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Selective Flotation of Zinc(II) and Silver(I) Ions from Dilute Aqueous Solutions

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ABSTRACT

An experimental investigation is presented of the batch competitive flotation of zinc(II) and silver(I) ions from dilute aqueous solutions with sodium dodecylsulfate and ammonium tetradecylsulfonate as anionic surfactants and with cetylpyridinium chloride as a cationic surfactant. The sequence of growing affinity of metal cations to anionic surfactants is the same as the sequence of ionic potential values of the studied cations: $Ag^+ < Zn^{2+}$. The presence of potassium sulfate in aqueous solution has a negative influence of Zn^{2+} foam separation with a anionic surfactant which is due to competition for the surfactant between Zn^{2+} and K^+ cations. Also, the effect of inorganic ligands (i.e., thiosulfates, thiocyanates, and cyanides) on the selectivity of ion flotation of Zn(II) and Ag(I) is established. Results are discussed in terms of the complex species of zinc(II) and silver(I). At a total $S_2O_3^{2-}$ concentration of 3×10^{-6} M, the silver(I) is floated as a mixture of anions $[Ag(S_2O_3)]^-$ and $[Ag(S_2O_3)_2]^{3-}$, whereas zinc(II) remains in the aqueous phase as Zn^{2+}. At total concentrations of SCN^- from 1×10^{-4} to 2×10^{-3} M, silver(I) is floated as a mixture of $[Ag(SCN)_2]^-$ and AgSCN species. Partial separation of zinc(II) from silver(I) can be achieved in the presence of CN^- ligands at total concentrations varying from 2.5×10^{-4} to 1.0×10^{-3} M. The affinity of the studied cyanide complexes to cetylpyridinium chloride follows the order $[Ag(CN)_2]^- < [Zn(CN)_4]^{2-} + [Zn(CN)_3]^-$.

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INTRODUCTION

Ion flotation, one of the adsorptive bubble separation techniques, involves the removal of surface inactive ions from aqueous solutions by the introduction of a surfactant and the subsequent passage of gas bubbles through the solution. As a result of the process, a solid known as the sublate appears on the gas/aqueous solution surface. Higher concentrations of floated ions and surfactant may lead to precipitation of sublate in the solution. If the species to be floated is a cation or an anion (generally named *colligend*) which forms a soluble complex or an ion pair with the opposite charged surfactant ion, the process is named foam fractionation. Foam fractionation is often confused with ion flotation and sometimes these two processes occur simultaneously.

The ion flotation selectivity for inorganic cations has been presented in several papers. Walling et al. (1) found the following selectivity order of the foam separation process: $H^+ < Na^+ < K^+ < NH_4^+$, and $Mg^{2+} < Ca^{2+}$. Huang and Talbot (2) determined another selectivity order of foam fractionation: $Cu^{2+} < Cd^{2+} < Pb^{2+}$. Kubota and Hayashi (3) found the following selectivity order from continuous foam fractionation: $Na^+ < Cd^{2+} < Cr^{3+}$. Grieves et al. (4) determined separation selectivity coefficients for Li^+, K^+, Rb^+, and Cs^+ versus Na^+ equal to 0.92, 1.10, 1.51, and 1.65, respectively. Walkowiak (5) found the following foam separation selectivity sequences toward anionic surfactants: $Mn^{2+} < Zn^{2+} < Co^{2+} < Fe^{3+} < Cr^{3+}$, and $Ag^+ < Cd^{2+} < In^{3+}$. For flotation of cations (where no sublate was formed in the bulk solution), the good agreement between selectivity sequences of studied metal cations with anionic surfactants and the ionic potentials of those cations was found (5).

A series of batch foam fractionation experiments of oxy-, cyanide-, and chloride-complex anions from multimetal aqueous solution was conducted. A first study was carried out (6, 7) with oxy-complexes and the following separation selectivity sequence was established at pH 6.0 with hexadecyldimethylbenzylammonium chloride as a cationic surfactant: $MoO_4^{2-} \geq ReO_4^- > HCrO_4^- = WO_4^{2-} > H_2VO_4^-(VO_3^-)$. Another study was reported (8) with the cyanide complex anions of Zn(II), Cd(II), Hg(II), and Au(III) which led to the following selectivity sequence: $[Au(CN)_4]^- > Hg(CN)_4]^{2-} > Cd(CN)_4]^{2-} > Zn(CN)_4]^{2-}$. From continuous flow, foam fractionation of cyanide complex anions of silver(I), gold(I), zinc(II), cadmium(II), and mercury(II) the selectivity followed the sequences (9, 10): $[Au(CN)_2]^- > [Ag(CN)_2]^-$ and $[Hg(CN)_4]^{2-} > [Cd(CN)_4]^{2-} > [Zn(CN)_4]^{2-}$. The separation selectivity for chloride complexes at $[Cl^-] = 3.0$ M was (11): $[AuCl_4]^- > [HgCN_4]^{2-} > [CdCl_4]^{2-} + [CdCl_3]^- > [ZnCl_4]^{2-} + [ZnCl_3]^-$. Moreover, the selectivity of flotation of chloride complexes of palladium(II), platinum(IV), and gold(III) were studied using cetyltrimethylammonium chloride as a cationic surfactant (12). From flotation curves of competitive ion flotation, the following sequence of flotation selectivity was established: $[AuCl_4]^- > [PtCl_6]^{2-} > [PdCl_4]^{2-}$. A general criterion of anions affinity for cationic surfactants can be formulated: A higher affinity to the surfactant is observed for less hydrated anions. The ion–water interactions were found to govern the affinity of an anion to a cationic surfactant (12).

