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Removal of Cadmium, Lead, Mercury, Tin, Antimony, and Arsenic from Drinking and Seawaters by Colloid Precipitate Flotation

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ABSTRACT

The removal of Cd(II), Pb(II), Hg(II), Sn(II), Sn(IV), Sb(III), Sb(V), As(III), and As(V) from aqueous solutions by colloid precipitate flotation using sodium sulfide as the coagulent and oleic acid (HOL) as the surfactant has been investigated. The complete flotation (about 100%) of these elements was attained at pH values of 5.5–6.5, 3–6.5, ≤1, 1–4, 0.5–3, and ≤2, respectively. The effects of some other factors, such as surfactant and coagulent concentrations, sequence of adding reagents, some selected foreign ions, ionic strength, and temperature, on the floatability of these elements have been studied. It was found that both temperature and ionic strength have no appreciable effect on the flotation efficiency of the metal ions investigated. The method was successfully applied to remove completely these metal ions added to 1 L samples of drinking and seawaters at the optimum conditions for each element. Moreover, the mechanism of flotation is proposed.

INTRODUCTION

Cadmium is one of the more toxic trace metals. The effects of acute cadmium poisoning are manifested in a variety of symptoms, including high blood pressure, kidney damage, and destruction of red blood cells. Lead is considered to be a serious cumulative body poison. Mercury is considered to be a serious heavy metal pollutant. Tin has so far received little attention as an environmental pollutant in natural waters. Antimony and arsenic have long been recognized as toxicants (1, 2). Therefore, from the viewpoints of environmental chemistry, geochemistry, marine biology, and limnology, it is important to establish a rapid, simple, sensitive, and accurate method for the removal of these elements from water.

Various separation techniques are available which include volatilization, liquid–liquid extraction, selective dissolution, precipitation, electrochemical deposition and dissolution, sorption, ion exchange, liquid chromatography, flotation, freezing, and zone melting (3). Although some of these techniques are simple, rapid, quantitative, and selective under proper conditions, they are frequently time-consuming and troublesome (3). To compensate for these difficulties, the precipitate flotation technique was the choice for this investigation.

The precipitate flotation technique has received attention from a number of investigators, beginning with the work of Skarylev and Mokrushin (4) and that of Baarson and Ray (5). A number of studies have been carried out (6–16) including an excellent group of papers by Pinfold et al. (17–20), by Rubin et al. (21, 22), and another by Grieves et al. (23, 24).

Although sodium sulfide is a typical example of a coagulent for precipitating metal sulfides (25), little work have been carried out by Ghazy and Kabil (26–29) for the flotation of heavy metal ions as sulfides. Therefore, the objective of the present work is to extend the application of colloid precipitate flotation to remove Cd(II), Pb(II), Hg(II), Sn(II), Sn(IV), Sb(III), Sb(V), As(III), and As(V) from drinking and seawaters by using sodium sulfide as the precipitant and oleic acid surfactant at the optimum conditions.

EXPERIMENTAL

Apparatus

All glassware was soaked overnight in a chromic mixture ($K_2Cr_2O_7$ + concentrated H_2SO_4), rinsed thoroughly with double distilled water, and dried in dust-free air.

A Perkin-Elmer Model 2380 atomic absorption spectrophotometer was used throughout. The optimum parameters used are presented in Table 1.

All the pH measurements were carried out with a Hanna Instruments Model 8519 digital pH meter, together with a combined glass electrode.

The flotation and separation cells were of two types (30). Type (a) was a graduated tube of 12 mm inner diameter and 290 mm length with a stopcock at the bottom. This cell was used to study the different factors affecting the separation of the investigated ions from aqueous solutions. Type (b) was a glass cylinder of 6 cm inner diameter and 45 cm length

TABLE 1
The Optimum Instrumental Parameters for AAS Determination of Cd(II) and Pb(II)

Parameter	Cd(II)	Pb(II)
Wavelength (nm)	228.8	217.0
Slit width (nm)	0.7	0.7
Lamp current (mA)	4	10.0
Fuel flow rate (L/min)	3	3
Air flow rate (L/min)	21	21
Observation height (cm)	0.9	2.1

with a stopcock at the bottom and a quick-fit stopper at the top. This cell was used to separate the studied ions from 1 L of drinking and seawaters.

