloid flotation. We investigated some of the theoretical aspects of flotation with mixed surfactant systems earlier (*6*, *7*), and these results encouraged us to carry out a study of the flotation of ferric hydroxide and of ferric–cupric hydroxide flocs with mixtures of sodium dodecylsulfate and several different cosurfactants (intermediate chain-length alcohols and octanoic acid) (*8*). That study concluded that surfactant costs could be greatly reduced without interfering with the effectiveness of the separations by the use of mixed surfactant systems.

Okamoto and Chou (*9*) and others showed the advantages of using chelating surfactants in ion flotation, and we found that a number of these chelating or complexing surfactants are effective in the flotation of precipitates and flocs; these include dodecyliminodiacetate (*10*), beta-hydroxy oximes (LIX reagents) (*11*), and monolaurylphosphate and monolauryldithiocarbamate (*12*). These have the advantage of being less easily displaced by adsorbable nonamphipathic ions, but they are also quite costly. It therefore seems advisable to see if these expensive surfactants can be extended with cheaper cosurfactants such as normal alcohols of intermediate chain-length.

The adsorbing colloid and/or precipitate flotation of lead (*13–15*), cadmium (*14*, *16*, *17*), and copper (*18–21*) have all been studied rather extensively; our references are representative, not exhaustive. It therefore seemed advisable to see if mixed surfactant systems containing chelating surfactants and extenders could be found which might provide some advantage over the earlier methods in terms of reduced interferences and possibly lower costs. Mumallah (*12*) previously investigated the use of dodecyl phosphate as a flotation agent; the foaming properties of this compound alone were inadequate, but he found that using Triton X-100 [an alkylbenzene poly(oxyethylene) alcohol] as a cosurfactant permitted good separations. Triton X-100 is rather expensive; however, the utility of dodecylphosphate for flotation would be greatly increased if a cheaper cosurfactant could be found.

The pK_a's of mono-*n*-butylphosphoric acid are 1.89 and 6.84, and the pK_a of di-*n*-butylphosphoric acid is 1.72 (*22*, *23*). One would expect the pK_a's of mono- and didodecylphosphoric acids to be quite similar to these values.

The objectives of the present study were to determine the critical micelle concentration (cmc) of dodecylphosphate, including the dependence of the cmc on pH, and to investigate the use of *n*-hexanol as a cosurfactant with sodium dodecylphosphate.

EXPERIMENTAL METHODS AND RESULTS

Lauryl alcohol (99%) was obtained from Aldrich. Phosphorus oxychloride (reagent grade) and *n*-hexanol (practical grade) were from

Eastman Kodak. Reagent grade NaOH, Na_2SO_4, NaH_2PO_4, Na_2SiO_3, $NaHC_2O_4$, $Fe(NO_3)_3$, $Cd(NO_3)_2 \cdot 4H_2O$, and $Pb(NO_3)_2$ were obtained from Fisher Scientific.

The batch foam flotation column has been described earlier (*8*). Infrared spectra were obtained on a Perkin-Elmer Model 727 instrument, and atomic absorption measurements were made on a Perkin-Elmer Model 403 atomic absorption spectrophotometer. Lead was analyzed for at 283.3 nm and cadmium at 228.8 nm with an air-acetylene flame. Conductivity measurements were made on a YSI Model 31 conductivity bridge.

Stock solutions (1000 mg/L) of Fe(III), Pb(II), and Cd(II) were prepared from the nitrates. *n*-Hexanol (200 ppm, v/v) was prepared by diluting the appropriate amount of *n*-hexanol to 1.000 L with deionized water and stirring for several hours. Stock solutions of phosphate, silicate, oxalate, and sulfate containing 1000 mg/L were made up from Na_2HPO_4, $NaSiO_3$, $NaHC_2O_4$, and Na_2SO_4 and deionized water. A 1000 mg/L stock solution of sodium dihydrogen dodecylphosphate was also made up.

