Insoluble Cross-Linked Starch Xanthate as a Selective Flocculant for Sulfide Minerals

S.C. Termes, R.L. Wilfong, and P.E. Richardson

Abstract—*This paper discusses recent Federal Bureau of Mines research on the selective flocculation of various minerals with insoluble cross-linked starch xanthate (ISX). ISX is insoluble, has a shelf life of more than a year when kept cold, and has a higher molecular weight due to cross-linking than the soluble, non-cross-linked starch xanthate used in previous flocculation studies on nonsulfide minerals.*

ISX readily flocculates a number of sulfide mineral fines, but no silicious gangue. Floc formation, floc size, and settling rate are dependent on pH. The pH dependence, however, differs for each mineral. Bornite has been selectively flocculated from bornite-quartz mixtures, and the bornite-containing flocs were separated from the unflocculated quartz using decantation and washing procedures.

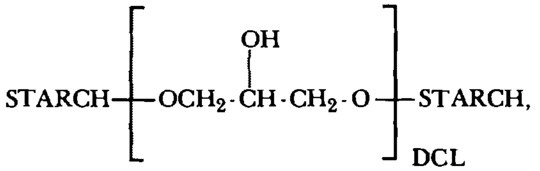
Introduction

Polymeric materials containing chelating groups of known reactivity towards specific mineral surfaces were proposed by Slater, Clark, and Kitchener (1969), Kitchener (1972), and Attia and Kitchener (1975) as selective flocculants for mineral fines. Because of the increasing interest in selective flocculation as a beneficiation technique for low-grade, highly disseminated ores, the US Bureau of Mines has been investigating the flocculation behavior of insoluble cross-linked starch xanthate (ISX). This material appeared to be attractive as a sulfide flocculant since xanthate groups are known to react selectively with sulfide minerals in flotation circuits; cross-linking of starch results in a high-molecular-weight polymeric backbone that generally increases floc-strength; ISX is prepared from easily renewable sources; and it is more stable and easier to handle than the soluble, alkali metal, non-cross-linked starch xanthates previously studied as flocculants by Chang (1954) and Brown (1953, 1955).

The insoluble ISX reported in this paper as a flocculant for sulfide minerals was developed by Wing and Doane (1976, 1977) at the US Department of Agriculture for removing heavy metal ions from waste waters.

Starches are composed of two polysaccharide fractions called amylose and amylopectin. The ratio of these fractions vary somewhat for each type of starch, such as corn, potato, rice, etc. Both amylose and amylopectin are made up of D-(+)-glucose units (Fig. 1a) joined in α-1, 4-glycoside linkages. While amylose chains are linear (Fig. 1b), amylopectin is a branched polymer with side chains attached through α-1, 6-glycoside linkages (Fig. 1c). Amylopectin dispersions in water are relatively stable, but amylose dispersions in water eventually precipitate out and, therefore, are regarded as metastable. The term "dispersion" rather than "solution" has been used to emphasize the colloidal nature of starch, amylose, and amylopectin molecules.

Cross-linking is a nondegradative reaction of starch where intermolecular bridges (cross-links) involving participation of the hydroxyl groups of starch are formed by reagents having multiple reactive sites, such as epichlorohydrin, formaldehyde, and phosphorous oxychloride. Cross-linking results in an increase in molecular weight. An epichlorohydrin cross-linked starch can be represented as



$$\text{STARCH}-\left[-\text{OCH}_2\cdot\overset{\text{OH}}{\overset{|}{\text{CH}}}\cdot\text{CH}_2\cdot\text{O}-\right]_{\text{DCL}}-\text{STARCH},$$

where DCL is the degree of cross-linking. Intramolecular bridges are also possible with these reagents, but are not generally referred to as cross-links.

Starch reacts readily with carbon disulfide in strong alkaline solutions to yield starch xanthate. The primary hydroxyl group (C-6) is involved mainly in the xanthation reaction (see Fig. 1). Alkali metal starch xanthates degrade readily in aqueous solutions, while alkaline-earth and heavy-metal salts are stable in water.

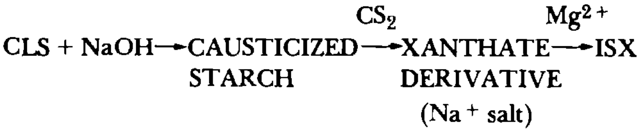
Experimental

Reagents

Unless otherwise specified, all chemicals used were reagent grade and all water was distilled and deionized. Commercial highly cross-linked starch (CLS) marketed as Vulca 90 from the National Starch and Chemical Co. was used in preparing the xanthate derivatives.

Synthesis and Characterization of ISX

ISX was prepared according to the method described by Wing and Doane (1977)



$$\text{CLS} + \text{NaOH} \longrightarrow \underset{\text{STARCH}}{\text{CAUSTICIZED}} \xrightarrow{\text{CS}_2} \underset{(\text{Na}^+\ \text{salt})}{\underset{\text{DERIVATIVE}}{\text{XANTHATE}}} \xrightarrow{\text{Mg}^{2+}} \text{ISX}$$

The synthesis proceeds readily with yields greater than 90%. The pale yellow, finely powdered ISX is stable for at least one year if kept in tightly closed glass containers and under refrigeration. At room temperature, the life of this product is a few months.