Reagents

All the reagents used were of BDH and analytical-reagent grades. Oleic acid stock solution was prepared from the food grade, with *d* 0.985, by dispersing in kerosene. Cadmium(II), lead(II), and mercury(II) stock solutions were prepared from $CdCl_2 \cdot H_2O$, $Pb(NO_3)_2$, and $HgCl_2$ by dissolving the requisite quantity in double-distilled water. Antimony stock solutions were prepared from Sb_2O_3 in dilute HCl and Sb_2O_5 in concentrated HCl. Arsenic stock solutions were prepared from $NaAsO_2$ and $Na_2HAsO_4 \cdot 12H_2O$ in double-distilled water. Tin stock solutions were prepared from $SnCl_2 \cdot 2H_2O$ by dissolving in concentrated HCl and completing with water to the desired volume, while $SnCl_4$ solution was prepared from high purity tin metal by heating with concentrated H_2SO_4, cooling, and completing to the required volume with dilute HCl. Sulfide stock solution was prepared daily from $Na_2S \cdot H_2O$ in double-distilled water. The pH of the solutions during experiments was controlled with HCl or HNO_3 and/or NaOH.

Procedure

For Aqueous Solutions

20 cm^3 of an aqueous solution containing defined concentrations of the metal ion, HCl or HNO_3 (for controlling pH), Na_2S, and HOL were introduced into the flotation cell (type a). The flotation cell was gently turned upside down 20 times by hand and then vigorously shaken 10 times to complete the flotation.

Concentrations of the investigated ions in the underlying solutions (and sometimes in the scum after elution with a suitable eluent) were determined by atomic absorption spectrometric measurements for cadmium and lead, complexometric measurements (31) for mercury, and iodimetric measurements (32) for tin, arsenic, and antimony. Arsenic(V) was also determined by the Volhard method (32).

The floatability F (%) was calculated by:

$$F = [(C_i - C_f)/C_i] \times 100$$

where C_i and C_f denote the concentrations of the metal ion before and after flotation, respectively.

Alternatively, the floatability F (%) can be calculated from the concentrations of the floated metal ions after elution (CdS and PbS by hot dilute HNO_3; HgS by aqua regia, Sb_2S_3, Sb_2S_5, SnS, and SnS_2 by warming with concentrated HCl, As_2S_3 by hot concentrated HNO_3; and As_2O_5 by ammonium hydroxide) using the following equation:

$$F = (C_f/C_i) \times 100$$

where C_i is the initial concentration and C_f is the concentration of the metal ion in the float.

For Drinking and Seawaters

Place 1 L of filtered uncontaminated water sample (taken up from different locations, and with the pH adjusted to the optimum value with HCl or HNO_3) into the flotation cell (type b), a defined concentration of Na_2S, and 5 mL oleic acid of the required concentration. Follow the previously described steps to complete the flotation.

Unless otherwise stated, the flotation measurements were carried out at room temperature, about 25°C.

To study the effect of temperature on flotation efficiency, the above steps were carried out in addition to either cooling or heating the solutions to the required temperature and pouring the solutions at time zero into the flotation cell jacketed with 1-cm fiberglass insulator.

RESULTS AND DISCUSSION

Effect of pH

An initial series of flotation experiments (using HOL surfactant, 6×10^{-3} mol·L^{-1}, and Na_2S precipitant, 4×10^{-4} mol·L^{-1}) was carried out in an effort to establish the optimum pH for the flotation of lead, cadmium, and mercury. The results are presented in Fig. 1. A maximum removal

