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To cite this Article Rao, S. R. , Finch, J. A. , Zhou, Z. and Xu, Z.(1998) 'Relative Flotation Response of Zinc Sulfide: Mineral and Precipitate', Separation Science and Technology, 33: 6, 819 — 833

To link to this Article: DOI: 10.1080/01496399808544878

URL: http://dx.doi.org/10.1080/01496399808544878

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SEPARATION SCIENCE AND TECHNOLOGY, 33(6), pp. 819–833, 1998

Relative Flotation Response of Zinc Sulfide: Mineral and Precipitate

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ABSTRACT

A study has been conducted to compare the floatability of fine zinc sulfide (ZnS) precipitates and sphalerite particles. The floatability of the precipitates was significantly poorer compared to sphalerite particles when xanthate was used as the collector. The floatability was improved by using dodecylamine as the collector, and the difference in floatability between the precipitates and sphalerite particles was reduced. The flotation kinetics of the precipitates was further improved significantly by incorporating a hydrodynamic cavitation tube in a conventional (mechanical) flotation cell. The improved kinetics was attributed to in-situ gas nucleation on the precipitates.

INTRODUCTION

Flotation continues to extend to nonmineral applications, including recycling of materials, soil remediation, and effluent treatment. For example, flotation has become the method of choice for deinking recycled paper pulp. Recovery of metal ions from industrial effluents represents another important area in which flotation is considered attractive. Direct ion flotation is one approach. However, the stoichiometric requirement of the collector, usually

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a long-chain surfactant, raises economic concerns. An alternative is to precipitate base metal ions, such as copper and zinc, as hydroxides or sulfides, followed by precipitate flotation. The precipitation-flotation approach reduces the collector consumption significantly in addition to giving high metal ion removal efficiency. There is an extensive knowledge base on sulfide mineral flotation, but relatively little work has been reported on sulfide precipitate flotation. One recognized difference is the fine size of the precipitates. Flotation of fine particles often requires a strong collector (i.e., one with a long hydrocarbon chain) and suitable hydrodynamics to promote collision with and attachment to bubbles.

In this communication we report a study of flotation characteristics of zinc sulfide both as minerals and precipitates. Two different collectors, dodecylamine (DDA), a long-chain cationic surfactant, and potassium isobutyl xanthate, a common sulfide flotation collector, were tested. The flotation was conducted using both a microflotation cell and a conventional mechanical flotation cell with and without a hydrodynamic cavitation reactor.

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EXPERIMENTAL

Materials and Chemicals

Mineral sphalerite was purchased from the Ward Minerals Establishment, Canada. A single mineral sphalerite was selected carefully and pulverized. Particles in the 250 to 100 mesh (63 to 149 μm) size range were collected for microflotation experiments, while a −400 mesh fraction was collected for zeta potential measurement. The prepared sphalerite sample was stored in a freezer at −4°C to minimize oxidation. Chemical analysis of the mineral sphalerite by atomic absorption showed a composition of 58.2% Zn, 0.38% Pb, 0.43% Fe, 0.14% Cu, and 6.5% acid insoluble solids. Reagent-grade zinc sulfate ($ZnSO_4 \cdot 9H_2O$) and sodium sulfide ($Na_2S \cdot 9H_2O$) (Fisher Scientific, Canada) were used to prepare zinc sulfide precipitates. Dodecylamine hydrochloride (DDA: $C_{12}H_{25}NH_2HCl$, Kodak Chemicals) and potassium isobutyl xanthate (KIBX: $C_3H_6OCS_2K$, American Cyanamid) were used as collectors. DDA was used as received while KIBX was purified using standard procedures (1) and stored in ether. Fresh collector stock solution (0.01 M) was prepared for each set of experiments. Reagent-grade sodium hydroxide (Fisher Scientific, Canada) and hydrochloric acid (BHP) were used as pH modifiers. All solutions and suspensions were prepared using nitrogenated, doubly distilled water.

ZnS Precipitation

The precipitates were prepared fresh, as required, by mixing 50 mL $ZnSO_4$ solution (20 g/L Zn) with 25 mL Na_2S solution (20 g/L S) at natural pH (pH

6). The precipitates with particle sizes around 30 μm were washed three times with nitrogenated water and then dispersed in 50 mL water to obtain a suspension containing 1.5 g ZnS (3% solids). After adjusting the pH to a given value, the suspension was conditioned with collectors for 5 minutes prior to flotation. For studies on the effect of activation, cupric ions (cupric sulfate) were added prior to the collector.