Removal of zinc(II) and silver(I) ions from aqueous solutions including wastewaters was studied recently. The application of liquid membranes for zinc(II) and silver(I) removal has been reviewed by Ho and Li (13), as well as by Hiratahi and Kasuga (14). The selective transport of Ag^+, Zn^{2+}, and other heavy metal ions, through supported liquid membranes was investigated by Saito (15). Silver(I) selective transport across polymer inclusion membranes containing new pyridyno- and bipyridynopodants was studied by Lamb and coworkers (16).

Adsorptive bubble separation techniques were also applied for zinc(II) and silver(I) removal from aqueous solutions. Removal of zinc(II) in the presence of chlorides, bromides, iodides, and thiocyanates by foam separation with cetyltrimethylammonium collector was investigated by Jurkiewicz (17). Ionic flotation of zinc(II) and other transition metal ions using fatty acids as surfactants was studied by Skrylev and coworkers (18). Another study reported (19) on the removal of zinc and other heavy metals of trace amounts from chelated solutions in electrolytic foam separation with sodium dodecylsulfate. Ion flotation of many metal ions, including Zn(II), and Ag(I), with cetylpyridinium chloride from mixed hydrochloric/nitric acid solutions was investigated by Hualing and Zhide (20). Silver(I) was floated from solution in 95–100% yield, while zinc(II) was only partly floated. He (21) reported on recovering of silver(I) from thiorea solution by ion flotation with sodium dodecylbenzenesulfonate. Silver recycling from a photographic bleach-fix bath by ionic flotation was studied by Jdid and coworkers (22).

Precipitate flotation of zinc(II) hydroxide from aqueous solutions using both cationic and anionic surfactants was studied by Jurkiewicz (23). Also, Zouboulis and coworkers (24) investigated precipitate flotation of zinc(II) hydroxide by dodecylamine. Precipitate flotation of zinc sulfide was studied by Stalidis et al. (25), as well as by Rao and Finch (26). Stalidis et al. (25) found the possibility of selective precipitate flotation of zinc and copper as sulfides from dilute aqueous solutions by the dissolved-air flotation technique. Zouboulis and coworkers (27) investigated zinc, nickel, and cadmium removal from dilute aqueous solutions by sorption onto nonliving anaerobically digested sewage sludge. The flotation was applied as an effective solid/liquid separation process using typical anionic and cationic surfactants.

Investigation of the selective flotation of silver(I) and zinc(II) ions from dilute aqueous solutions has a potential, practical meaning since silver often accompanies zinc in its ores (e.g., zinc ore deposit in southern Poland). Technologies for processing zinc minerals involve sulfuric acid leaching of oxidized mineral feed followed by electrowinning of zinc from the resultant aqueous solution. Therefore, streams of sulfate aqueous solutions of a wide range of concentrations containing Zn and Ag are generated in such industrial processes.

We now present an investigation on the selective separation of zinc(II) and silver(I) ions by flotation from dilute aqueous solutions using sodium dodecylsulfate and ammonium tetradecylsulfonate as anionic surfactants and cetylpyridinium chloride as a cationic surfactant. Flotation results are discussed in terms of the stability constants of thiosulfate, thiocyanate, and cyanide metal complexes.

EXPERIMENTAL

The initial aqueous solutions subjected to flotation were prepared with double distilled water of 1.5 $\mu\Omega^{-1}\cdot\text{cm}^{-1}$ conductivity at 25°C, and with the analytical grade $ZnSO_4$ and Ag_2SO_4. Known amounts of the sulfates of zinc(II) and silver(I) and 2.0–5.0 kBq of the respective gamma isotope (the analytical tracer) were added to 100 mL of each initial solution. For some experiments, known volumes of 0.1 M K_2SO_4, KSCN, KCN, and $K_2S_2O_3$ (analytical reagent grade) were added to the initial solutions of metals. To establish the ionic equilibrium, the initial solutions were aged for 6 hours before being floated.

The gamma radioactive isotopes, i.e., Zn-65 and Ag-110m, were used as sulfate aqueous solutions. They were of sufficiently high specific activity to neglect the effect of carrier concentration (17 GBq/g for Zn-65, and 5.4 GBq/g for Ag-110m). Radioactive isotopes were from the Atomic Energy Institute (Swierk, Poland).

Typical ionic surfactants were applied, i.e., sodium dodecylsulfate (DDSNa, BHD reagent) and ammonium tetradecylsulfonate ($TDSNH_4$, Fisher Scientific Co. reagent) as anionic surfactants and cetylpyridinium chloride (CPCl, Loba-Chemie reagent) as cationic surfactant. Surfactants were used as 0.050 M standard solutions in analytical reagent-grade ethanol. Addition of ethanol to the feed solution would slightly influence the course of flotation (20, 28). Therefore, the total volume of ethanol added in each experiment was kept constant, i.e., 0.5 mL, in order to neglect the above influence on our results. The concentration of surfactants in the initial aqueous solution was kept constant at 2.5×10^{-4} M throughout this investigation.

The flotation experiments were carried out in a glass column 45.7 cm high and 2.4 cm in diameter. The nitrogen gas was saturated with double distilled water, and the flow rate was maintained at 4 mL/minute through a sintered glass sparger of 20–30 μm nominal porosity. The initial volume of each feed solution was 100 mL. The temperature was maintained at 22 ± 2°C.

