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Adsorbing Colloid Flotation with Polyaluminum Chloride: A Powerful Technique for Removing Heavy Metals from Wastewater

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ABSTRACT

Adsorbing colloid flotation (ACF) possesses many distinct advantages for removing heavy metal ions from wastewater, but it is not much used for wastewater treatment because its separation efficiency is very poor for solutions with high concentrations of electrolyte. We demonstrate that this drawback can be eliminated by using polyaluminum chloride (PAC) as a coagulant. ACF with PAC was also tested for the removal of heavy metals from the wastewaters of a tannery and of an electroplating factory. ACF is shown to be a very promising technique for the removal of heavy metal ions from wastewater.

INTRODUCTION

Many kinds of separation techniques are used for wastewater treatment; foam separation techniques are among the less familiar. These techniques are based on the fact that surface-active materials tend to concentrate at gas–liquid interfaces. When air is bubbled through a solution, the surface-active material adsorbs on the rising bubbles, which then separate it from the solution. The substance to be removed (i.e., colligend), if not surface active, can be made so through union with, or adsorption of, a surfactant (such as sodium dodecyl sulfate, SDS). For instance, adsorbing colloid flotation (ACF, which is

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illustrated schematically in Fig. 1) involves addition of a coagulant to produce the floc. The dissolved material (such as heavy metal ions) is adsorbed onto the floc particle and/or coprecipitated with it. A surfactant (the collector) such as SDS is then added, adsorbs onto the floc, rendering it hydrophobic (surface active), and then the floc (with adsorbed material) is adsorbed on to the surface of the bubbles rising through the liquid. During the foam flotation process, these bubbles form a foam at the top of the solution (sometimes addition of a frother is required to maintain a stable foam). This foam is relatively rich in adsorbed material, so that enrichment or separation results.

Foam separation processes have been widely utilized by industry for the recovery of valuable minerals from ores (ore flotation). These techniques are also effective in the separation or concentration of surfactants, proteins, enzymes, microorganisms, and various metallic and nonmetallic ions (1); however, these industrial applications are rare. The extensive literature in the field has been reviewed by Lemlich (1, 2), Somasundaran (3), Grieves (4), Sebba (5), Mizuike (6), Clarke and Wilson (7, 8), Caballero et al. (9), and Huang (10).

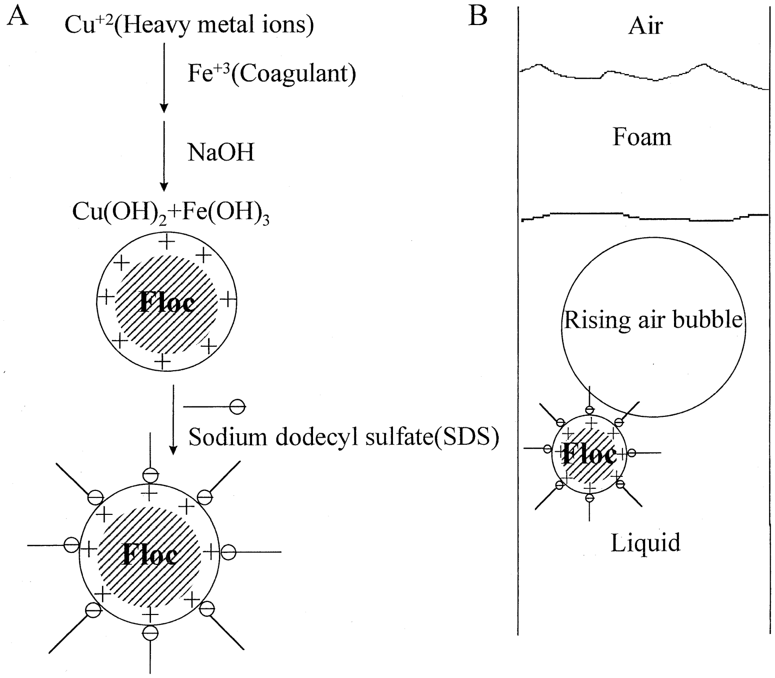

FIG. 1 Schematic representation of adsorbing colloid flotation (ACF) of heavy metal ions. (A) Fe(III) is used as the coagulant to produce the floc. Heavy metal ions are adsorbed onto or coprecipitated with the floc. The surfactant (SDS), with a hydrophobic part (—) and a hydrophilic part (○), is adsorbed onto the floc to render the floc hydrophobic. (B) The floc (with adsorbed material) is adsorbed on the surface of the bubble rising through the liquid. The bubbles form a foam at the top of the solution. This foam is relatively rich in adsorbed material, so that enrichment or separation results.

