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Simutaneous Removal of Heavy Metal Ions from Wastewater by Foam Separation Techniques

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Abstract

The objective of the present work is to extend the application of adsorbing colloid flotation techniques to remove mixtures of metal ions. The systems studied are: 1) Co(II) and Cr(VI); 2) Co(II), Ni(II), and Cr(VI); 3) Cr(VI), Cu(II), and Zn(II); 4) Cr(VI), Cu(II), Zn(II), and Ni(II); 5) Cd(II), Pd(II), and Cu(II). Ferric hydroxide and aluminum hydroxide were used as the coprecipitant, and sodium lauryl sulfate was used as the collector and frother. The ionic strength of the solution was adjusted with $NaNO_3$ or Na_2SO_4. It was found that all the heavy metals can be removed effectively by a single step foam flotation treatment.

INTRODUCTION

Foam separation techniques have been of considerable interest in recent years due in large part to their ready adaptability to the removal of traces of toxic heavy metals from industrial effluents. A number of excellent reviews on foam separation are available (*1–5*). Of particular interest to our group has been the extensive work of Wilson and his collaborators on heavy metal removal from wastewater on the laboratory scale (*6–10*) and for pilot-plant scale (*11*, *12*) adsorbing colloid flotation plants.

The objective of the present work is to extend the application of

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adsorbing colloid flotation to remove mixtures of metal ions simutaneously. The work involving mixture of metals is of significant importance because industrial wastewater is usually a complex mixture of varying compositions rather than a simple solution containing a single contaminant. This work will be helpful in defining the capabilities and limitations of adsorbing colloid flotation. Most of the publications on foam separation of heavy metals from water involve the removal of a single contaminant from aqueous solutions (*1–5*). Wilson and his collaborator (*13*) demonstrated the simultaneous removal of Cu(II), Pb(II), and Zn(II) by batch foam flotation with $Fe(OH)_3$ and sodium lauryl sulfate. An adsorbing colloid foam flotation pilot plant was used by Wilson's (*1*, *2*) group to remove Cu(II), Zn(II), and Cr(III) from aqueous mixtures of these ions. The simulated wastewater contained 20 mg/L of each of these ions. Residual concentrations of the ions were less than 0.1 mg/L (Cu), 0.5 mg/L (Zn), and 0.2 mg/L (Cr) under optimum conditions.

The simulated wastewaters investigated by our group are 1) Co(II) and Cr(VI); 2) Co(II), Ni(II), and Cr(VI); 3) Cr(VI), Cu(II), and Zn(II); 4) Cr(VI), Cu(II), Zn(II), and Ni(II); 5) Cd(II), Pb(II), and Cu(II). The details of the first two systems were presented in our recent report (*14*) and will only be briefly reviewed here. We are particularly interested in removing Cr(VI) instead of Cr(III) because chromium in industrial wastes exists predominantly in the hexavalent form as chromate (CrO_4^{2-}) and dichromate ($Cr_2O_7^{2-}$).

It was found that all the heavy metal ions can be removed simutaneously from the systems studied by a single step foam flotation treatment.

EXPERIMENTAL

The batch foam flotation system used was similar to that described earlier (*14*, *15*). Figure 1 depicts the apparatus used for the batch separations. A soft glass column 90 cm in length with an inside diameter of 3.5 cm was used for the flotation. There was a side arm with a rubber septum near the bottom to inject the collector. 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. The gas sparger was a commercially available gas dispersion tube. A lipped side arm near the top of the column served as a foam outlet. Compressed air was generated from an air pump. The air flow rate was adjusted with a Hoke needle valve with micrometer control and measured with a soap film flowmeter. The air was purified by passing it through glass wool to remove

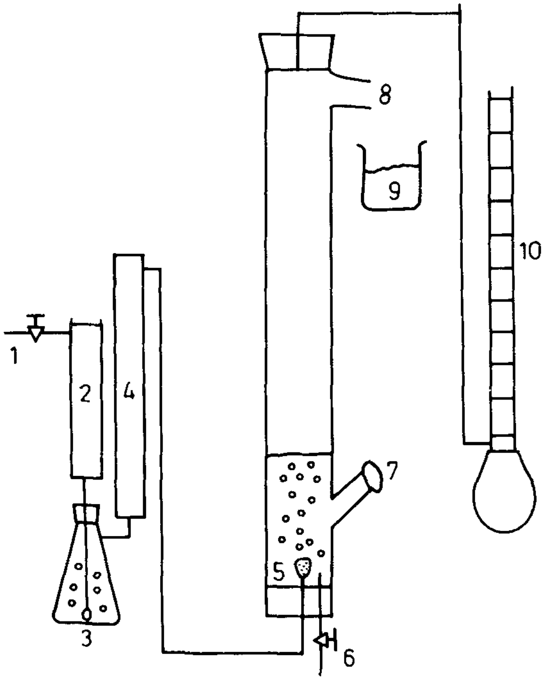

FIG. 1. The apparatus for foam separation. 1) Air needle valve, 2) Ascarite tube for CO_2 removal, 3) humidifier, 4) glass wool column, 5) fritted glass sparger, 6) drain, 7) reagent syringe, 8) foam discharge port, 9) discharged foam, 10) soap film flowmeter.

particulates, through Ascarite to remove carbon dioxide, and through distilled water for controlled rehumidification.