The time dependence of the concentration of each metal in the bulk solution (c) was recorded continuously during an ion flotation experiment by means of radiotracers and gamma radiation spectrometry following a procedure described previously (29). The c/c_i (c_i = initial metal ion concentration) vs time curves allowed the calculation of the maximal flotation percent, $(1 - c_r/c_i)100$, where c_r is the metal ion concentration in the residual solution when foaming ceases. Also, the degree of separation as the ratio of the maximal flotation percentages for both separated metals was calculated.

RESULTS AND DISCUSSION

The study of batch ion flotation first involved the separation of Zn(II) and Ag(I) cations with anionic surfactants. The kinetic curves for flotation of the studied cations with the anionic surfactants, ammonium tetradecylsulfonate and sodium dodecylsulfate, are shown in Fig. 1. The pH in the initial aqueous solutions for all cation flotations was kept constant and equal to 6.2. Zinc(II) cations floated well for both surfactants (Curves 1 and 2) while silver(I)

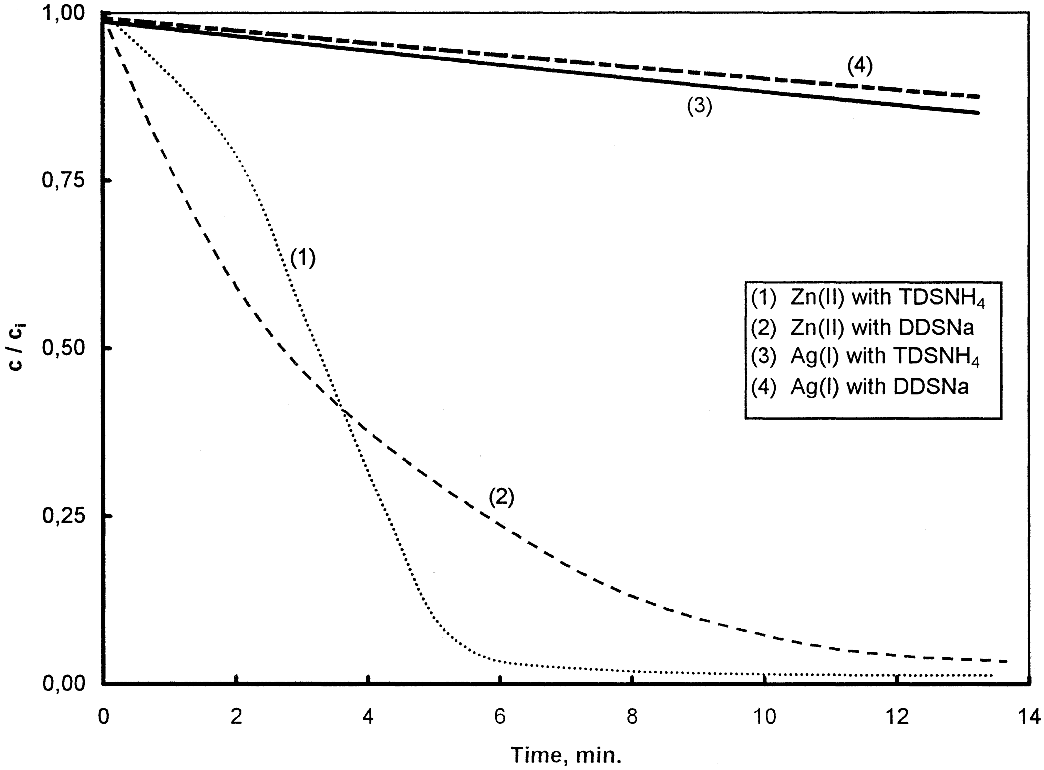

FIG. 1 Competitive flotation of zinc(II) and silver(I) cations with anionic surfactants. $c_{Zn(II)} = c_{Ag(I)} = 1.0 \times 10^{-6}$ M.

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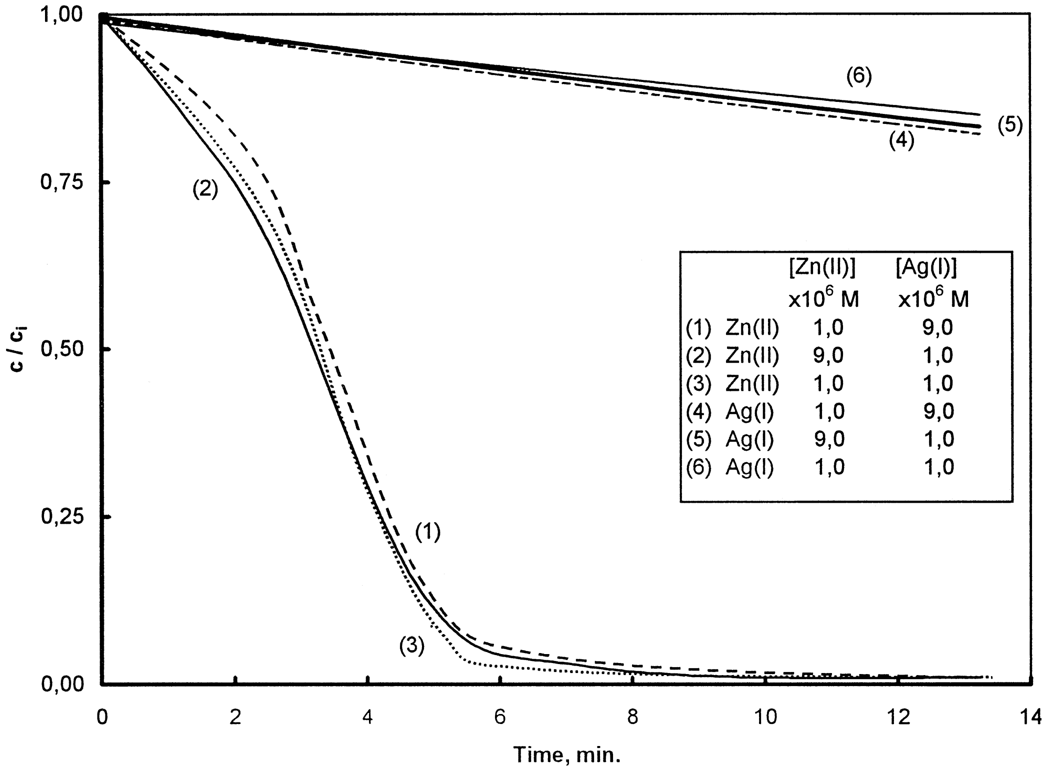
floated very poorly (Curves 3 and 4). The maximal flotation percent for zinc(II) cations was 99 after 6 minutes for $TDSNH_4$ and 96 after 10 minutes for DDSNa. The degrees of separation for $TDSNH_4$ and DDSNa were 6.9 and 8.4, respectively.