Microflotation

A microflotation cell was constructed after the model described by Siwik and coworkers (2) (Fig. 1). The slurry was introduced through an inlet into

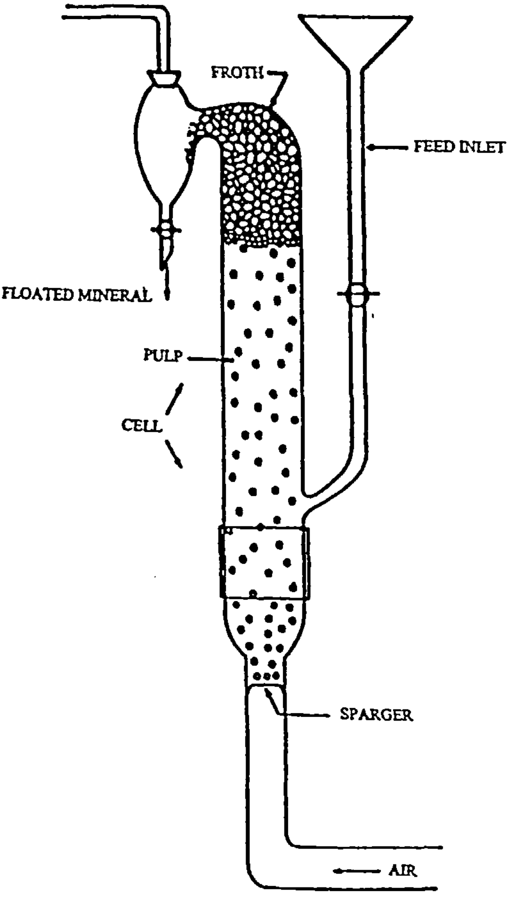

FIG. 1 Schematic diagram of the microflotation cell used in the present work.

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the cell with a constant air flow at 74 mL/min through a fritted glass sparger at the bottom of the cell to keep the solids from settling. The air bubbles produced as such from a compressed air source were used as carriers for solids flotation, and the air/solids ratio was kept constant for all the experiments. The pulp level was set 3 cm below the lip of the cell when DDA was used (as this reagent produced a voluminous froth), but only 0.8 cm when KIBX was used. Whenever xanthate was used, 10 ppm frother was added prior to turning on the air. The solids floated to the top of the pulp, and the froth overflowed into a separatory funnel where they were drawn into a beaker and recovered. Flotation was continued for 3 minutes, and the solids in both floats and tails were filtered on a weighed filter circle fixed on a funnel, and washed with acetone to facilitate drying at temperatures below 60°C. Recovery was calculated from the solid mass in floats and tails.

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Zeta Potential

A Lazer Zee meter (Model 510 Penkem, New Jersey) was used. About 1 g of fine sphalerite or freshly prepared zinc sulfide precipitates were mixed with 500 mL KCl (5 mM) solutions for 5 minutes with a magnetic stirrer while the suspension was bubbled with nitrogen to maintain an inert environment. Approximately 30 mL of the suspension was drawn to fill the microelectrophoresis cell, and the zeta potential was measured under an electric field strength of 100/120 V/mm. Three measurements were conducted for each pH and reported as an average with an accuracy of ±3 mV.

Sedimentation

To evaluate the effect of coagulation induced by collectors on the floatability, settling characteristics were investigated. A suspension of 3 g/L solids was used in a 250-mL stoppered, graduated cylinder. After thorough mixing, solids settling was followed by measuring the time the mud line (i.e., solid–liquid interface) took to pass selected reference marks.

E_p Measurement

To distinguish the effect of different physicochemical parameters, the redox potential (ORP), more precisely the pulp potential, was measured using a combination ORP electrode (gold in combination with Ag-AgCl, Cole-Parmer). The measured value was reported as E_p which can be readily converted to E_h by adding 222 mV (standard redox potential of Ag-AgCl). However, the use of E_p is preferred for our purposes because it is loosely defined in the present case, in distinction to rest potential.