The continuous flow foam flotation system used is shown in Fig. 2. The simulated wastewater was premixed with $FeSO_4$ [or $Fe(NO_3)_3$], NaOH, and sodium lauryl sulfate (NLS). These chemicals were added to each batch of simulated wastewater in a well-stirred storage tank from which the resulting slurry was pumped to the column. A Pyrex glass column 110 cm in length with an inside diameter of 7.5 cm was used for the continuous flow stripping column flotation system. The slurry was pumped into the column through a spider-shaped dispersion head 35 cm below the top of the column. The liquid cascaded down through six baffles with 10 cm separation between each baffle in the stripping section of the column, and was discharged from the bottom of the column. Air bubbles were formed by the glass frit air dispersion head, generating foam which rises through the baffles in the stripping section of the

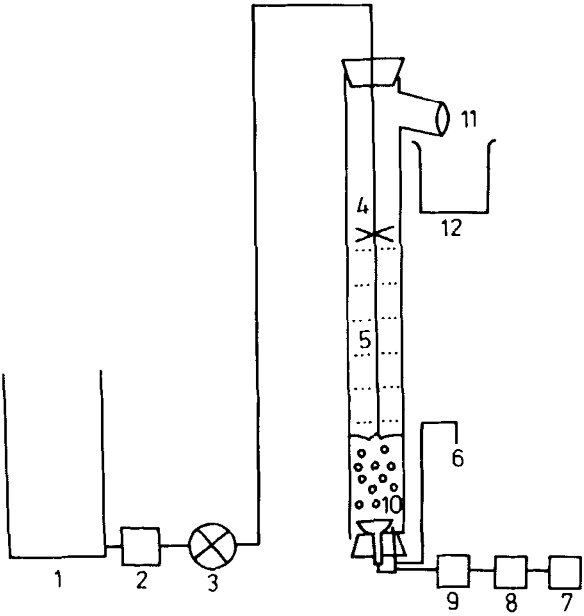

FIG. 2. Schematic diagram of continuous flow foam flotation system. 1) Waste tank, 2) pump, 3) influent flowmeter, 4) flow dispersion head, 5) baffles, 6) effluent outlet, 7) compressed air tank, 8) air pressure regulator, 9) air flow controller, 10) air diffuser, 11) foam outlet, 12) clarifier.

column, past the influent dispersion head, through the foam drainage section of the column. A lipped side arm near the top of the column served as a foam outlet.

Laboratory-Grade NLS was used as the collector and frother without further purification. Reagent grade $Fe(NO_3)_3 \cdot 9H_2O$, $FeSO_4 \cdot 7H_2O$, $Al(NO_3)_3 \cdot 9H_2O$, $CoCl_2 \cdot 6H_2O$, $Na_2Cr_2O_7 \cdot 2H_2O$, $NiCl_2 \cdot 6H_2O$, $Cu(NO_3)_2 \cdot 3H_2O$, $Zn(NO_3)_2 \cdot 4H_2O$, $Cd(NO_3)_2 \cdot 4H_2O$, $Pb(NO_3)_2$, NaOH, $NaNO_3$, and Na_2SO_4 were used for sample preparation.

The pH measurements were made with a Radiometer PHM63 digital pH meter. Concentrations of metal ions were measured with a Perkin-Elmer 5000 atomic absorption spectrophotometer.

Zeta potentials of particles were measured with a Zeta Meter (Zeta-Meter, Inc.) consisting of a cell across which a potential can be applied which will cause the charged particles to move. The time needed for a colloid particle to pass across a certain distance was measured. Ten to 20 particles were tracked. The average velocity of the particles is calculated at a known applied voltage to determine the zeta potential. A graph prepared from the Helmholtz-Smoluchowski formula was used to determine the zeta potential (*16*).

RESULTS AND DISCUSSIONS

Foam Separation of Co(II) and Cr(VI), Batch Type

It was attempted to remove chromium species and Co(II) together by reducing Cr(VI) with $FeSO_4$, followed by flotation under various pH conditions. The simulated wastewater contains 50 ppm each of Co(II) and Cr(VI) initially. The results are shown in Fig. 3 (*14*). Both Co(II) and