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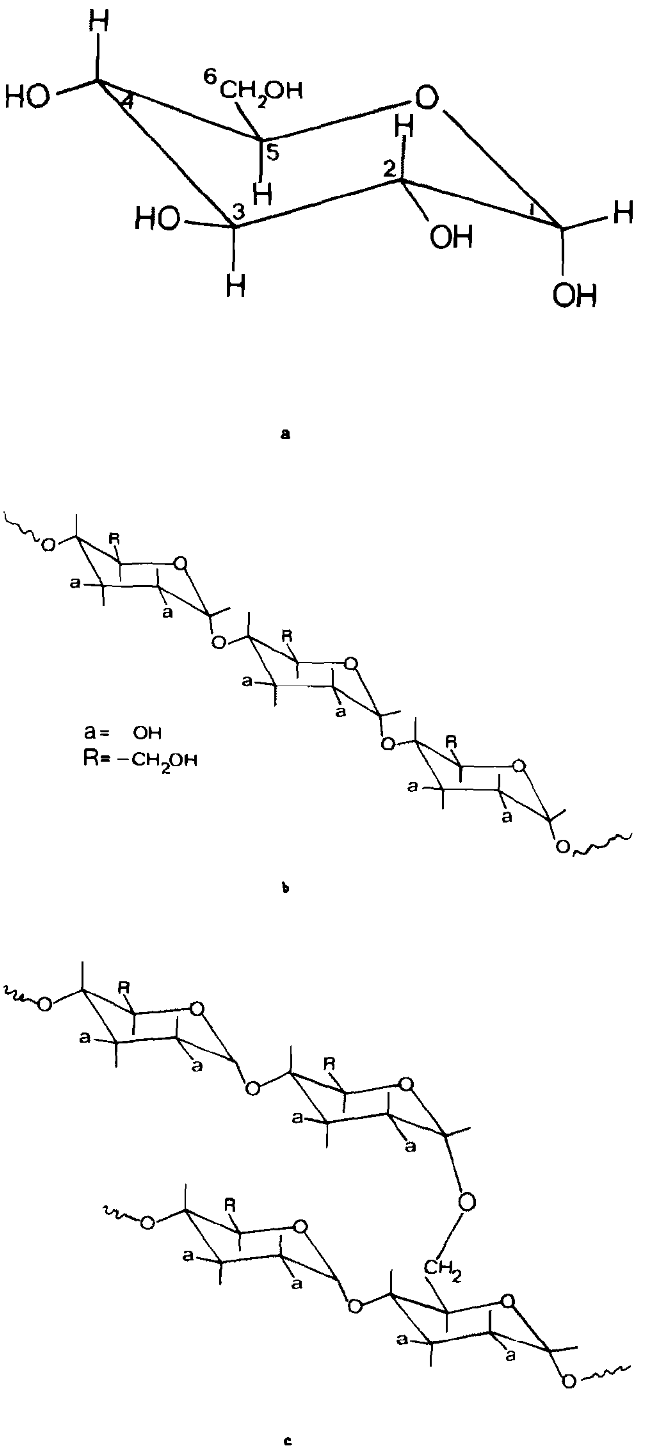

a

b

c

Fig. 1—Starch components: (a) D-(+)-glucose unit, (b) amylose, (c) amylopectin

The degree of substitution as determined by the acidimetric method of Swanson and Buchanan (1978) was between 0.2 and 0.4, which is within the range recommended by Wing and Doane (1976, 1977) for effective removal of heavy metal ions. Addition of ISX to water or solutions containing heavy metal ions produces an increase in pH.

Mineral Preparation

Mineral specimens were obtained from Ward's Natural Science Establishment, New York. Hand picked crystals were dry ground to -38 μm (-400 mesh) with an agate mortar and pestle. X-ray diffraction analysis showed that the samples were essentially monomineralic. The flocculation and zeta potential experiments reported in this paper were done with the mineral ground just before each experiment to minimize oxidation.

Flocculation Experiments

Flocculation tests were performed in 100-ml (3.4-fl oz) graduated cylinders fitted with ground glass stoppers. Unless otherwise specified, 3-g (0.1-oz) samples in 80 ml (2.7 fl oz) of water were used in all experiments. Dilute HCl and NaOH solutions were used to adjust pH to the desired values before flocculant addition and for any subsequent adjustments. The cross-linked starch and ISX were dispersed by stirring the solid in water —0.1 g (0.004 oz) of the starch in 20 ml (0.7 fl oz) of water — immediately before adding to the mineral pulp. Mixing was done manually by shaking and inverting the closed cylinder for one minute, after which the cylinder was set on a bench and visible changes were recorded. To determine the effectiveness of ISX as a flocculant, runs were made in the absence of any starch (acid-base additions only) and in the presence of CLS.

Other Measurements

Zeta potentials were measured with a Zeta-Meter, and turbidities were measured with a HACH ratio turbidimeter. Turbidities above the 200-NTU limit were calculated by extrapolating the readings of dilute aliquots.