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There are many types of foam separation techniques. Among these, ACF is the most promising for wastewater treatment. ACF appear to possess many distinct advantages for removing heavy metal ions from wastewater (7, 8): low energy requirements, low residual concentrations of metals, rapid operation, small space requirements, flexibility of application to various metals at various scales, production of small volumes of sludge highly enriched with the contaminant, and moderate cost. The chemical and capital costs of treatment by ACF were compared with those of lime precipitation; the results favored ACF significantly (7, 8, 11).

ACF has one distinct disadvantage: the separation efficiency is very poor for solutions with high concentrations of electrolytes (7–14). Furthermore, the operating parameters may have to change with variation of the composition of the wastewater. Because industrial wastewater is typically a complex mixture with varied composition and high concentrations of electrolyte, these drawbacks restrict the application of ACF for wastewater treatment, and they are the major reasons why this technique is not much used for wastewater treatment, despite its advantages.

Fe(III) and Al(III) salts are the coagulants most commonly used (6–18) for metal ion removal by ACF. An anionic surfactant such as sodium dodecyl sulfate (SDS) is generally used as the collector and the frother. SDS is physically adsorbed on the floc surface through coulombic interactions (7, 13). Wilson et al. (7, 8, 11, 15–18) carried out several studies on the removal of heavy metals by foam separation. These mainly concerned ACF of heavy metal ions with Fe(III) or Al(III) salts. We have shown (13, 14) that the electrolyte tolerance of ACF of heavy metal ions from wastewater with iron(III) hydroxide floc can be improved when a mixture of sodium oleate and SDS is used as the collector and frother.

Polyaluminum chloride (PAC) is widely used for wastewater treatment with conventional chemical precipitation. However, it has never been used with ACF. Here we demonstrate that when PAC is used as the coagulant, the electrolyte tolerance of ACF in the treatment of wastewater containing heavy metal ions (such as Cu, Zn, Pb, Cr, Co) can be improved significantly, and the operating parameters of ACF are no longer sensitive to variations in the composition of the wastewater. The applicability of ACF for the removal of heavy metals from wastewater is thus greatly extended.

EXPERIMENTAL

The batch foam flotation system used was similar to that described earlier (12–14). Air bubbles were generated by passing compressed air through a gas sparger (pore size 25–50 μm). A glass column (length 60 cm and inside diameter 3.5 cm) was used for flotation. The bottom of the column was closed with a rubber stopper with holes for a gas sparger and a stopcock to take samples and to drain the column. A lipped side arm near the top of the column served as the foam outlet. Sodium dodecyl sulfate (SDS) and sodium oleate (laboratory grade) were used as collectors. PAC (technical grade) was used as the coagulant to remove heavy metal ions from wastewater by ACF. $Zn(NO_3)_2 \cdot 6H_2O$, $Pb(NO_3)_2$, $Cu(NO_3)_2 \cdot 3H_2O$, $Ni(NO_3)_2$, $K_2Cr_2O_7$, NaOH, $NaNO_3$, and $NaHSO_3$ (reagent grade) were used for sample preparation. The initial concentrations of heavy metal ions (Cu, Zn, Pb, Cr, Co) in the synthetic wastewater were 50 $mg \cdot L^{-1}$ unless specified otherwise. The ionic strength of the synthetic wastewater was adjusted with $NaNO_3$. ACF was also tested for removal of heavy metals from the wastewaters of a tannery and of an electroplating factory.

The rate of airflow was adjusted with a needle valve (Hoke) with micrometer control and measured with a soap film flowmeter. The air was purified by passage through glass wool to remove particulates, through Ascarite to remove carbon dioxide, and through distilled water to control humidity.

ACF of Cr(VI) was performed by reducing Cr(VI) (50 $mg \cdot L^{-1}$) with $NaHSO_3$ (120 $mg \cdot L^{-1}$) at pH 2.5–3.0 (19). The solution was stirred for 10 minutes to allow the reaction to proceed to completion before treatment by foam flotation. All experiments were run using 250 mL of solution. The duration of flotation was 10 minutes for all runs unless specified otherwise.

Measurements of pH were made with a digital pH meter (Radiometer pHM 82). The initial pH values of the runs are listed in Tables 1–4 and 6. Concentrations of heavy metals were determined with an atomic absorption spectrophotometer (Perkin-Elmer 5000). Each datum reported is the arithmetic average of three runs.