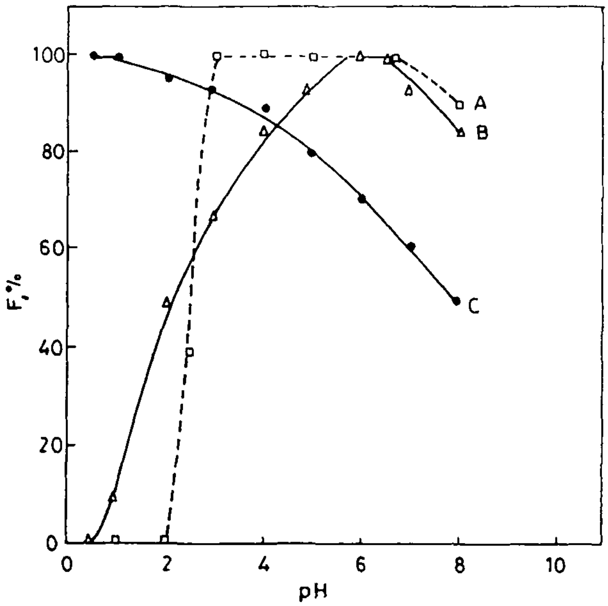

FIG. 1 Effect of pH on the floatability of 1 × 10^{-4} mol·L^{-1}; A, Pb(II); B, Cd(II); and C, Hg(II) using Na_2S, 4 × 10^{-4} mol·L^{-1} and HOL, 6 × 10^{-3} mol·L^{-1}.

of Hg(II) of about 100% was obtained at $pH \leq 1$. This may be attributed to the stability of HgS in highly acidic solutions. The flotation of Hg(II) decreases at higher pH values owing to the dissociation of HOL surfactant. The maximum flotation efficiency (about 100%) of both Pb(II) and Cd(II) was obtained at pH ranges of 3–6.5 and 5.5–6.5, respectively. At lower pH values the flotation of Pb(II) and Cd(II) is lower, indicating inefficient or no flotation of the precipitate. This was possible because of the solubility of PbS and CdS in strongly acidic solutions.

Another series of experiments were conducted (using HOL surfactant, 6 × 10^{-4} mol·L^{-1} and Na_2S precipitant, 5 × 10^{-4} mol·L^{-1}) to elucidate the optimum pH for the separation of Sn(II), Sn(IV), Sb(III), Sb(V), As(III), and As(V). The results are shown in Fig. 2. As can be seen, the maximum flotation efficiency (about 100%) of Sn(II) and Sn(IV) (curve A), Sb(III) and Sb(V) (curve B), and As(III) and As(V) (curve C) is obtained at pH ranges of 1–4, 0.5–3 and ≤ 2, respectively. The maximum floatability of each of these elements is obtained in acidic region.

Comparing Figs. 1 and 2, it seems that there is a reasonable relationship between the solubility product of the metal sulfides and the pH of their

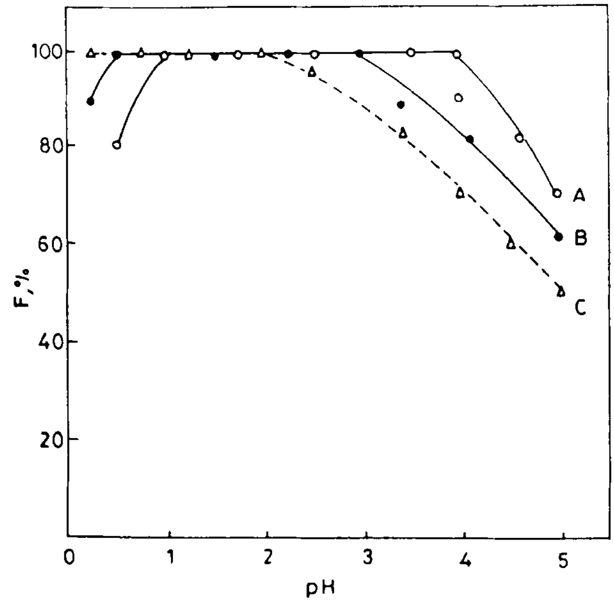

FIG. 2 Effect of pH on the floatability of 1 × 10^{-4} mol·L^{-1}; A, Sn(II) and Sn(IV); B, Sb(III) and Sb(V); and C, As(III) and As(V) using Na_2S, 5 × 10^{-4} mol·L^{-1} and HOL, 6 × 10^{-4} mol·L^{-1}.