Varying the Zn(II)/Ag(I) concentration ratio from 0.11 to 9.0 gave similar kinetic curves for both cations (Figs. 2 and 3). A final series of flotation experiments for zinc(II) and silver(I) cations was made in order to determine the influence of potassium cations added to the feed solution as potassium sulfate (Fig. 4). Based on the kinetic curves of flotation, the presence of K_2SO_4 in an aqueous solution has a negative influence on Zn^{2+} removal with an anionic surfactant because the increase of potassium sulfate concentration causes competition for the surfactant between colligend Zn^{2+} and the added potassium cations. The removal of silver(I) remains at the 10–11% level.

According to the values of stability constants (30) of metal ions in aqueous solutions, zinc(II) and silver(I) totally remain in the form of Zn^{2+} and Ag^+ cations at pH 6.2. The course of flotations shows the higher affinity of diva-



		[Zn(II)] x10^6 M	[Ag(I)] x10^6 M
(1)	Zn(II)	1,0	9,0
(2)	Zn(II)	9,0	1,0
(3)	Zn(II)	1,0	1,0
(4)	Ag(I)	1,0	9,0
(5)	Ag(I)	9,0	1,0
(6)	Ag(I)	1,0	1,0

FIG. 2 Competitive flotation of zinc(II) and silver(I) cations with $TDSNH_4$ vs concentrations of Zn(II) and Ag(I).

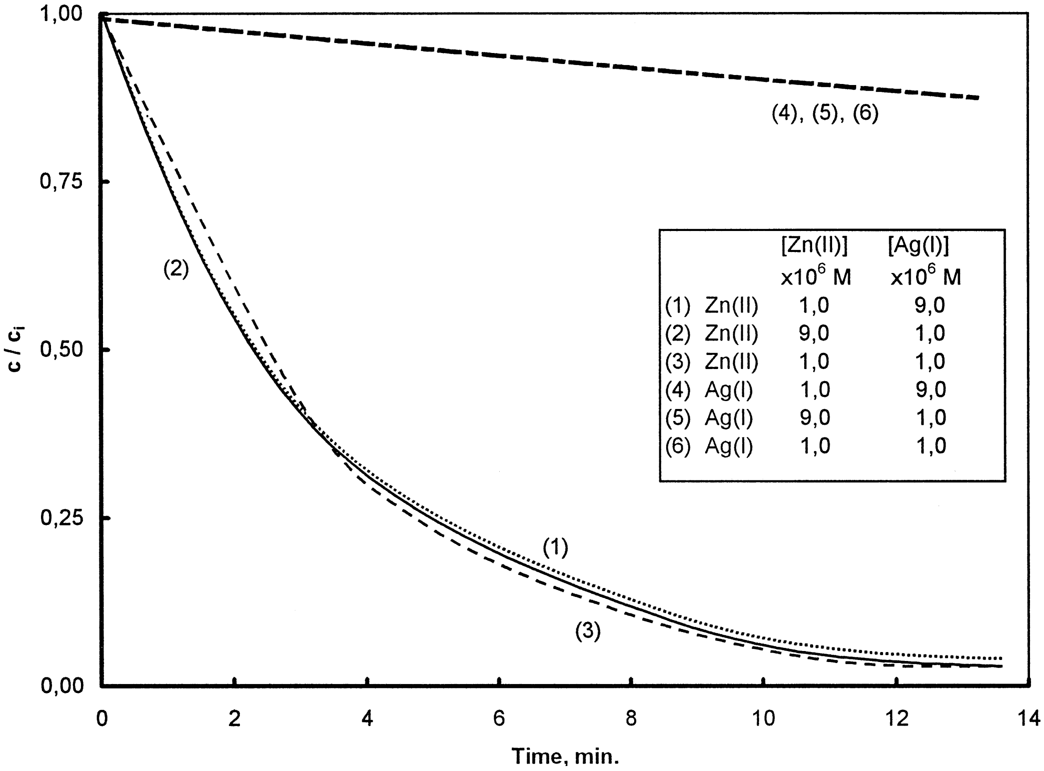


FIG. 3 Competitive flotation of zinc(II) and silver(I) cations with DDSNa vs concentrations of Zn(II) and Ag(I).

lent metal cations, i.e., Zn^{2+}, to anionic surfactants, while the monovalent metal cations, i.e., Ag^{+}, exhibit a lower affinity to these surfactants. The sequence of increasing affinity of metal cations to anionic surfactants follows the sequence of their ionic potentials:

$$\begin{array}{ccc} Ag^{+} & < & Zn^{2+} \\ 0.78 & & 2.25 \end{array}$$

Our results confirm the selective foam separation model proposed by Jorne and Rubin (31), which is based on the Gouy–Chapman diffusion double layer theory with the restriction that the size of hydrated cations determines their closest approach to the surface.