The preparation of monolauryl phosphate was carried out according to the procedures described by Christiansen (*24*) and Plimmer and Burch (*25*) with slight modifications as suggested by Mumallah (*12*). *n*-Dodecanol (0.1 mol) was added to 60 mL of anhydrous ethyl ether and stirred. Slightly more than 0.1 mol of $POCl_3$ was then slowly added with stirring, and the solution allowed to reflux with stirring for 2 h. Stirring was continued for an additional 30 min to liberate HCl. The mixture was cooled to room temperature and the ether evaporated under vacuum (water aspirator). Approximately 100 mL of 1.0 *M* aqueous NaOH was then slowly added to the oily residue; the mixture was chilled in an ice bath during this step. Excess liquid was then decanted and discarded and the white, soapy solid was collected and dried at room temperature for several days. It was then placed in a desiccator ($CaCl_2$) and dried to constant weight.

The infrared spectrum of monolauryl (dodecyl) phosphate (SDP) was recorded from a Nujol mull and from a KBr disk. Observed absorbances were in agreement with those observed by Mumallah (*12*), Ferraro et al. (*26*), and Thomas (*27*). No absorbance was observed in the 3200 cm^{-1} region which would indicate the presence of water, in agreement with Mumallah's finding (*12*); the absence of water was further verified by equivalent weight determination. Absorption bands observed (cm^{-1}) are as follows: 2955, 2918, 2850, 1140, 1070, 958 (strong); 1200 (broad and strong); 1025 (broad and very strong); 1460, 890, 783 (medium); and 1040 and 995 (shoulders).

The equivalent weight of the prepared SDP was determined by titration with 0.0914 *N* NaOH. Approximately 0.4000 g SDP was weighed out and placed in 250 mL of hot deionized water; the mixture was then stirred for 30 min, by which time it was near room temperature. The solutions remained turbid, but no solid particles were visible. The initial pH of the solutions was approximately 3.2. A typical titration curve is shown in Fig. 1. The first equivalence point is at approximately pH 5.1; the second, at pH 9.1. About 8 mL NaOH was required to reach the first equivalence point, with an additional 17 mL needed to reach the second. Evidently the preparation contained both dihydrogen laurylphosphate and sodium hydrogen laurylphosphate. The average molecular weight of the sample was calculated from the difference between the volume of NaOH required to reach the second equivalence point; this was 279 ± 5. The molecular weights of the two components are 266.33 and 288.33. We conclude that the sample was roughly a 50–50 mixture of the two compounds and that it contained little or no water, in agreement with the infrared findings.

The melting range of the SDP was 90–94°C. Critical micelle concentrations of SDP at varioius pH's were determined by conductivity titration.

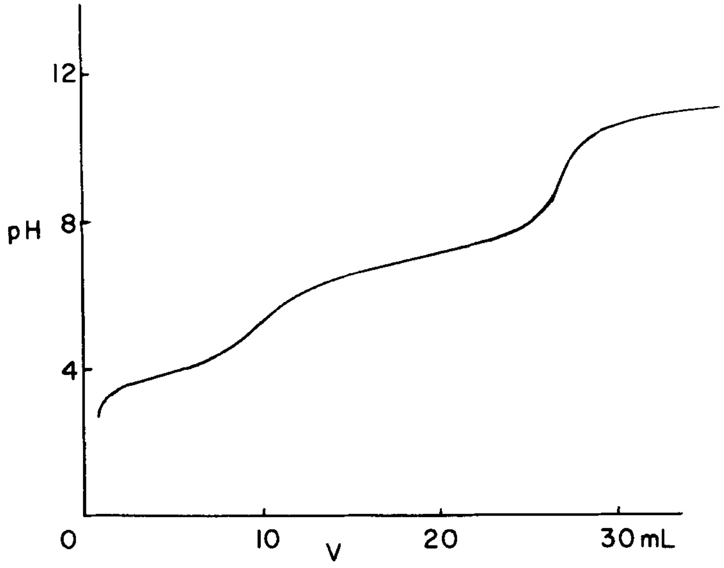

FIG. 1. Titration curve of sodium dodecylphosphate. Sample weight = 0.4081g; normality of NaOH = 0.0914. The first equivalence point is observed at a titrant volume of 8.95 mL and a pH of 5.2; the second, at a titrant volume of 25.30 mL and a pH of 9.0.