maximum floatability. The metal sulfides having the lowest solubility product float efficiently at lower pH values. As an example, HgS with $K_{sp} = 6.44 \times 10^{-53}$ floats at pH ≤ 1 while CdS with $K_{sp} = 1.40 \times 10^{-29}$ floats at the pH range 5.5–6.5 and so on. Moreover, the flotation of the sulfides of these elements generally decreases in the alkaline medium, which may be due to the formation of oleate ions and excessive foams distributed through the whole bulk of the floating medium.

Effect of Surfactant Concentration

In order to find the most suitable concentration of HOL surfactant for removing 1 × 10^{-4} mol·L^{-1} Cd(II), Pb(II), and Hg(II) from aqueous solutions, several experiments were conducted using Na_2S, 4 × 10^{-4} mol·L^{-1}, at the optimum pH. The results are shown in Fig. 3. As can be seen, the flotation reaches its maximum value at HOL concentration ranges from 1 × 10^{-5} to 7.5 × 10^{-3} mol·L^{-1} for Hg(II), 6–7 × 10^{-3} mol·L^{-1} for Pb(II), and 6.0–6.5 × 10^{-3} mol·L^{-1} for Cd(II). Therefore,

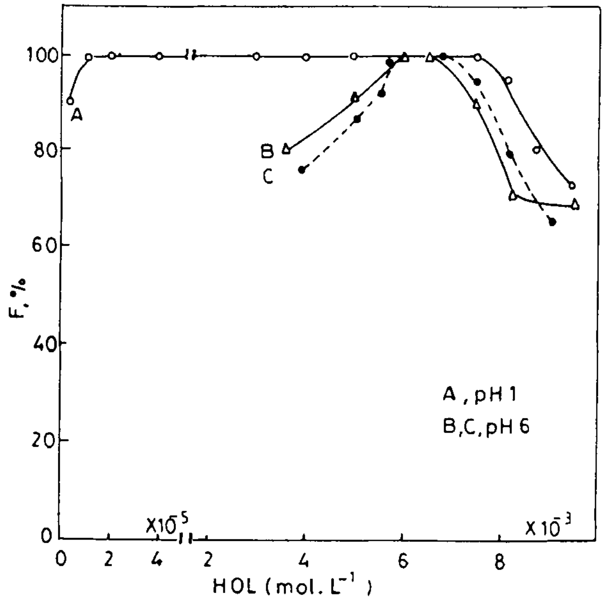

FIG. 3 Effect of surfactant concentration on the floatability of 1 × 10^{-4} mol·L^{-1}; A, Hg(II); B, Cd(II); and C, Pb(II) using Na_2S, 4 × 10^{-4} mol·L^{-1}.

6 × 10^{-3} mol·L^{-1} HOL was used for all other flotation measurements of these elements.

Another series of experiments were also performed at different concentrations of HOL to float 1 × 10^{-4} mol·L^{-1} of Sn(II), Sn(IV), Sb(III), Sb(V), As(III), and As(V) from aqueous solutions using Na_2S, 5 × 10^{-4} mol·L^{-1} at the optimum pH (Fig. 4). The maximum floatability of these elements is attained at HOL concentration ranges from 1 × 10^{-5} to 6.25 × 10^{-3} mol·L^{-1} for Sn(II), 1 × 10^{-5} to 6.75 × 10^{-3} mol·L^{-1} for Sb(III) and As(III), 1 × 10^{-5} to 7.25 × 10^{-3} mol·L^{-1} for Sb(V) and As(V), and 1 × 10^{-5} to 7.75 × 10^{-3} mol·L^{-1} for Sn(IV). The suitable HOL concentration chosen for other flotation measurements of these elements was 6 × 10^{-4} mol·L^{-1}.