X-ray Diffraction

A powder x-ray diffractometer (Philips BW 1710 diffractometer) was used to obtain diffraction patterns of mineral sphalerite and zinc sulfide precipitates. An x-ray of CuKα was used as the radiation source, and the diffraction pattern was collected at an angle resolution (2θ) of 0.02°. The diffraction pattern is presented without further data processing. The peak angle value, however, was obtained by processing raw data using a computer program provided with the instrument, in which case the noise was averaged out prior to data processing.

RESULTS AND DISCUSSION

Precipitate Characterization

The zinc sulfide precipitates formed were washed with acetone, air dried at room temperature (22°C), and characterized by atomic absorption (AA) for chemical composition and x-ray diffraction (XRD) for crystallinity. The AA analysis showed a Zn concentration of 65.4%, equivalent to a 1:1 Zn-to-S ratio, i.e., suggesting a ZnS stoichiometry with a purity of >99.95% ZnS. The XRD pattern of zinc sulfide precipitates and mineral powders (for comparison) is shown in Fig. 2. The main peak positions with relative intensity for both samples as well as for the standard are given in Table 1. As shown, the pattern of mineral matched the standard (vertical lines). For precipitates, only a few broad bands centered at ca. $2\theta = 28.3°, 47.5°, 56.2°, 69.8°, 77.5°$, 89.2°, and 96.2° were observed. These bands correspond to the intense bands of sphalerite. The broad nature of the bands suggests that the precipitates are less crystalline than the sphalerite. It should be noted that sphalerite and wurtzite have the same chemical composition and x-ray diffraction pattern, although the former is in the cubic, hextetrahedral class while the latter is in the hexagonal, trigonal trapezohedral class. The crystal structure difference causes a change in solubility between the two minerals.

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Flotation with Xanthate

The microflotation results for precipitates using KIBX in the presence and absence of cupric ions as a function of pH (Fig. 3) show that the precipitates were nonfloatable at a xanthate dosage of 0.5 g/kg. The recovery fluctuated around 20%, mainly due to entrainment in feed water reporting to the concentrate (ca. 40% water was recovered). In tests with frother only, a recovery of 37% (dashed line in Fig. 3) was obtained with a water recovery of 70%. Clearly, water recovery has to be considered in evaluating the true floatability of precipitates. In subsequent flotation tests, water recovery was kept by con-

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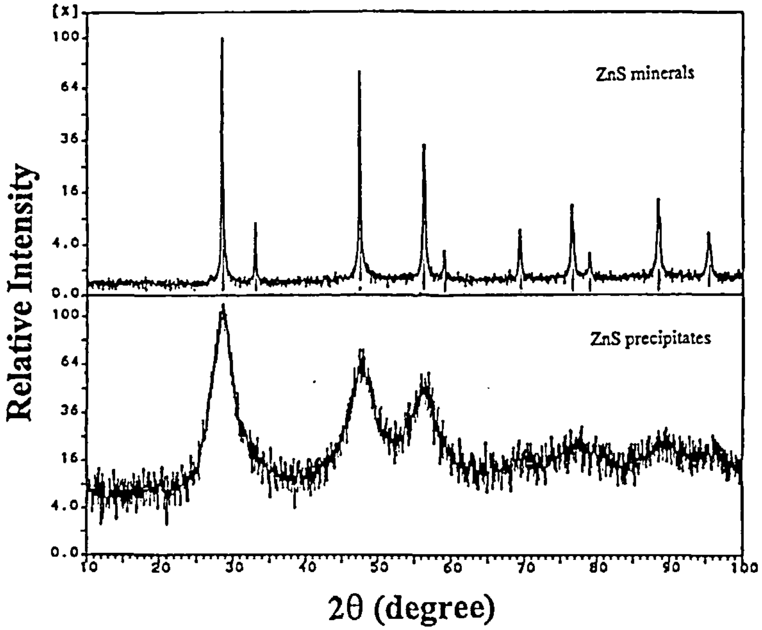

FIG. 2 X-ray diffraction patterns obtained with zinc sulfide precipitates and minerals.

trolling the pulp level and make up water at ca. 40 and 70% when KIBX and DDA were used as collectors, respectively.