The second part of our experiments involved the competitive flotation of zinc(II) and silver(I) ions with cetylpyridinium chloride as a cationic surfactant from feed solutions containing inorganic ligands, i.e., thiosulfates, thiocyanates, and cyanides. The results are shown in Figs. 5, 6, and 7.

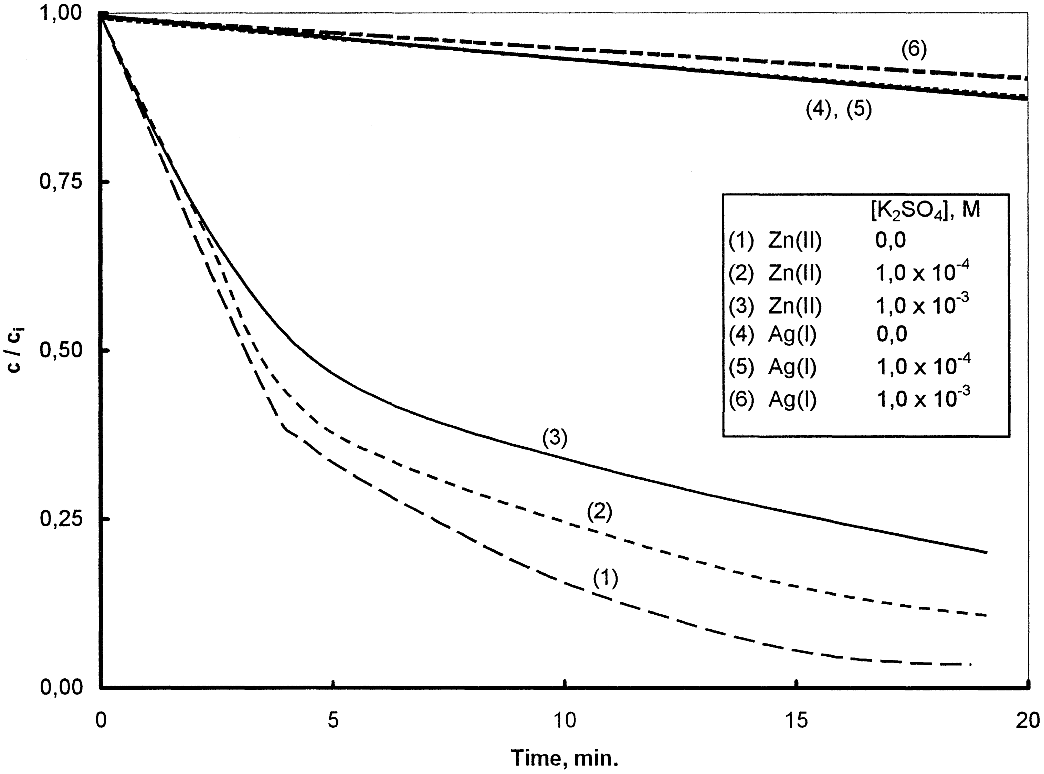


FIG. 4 Competitive flotation of zinc(II) and silver(I) cations with DDSNa from feed solutions containing K_2SO_4. $c_{Zn(II)} = c_{Ag(I)} = 1.0 \times 10^{-6}$ M.

To explain the results of ion flotation from solutions of inorganic ligands, the contributions of the complex species formed are needed. In Fig. 8 the percent molar contributions of thiosulfate, thiocyanate, and cyanide complex species for silver(I) and zinc(II) versus the ligand concentrations are shown. The percent contributions of the mentioned complexes for the chosen concentrations of ligands at which the flotation experiments were performed are given in Table 1. α_0 is the percent molar contribution of uncomplexed cations, i.e., Zn^{2+} and Ag^+. α_1, α_2, α_3, and α_4 are the percent molar contributions of complexed ions with 1, 2, 3, and 4 ligands, respectively.

At concentration of thiosulfate equal to 3×10^{-6} M, silver(I) remains in the form of an anionic species, i.e., $[Ag(S_2O_3)]^-$ and $[Ag(S_2O_3)_2]^{3-}$. The percent molar contribution for these complex anions is 87 and 12, respectively. Zinc(II) remains as Zn^{2+} cations for the same thiosulfate concentration. This explains the results of flotation of zinc(II) and silver(I) ions from solutions containing thiosulfates (Fig. 5). The maximal flotation percent of silver reaches 91 ÷ 96. The decrease in the maximal percent flotation of silver(I) with decreasing pH values can be explained in terms of decomposition of thio-