From Figs. 3 and 4 it is noted that the maximum floatability of the metal sulfides is obtained at a HOL concentration which is about 10% of that of the investigated elements except for Cd and Pb. This is in good agreement with the literature data (23) reporting that the precipitate flotation may require a quantity of surfactant only 5 to 20% of the stoichiometric

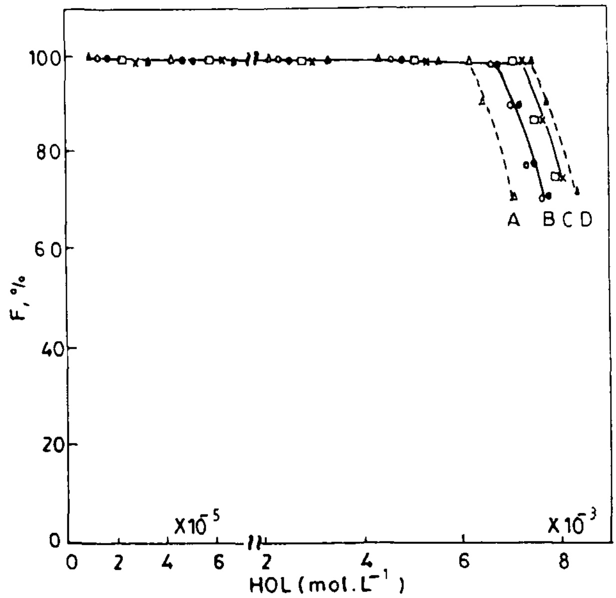

FIG. 4 Effect of surfactant concentration on the floatability of 1 × 10^{-4} mol·L^{-1}; A, Sn(II); B, Sb(III) and As(III); C, Sb(V) and As(V); and D, Sn(IV) using Na_2S, 5 × 10^{-4} mol·L^{-1} at pH 1.5.

quantity. Moreover, the flotation efficiency decreases at higher concentrations of HOL, which may be attributed to the fact that the surfactant changes the state of suspension of the particles from coagulation precipitation through coagulation flotation to redispersion with an increase in the amount of the surfactant added (33).

Effect of Sulfide Concentration

Figure 5 shows the floatability of 1 × 10^{-4} mol·L^{-1} of Cd(II), Pb(II), and Hg(II) from aqueous solutions using different concentrations of Na_2S and a defined concentration of HOL, 6 × 10^{-3} mol·L^{-1}, at the optimum pH for each element. The maximum floatation efficiency of each element is achieved when the stoichiometric ratio between the metal and sulfide ions is verified.

A similar trend is obtained on floating 1 × 10^{-4} mol·L^{-1} of Sn(II), Sn(IV), Sb(III), Sb(V), As(III), and As(V) from aqueous solutions under the optimum conditions set by the legend of Fig. 6. However, the flotation efficiency of these elements (Figs. 5 and 6) decreases at higher concentra-

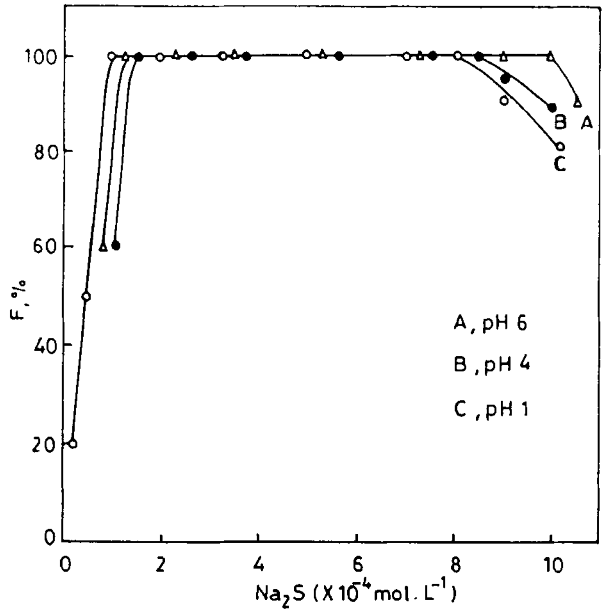

FIG. 5 Effect of coagulent concentration (Na_2S) on the floatability of 1 × 10^{-4} mol·L^{-1}; A, Cd(II); B, Pb(II); and C, Hg(II) using HOL, 6 × 10^{-3} mol·L^{-1}.