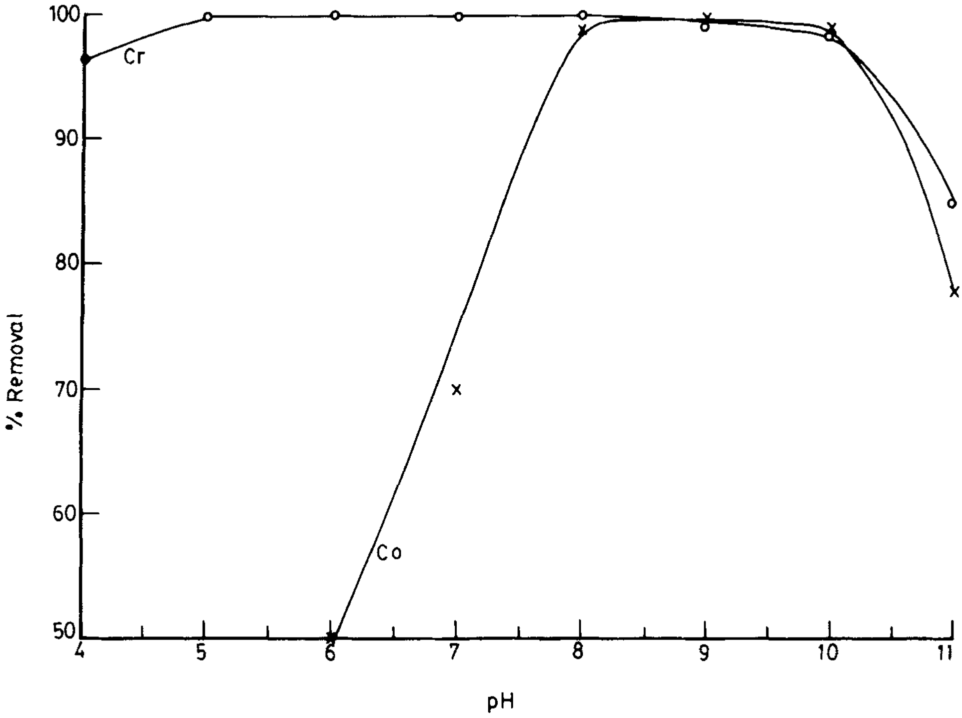

FIG. 3. Effect of pH on flotation of Co(II)-Cr(VI) with Fe(II). Fe(II) dosage = 160 ppm, duration of runs = 10 min, (O) percent removal of chromium, (X) percent removal of Co(II).

chromium species could be removed from the solution simultaneously at pH 8 to 9, and the efficiency was over 99% for both ions.

Foam Separation of Co(II), Ni(II), and Cr(VI), Batch Type

The simulated wastewater contained 50 ppm each of Co(II), Ni(II), and Cr(VI) initially. A series of experiments was carried out by reducing Cr(VI) with $FeSO_4$ to Cr(III), and the floc containing $Cr(OH_3)$, $Fe(OH)_3$, $Co(OH)_2$, and $Ni(OH)_2$ was then removed from the solution by foam flotation. The results are shown in Fig. 4. It is interesting to note that the effective pH range for Cr(VI) removal was 4.5 to 7.0 when the solution contained Cr(VI) alone. When the solution contained Ni(II), Co(II), and Cr(VI), the effective pH range for Cr(VI) removal was extended to 5 to 10.

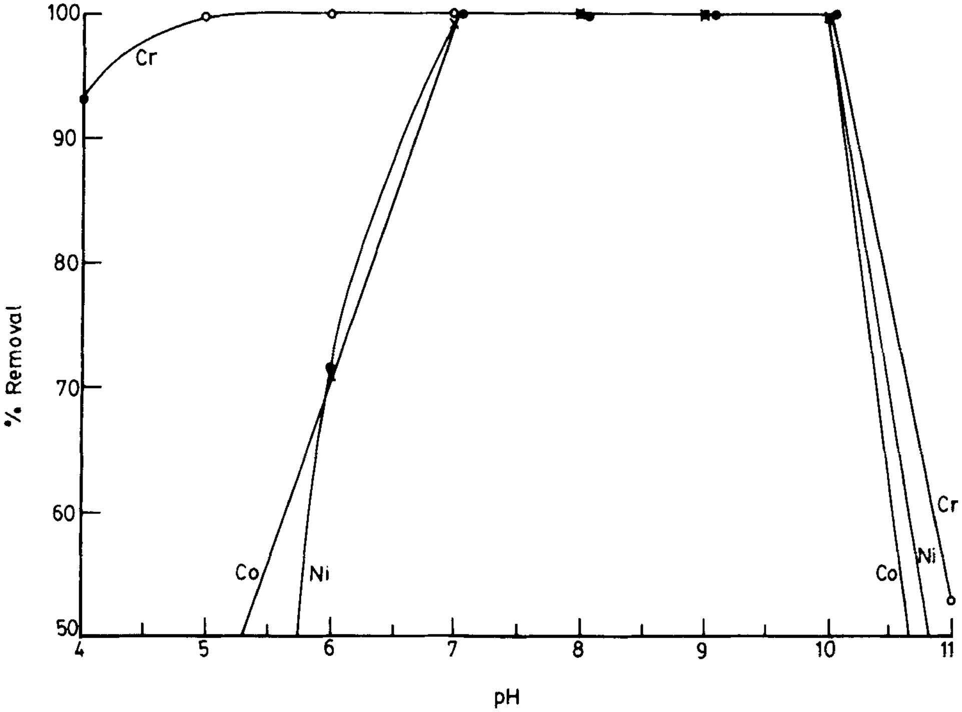

FIG. 4. Effect of pH on flotation of Co(II)-Ni(II)-Cr(VI) with Fe(II). Fe(II) dosage = 160 ppm, duration of runs = 10 min, (○) percent removal of chromium, (×) percent removal of Co(II), (●) percent removal of Ni(II).