As shown in Fig. 3, when the xanthate dosage was increased to 1 g/kg, a slight increase in recovery to ca. 40% was observed with Cu^{2+} activation,

TABLE 1

Angle and Relative Intensity of Peaks from X-ray Diffraction Pattern ZnS Precipitates and Minerals Along with the Values Taken from the Computer Database

Sample	2θ (degree) (relative intensity, %)									
Standard	28.5	30.6	47.5	56.4	59.0	73.0	76.1	79.1	88.7	95.9
	100	98	86	52	2	16	7	12	15	6
Mineral	28.5	33.1	47.4	56.3	59.9	69.3	76.6	79.0	88.4	95.3
	100	11	44	18	1	2	5	1	5	2
Precipitate	28.5	Nil	47.4	56.3	Nil	69.3	76.6	Nil	88.4	95.3
	100		75	59		26	31		29	23

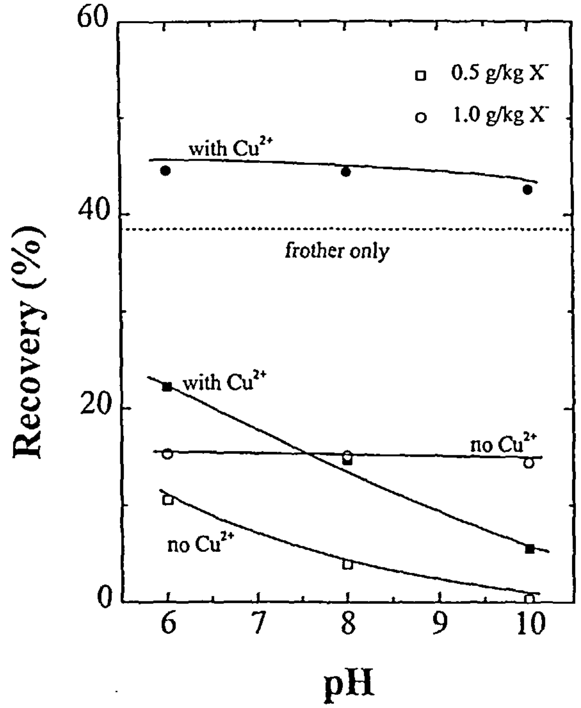

FIG. 3 Flotation recovery of zinc sulfide precipitates as a function of pH in the presence (filled: 200 ppm) and absence (open) of cupric ions with xanthate as collector. Squares and circles are for 0.5 and 1 g of xanthate per kg solids, respectively, and the dotted line is the flotation recovery with frother only (at 70% water recovery).

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although flotation remained insensitive to pH over the pH range studied. Here, cupric ions exchange with zinc ions to form a surface which has similar flotation characteristics to copper sulfide.

Compared to precipitates, sphalerite mineral was more floatable with KIBX as shown in Fig. 4. In the absence of activation, about 60% was floated with KIBX near neutral pH (as compared to ca. 12% in the absence of collector), decreasing with increasing pH. This decrease appears to result from competition between hydroxides and xanthate anions for the surface zinc sites. It should be noted that uncontaminated sphalerite is unfloatable with xanthate. The observed floatability cannot be accounted for by collectorless flotation since only 12% recovery was obtained without using a collector. Rather, it appears to be caused by a small amount of impurities (possibly Cu and Pb) present in the sphalerite samples. The important message is that the recovery

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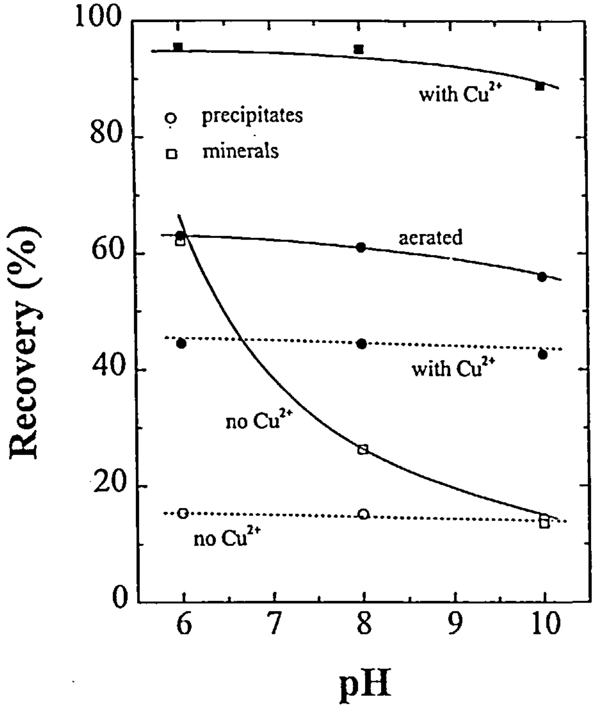