tions of Na_2S. This may be attributed to (a) the reaction between excess Na_2S and HOL (consuming the amount of HOL surfactant available for flotation) and/or (b) the solubility of some metal sulfides in an excess of Na_2S, e.g., HgS is appreciably soluble in sodium sulfide (34) while Cd(II) and Pb(II) show solubility increases with increasing sulfide concentration (35).

Order of Addition of Reagents

In order to compensate for some problems such as the formation of excessive foaming in the bulk solution, the evolution of H_2S gas, the dispersion of metal sulfide precipitates in the flotation pulp, and the formation of white emulsion in the mother liquor originating from oleate ions, an extensive series of experiments was performed (under the optimum conditions) to study the effect of the order of adding reagents on the floatation efficiency of the metal ions. It has been found that the best sequence for adding reagents is in the order metal ion, HNO_3 or HCl and/or NaOH (for controlling pH), Na_2S, and HOL surfactant.

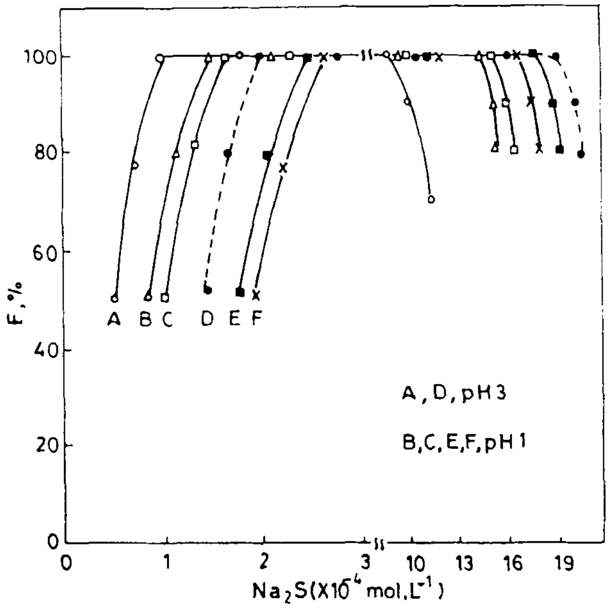

FIG. 6 Effect of coagulent concentration (Na_2S) on the floatability of 1 × 10^{-4} mol·L^{-1}; A, Sn(II); B, Sb(III); C, As(III); D, Sn(III); E, Sb(V); and F, As(V) using HOL, 6 × 10^{-4} mol·L^{-1}.

Effect of Temperature

The effect of temperature on the floatability of the metal ions studied was investigated in a wide range of temperatures from 10 to 80°C under the optimum conditions. A change in temperature has no appreciable effect on the flotation efficiency of the metal ions except for decreasing the time of flotation at higher temperatures. An increase in temperature promotes collection by speeding up the thinning of the hydration layers between the aggregates and bubbles, thereby increasing the probability of attachment, which leads to a high flotation efficiency.

Effect of Ionic Strength

Table 2 shows the effect of greater increases in ionic strength and of varying the ionic strength with different salts. The optimum conditions for the flotation of the metal ions investigated are presented in the column head of Table 2. Clearly, the valency and the type of the cation or anion could be taken into account by the ionic strength. In particular, there was

TABLE 2

Effect of Salt Variation and Ionic Strength on the Floatability of 1 × 10^{-4} mol·L^{-1} of Cd, Pb, Hg (using Na_2S, 4 × 10^{-4} mol·L^{-1}, and HOL, 6 × 10^{-3} mol·L^{-1}), Sn, Sb, and As (using Na_2S, 5 × 10^{-4} mol·L^{-1}, and HOL, 6 × 10^{-4} mol·L^{-1}) at 25°C