A 75-mL portion of deionized water was adjusted to the desired pH by the addition of small amounts of 0.1 *N* HCl and NaOH; 1 mL portions of a standard solution of SDP were then added from a 50-mL buret and the conductivity of the solution was measured after each addition. Titrant was added until well beyond the point at which the change in conductivity per milliliter of added titrant decreased. A typical plot of conductivity versus SDP concentration is shown in Fig. 2. The cmc was then determined by fitting least-squares straight lines to the two linear segments of the conductivity plot and determining the concentration of SDP at their point of intersection. The results are given in Table 1.

It was possible to determine cmc's for SDP only at pH's below 4.0. The cmc increased with increasing pH in this range. The low values observed are in the range commonly found for nonionic surfactants or mixtures of ionic and nonionic surfactants, suggesting that an appreciable fraction of the SDP is present in the micelles in the form of the undissociated acid. As the pH was increased up toward its value at the first equivalence point, a fluffy white precipitate was formed, indicating a rather low solubility for sodium hydrogen laurylphosphate. This low solubility prevented the

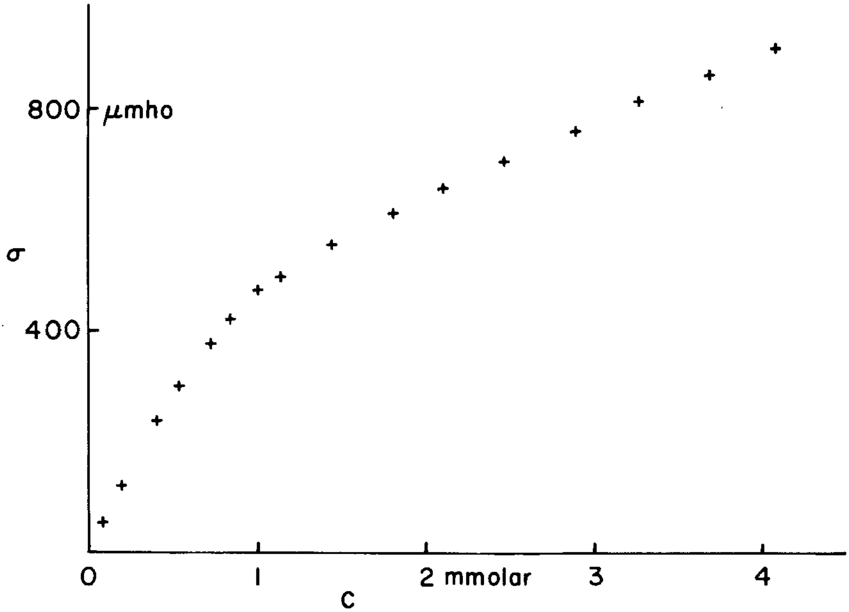

FIG. 2. Determination of critical micelle concentration of sodium dodecylphosphate by conductivity titration. pH of solution = 3.0. The cmc is calculated to be 0.787 m*M*.

TABLE 1
Critical Micelle Formation of Sodium Dodecyl Phosphate

pH	cmc[a]	Observation
2.8	0.616	—
3.0	0.680	—
3.5	0.710	—
4.0	—	Precipitated

[a]All cmc's are reported in mmol/L.

determination of cmc values in the pH range 4–9. At pH's above 9.5, SDP is again soluble, the dodecylphosphate presumably present as the dianion. Conductivity titrations carried out at these high pH's did not show any breaks, indicating that the coulombic repulsions of the doubly charged anions are sufficiently large to prevent micelle formation.

Several conductivity titration runs were made in which low concentrations of various normal alcohols were added to the SDP titrant solution in an effort to suppress precipitate formation. It was found that, among the alcohols from methanol to *n*-octanol, *n*-propanol and *n*-butanol at concentrations of 100 mg/L seemed to be the most effective in reducing precipitate formation; even these did not completely prevent it.

FOAM FLOTATION STUDIES

The ability of SDP to foam was tested at various pH's, and the results are in agreement with those reported by Mumallah (*12*). Good foam was generated in the flotation column when SDP was present alone in the solution at pH's below 3.5. Foam did not foam or was sparse at pH's above 3.5.