FIG. 4 Flotation recovery of zinc sulfide precipitates (circles) and minerals particles (squares) as a function of pH in the presence (filled: 200 ppm) and absence (open) of cupric ions using xanthate (1 g/kg) as collector. The solid line labeled by aerated indicates the precipitate flotation recovery after aeration.

in the case of unactivated sphalerite is significantly higher than the recovery of zinc sulfide precipitates in the presence of a significantly higher concentration of cupric ions (see Fig. 4). With Cu^{2+} activation (6 g/kg), the recovery of sphalerite increased to ca. 90% regardless of pH, which resembles the floatation characteristics of copper sulfide using xanthate as collector.

The general observation by comparison between Figs. 3 and 4 is that mineral sphalerite is more floatable with xanthate than the precipitate. This difference may be related to particle size, crystallinity, and/or the chemical environment of the two flotation systems. The precipitates were fine, which often results in a low flotation rate. It is also possible that more reducing conditions exist in the case of precipitate flotation than for mineral flotation due to the use of Na_2S and nitrogenated water in precipitation. To evaluate this, the pulp potential (E_p) was measured, and the value for precipitate suspension at pH 8 was ca. 360 mV, which was significantly lower than that for the mineral sphalerite suspension (ca. 440 mV). In an attempt to isolate the pulp potential effect, precipitate flotation was also conducted using a vigorously aerated slurry which had an E_p of ca. 450 mV. The recovery of precipitates (Fig. 4) was indeed improved substantially (from ca. 40 to 60%), but remained significantly lower than the recovery of mineral sphalerite (60 vs 90%). It appears that the particle size and crystallinity may be the major factors contributing to low floatability of precipitates compared to mineral. A common feature in Figs. 3 and 4 is that cupric ions increased floatability and reduced dependency on pH.

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Flotation with Dodecyl Amine

As shown in Fig. 5, the recovery of precipitates at pH 6 increased significantly using DDA compared to xanthate plus Cu^{2+} activation, becoming comparable to that of mineral. However, the floatability with DDA was strongly pH dependent, decreasing with increasing pH. The significant drop in recovery at pH 10 is similar to observations in oxide flotation, which has been attributed to the precipitation of neutral amine molecules, reducing its surface activity (3–6). It is interesting to note that at high DDA concentration, the recovery declined, possibly due to reversed orientation of a second adsorbed layer of DDA, resulting in reduced hydrophobicity. It is well known that DDA reacts with a surface mainly by electrostatic attraction which in turn induces two-dimensional hydrophobic association of adsorbed surfactant on the surface. In this case, pulp potential is expected to play little role in collector adsorption and flotation. Hydrophobic coagulation of fine precipitates induced by surfactant may contribute to the particle size effect on floatability, which was less significant when xanthate was used as collector.

Electrokinetics

The zeta potentials of precipitates and sphalerite are shown in Fig. 6. The isoelectric point (iep) value of ca. pH 3 for sphalerite agrees with those reported previously (5, 6). The zeta potential curve for zinc sulfide precipitates shifted toward high pH direction with an iep at ca. pH 8. The higher iep, also reported by Duran et al. (7), may be related to a higher solubility of less crystalline precipitates or to a slightly high solubility of wurtzite which may be the product of zinc sulfide precipitates. Zinc ions released from the precipitates may re-adsorb, probably as monohydroxide cations. This hypothesis was supported by the observed increase in iep of sphalerite (to ca. pH 4) in the presence of 2 ppm Zn^{2+}.