Ionic strength (mol·L^{-1})	Salt	Floatability (%)								
		Cd(II), pH 6.0	Pb(II), pH 6.5	Hg(II), pH 1.0	Sn(II), pH 3.0	Sn(IV), pH 3.0	Sb(III), pH 2.5	Sb(V), pH 2.5	As(III), pH 1.0	As(V), pH 1.0
0.01	NaCl	99.95	99.90	99.98	99.00	99.50	99.95	99.40	99.00	99.45
	KCl	99.90	99.92	99.95	99.20	99.55	99.93	99.45	99.20	99.65
	$MgCl_2$	99.82	99.50	99.10	99.15	99.15	99.60	99.70	99.10	99.45
	$CaCl_2$	98.00	99.00	99.20	99.10	99.12	99.50	99.65	99.20	99.35
	Na_2SO_4	99.50	99.60	99.90	99.30	99.25	99.65	99.72	99.25	99.50
	$MgSO_4$	99.10	99.20	99.80	99.20	99.20	99.30	99.50	99.12	99.32
	$CaSO_4$	98.50	98.60	99.50	99.15	99.15	99.10	99.40	99.11	99.13
0.05	NaCl	99.90	99.82	99.95	98.80	99.40	99.70	99.30	99.00	99.30
	KCl	99.82	99.74	99.90	98.75	99.30	99.65	99.22	98.80	99.20
	$MgCl_2$	98.00	97.90	99.80	98.65	99.20	99.30	99.15	98.70	99.10
	$CaCl_2$	97.90	97.60	99.80	98.70	99.20	99.25	99.20	98.65	99.00
	Na_2SO_4	98.50	98.70	99.85	99.00	99.13	99.20	99.15	98.90	99.10
	$MgSO_4$	98.40	98.30	99.00	98.90	99.00	99.13	99.00	98.90	99.00
	$CaSO_4$	97.80	97.20	99.00	98.92	99.10	99.10	99.00	98.70	99.00
0.50	NaCl	93.00	96.00	99.00	98.20	99.00	99.10	98.15	97.90	98.50
	KCl	99.80	99.60	99.00	98.60	99.20	98.20	98.20	97.85	98.00
	$MgCl_2$	97.20	98.20	97.90	98.20	99.15	98.30	98.90	98.00	98.00
	$CaCl_2$	97.00	96.50	97.80	98.10	99.10	97.90	98.50	98.10	98.10
	Na_2SO_4	97.10	96.80	97.90	98.00	99.10	98.00	98.60	98.00	97.90
	$MgSO_4$	97.20	96.90	98.00	98.30	98.90	98.13	98.25	98.00	97.85
	$CaSO_4$	97.30	96.80	98.20	98.00	98.15	98.50	98.13	97.90	97.20

no evidence of a specific reaction between Cl^-, SO_4^{2-} and the floated metal ions (except between Cl^-, SO_4^{2-}, and Pb^{2+}, which did not affect the floatability of PbS). It is evident that a change in ionic strength has no appreciable effect on the flotation efficiency of the metal ions investigated. This is in good agreement with literature data which states that precipitate flotation is completely independent of ionic strength (22). However, a slight decrease (about 3%) was obtained in the flotation efficiency of Cd^{2+} and Pb^{2+} (especially in the case of Mg and Ca salts). This may be attributed to the reaction between Mg^{2+}, Ca^{2+} and oleate ions, obtained at pH ≥ 5.2 (36) and forming white precipitates, which leads to a decrease in the HOL surfactant available for flotation.

Effect of Some Foreign Ions

By following the proposed procedure, the effects of various foreign ions on the separation of the metal ions studied from aqueous solutions were investigated under optimum conditions. It has been found that the following anions (present in a 200-fold concentration of the investigated metal ions), oxalate, citrate, tartarate, chloride, bromide, nitrate, and sulfate, and the following cations (present in a 100-fold concentration), K^+, Na^+, Fe^{3+}, Al^{3+}, Ag^+, Cu^{2+}, Bi^{3+}, Mg^{2+}, Ca^{2+}, Co^{2+}, Ni^{2+}, Mn^{2+}, Ba^{2+}, Sr^{2+}, and Zn^{2+}, have no effect on the flotation efficiency. This may be due to the fact that the separation of the metal ions investigated as sulfides was carried in acidic region which may prevent the formation of sulfides of most of the foreign cations. However, if some foreign cations (such as Ag^+, Bi^{3+}, and Cu^{2+}) interfere to some extent, their effect was eliminated by adding a slight excess of the sulfide precipitant.