At pH 7 to 10, all these three metal ions were removed with over 99% effeciency. Best removal efficiency was achieved at pH 9.0, being over 99.8% for all three metal ions (*14*).

Foam Separation of Co(II), Ni(II), and Cr(VI), Continuous Flow Type

The simulated wastewater containing 50 ppm each of Co(II), Ni(II), and Cr(VI) was treated with $FeSO_4$ to reduce Cr(VI) to Cr(III). The pH of the solution was adjusted with NaOH solution. Sodium lauryl sulfate was used as the collector and frother. These chemicals were added to each batch of simulated wastewater in a well-stirred storage tank, from which the resulting slurry was pumped continuously to the column. All the experiments were conducted at pH 9.0, which was found to be the optimum pH for separation from batch-type experiments. The air flow rate was set at 210 mL/min, which was the maximum flow rate that could be set by the flow controller being used. When the influent flow rate was maintained at 150 mL/min, effluent cobalt levels below 0.03 ppm, nickel levels below 0.02 ppm, and chromium levels below 0.05 ppm were achieved with 160 ppm Fe(II) and 100 ppm NLS dosage. It was found that the maximum ultimate removal of the mixed metal ions was not significantly affected by the ionic strength variation (adjusted with $NaNO_3$) provided that the ionic strength of the solution was no greater than 0.1 *M* in $NaNO_3$. Residual metal ions of less than 0.05 ppm could be achieved (*14*).

Foam Separation of Cu(II), Zn(II), and Cr(VI), Batch Type

The simulated wastewater containing 20 ppm each of Cu(II), Zn(II), and Cr(VI) was treated with 64 ppm Fe(II) to reduce Cr(VI) to Cr(III). The floc was then removed by foam flotation. The effect of pH on separation is shown in Fig. 5. Best removal efficiency was achieved at pH 7.5. The residual metal concentations were 0.08 ppm (Cu), 0.61 ppm (Zn), and 0.43 ppm (Cr) after 10 min foam flotation treatment. It was found that the separation efficiency of the metal ions in this system was very easily affected by the presence of a neutral salt (such as $NaNO_3$ or Na_2SO_4). However, if 20 ppm Ni(II) was also added to the system before pH adjustment, good separation could be achieved from solutions containing 0.2 *M* $NaNO_3$ or 0.05 *M* Na_2SO_4.

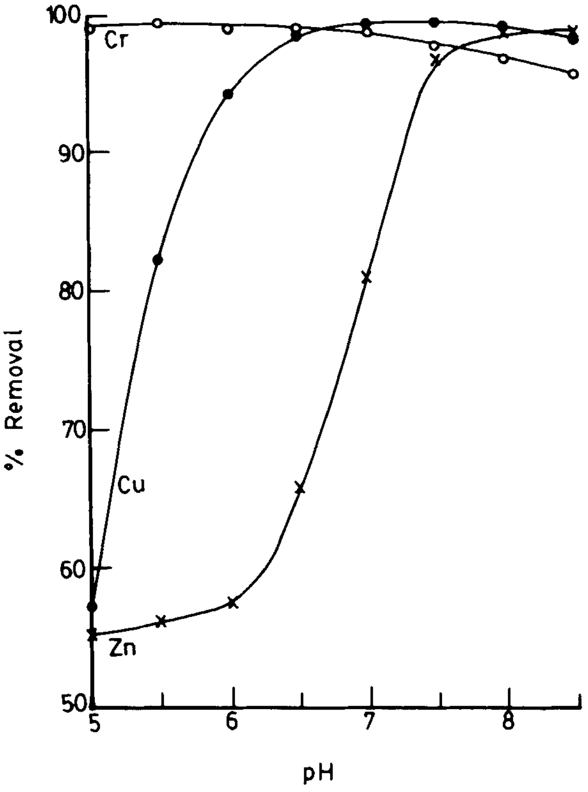

FIG. 5. Effect of pH on flotation of Cu(II)-Zn(II)-Cr(VI) with Fe(II). Fe(II) dosage = 64 ppm, duration of runs = 10 min, (O) percent removal of chromium, (X) percent removal of Zn(II), (●) percent removal of Cu(II).