Results

Single Mineral Pulps

Effect of pH: A series of preliminary tests were conducted to assess the ability of ISX to flocculate single mineral suspensions and to determine the effect of pH on flocculation behavior. In these tests, the height of settled solids after a fixed five-minute resting period was used as an indication of flocculation. It should be noted that this height is not a direct measure of the quantity of mineral removed from the suspension, but it allows comparison of changes produced by different reagents (ISX, CLS, acid-base addition). By comparing the flocculation response of a mineral to different reagents — acid/base, CLS, ISX — the height of the settled solid bed is an indication of floc formation.

Figures 2 through 6 show results of the flocculation tests on freshly ground fines of covellite, bornite, pyrite, chalcopyrite, and quartz as a function of pH in the presence of ISX, CLS, and acid-base additions. In these experiments, ISX and CLS were added at the natural pH (pH_n) of the pulp. This was the pH value of the mineral suspension in water in the absence of any reagent. It was observed that within a two-minute period the pulp had reached a steady pH value, and this value was taken as pH_n.

It can be seen that ISX has a pronounced effect on floc formation on covellite (Fig. 2), bornite (Fig. 3), pyrite (Fig. 4); a moderate effect on chalcopyrite (Fig. 5); and no effect on quartz (Fig. 6). Cross-linked starch induced partial

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Note: References to specific brand names and suppliers is made for identification purposes only and does not imply endorsement by the US Bureau of Mines.

floc formation on pulps of bornite and pyrite, and had little effect on other minerals. The pH ranges of maximum visual clarification after a five-minute period are indicated in the figures by broken lines and vary for each sulfide mineral.

Data in Figs. 2 through 6 were obtained by adding ISX at the natural pH_n of the mineral pulp. ISX addition at pH values lower and higher than pH_n slightly affected the settling behavior of the sulfide minerals. Table 1 summarizes results of adding ISX at various pH values for covellite, bornite, pyrite, and chalcopyrite. For bornite, the pH of ISX addition has essentially no effect, whereas for other minerals there is a slight shift in the pH range where good flocculation and clarification occur.

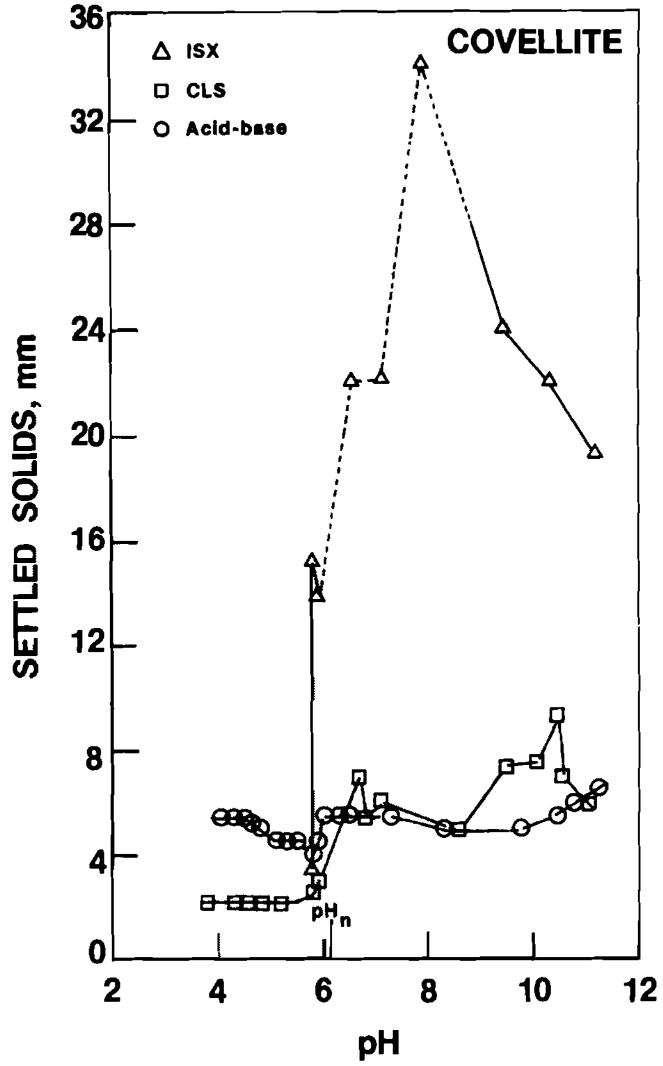

Fig. 2—Covellite settling

Table 1—ISX Addition at Different Pulp pHs

Mineral	pH of ISX addition	pH after ISX addition	Region of rapid settling and clarification
Covellite	2.7	5.6	7-8
	5.1	6.0	6-7
	6.1 (pH_n)	9.5	6-9
	10.9	11.0	5-7
Bornite	5.0	8.0	8-10
	6.9 (pH_n)	10.0	8-9
	10.0	11.0	8-9
Pyrite	3.3	9.0	7-11
	6.5 (pH_n)	10.0	6-10
	11.6	11.6	6-10
Chalcopyrite	3.2	10.3	5-6
	5.6 (pH_n)	11.3	6
	9.4	10.9	No clarification