In the presence of DDA, the zeta-potential of precipitates remained the same within experimental error (Fig. 7). Flotation results suggest that DDA

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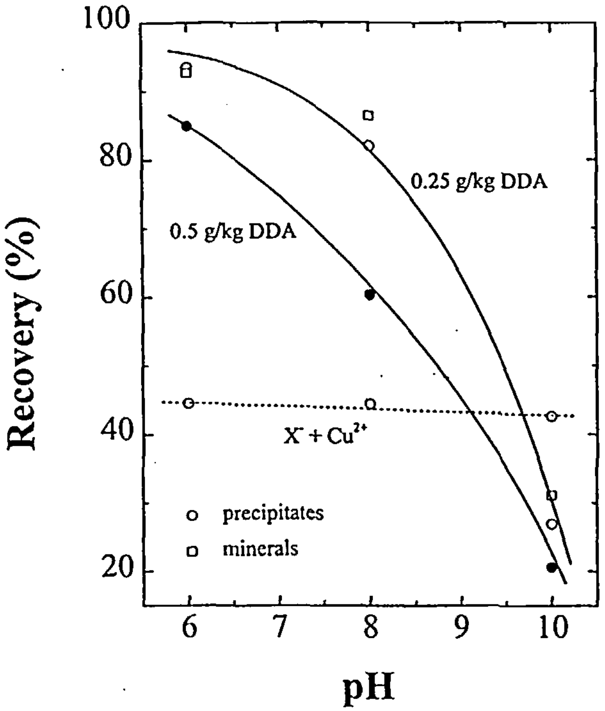

FIG. 5 Flotation recovery of zinc sulfide precipitates (circles) and mineral particles (squares) as a function of pH using dodecyl amine as collector. Open and filled symbols are for 0.25 and 0.5 g of DDA per kg solids, respectively, and the dotted line labeled $X^- + Cu^{2+}$ represents the precipitate flotation recovery with xanthate (1 g/kg) activated by cupric ions (200 ppm).

has adsorbed. Simple electrostatic adsorption alone cannot account for the increased floatability of precipitates observed near neutral pH compared to alkaline pH. At neutral pH, ZnS precipitates are positively charged, and hence electrostatic repulsion should exist, hindering adsorption of (cationic) DDA. A possible explanation is the formation of Zn–DDA complexes on the precipitates whose charges are compensated by S^{2-} acting as counterions. Another possible mechanism is ion exchange: DDA adsorbing through ion exchange with surface $ZnOH^+$ species. The adsorbed DDA molecules were most likely in the form of two-dimensional aggregates which were in a more energitically favorable state. Therefore, an additional non-DLVO (Derjaguin-Landau-Verwey-Overbeek) attraction operates between an air bubble and particles, which contributes to the observed flotation. This non-DLVO force has been discussed recently in detail, e.g., by Lafrance and Grasso (8).

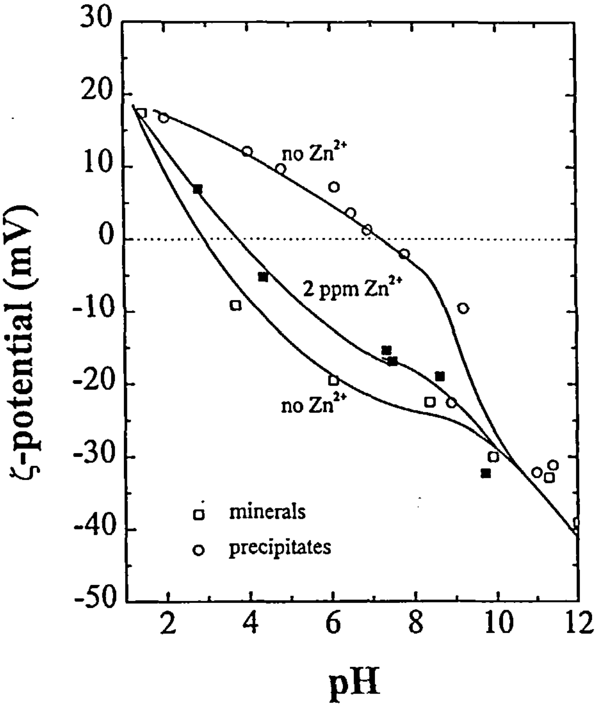

FIG. 6 Zeta potential of zinc sulfide precipitates (circles) and mineral particles (squares) as a function of pH in the presence (filled) and absence (open) of zinc ions (2 ppm).

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Settling Characteristics

The settling response of precipitates in the presence and absence of DDA at pH 6 and 10 is shown in Fig. 8. Two features were observed: 1) the precipitates settled faster at higher pH and 2) DDA increased the settling rate significantly at pH 10 but had no effect at pH 6. Observation 1) seems to be contrary to classical colloidal stability theory (i.e., DLVO theory) which would predict a stronger repulsive force for ZnS precipitates at pH 10 compared to pH 6, yet a less stable suspension was observed. A similar phenomenon has been observed with mineral sphalerite particles (9, 10). The exact cause for this unexpected result is not clear, but it may be related to either charge correlation or hydrophobic interactions. A detailed discussion of mechanisms is beyond the scope of this communication. However, hydrophobic interaction is anticipated between DDA adsorbed particles, which was addressed in the previous paragraph. Extended discussions on hydrophobic interaction can be found elsewhere (8, 11–13).