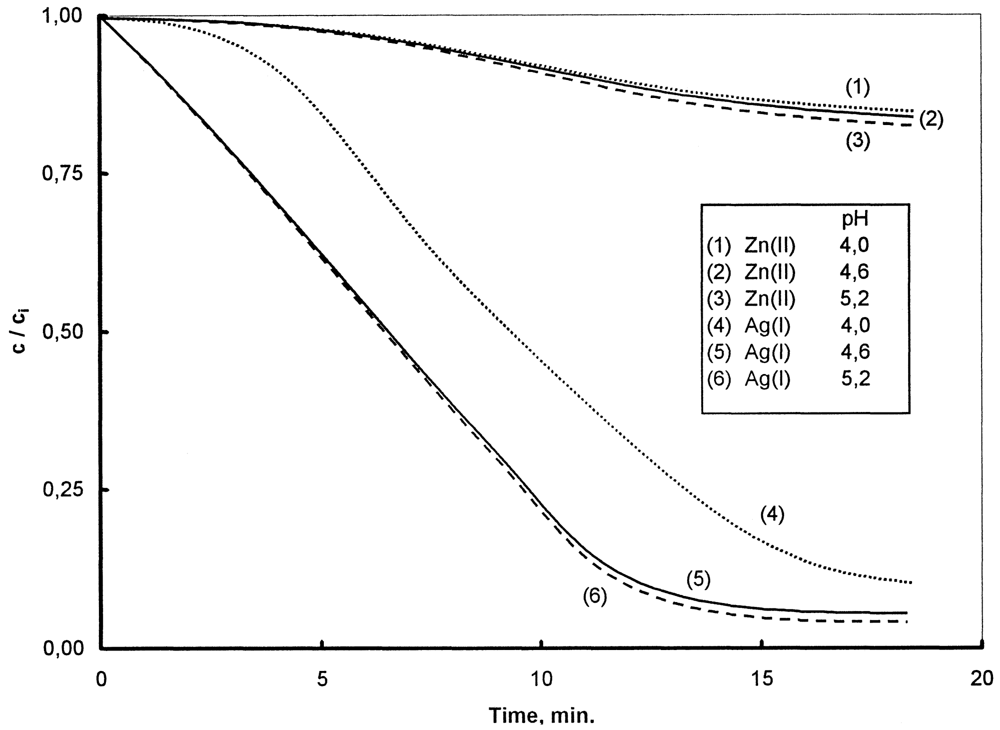


FIG. 5 Competitive flotation of zinc(II) and silver(I) ions with CPCl from feed solutions containing thiosulfates. $c_{\mathrm{Zn(II)}} = c_{\mathrm{Ag(I)}} = 1.0 \times 10^{-6}$ M, $[K_2S_2O_3] = 3.0 \times 10^{-6}$ M.

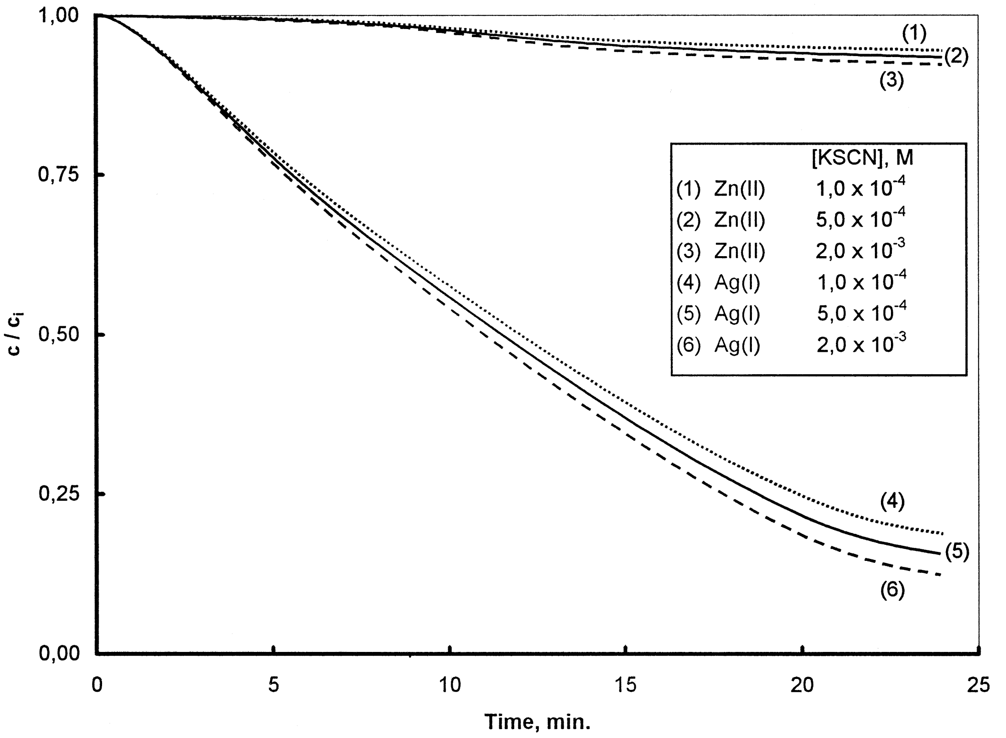


FIG. 6 Competitive flotation of zinc(II) and silver(I) ions with CPCl from feed solutions at different concentrations of thiocyanates. $c_{\mathrm{Zn(II)}} = c_{\mathrm{Ag(I)}} = 1.0 \times 10^{-6}$ M.