The foam flotation of lead was carried out as follows. A 25-mL portion of the 1 g/L Pb(II) stock solution was diluted to about 300 mL with deionized water; 50 mL of the 1 g/L Fe(III) stock solution and 375 mL of 200 mg/L *n*-hexanol stock solution were added. The pH of the solution was then adjusted to the desired value by the addition of 0.1 *N* NaOH and HCl, the solution diluted to 950 mL, mixed, and allowed to stand for about 5 min. Then 50 mL of 1.00 g/L SDP solution was added to the gently stirring solution, and the solution allowed to stand for another 5 min. The air flow rate through the column was adjusted to 180 mL/min, and the sample transferred to the column. After 1 or 2 min of aeration, 30 mL samples were taken and acidifed with 2 drops of concentrated HNO_3 to prevent lead deposition on the sample bottles.

The results for the flotation of Ph(II) are reported in Tables 2–5. It was found that 50 mg/L SDP, 50 mg/L Fe(III), and 75 mg/L *n*-hexanol were required to remove 25 mg/L Ph(II) adequately. When there was no interfering ion present in the solution (Table 2, Column 1), flotation in the pH range 6.5–9.5 yielded residual lead concentrations below 0.3 mg/L after 1 min of flotation. At pH's above 9.5 or below 6.5, residual lead concentrations were greater than 10 mg/L. At the lower pH's, foam was formed and some removal did occur; at pH's above 9.5 there was no removal whatsoever of the precipitate despite the fact that copious foam was produced. In the pH range 6.5–9.5, a ratio of *n*-hexanol to SDP of 3:2 and an alcohol-lead ratio of 3:1 provided optimal results. After the solution became clear, usually in about 1 min, no further lead removal was observed. In most of the runs the foam collapsed roughly 3 min after the start of the run, allowing most of the removed floc to reenter the solution. This would present no serious problem, since in a continuous flow apparatus the foam would already have been removed by this time.

The presence of sulfate had little effect on the residual concentrations of lead, even when the sulfate concentration was as high as 0.2 *M*. With 0.2 *M* sulfate concentrations, residual lead concentrations less than 1 mg/L were obtained in the pH range of 7–8 after 1 min of flotation. Evidently dodecylphosphate is more effective at competing with sulfate for adsorption sites than is dodecylsulfate. Rubin and Lapp (*13*) report lead removals of 55% from solutions containing 0.2 *M* sulfate with dodecylsulfate at pH 8.2; SDP/*n*-hexanol provides better than 95% removal under these conditions.

TABLE 2

Floc Foam Flotation of Lead with $Fe(OH)_3$ of SDP/*n*-Hexanol Residual Lead (ppm) (1 min)[a]

	Added Na_2SO_4 (*M*)				
pH	0	0.025	0.050	0.100	0.200
4.0	>10	>10	>10	>10	5.4
5.0	>10	>10	8.2	>10	5.8
6.0	1.1	1.9	0.2	2.7	3.7
7.0	0.3	0.6	0.3	0.4	1.4
10.0	NR[b]	—	—	—	—

[a] All runs were made with 25 ppm Pb, 50 ppm Fe(III), 50 ppm SDP, and 75 ppm *n*-hexanol. Initial sample volume = 1 L, air flow rate = 180 mL/min.

[b] NR = no removal.

When oxalate is present as an interfering anion (see Table 3), it increases residual lead even when present at relatively low concentrations. The optimum pH range was found to be 6.5–9.5; when 25 mg/L oxalate was present and the pH was not within this range, no foam was formed and no removal occurred, even after 10 min of flotation. With 25 mg/L oxalate and a pH in the range 6.5–9.5, residual lead concentrations below 2 mg/L were produced after 1 min of flotation. Later the foam collapsed, as with the sulfate runs. Quite substantial removal of lead was observed in the pH range 7.5–9.5 up to 100 mg/L oxalate, above which no foam was formed and no lead removal was observed.

Phosphate (Table 4) inhibits lead removal at even lower concentrations than does oxalate, but appears to increase the foaming ability of the SDP. Only in the pH range 4–5 were residual lead concentrations less than 3 mg/L in the presence of 75 mg/L phosphate. At phosphate concentrations of 25 mg/L, residual lead concentrations below 1 mg/L were found in the pH range 4.5–6.5.