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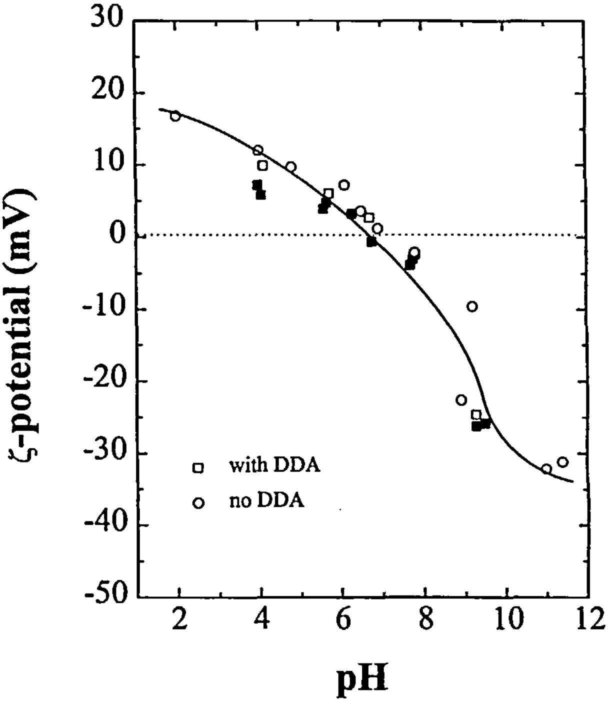

FIG. 7 Zeta potential of zinc sulfide precipitates in the presence (squares) and absence (circles) of DDA as a function of pH: open and filled squares are for 0.01 and 0.1 mM DDA, respectively.

Observation 2) is anticipated as the electrostatic attraction between cationic DDA molecules and negatively charged precipitates at alkaline pH enhances DDA adsorption on the precipitates, inducing hydrophobic coagulation. Following this reasoning, it is not clear why floatability decreased at alkaline pH (Fig. 5). This apparent contradiction suggests the ZnS precipitate-flotation system is quite complex, and thus should stimulate further fundamental investigations.

The above shows that the poor floatability of ZnS precipitates with xanthate is not solely related to the fine size of precipitates, but also to unfavorable surface properties and/or flotation pulp chemistry. Evidently different flotation conditions have to be established for effective removal or recovery of metal precipitates when using xanthate as collector. In contrast, long-chain amine collectors appear to be promising.

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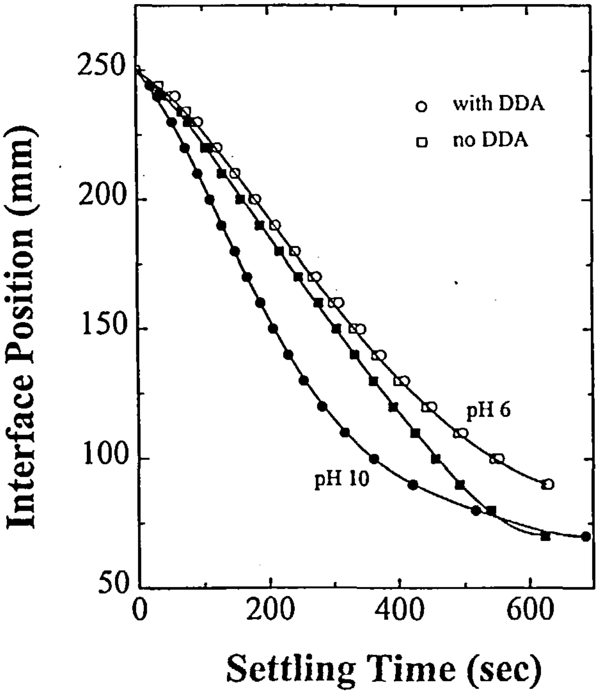

FIG. 8 Settling characteristics of zinc sulfide precipitates at pH 6 (open) and 10 (filled) in the presence (circles) and absence (squares) of DDA.