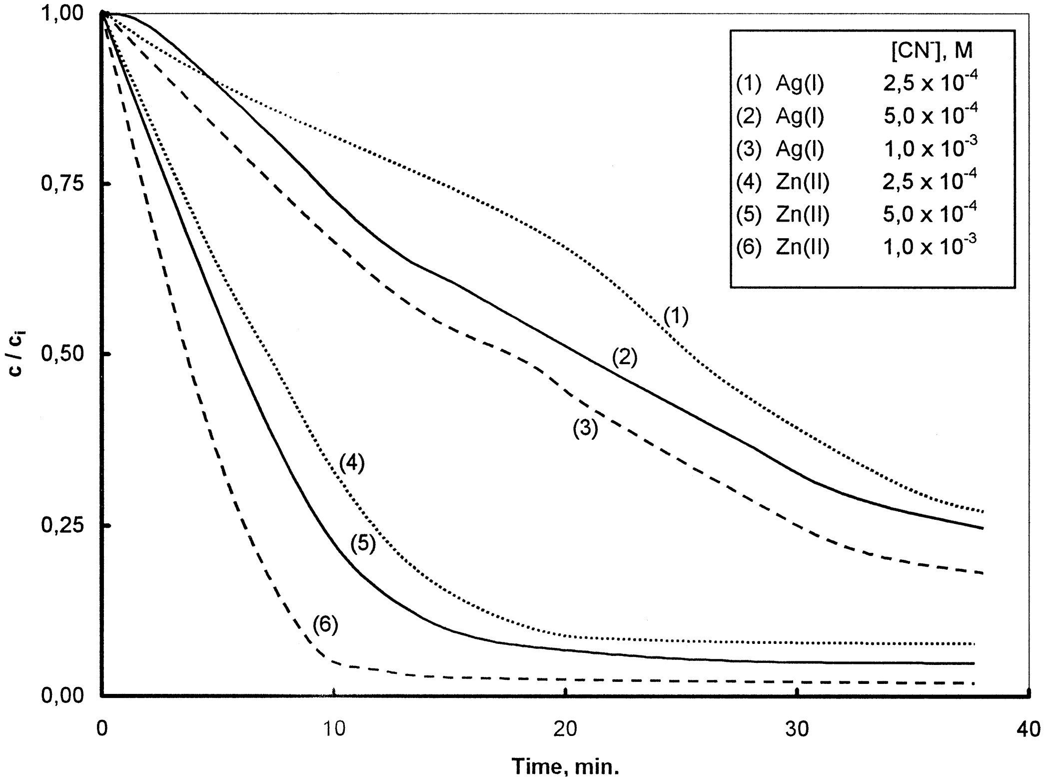


FIG. 7 Competitive flotation of zinc(II) and silver(I) ions with CPCl from feed solutions containing cyanides. $c_{\mathrm{Zn(II)}} = c_{\mathrm{Ag(I)}} = 1.0 \times 10^{-6}$ M.

sulfate according to the following reaction:

$$S_2O_3^{2-} + 2H^+ = SO_2 + S + H_2O$$

The maximal flotation percent of zinc(II) ions is from 15 to 17. The degree of separation for Ag/Zn is from 5.9 to 5.4.

Figure 6 provides ion flotation curves for zinc(II) and silver(I) from solutions of thiocyanates versus their concentrations. The pH for the system studied [i.e., containing Zn(II), Ag(I) and SCN^- ions] was kept constant and equal to 6.2. The maximal flotation percent for silver(I) ions reaches 82–88 whereas for zinc(II) it is only 6–8. The degree of separation for Ag/Zn is from 11.5 to 14.4. The molar contribution of silver(I) anionic species, i.e., $[Ag(SCN)_2]^-$, at thiocyanate concentrations of 1×10^{-4}, 5×10^{-4}, and 2×10^{-3} M is 33.3, 56.5, and 84.2, respectively. These molar contributions are much lower than the maximal percent flotation of silver(I) (Fig. 6). A possible explanation of this disagreement is to assume that AgSCN species are also floated since the sum of percent molar contributions of AgSCN and $[Ag(SCN)_2]^-$ species for

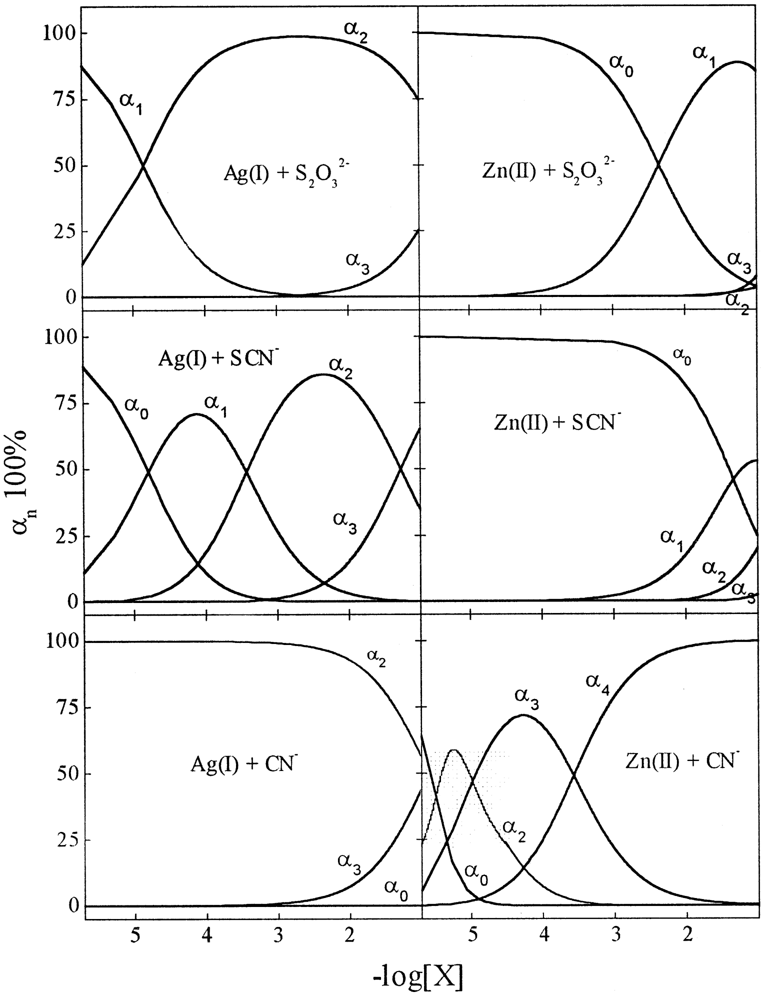


FIG. 8 Percent molar contributions ($\alpha_n \times 100\%$) of thiosulfate, thiocyanate, and cyanide complex species for silver(I) and zinc(II) at different concentrations of ligands (X).

the ligand concentration studied reaches 95–100. Zinc(II) is not floated since it remains as Zn^{2+} cations at our experimental conditions.