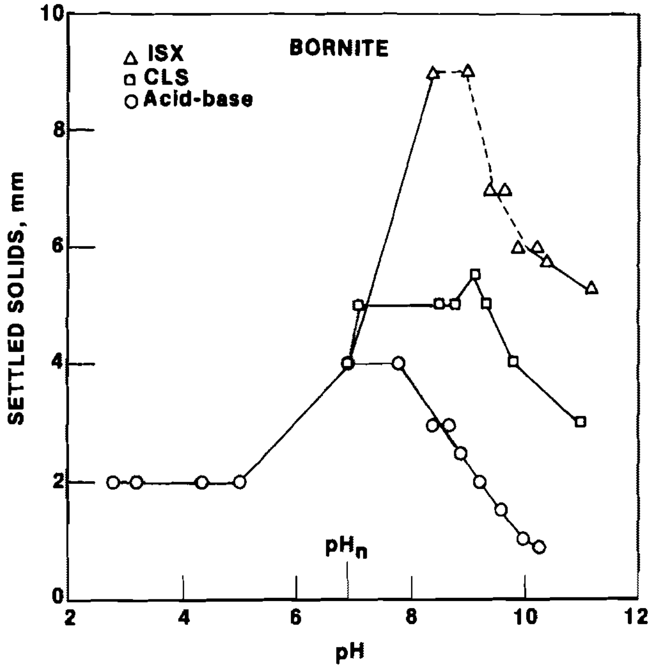

Fig. 3—Bornite settling

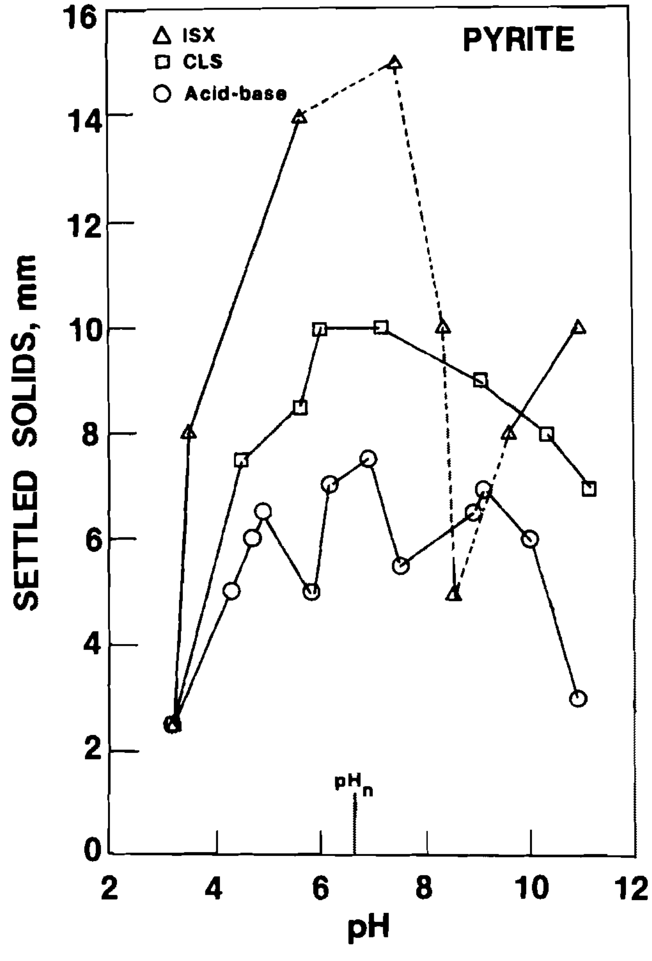

Fig. 4—Pyrite settling

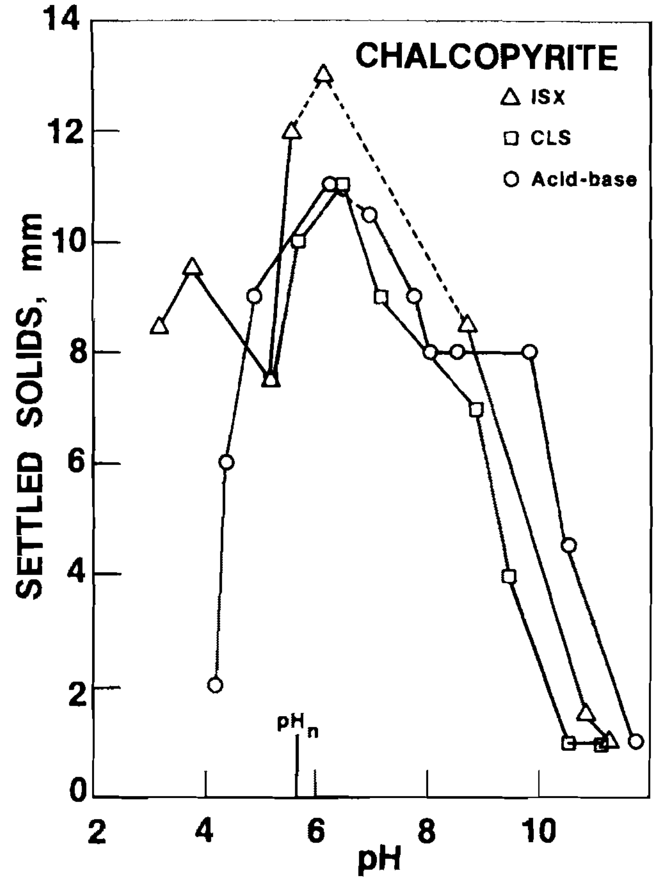

Fig. 5—Chalcopyrite settling

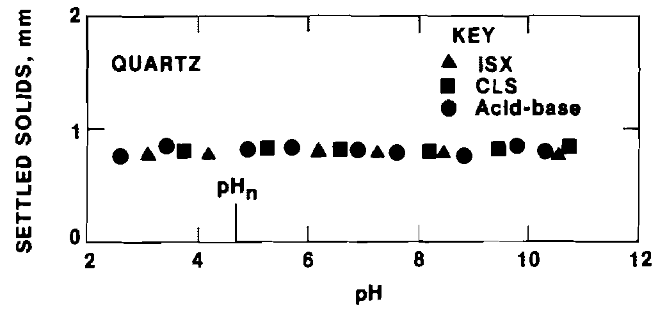

Fig. 6—Quartz settling

Characteristics of ISX-Induced Flocs: ISX addition to bornite at pH$_n$ produces rapid formation of small flocs in the size range of 0.5 - 1 mm (0.02 - 0.04 in.). During the presedimentation period, these small flocs further aggregate in the supernatant in several minutes to form flocs as large as 7 - 8 mm (0.28 - 0.32 in.), which then fall rapidly through the suspension. Floc growth is greatly enhanced if the cylinder is subjected to rotating motions. Although large flocs appear to break into smaller fragments upon shaking, reagglomeration into larger flocs occurs continuously. Even if the flocs are separated from the supernatants and redispersed in water, reagglomeration into larger flocs is observed.

Covellite and pyrite form flocs in the size range of 2 - 3 mm (0.08 - 0.12 in.), and chalcopyrite in the range of 0.5 - 1 mm (0.002 - 0.04 in.). However, in contrast to bornite, the flocs of these minerals readily break apart upon shaking or washing and show little tendency to reform.

All ISX-flocculated minerals filtered readily. The solids dewatered well, leaving clustered particles on the filter. This is in contrast with nontreated samples (acid-base only), which produce slimy residues, and the CLS-treated samples, where the settled solids take longer to filter or dewater.

Flocculant Dosage and Pulp Density: Because ISX induces covellite and bornite to form large and rapidly settling flocs, these minerals were chosen to study the effect of mineral pulp concentration and flocculant dosage. The desired weight of mineral was suspended in 80 ml (2.7 fl oz) of water. The turbidity was above the instrument's range (200 NTU), and no aggregation or fast settling was evident during the experiment, except when ISX was added. A known amount of ISX, suspended in 20 ml (0.7 fl oz) of water, was added. The cylinder was shaken for one minute and then kept stationary for three hours, after which the top 30 ml (1 fl oz) of the supernatant was carefully withdrawn with a pipet. The turbidity and total copper content (which takes into account both copper in covellite and copper from dissolution of covellite) of the withdrawn supernatant as a function of the ratio of ISX to covellite are shown in Figs. 7 and 8, respectively.