RESULTS AND DISCUSSION

Adsorbing Colloid Flotation of Cu(II) with PAC

The effects of pH, initial PAC concentration and initial SDS concentration on the separation of Cu by ACF are shown in Table 1. Residual Cu concentrations of less than 1 $mg \cdot L^{-1}$ were achieved over a pH range of 8.0 to 10.0. As little as 10 $mg \cdot L^{-1}$ of PAC was required for an effective separation. Too large a dose of PAC (100 $mg \cdot L^{-1}$) resulted in poor separation; a stable foam layer was not formed and redispersion of the floc occurred during flotation under these conditions. The residual Cu concentration decreased somewhat with increasing SDS concentration. The effect of concentration of electrolyte and pH on the separation of Cu by ACF is shown in Table 2. Residual Cu concentrations of less than 1 $mg \cdot L^{-1}$ were achieved from solutions containing $NaNO_3$ (0–1.5 M) at pH 8.5–9.0. Separation was poor for solutions with both higher pH (9.5 or greater) and also a large concentration of electrolyte

TABLE 1
Residual Cu Showing Effects of pH, PAC Concentration, and SDS Concentration on ACF of Cu[a]

pH	PAC ($mg \cdot L^{-1}$)	SDS ($mg \cdot L^{-1}$)	Residual Cu ($mg \cdot L^{-1}$)
8.0	10	80	0.35 ± 0.04
8.5	10	80	0.16 ± 0.03
9.0	10	80	0.14 ± 0.02
9.5	10	80	0.15 ± 0.02
10.0	10	80	0.61 ± 0.13
9.0	10	80	0.14 ± 0.02
9.0	20	80	0.24 ± 0.03
9.0	50	80	0.43 ± 0.06
9.0	100	80	5.35 ± 0.75
9.0	10	40	1.44 ± 0.04
9.0	10	50	1.01 ± 0.27
9.0	10	60	0.71 ± 0.19
9.0	10	70	0.51 ± 0.08
9.0	10	80	0.14 ± 0.02

[a] All runs were made with Cu(II) (50 $mg \cdot L^{-1}$) initially, no $NaNO_3$ added, rate of air flow = 70 $mL \cdot min^{-1}$, and duration of flotation = 10 minutes.

(1.2–1.5 M $NaNO_3$) due to the decreased surface potential of the floc at higher pH and larger concentration of electrolyte; the anionic surfactant (SDS) can no longer be adsorbed on the surface of the floc to render the surface hydrophobic (7, 8, 13, 14). Similar effects were observed during ACF of other heavy metal ions (Zn, Pb, Cr, Co) with PAC.

TABLE 2
Residual Cu ($mg \cdot L^{-1}$) Showing Effects of Concentration of Electrolyte and pH on ACF of Cu[a]

	$NaNO_3$ (M)		
pH	0	1.2	1.5
8.5	0.16 ± 0.03	0.41 ± 0.02	0.53 ± 0.04
9.0	0.14 ± 0.02	0.50 ± 0.06	0.59 ± 0.11
9.2	—	1.43 ± 0.08	2.36 ± 0.46
9.5	0.15 ± 0.02	1.62 ± 0.17	5.59 ± 1.32
9.7	—	5.31 ± 0.78	>10

[a] Initial Cu(II) = 50 $mg \cdot L^{-1}$, PAC = 10 $mg \cdot L^{-1}$, SDS = 80 $mg \cdot L^{-1}$, and duration of flotation = 10 minutes.

TABLE 3
Residual Zn Showing Effects of pH, PAC Concentration, and SDS Concentration on ACF of Zn[a]

pH	PAC ($mg\cdot L^{-1}$)	SDS ($mg\cdot L^{-1}$)	Residual Zn ($mg\cdot L^{-1}$)
8.0	50	90	>10
8.5	50	90	2.56 ± 0.13
9.0	50	90	0.88 ± 0.08
9.5	50	90	1.03 ± 0.12
10.0	50	90	4.05 ± 0.34
9.0	30	90	5.62 ± 1.07
9.0	50	90	0.88 ± 0.08
9.0	60	90	0.11 ± 0.02
9.0	70	90	0.30 ± 0.05
9.0	80	90	4.92 ± 0.80
9.0	90	90	5.44 ± 0.98
9.0	60	80	3.75 ± 1.50
9.0	60	90	0.11 ± 0.22
9.0	60	100	0.07 ± 0.01
9.0	60	110	0.08 ± 0.02

[a] All runs were made with Zn(II) (50 $mg\cdot L^{-1}$) initially, $NaNO_3$ added, rate of air flow = 85 $mL.min^{-1}$, and duration of flotation = 10 minutes.