Foam Separation of Cu(II), Zn(II), Ni(II), and Cr(VI), Batch Type

The simulated wastewater contains 20 ppm each of Cu(II), Zn(II), Ni(II) and Cr(VI) initially. Cr(VI) was reduced with $FeSO_4$, and the floc was then removed from the solution by foam flotation. The effect of pH on separation is shown in Fig. 6. Good separation was achieved over a wide pH range (pH 7.0 to 9.5). Over 99.6% of all metal ions were removed under optimum pH (8.5 to 9.0). At pH below 7.0, the residual nickel level was somewhat high, probably due to the high solubility of nickel hydroxide. However, nickel is not a very toxic heavy metal ion, and high separation efficiency for nickel is not absolutely required. It was found

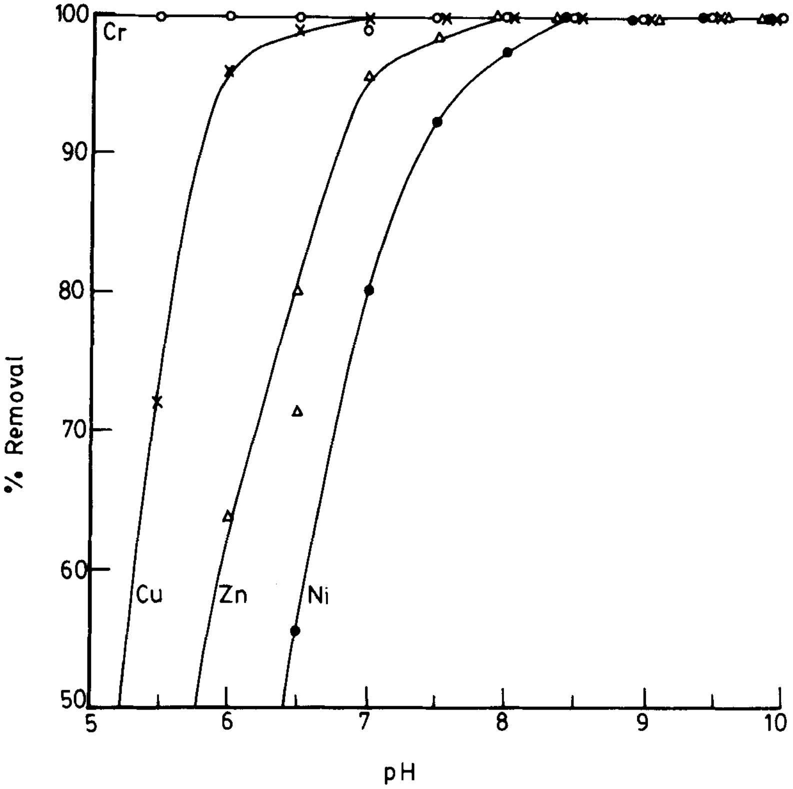

FIG. 6. Effect of pH on flotation of Cu(II)-Zn(II)-Ni(II)-Cr(VI) with Fe(II). Fe(II) dosage = 64 ppm, duration of runs = 10 min, (○) percent removal of chromium, (×) percent removal of Cu(II), (●) percent removal of Ni(II), (△) percent removal of Zn(II).

that the separation efficiency was not significantly affected by the neutral salt provided that the salt concentration of the solutions was no greater than 0.5 *M* $NaNO_3$ or 0.08 *M* Na_2SO_4. If 20 ppm Al(III) was added to the solution before pH adjustment, the separation was effective from solutions containing 1.0 *M* $NaNO_3$ or 0.1 *M* Na_2SO_4, as shown in Table 1. The effect of aluminum as the activator for foam flotation from solution with high ionic strength was probably due to the increase in surface protential of the floc by the aluminum species, as we proposed earlier (*15*).

TABLE 1
Effect of Ionic Strength and Al(III) on Flotation of Cr-Cu-Ni-Zn[a]

					Residual metal concentration (ppm)			
$NaNO_3$ (*M*)	Na_2SO_4 (*M*)	Al(III) (ppm)	pH	NLS (ppm)	Cr	Cu	Ni	Zn
0	0	0	7.0	50	0.08	0.03	1.83	0.46
0	0	0	7.5	50	0.07	0.02	1.05	0.14
0.5	0	0	7.0	50	0.45	0.35	>10	6.56
0.5	0	0	7.5	50	0.44	0.19	7.11	2.26
1.0	0	0	7.0	50	0.98	1.08	>10	6.62
1.0	0	0	7.5	50	1.27	0.85	8.75	3.01
1.0	0	0	7.0	60	1.35	1.44	>10	5.88
1.0	0	0	7.5	60	1.06	0.51	4.86	1.39
1.0	0	20	7.0	60	0.13	0.10	2.43	0.81
1.0	0	20	7.5	60	0.19	0.13	0.83	0.17
0	0.08	0	7.5	50	0.35	0.11	3.20	1.12
0	0.10	0	7.0	50	4.18	2.70	9.75	5.32
0	0.10	0	7.5	50	0.96	0.53	4.43	1.49
0	0.10	20	7.0	60	0.06	0.05	2.83	1.34
0	0.10	20	7.5	60	0.06	0.03	1.05	0.24

[a] Fe(II) = 64 ppm, duration of run = 10 min.