In experiments where 0.5 and 1 g (0.02 and 0.04 oz) of covellite in 100 ml (3.4 fl oz) of water were used, weight ratios of ISX to covellite below 0.1 produce the clearest supernatants and the best copper removal. A ratio of 0.05, for instance, corresponds to a dosage of 5×10^{-2} (2×10^{-3} oz) of ISX to 1 g (0.04 oz) of covellite. At lower ISX dosages, flocs formed nearly instantaneously with ISX addition. For dosages above 0.1 g (0.004 oz), no flocs were observed and some foaming and the odor of CS_2 from decomposition of the xanthate were frequently noted. The increase in turbidity may be due to ISX in itself. It is also possible that at higher dosages, ISX may be acting as a dispersant. Although not shown, optimal ISX dosages for bornite fell within the same range as for covellite.

Zeta Potentials: To gain some insight into the reaction mechanisms responsible for flocculation, zeta potential measurements were carried out on suspensions of ISX and CLS (Fig. 9) and each of the untreated sulfides (Fig. 10). In the 3 - 11 pH range, the zeta potential of CLS is positive and its magnitude increases with increasing pH. ISX is negatively charged with a zeta potential of about − 8 mV, essentially independent of pH.

In the pH range of best flocculation by ISX, pyrite and ISX are oppositely charged with zeta potential values less than 14 mV. Attractive electrostatic forces may be, in part, responsible for pyrite flocculation by ISX (Healy, 1979). Since repulsive energy barriers between particles become more significant only at zeta potential values greater than 14 mV, other contributions, such as system hydrodynamics and adsorption energy, may be more predominant in the flocculation of covellite, bornite, and chalcopyrite by ISX. The strength of the bond formed between xanthate groups in ISX and lattice metal sites could, for example, account for the flocculation of these minerals, with bornite likely forming the strongest bonds.