Adsorbing Colloid Flotation of Zn(II) with PAC

The effects of pH, initial PAC concentration, and initial SDS concentration on the separation of Zn by ACF are shown in Table 3. The separation of Zn are poor at pH less than 8.5 (PAC = 50 $mg\cdot L^{-1}$) due to the incomplete co-precipitation of Zn ion with the floc. Too large a dose of PAC (80–100 $mg\cdot L^{-1}$) also resulted in poor separation. The effects of the concentration of electrolyte and pH on the separation of Zn by ACF is shown in Table 4. A

TABLE 4
Residual Zn ($mg\cdot L^{-1}$) Showing Effects of Electrolyte Concentration and pH on ACF of Zn[a]

	$NaNO_3$ (M)			
pH	1.2	1.5	1.8	2.0
8.5	2.76 ± 0.13	4.16 ± 0.78	—	—
8.8	1.44 ± 0.26	2.84 ± 0.40	—	—
9.0	1.16 ± 0.21	1.94 ± 0.12	3.83 ± 0.78	6.90 ± 1.81
9.2	0.21 ± 0.03	0.57 ± 0.29	1.10 ± 0.05	1.15 ± 0.13
9.5	0.16 ± 0.04	0.26 ± 0.07	0.69 ± 0.12	1.69 ± 0.16

[a] PAC = 60 $mg\cdot L^{-1}$ and SDS = 100 $mg\cdot L^{-1}$.

residual Zn concentration of less than 1 mg·L^{-1} was achieved from a solution containing 1.8 M $NaNO_3$.

Electrolyte Tolerance of ACF of Heavy Metals

Electrolyte tolerance of ACF of heavy metals is summarized in Table 5. Since the separation efficiency and the electrolyte tolerance of ACF are much better than those of precipitate flotation, we compared the results of ACF using PAC with those using Fe(III) or Al(III). The electrolyte tolerance of a flotation system is defined as the largest concentration of $NaNO_3$ in a solution from which heavy metal is removed by foam flotation to a residual heavy metal concentration smaller than specified levels (Cu, Zn, Pb, and Co, 1 mg·L^{-1}; Cr, 0.5 mg·L^{-1}). Typical residual levels of heavy metals were 0.1 to 0.3 mg·L^{-1} after the wastewater was treated by ACF with PAC. The electrolyte tolerances of ACF shown in Table 5 using Fe(III) and Al(III) as the coagulant can be found from the literature (7–18) and have been rechecked in our laboratory.

The separation efficiency of ACF with Fe(III) or Al(III) floc is very poor for solutions with high concentrations of electrolyte, due in large part to the decreased surface potential of the floc at large concentrations of electrolyte. The reason that the electrolyte tolerance of ACF of heavy metals using PAC as the coagulant is much greater than that using either an Fe(III) or an Al(III) salt is explained as follows. The surface of the floc produced from PAC (containing a complex such as $Al_{13}O_4(OH)_{24}(H_2O)_{12}^{7+}$) is more positively charged than the floc produced from Al(III) or Fe(III) salts (20, 21); and therefore the surface of the floc produced from PAC can remain positively charged (for an-

TABLE 5
Electrolyte Tolerance ($NaNO_3$, M) Showing Effects of Coagulant and Collector on Electrolyte Tolerance of ACF[a]

	Colligend and collector			
Heavy metal	PAC[b]/SDS	PAC[b]/(SDS and oleate)	Fe[c]/SDS	Al[d]/SDS
Cu	1.5	—	0.4	0.05
Zn	1.8	—	0.08	0.2
Pb	0.5	—	0.2	0.1
Cr	1.0[e]	1.6	0.1	0.02
Co	0.05	1.7	1.0	1.0

[a] Duration of flotation = 10 minutes (except for Pb, which was 15 minutes).

[b] PAC = 10 mg·L^{-1} (Cu), 60 mg·L^{-1} 1 (Zn), 50 mg·L^{-1} (Pb), 100 mg·L^{-1} (Cr), and 40 mg·L^{-1} (Co).

[c] Fe(III) = 100 mg·L^{-1}.