Flotation with a Cavitation Reactor

The above study suggests that only limited improvement in zinc sulfide precipitates flotation can be obtained by adjusting physicochemical conditions, mainly due to the limitation in hydrodynamics of fine precipitates. To promote the flotation kinetics of fine particles, an in-situ gas nucleation reactor based on hydrodynamic cavitation principle was tested using a mechanical flotation cell as schematically shown in Fig. 9. Continuous flotation was conducted by pumping the slurry (0.2% w/w ZnS precipitates) through a cavitation tube into a 5-L laboratory mechanical miniflotation cell (14). The cavitation tube was made from glass with an inner nozzle diameter of 1.30 mm (15). The concentrate and tails were collected simultaneously for a given period of time and weighed to obtain the feed flow velocity through the nozzle and the retention time (τ) of slurry in the flotation cell. Recovery was

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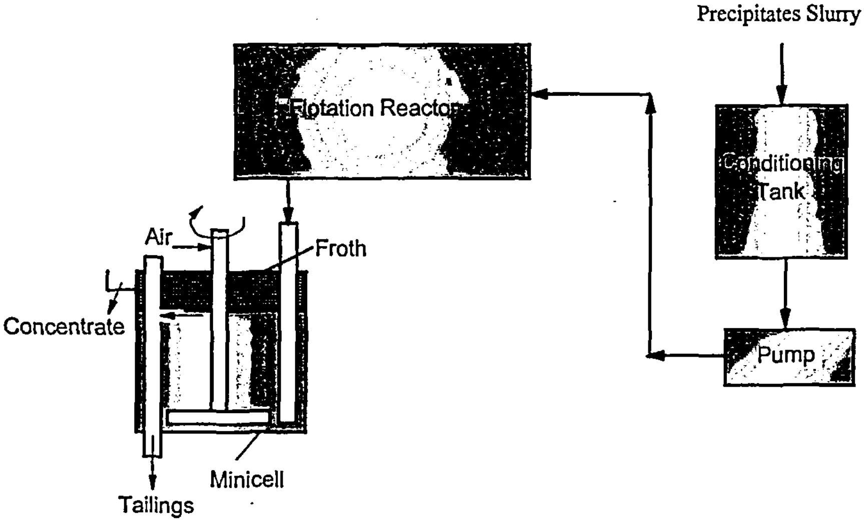

FIG. 9 Schematic diagram of setup for continuous laboratory flotation tests.

calculated from the dry weight of the concentrate and tailings, respectively. Assuming perfect mixing and first-order kinetics, the flotation rate constant (k) was obtained by solving the following equation for a given recovery and retention time:

$$R_c = \frac{k\tau}{1 + k\tau} \tag{1}$$

As a baseline test for comparison, a precipitate slurry was fed directly to the miniflotation cell. Flotation was conducted using 2 L/min air at pH 8.4 with 0.1 mM DDA as collector and 20 ppm MIBC as frother (to produce a more stable froth and later to give enhanced bubble formation by cavitation). The flotation rate constant in the baseline test was ca. 0.14 min^{-1}. (It should be noted that flotation chemistry was not optimized in the flotation tests to investigate the role of hydrodynamic cavitation.) Under the same chemical conditions, the slurry fed through the cavitation reactor at a linear flow velocity of ca. 17 m/s caused an increase in flotation rate constant to 0.23 and 0.31 min^{-1}, depending on whether the slurry exiting the cavitation reactor was fed into or above the pulp in the minicell. The significant improvement in flotation kinetics of fine precipitates by hydrodynamic cavitation is evident, and the mechanism of improved flotation kinetics has been discussed elsewhere (16).

SUMMARY AND CONCLUSIONS

The present study has shown that the floatability of ZnS precipitates is in general poorer than that of mineral sphalerite when xanthate is used as the collector. The differences appear to result from fine size and less crystallinity of precipitates. The floatability of precipitates was improved by using dodecylamine or by exploiting hydrodynamic cavitation.

ACKNOWLEDGMENTS

The authors acknowledge financial support from the Natural Sciences and Engineering Research Council of Canada under Strategic Grant NSERC-STR0149414. The in-kind support from Cominco, Inco, and Noranda is also acknowledged.

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Received by editor March 28, 1997
Revision received August 1997