Mechanism of Flotation

In this precipitate flotation the fundamental process may be the interaction of the surfactant with the sulfide surfaces. This process renders the surface hydrophobic and so enables the sulfide precipitates to be held at the air–water interface.

The flotation pulp consists of sulfide precipitates in an aqueous solution of electrolytes, and an electrical double layer is formed at the solid–solution interface. The surfactant–precipitate interaction takes place in this double layer and is, therefore, electrochemical in nature.

Colloidal systems (such as metal sulfides) have an ion-exchange capacity (37), and the presence of ionic points on their surfaces originates from the solvation of the electrical double layer (38). Both the undissociated HOL molecules, obtained at $pH \leq 5.2$ (36), with polar carboxylic groups and oleate ions, obtained at $pH \geq 5.2$, combine electrostatically with sulfide precipitates, and the hydrophobic aggregates float on air bubbles to the surface of the solution (26–29).

Application

In order to investigate the applicability of this method to drinking and seawater, the recoveries of known amounts of cadmium, lead, mercury, tin, antimony, and arsenic added to drinking water and natural seawater samples taken up at two different locations were examined by the above procedure. The analyses were carried out on 1000-mL aliquots of clear, uncontaminated drinking and seawaters after adjusting the pH with HCl or HNO_3 immediately after sampling. Table 3 presents the recovery data

TABLE 3

Recovery of Cadmium, Lead, Mercury, Tin, Antimony, and Arsenic Added to 1 L Drinking and Seawater Samples

		Drinking water		Seawater	
Element	pH	Added (μg)	Mean recovery (%)	Added (μg)	Mean recovery (%)
Cd(II)	6	55	99.5[a]	55	99.0[c]
		75		75	
		55	99.2[b]	55	98.5[d]
		75		75	
Pb(II)	6	105	99.3[a]	105	99.2[c]
		150		150	
		105	99.0[b]	105	98.9[d]
		150		150	
Hg(II)	1	100	99.0[a]	100	98.9[c]
		160		160	
		100	99.2[b]	100	98.8[d]
		160		160	
Sn(II)	3	60	99.5[a]	60	99.1[c]
		80		80	
		60	99.6[b]	60	99.1[d]
		80		80	
Sn(IV)	3	60	99.2[a]	60	99.0[c]
		80		80	
		60	99.3[b]	60	99.1[d]
		80		80	
Sb(III)	2	65	99.0[a]	65	98.9[c]
		85		85	

(continued)

TABLE 3 Continued

		Drinking water		Seawater	
Element	pH	Added (μg)	Mean recovery (%)	Added (μg)	Mean recovery (%)
		65 85	98.9[b]	65 85	98.8[d]
Sb(V)	2	65 85	99.1[a]	65 85	98.9[c]
		65 85	99.0[b]	65 85	98.7[d]
As(III)	1	30 50	99.5[a]	30 50	99.0[c]
		30 50	99.4[b]	30 50	99.1[d]
As(V)	1	30 50	99.1[a]	30 50	99.0[c]
		30 50	99.2[b]	30 50	99.0[d]

[a] This sample was taken from drinking water of Mansoura City.
[b] This sample was taken from drinking water of Manzalah City.
[c] This sample was taken from the Mediterranean Sea (Alexandria).
[d] This sample was taken from the Mediterranean Sea (Gamasa).

of the elements investigated. These results indicate that the analytical process had a satisfactory recovery of cadmium and lead at pH 6, of mercury and arsenic at pH 1, of tin at pH 3, and of antimony at pH 1.

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