Foam Separation of Cd(II), Pb(II), and Cu(II), Batch Type

The simulated wastewater contains 20 ppm each of Cd(II), Pb(II), and Cu(II) initially. The results of precipitate flotation of the mixed metal hydroxides are shown in Fig. 7. Best removal efficiency was achieved at pH 11.0. The residual metal ion concentrations in the solution were 0.14 ppm (Cd), 0.17 ppm (Cu), and 0.9 ppm (Pb), respectively. Cadmium is highly toxic, and residual cadmium levels below 0.1 ppm are generally required. The residual cadmium level in the effluent after the solution was treated with precipitate flotation was somewhat higher than that considered safe to be discharged. The effect of ionic strength variation is shown in Table 2. Residual copper levels were not affected by increasing the Na_2SO_4 concentration. However, the residual cadmium levels increased a little bit, and the residual lead levels increased significantly in the presence of Na_2SO_4. The increase of the residual lead levels in the presence of Na_2SO_4 was probably due in large part to the decrease in coprecipitation of lead ion with the mixed metal hydroxides, and therefore, lead in the form of soluble lead ion (or lead sulfate) was not removed from the solution by flotation.

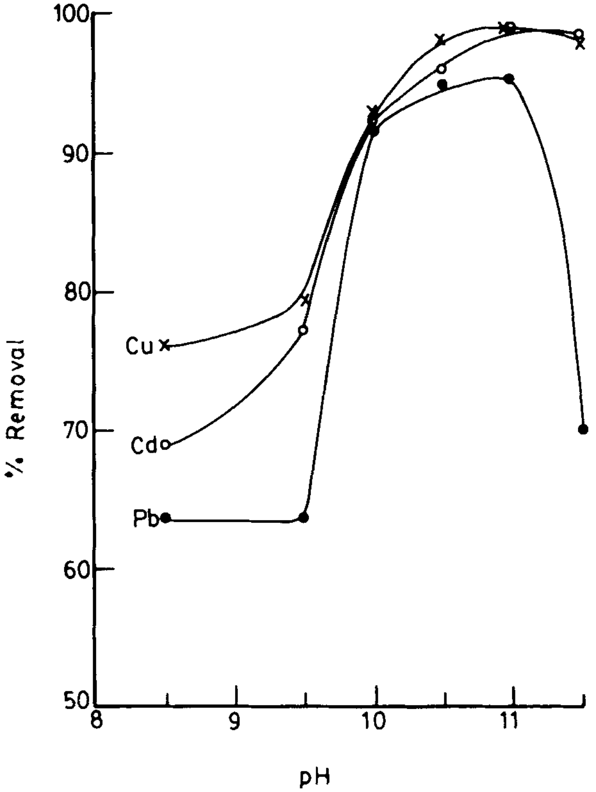

FIG. 7. Effect of pH on precipitate flotation of the mixed metal hydroxides of Cd(II)-Pb(II)-Cu(II). Duration of runs = 10 min, (O) percent removal of Cd(II), (X) percent removal of Cu(II), (●) percent removal of Pb(II).

TABLE 2
Effect of Sulfate on Precipitate Flotation of Cd-Cu-Pb Hydroxide[a]

Na_2SO_4 (*M*)	Residual metal concentration (ppm)		
	Cd	Cu	Pb
0.00	0.14	0.17	0.9
0.02	0.20	0.10	3.9
0.05	0.15	0.11	3.8
0.10	0.23	0.15	3.1
0.20	0.20	0.11	3.0

[a] pH = 11.0, duration of run = 10 min.

Precipitate flotation was not very successful in removing all the metals simutaneously from the solution. Removing the heavy metals by adsorbing colloid flotation with $Al(OH)_3$ or $Fe(OH)_3$ was then attempted. The effects of pH on adsorbing colloid flotation with $Al(OH)_3$ are shown in Fig. 8. The separation efficiency was better than that of precipitate flotation. Furthermore, the optimum pH range for separation was 9.5 to 10.0, which was lower than that of precipitate flotation (pH 11.0). The residual metal levels were 0.08 ppm (Cd), 0.08 ppm (Cu), and 0.3 ppm (Pb) after 10 min treatment under optimum pH (pH 10.0). The separation efficiency was very sensitive to the ionic strength variation. The floc cannot be removed from the solution containing 0.02 *M* Na_2SO_4,