Selective Flocculation of Bornite from Mixtures with Quartz: Based on the observation that ISX formed the strongest flocs with bornite but did not flocculate quartz, bornite-quartz mixtures were chosen to assess the capability of ISX as a selective flocculant in binary mixtures. Adding ISX to 50% mixtures of bornite with quartz results in the rapid formation of large, dark flocs and a whiter, turbid supernatant. Nonflocculated quartz particles were also seen deposited around the flocs. X-ray diffraction of the solids separated from the supernatant and settled floc fractions showed the supernatant was enriched in quartz and the flocs enriched in bornite.

To separate flocculated bornite from unflocculated quartz, the quartz-rich supernatants were first decanted. The flocs were redispersed in water and recovered by filtration. This material was again redispersed in 150 - 200 ml (5 - 7 fl oz) of water and subjected to a swirling motion to momentarily break up large flocs, which allowed the release of entrapped quartz into the supernatant. The supernatant was decanted, and the described procedure was repeated twice.

It was observed that the pH of floc-containing supernatants remained in the 9 - 10 range during washing of the flocs. The results of chemical analyses of the solids recovered from the combined supernatant and the floc fractions are listed in Table 2. These experiments refer to mixtures containing 50% bornite; solid contents of 3 g (0.1 oz) in 100 ml (3.4 fl oz) of water; and starch dosage of 0.05 g (0.002 oz). Mixing time was one minute, and time elapsed between flocculation and separation is indicated in Table 2.

Tests one to three established that the washed floc fractions were considerably enriched in bornite, with recoveries of 60 - 65% and grades of 76 - 92%. Likewise, the unflocculated solids recovered from the supernatant and from the wash waters were enriched in quartz, with recoveries of 49 - 84% and corresponding grades of 83 - 87%. Recoveries do not total 100%, owing to transfer losses incurred in several stages of washing, decantation, and filtration.

For comparison, data are given for the solid contents of settled and supernatant fractions obtained without a reagent (test 4) and with cross-linked starch (test 5). As expected, there is essentially no selective separation when NaOH (test 4) is used to adjust the pH to values near that of ISX-treated pulps, and the settled fractions were slimy and difficult to dewater. With CLS, separation was observed, with the settled fraction containing 62% bornite and the supernatant 74% quartz. According to Fig. 3, CLS flocculates bornite, accounting for the enrichment in bornite in the settled fraction. However, in contrast to ISX-bornite flocs, those formed with cross-linked starch were small, fragile, and easily resuspended, making it difficult to carry on the described post-flocculation separation. Although the effect of Mg^{2+} released from ISX into water was not directly studied for the dosage and volume used here, the amount of Mg^{2+} would be less than 10^{-4} moles per liter.