Silicate (Table 5) interfered with lead removal the most drastically of the four interfering anions tested. When more than 50 mg/L silicate was present in the solution, no foam formed and no lead removal resulted at any pH after 10 min of flotation. When 50 mg/L silicate was present, residual lead concentations were reduced to 1 mg/L after 1 min, but only when the pH of the solution was approximately 6.0. At pH's below 5.5 or greater than 6.5, some foam was formed, but residual lead concentrations were greater than 10 mg/L. When 25 mg/L silicate was present, most of the lead was removed within 2 min in the pH range 4.5–

TABLE 3
Floc Foam Flotation of Lead with $Fe(OH)_3$ and SDP/*n*-Hexanol Residual Lead (ppm)[a]

	Added C_2O_4 (ppm)				
pH	25	50	75	100	150
5.0	NF/NR[b]	—	—	—	—
6.0	NF/NR	—	—	—	—
7.0	1.7	1.4	3.8	NF/NR	—
8.0	0.6	0.7	1.0	1.3	NF/NR
9.0	0.7	0.8	0.6	0.4	NF/NR
10.0	NF/NR	—	—	—	—

[a] All runs were made with 25 ppm Pb, 50 ppm Fe(III), 50 ppm SDP, and 75 ppm *n*-hexanol. Initial sample volume = 1 L, air flow rate = 180 mL/min.

[b] NF/NR = no foam and no removal.

TABLE 4
Floc Foam Flotation of Lead with $Fe(OH)_3$ and SDP/*n*-Hexanol Residual Lead (ppm) (3 min)[a]

pH	Added phosphate (ppm)			
	25	50	75	100
4.0	4.1[b]	3.6	0.5	1.7
5.0	0.7[b]	1.8	2.7	5.9
6.0	0.1	>10	NF/NR	—
7.0	3.7	NF/NR[c]	—	—
8.0	1.9	NF/NR	—	—
9.0	>10	NF/NR	—	—

[a] All runs were made with 25 ppm Pb, 50 ppm Fe(III), 50 ppm SDP, and 75 ppm *n*-hexanol. Initial sample volume = 1 L, air flow rate = 180 mL/min.
[b] Flotation time = 1 min.
[c] NF/NR = no foam and no removal.

9.5, but residual lead concentrations less than 1 mg/L were obtained only in the pH range 6.0–8.0.

The procedure for the coprecipitation of $Cd(OH)_2$ with ferric hydroxide and flotation with SDP and *n*-hexanol was the same as described above for the removal of lead. The results are given in Tables 6 and 7. Cadmium was removed by this flotation procedure only at pH's in the range 8.5–9.5. When the solution pH was outside of this range, no foam was formed and

TABLE 5
Floc Foam Flotation of Lead with $Fe(OH)_3$ and SDP/*n*-Hexanol Residual Lead (ppm)[a]

pH	Added SiO_3 (ppm)		
	25	50	75
4.0	NF/NR	—	—
5.0	8.4[b]	>10	—
8.0	1.3[b]	1.0	NF/NR
7.0	0.4	>10	NF/NR
8.0	1.3	NF/NR	—
9.0	6.5	—	—
10.0	NF/NR[c]	—	—

[a] All runs were made with 25 ppm Pb, 50 ppm Fe(III), 50 ppm SDP, and 75 ppm *n*-hexanol. Initial sample volume = 1 L, air flow rate = 180 mL/min.
[b] Time = 1 min.
[c] NF/NR = no foam and no removal.

TABLE 6
Floc Foam Flotation of Cadmium with $Fe(OH)_3$ and SDP/*n*-Hexanol Residual Cd (ppm) (1 min)[a]

	Added Na_2SO_4 (*M*)				
pH	0	0.025	0.050	0.100	0.200
8.0	NF/NR[b]	—			
8.5	>10	—			
9.0	2.6	2.5	3.5	>10	>10
9.5	2.2	0.4	1.6	2.3	>10
10.0	NF/NR	—			

[a] All runs were made with 25 ppm Pb, 50 ppm Fe(III), 50 ppm SDP, and 75 ppm *n*-hexanol. Initial sample volume = 1 L, air flow rate = 180 mL.

[b] NF/NR = no foam and no removal.

residual cadmium concentrations were unchanged from the initial values. Residual cadmium concentrations were approximately 2 mg/L when the solution pH was 8.5 and no interfering anions were present. The presence of very low concentrations (25 mg/L or less) of oxalate, silicate, and phosphate was found to prevent any foam formation or cadmium removal at all of the pH's tested. Concentrations of sulfate up to 0.05 *M* did not significantly alter flotation characteristics; residual cadmium concentrations of about 2 mg/L were obtained.