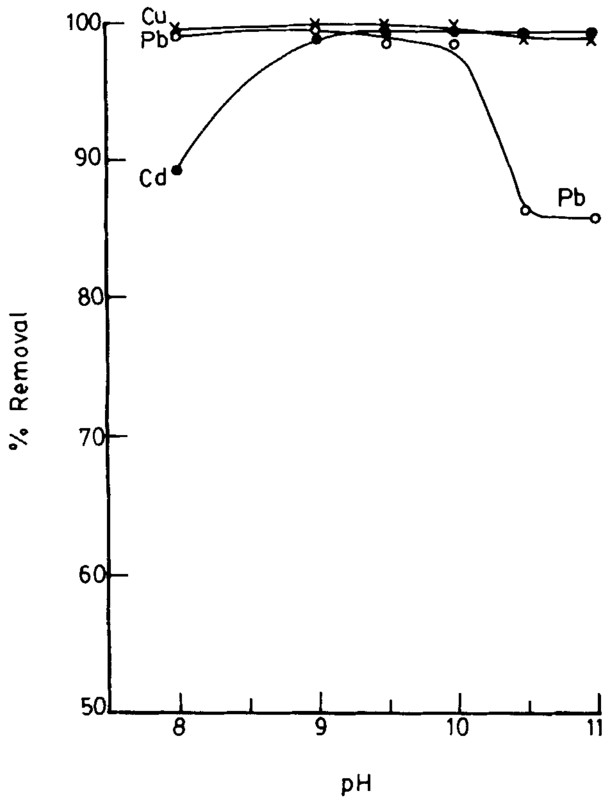

FIG. 8. Effect of pH on flotation of Cd(II)-Pb(II)-Cu(II) with Al(III). Al(III) dosage = 100 ppm, (O) percent removal of Pb(II), (X) percent removal of Cu(II), (●) percent removal of Cd(II).

probably due to the decrease of the surface potential of the floc by Na_2SO_4. It was found that the effect of ionic strength variation on separation efficiency can be compensated somewhat with the aid of zinc ion as the activator. The pH of the solution was adjusted to a value somewhat higher than 9.5, and after zinc solution was added, the pH value dropped to 9.5, and then the floc was removed by flotation. The results are shown in Table 3. It was found that good separation efficiency could be achieved from the solution containing 0.1 *M* Na_2SO_4 when Zn(II) was used as the activator.

Adsorbing colloid flotation using $Fe(OH)_3$ as the coprecipitant was also attempted. The effect of pH on separation efficiency is shown in Fig. 9. The residual cadmium level was somewhat higher than 0.1 ppm after 10 min flotation under the optimum pH range (9.5 to 10.0). At pH 10.5, the floc could not be removed from the solution, which resulted in very poor separation. The zeta potentials of the floc at various pH were shown in Table 4. The zeta potential of the floc decreases with increasing pH value of the solution. At pH 10.5, the zeta potential of the floc was highly negative, such that the negatively charged surfactant could not be adsorbed effectively on the surface of the floc to render the floc hydrophobic; this may account for the poor separation at pH 10.5.

The efficiency of removal can be improved somewhat [with residual Cd(II), Cu(II), and Pb(II) levels less than 0.1 ppm] when a suitable amount of aluminum(III) was also added to the system before pH adjustment. The results are shown in Table 5. The improvement in separation with the aid of Al(III) was probably due to the enhancement in coprecipitation of the metal ions with the floc by the aluminum hydroxide. Note that an overdosage of Al(III) resulted in very poor separation. The zeta potentials of the floc produced from the solution

TABLE 3

Effect of Sulfate on Flotation of Cd-Cu-Pb with $Al(OH)_3$ and Zn(II)[a]

Na_2SO_4 (*M*)	Residual metal concentration (ppm)[b] Cd	Cu	Pb
0.02	0.03	0.01	0.1
0.05	0.02	0.02	0.1
0.10	0.05	0.05	0.2
0.20	0.12	0.21	0.4

[a] pH = 9.5, initial Al(III) = 150 ppm, initial Zn(II) = 40 ppm.

[b] Residual Zn(II) less than 0.3 ppm.