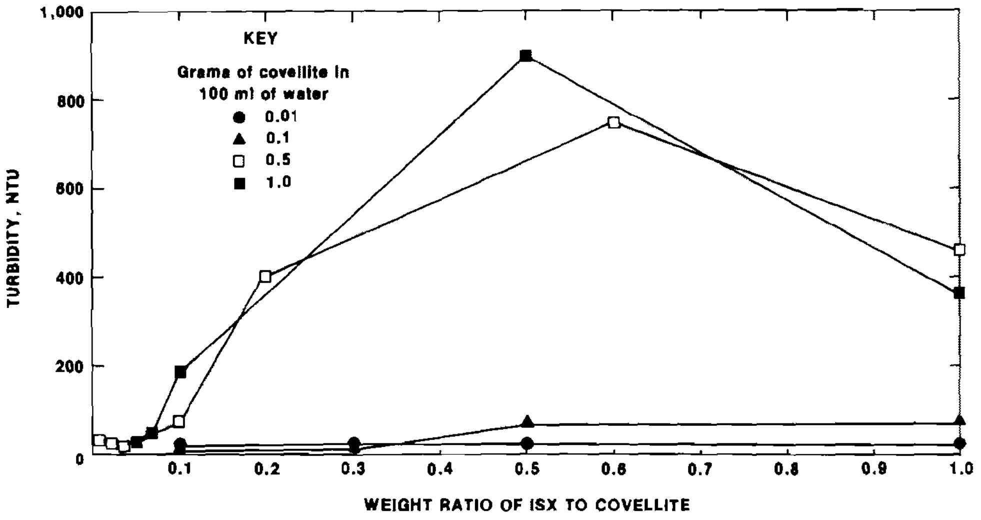

Fig. 7—Turbidity as a function of the weight ratio of ISX to covellite

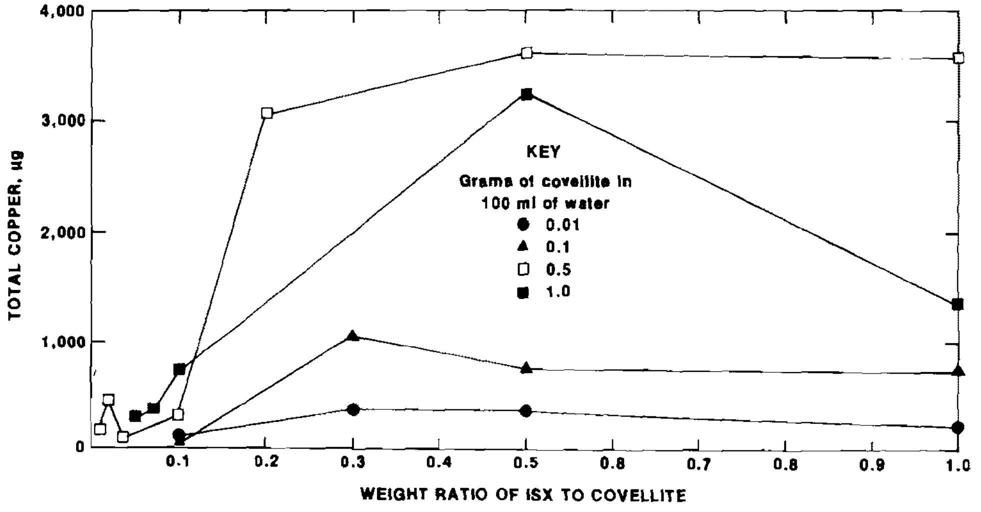

Fig. 8—Total copper content as a function of the weight ratio of ISX to covellite

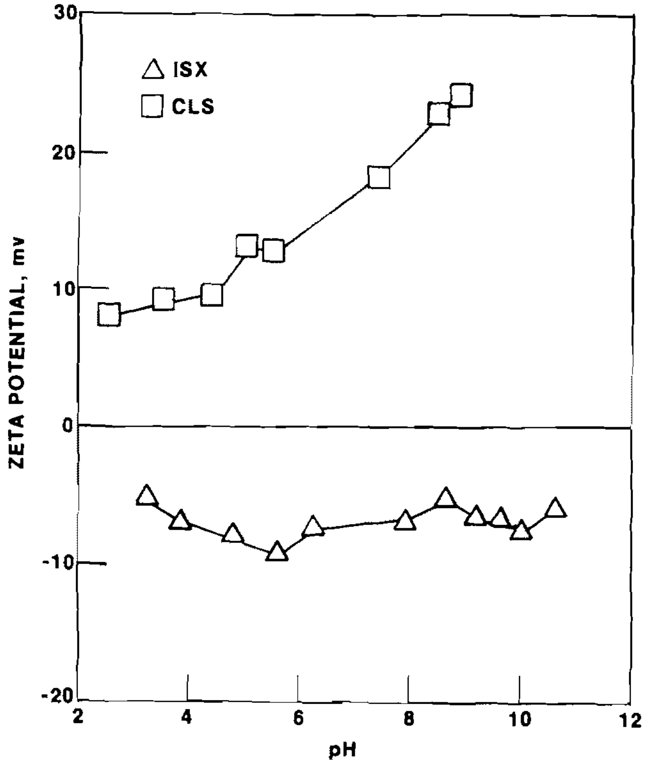

Fig. 9—Zeta potential of ISX and CLS

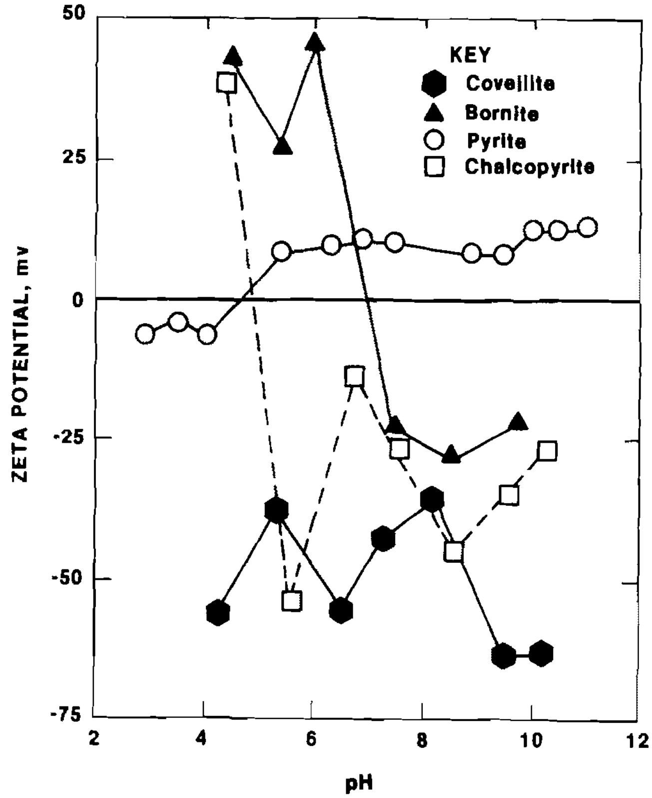

Fig. 10—Zeta potential of minerals

Table 2—Selective Flocculation of Bornite From Quartz

Test	Reagent	pH Initial	pH Final	Standing time (min)	Fraction analyzed	Recovery[c] (%) Bornite	Recovery[c] (%) Quartz	Grade[c] (%) Bornite	Grade[c] (%) Quartz	Separation Index[d]
1	ISX	8.1	10.3	150	F[a]	65	6	92	—	
					UF[b]	15	71	—	83	0.48
2	ISX	7.5	10.0	90	F	65	21	76	—	
					UF	10	49	—	83	0.48
3	ISX	8.0	9.0	30	F	60	11	85	—	
					UF	13	84	—	87	0.47
4	NaOH	8.1	10.0	30	Settled	72	68	51	—	
					Supernatant	6	7	—	54	0.26
5	CLS	8.0	8.1	30	Settled	70	43	62	—	
					Supernatant	2	56	—	74	0.44