[d] Al(III) = 100 mg·L^{-1}.

[e] Cr(VI) was reduced to Cr(III) by $NaHSO_3$ before ACF treatment.

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ionic surfactant adsorption) at relatively higher concentrations of electrolyte. For instance, the electrolyte tolerance of ACF of Cu(II) with PAC is 1.5 M; the corresponding values using an Al(III) salt or an Fe(III) salt are 0.05 and 0.4 M, respectively (SDS was used as the collector in all cases). The electrolyte tolerances of ACF of Zn and Pb using PAC are also much greater than those using Fe(III) or Al(III) salts. The electrolyte tolerances of ACF of Cr(III) and Co(II) using PAC as the coagulant and SDS as the collector are 0.05 and 1.0 M, respectively. However, much higher electrolyte tolerances (1.6 M or greater) can be achieved when a mixed surfactant (SDS and oleate) is used as the collector. This is presumably due to chemisorption of oleate ion to the surface of the floc (13, 22–24).

Dependence of Operation Parameters on Composition of Wastewater

When PAC is used as the coagulant, the operating parameters of ACF are no longer sensitive to variations of the composition of the wastewater. As shown in Table 6, the same operating parameters can be used to treat wastewater samples containing Cu(II) from 20 to 150 $mg\cdot L^{-1}$ and $NaNO_3$ from 0 to 1.5 M. Another sample containing Cu, Zn, and Ni (50 $mg\cdot L^{-1}$ each) was treated by ACF with PAC. These were removed simultaneously by ACF to residual concentrations of less than 0.3 $mg\cdot L^{-1}$.

Tests on Wastewater

ACF with PAC was tested to remove heavy metal ions from tannery wastewater and electroplating wastewater. Initial concentrations of Cr(III) in

TABLE 6

Residual Cu in the Effluent after Wastewaters [with varied concentration of electrolyte ($NaNO_3$, M) and initial Cu concentration] Treated by ACF under the Same Operation Parameter[a]

pH	$NaNO_3$ (M)	Initial Cu ($mg\cdot L^{-1}$)	Residual Cu ($mg\cdot L^{-1}$)
8	0.0	20	0.11 ± 0.05
8	1.5	20	0.14 ± 0.02
8	0.0	150	0.30 ± 0.10
8	1.5	150	0.52 ± 0.03
9	0.0	20	0.09 ± 0.01
9	1.5	20	0.15 ± 0.02
9	0.0	150	0.06 ± 0.01
9	1.5	150	0.54 ± 0.10

[a] PAC = 10 $mg\cdot L^{-1}$, SDS = 100 $mg\cdot L^{-1}$, rate of air flow = 110 $mL\cdot min^{-1}$, and duration of flotation = 10 minutes.

the tannery wastewater samples were in the 5–11 mg·L^{-1} range. Residual Cr(III) in the effluents was less than 0.3 mg·L^{-1} (PAC, 30 mg·L^{-1}; SDS, 80 mg·L^{-1}; oleate, 20 mg·L^{-1}; pH 5.0; sample volume, 250 mL; air flow rate, 200 mL/min; duration of run, 10 minutes; $n = 3$). Another sample which containing Cr(III) (24 mg·L^{-1}), COD (878 mg·L^{-1}), and suspended solid (480 mg·L^{-1}) was initially treated by flotation with PAC (30 mg·L^{-1}) at pH 5.0; the effluent contained Cr(III) (0.9 mg·L^{-1}), COD (418 mg·L^{-1}), and suspended solid (31 mg·L^{-1}). The Ni concentration in the electroplating wastewater was reduced from 43 to 0.4 mg·L^{-1} by ACF treatment with PAC (PAC, 100 mg·L^{-1}; SDS, 80 mg·L^{-1}; oleate, 20 mg·L^{-1}; pH 10; sample volume, 250 mL; air flow rate, 150 mL/min; duration of run, 10 minutes; $n = 3$).

Our work virtually eliminates the greatest drawback of ACF for wastewater treatment. ACF is shown to be a very promising technique for the removal of heavy metal ions from wastewater.

CONCLUSION

When PAC is used as the coagulant, the electrolyte tolerance of ACF in the treatment of wastewater containing heavy metal ions (such as Cu, Zn, Pb, Cr, Co) can be improved significantly, and the operating parameters of ACF are no longer sensitive to variations in the composition of the wastewater. The applicability of ACF for the removal of heavy metals from wastewater is thus greatly extended.

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