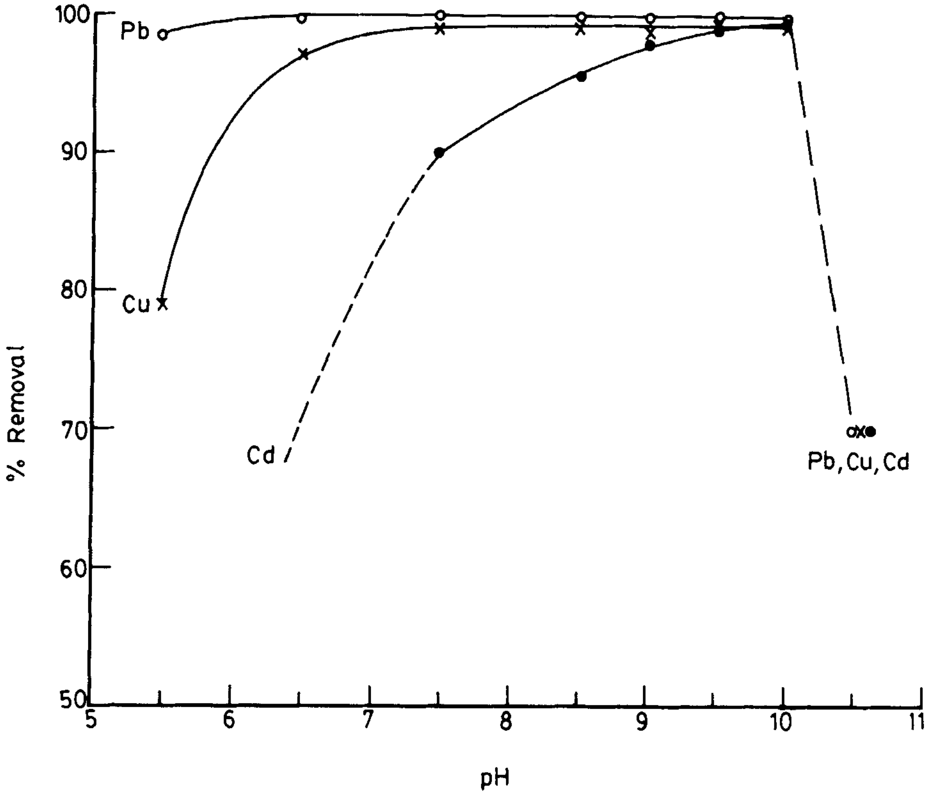

FIG. 9. Effect of pH on flotation of Cd(II)-Pb(II)-Cu(II) with Fe(III). Fe(III) dosage = 100 ppm, duration of runs = 10 min, (O) percent removal of Pb(II), (X) percent removal of Cu(II), (●) percent removal of Cd(II).

with various dosage of Al(III) are shown in Table 6. At pH 9.5, the zeta potential of the floc decreased with increasing Al(III) dosage, such that the floc could not be removed from the solution when too much Al(III) was added to the solution. The separation efficiency decreased with increasing ionic strength of the solution, as shown in Table 7. The

TABLE 4

Effect of pH on the Zeta Potential of the Floc Containing Fe, Cd, Cu, and Pb[a]

pH	7.5	8.5	9.5	10.5
Zeta potential (mV)	45 ± 7	35 ± 6	22 ± 4	−26 ± 4

[a] Fe(III) = 100 ppm, Cu(II) = 20 ppm, Cd(II) = 20 ppm, Pb(II) = 20 ppm.

TABLE 5
Effect of Al(III) Dosage on Flotation of Cd-Cu-Pb with $Fe(OH)_3$[a]

Al(III) (ppm)	Residual metal concentration (ppm)			
	Cd	Cu	Pb	Al
10	0.08	0.07	0.1	4.4
20	0.10	0.06	0.1	10.2
50	0.11	0.04	0.2	26.4
100	>5	>5	>5	>50

[a] pH = 9.5, initial Fe(III) = 100 ppm, duration of runs = 10 min.

TABLE 6
Effect of Al(III) Dosage on the Zeta Potential of the Floc Containing Fe-Cd-Cu-Pb[a]

Al(III) (ppm)	10	20	50	100
Zeta potential (mV)	18 ± 2	16 ± 1	11 ± 2	0 ± 1

[a] Fe(III) = 100 ppm, pH = 9.5.

residual cadmium level was higher than 0.1 ppm from the solution containing 0.02 *M* Na_2SO_4. The effect of sulfate on separation efficiency could be compensated somewhat with the aid of Zn(II) as the activator. The results are shown in Table 8. Fe(III) and Al(III) were added to the solution first, the pH of the solution was adjusted to 10.3 with NaOH solution, a solution of Zn(II) was added (which causes the pH of the solution to decrease somewhat to a level of about 9.5), and the floc was then removed by flotation. Effective removing efficiency can be achieved from a solution containing 0.1 *M* Na_2SO_4.

CONCLUSION

It was found that all the toxic heavy metal ions can be removed simutaneously from the systems being studied by a single step foam flotation treatment. The separations were effective from solutions containing a high concentration of neutral salts. The rate of separation was very fast; a time of less than 10 min was required for effective batch foam separation. Adsorbing colloid flotation proved to be a powerful technique for heavy metal ions removal from wastewater.

TABLE 7
Effect of Sulfate on Adsorbing Colloid Flotation of Cd-Cu-Pb with $Fe(OH)_3$ and Al(III)[a]

Na_2SO_4 (*M*)	Al(III) (ppm)	Residual metal concentration (ppm)		
		Cd	Cu	Pb
0.01	10	0.03	0.08	0.2
0.01	20	0.10	0.07	0.2
0.02	10	0.25	0.18	0.3
0.02	20	0.37	0.20	0.4

[a] pH = 9.5, Fe(III) = 100 ppm, duration of run = 10 min.

TABLE 8
Effect of Sulfate and Zn(II) on Adsorbing Colloid Flotation of Cd-Cu-Pb with $Fe(OH)_3$ and Al(III)[a]

pH	Na_2SO_4 (*M*)	Reagent dosage (ppm)		Residual metal concentration (ppm)[b]		
		Al(III)	Zn(III)	Cd	Cu	Pb
9.78	0	5	16	0.04	0.05	0.2
9.45	0.05	10	20	0.08	0.04	0.2
9.46	0.10	20	32	0.08	0.06	0.2
9.57	0.20	30	32	0.13	0.07	0.2

[a] Fe(III) = 150 ppm, duration of run = 10 min.
[b] Residual zinc less than 1 ppm.

Acknowledgment

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