A number of runs were made in an effort to optimize the ratio of *n*-hexanol to SDP, but these provided no better results than those reported. Evidently this technique is not satisfactory for the removal of cadmium.

The last metal studied was Cu(II). The procedure used for its

TABLE 7
Floc Foam Flotation of Cadmium with $Fe(OH)_3$ and SDP/*n*-Hexanol Residual Cd (ppm)[a]

	Added anion (ppm)					
	C_2O_4		PO_4		SiO_3	
pH	25	50	25	50	25	50
8.5	NF/NR[b]	—	NF/NR	—	NF/NR	—
9.0	>10	—	NF/NR	—	NF/NR	—
9.5	>10	—	NF/NR	—	NF/NR	—

[a] All runs were made with 25 ppm Pb, 50 ppm Fe(III), 50 ppm SDP, and 75 ppm *n*-hexanol. Initial sample volume = 1 L, air flow rate = 180 mL/min.

[b] NF/NR = no foam and no removal.

precipitation and flotation was the same as that described above for the removal of Pb(II) and Cd(II). As seen in Table 8, Cu(II) was floated from solution by SDP/*n*-hexanol only when the pH was kept between 9.0 and 10.5. When the solution pH was less than 9, foam was sometimes generated but removal was minimal; at pH's greater than 10.5, no foam was generated. When the pH was between 9.0 and 10.5 and no interfering anions were present, good removals were obtained, with the lowest residual Cu concentration, 0.8 mg/L, occurring at pH 10.0.

Concentrations of interfering anions used were 0.05 *M* SO_4^{2-}, 25 mg/L $C_2O_4^{2-}$, 25 mg/L SiO_3^{2-}, and 25 mg/L PO_4^{3-}. These concentrations were employed because they had been found to cause significant interference in the removal of Pb and Cd, but did allow some removal. In the presence of sulfate, phosphate, or oxalate, residual Cu(II) concentrations below 2 mg/L were observed at pH 10.0. In the presence of silicate, the minimum Cu(II) concentration, 4.2 mg/L, was obtained at pH 9.5. As in the earlier work, removed floc that sometimes tended to reenter the solution's foam collapsed, although this does not present a significant problem.

CONCLUSIONS

Sodium dodecylphosphate (SDP) was found to form micelles only at pH's below approximately 4; in the pH range 4–8.5 the surfactant is not sufficiently soluble to permit determination of the cmc. At pH's above 8.5, where the ion is expected to be double charged, conductivity measurements did not indicate cmc's.

TABLE 8

Floc Foam Flotation of Copper with $Fe(OH)_3$ and SDP Residual Copper (ppm) (2 min)[a]

	Added anion concentration				
pH	0	SO_4^{2-} (0.05 *M*)	$C_2O_4^{2-}$ (25 ppm)	PO_4^{3-} (25 ppm)	SiO_3^{2-} (25 ppm)
9.5	4.7	>10	3.0	>10	4.2
10.0	0.8	1.7	1.4	1.0[b]	>10
10.5	5.5	6.5	>10	NF/NR[c]	NF/NR

[a] All runs were made with 50 ppm Cu, 50 ppm Fe(III), 50 ppm SDP, and 75 ppm *n*-hexanol. Initial sample volume = 1 L, air flow rate = 180 mL/min.

[b] Samples taken at 4 min.

[c] NF/NR = no foam and no removal.

A number of *n*-alcohols were tested for use as cosurfactants with SDP; *n*-hexanol was found to be most satisfactory, with a 3:1 ratio of *n*-hexanol to SDP yielding optimal results. The adsorbing colloid flotation of lead, cadmium, and copper with SDP/*n*-hexanol surfactant and ferric hydroxide as the carrier floc was studied; lead and copper were readily removed, while cadmium removal was less satisfactory. The presence of interfering anions at concentrations which cause flotation with sodium dodecylsulfate to fail is tolerated by the SDP/*n*-hexanol system, but it, too, fails at sufficiently high concentrations of sulfate, oxalate, phosphate, or silicate. The use of relatively cheap *n*-hexanol as an extender for the substantially more expensive surfactant SDP should markedly improve the economics of flotation with this surfactant.

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