Figure 7 represents the flotation curves of metals from cyanide aqueous solutions at concentrations varying from 2.5×10^{-4} to 1.0×10^{-3} M. To prevent hydrolysis of cyanide anions, the pH was maintained at 11.0. As can be seen from this figure, contrary to the other ligands, both metals are removed with high efficiency, i.e., with maximal flotation percentages of 73 ÷ 82 for silver(I) and 92 ÷ 98 for zinc(II). The flotation efficiency is higher at a higher CN^- ligand concentration. Based on the molar contributions of the existing metal species, silver(I) is present in the aqueous solution only as $[Ag(CN)_2]^-$

TABLE 1
Percent Contributions of Thiosulfate, Thiocyanate, and Cyanide Complex Species of Zinc(II) and Silver(I) [30]

System	Ligand concentration (M)	$\alpha_0 \cdot 100\%$	$\alpha_1 \cdot 100\%$	$\alpha_2 \cdot 100\%$	$\alpha_3 \cdot 100\%$	$\alpha_4 \cdot 100\%$
Ag(I) + $S_2O_3^{2-}$	3×10^{-6}	0.0	87.5	12.5	0.0	0.0
Zn(II) + $S_2O_3^{2-}$		100.0	0.0	0.0	0.0	0.0
Ag(I) + SCN^-	1×10^{-4}	4.9	61.3	33.3	0.0	0.0
Zn(II) + SCN^-		99.6	0.4	0.0	0.0	0.0
Ag(I) + SCN^-	5×10^{-4}	1.4	42.1	56.5	0.0	0.0
Zn(II) + SCN^-		98.9	1.1	0.0	0.0	0.0
Ag(I) + SCN^-	2×10^{-3}	0.2	15.6	84.2	0.0	0.0
Zn(II) + SCN^-		96.0	4.0	0.0	0.0	0.0
Ag(I) + CN^-	2.5×10^{-4}	0.0	0.0	100.0	0.0	0.0
Zn(II) + CN^-		0.0	0.0	2.1	50.7	47.1
Ag(I) + CN^-	5×10^{-4}	0.0	0.0	100.0	0.0	0.0
Zn(II) + CN^-		0.0	0.0	0.7	34.7	64.6
Ag(I) + CN^-	1×10^{-3}	0.0	0.0	100.0	0.0	0.0
Zn(II) + CN^-		0.0	0.0	0.2	21.1	78.7

whereas 96.0 to 99.8% of zinc(II) is in the form of $[Zn(CN)_4]^{2-}$ + $[Zn(CN)_3]^-$ complex anions. This means that the affinity of $[Ag(CN)_2]^-$ for cetylpyridinium chloride is lower than to a mixture of $[Zn(CN)_4]^{2-}$ and $[Zn(CN)_3]^-$ anions.

Some flotation curves (Fig. 1, Curve 1; Fig. 2, Curves 1 and 2; Fig. 5, Curve 4; Fig. 7, Curves 1 to 3) differ from others. A characteristic inflection appearing on these curves separates a low rate period from a higher rate part of a kinetics curve. A similar feature was observed for $[Co(CN)_6]^{3-}$ (32), $[PdCl_4]^{2-}$ (33), and $[PtCl_6]^{2-}$ (34) when a sublate formed in the bulk solution.

CONCLUSIONS

Zinc(II) and silver(I) can be separated from diluted weakly acidic aqueous solutions by flotation with anionic surfactants since Zn^{2+} cations exhibit a much higher affinity for a surfactant than do Ag^+ cations. The separation of both studied metal ions is also possible from solutions containing thiosulfate or thiocyanate ligands. For the first ligand at total concentration 3×10^{-6} M, the silver is floated as a mixture of $[Ag(S_2O_3)]^-$ and $[Ag(S_2O_3)_2]^{3-}$ species, whereas zinc(II) remains in the aqueous phase as a simple divalent cation. For SCN^- ligands at concentrations from 1×10^{-4} to 2×10^{-3} M, silver(I) is floated as $[Ag(SCN)_2]^-$ and AgSCN species. The partial separation of zinc(II) and silver(I) can also be achieved from solutions containing CN^- ligands at concentrations ranging from 2.5×10^{-4} to 1.0×10^{-3} M, where $[Zn(CN)_3]^- + [Zn(CN)_4]^{2-}$ anions are floated much better (with a maximal flotation percent equal to 92 ÷ 98) than $[Ag(CN)_2]^-$ anions (with a maximal flotation percent equal to 73 ÷ 82).

In the light of our study, it appears that the ion flotation technique is an effective method for the separation of zinc(II) and silver(I) ions from dilute aqueous solutions. Removal of silver(I) from aqueous sulfate solutions containing zinc(II) requires a cationic surfactant and the introduction of thiosulfate or thiocyanate ligands to the feed solution. Moreover, the first ligand is unstable in acidic solution.

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