[a] — *Flocs.*

[b] — *Unflocculated.*

[c] — *Recoveries and grades were calculated from analytical data for Cu, Fe, and Si.*

[d] — *Separation Index* = $\frac{\text{(\% bornite recovered in flocs + \% quartz in unflocculated fraction)} - 100}{100}$

From Sresty and Somasundaran (1980).

Discussion

Selective flocculation of sulfide minerals from mixtures with quartz has been reported in the literature. Usoni and coworkers (1968) studied the selective flocculation of galena, pyrite, and sphalerite with hydrolyzed polyacrylonitrile, polyacrylamides, and carboxymethylcellulose as a function of pH. They found that the best results, in terms of recovery, were given by polyacrylamide flocculants. Yarar and Kitchener (1970) investigated the selective flocculation of galena from quartz with polyacrylamides of variable degrees of anionic character. It was found that floc size, total solids content, ratio of components, and number of stages involved in the operation influenced recoveries and grades. Attia and Kitchener (1975) reported that cellulose xanthate caused strong flocculation of galena, moderate flocculation of pyrite and chalcopyrite, weak flocculation of sphalerite, and had no effect on quartz. They did, however, not report any selective flocculation experiments of these minerals from quartz. The use of hydroxypropylcellulose xanthate to selectively flocculate chalcopyrite from quartz was studied by Sresty and Somasundaran (1980). They emphasized the importance of cleaning the flocculated product to improve separation.

Recoveries of the sulfide mineral with polyacrylamide polymers, as reported by Usoni and coworkers (1968) and Yarar and Kitchener (1970), were greater than 98%. Grades obtained with these polymers varied: 98% or above for galena in Yarar and Kitchener's work compared to about 81% for pyrite, and about 73% for sphalerite in Usoni and coworkers' study. The separation indexes calculated for bornite-quartz separations with ISX (about 0.5, Table 2, Tests 1 - 3) were lower than those obtained by Sresty and Somasundaran (1980) for chalcopyrite-quartz separations with hydroxypropylcellulose xanthate (about 0.75).

Lower separation indexes are due to the term involving bornite recovery (60 - 65%) rather than to quartz grade terms

(83 - 87%). Lower bornite recoveries may result from the somewhat less efficient separation method (decantation) than the method employed by Sresty and Somasundaran (vaccum siphoning of the supernatant). Experiments with other minerals and flocculants have shown that flocculation followed by elutriation improve recovery (Termes and Wilfong, 1983).

One of the most interesting questions concerning the ability of ISX to flocculate sulfides is the manner in which ISX interacts with the mineral surface. As mentioned in the introduction, flocculants with functional groups that could chelate the mineral's metal component(s) are particularly attractive as flocculants, since one would expect such a bonding mechanism to be more selective than that of flocculants that adsorb by electrostatic attraction. It is well known that heavy metal ions form covalent bonds with xanthate groups. Covalent bond formation between xanthate group and surface metal component(s) is believed to also occur in the alkyl xanthate flotation of a number of sulfide minerals (Poling, 1976). The much larger and stronger flocs formed with ISX, when compared with those formed with crosslinked starch alone, also support the involvement of the xanthate group in the flocculation mechanism.

Because of the xanthate group's strong tendency to form covalent bonds, it is likely that the flocculation of bornite, covellite, and chalcopyrite particles involves, at least to some extent, covalent bonding of ISX to a metal site through the xanthate groups. The fact that bornite forms the largest and strongest flocs with ISX suggests that bonding between the xanthate group and the lattice metal component(s) is stronger for this mineral than for covellite and chalcopyrite. Selectivity may thus be determined by the relative bond strength between the chelating group and the surface metal component(s) in a given mineral. Factors other than the nature of the chelating group, such as any electronic or stereochemical contributions from the polymer backbone, may also influence flocculant selectivity, as indicated by Kitchener (1972). Similar factors are observed in the flotation of sulfide minerals with alkyl xanthates, where the length of the alkyl chain can affect selectivity.

Summary

Insoluble cross-linked starch xanthate flocculates bornite, covellite, pyrite, and chalcopyrite fines, but not quartz. The flocculation behavior — floc formation and clarification of supernatants — depends slightly on the pH at which ISX is added, and more strongly on subsequent pH adjustments.

ISX-induced bornite flocs are sufficiently strong to resist postflocculation washing treatments. These findings have been used to selectively flocculate and separate bornite from synthetic bornite-quartz mixtures, on a small laboratory scale. Bornite grades are in the range of 76 - 92%. ■

Acknowledgements

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