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THE EFFECT OF OXIDATION ON SULFIDE MINERAL FLOTATION

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Abstract. Oxidized sulfide minerals, commonly formed by weathering, increasingly are becoming important resources as primary sulfide deposits are depleted. Oxidation, in general, reduces selectivity and recovery in flotation. The objective of this research was to determine the mechanisms of oxidation and collector adsorption in sulfide systems as a step towards developing improved technologies for beneficiation of oxidized sulfides. Data from microcell flotation tests are combined with electrochemical and spectrophotometric measurements. The adsorption of ethyl xanthate (EX) on oxidized sulfides was characterized using AC impedance techniques and correlated to flotation response. The floatability of chalcocite, chalcopyrite and pyrite was dependent on the extent of oxidation. Oxidation for 1-3 d at 65° C inhibited flotation of chalcocite and chalcopyrite even after the addition of a large amount (1×10^{-3} M) of collector. Heavily oxidized (27 d) chalcocite remained unfloatable. However, longer oxidation of pyrite and chalcopyrite restored the floatability. Oxidation resulted in an electrically resistive, nonporous surface layer.

Introduction

Flotation often is used to separate sulfide minerals from gangue and to separate sulfide minerals from each other. Although some sulfides become inherently floatable following slight oxidation (Trahar, 1984; Guy, 1985; Woods, 1986; Hayes, 1987), oxidation generally reduces both the selectivity and the recovery in sulfide flotation.

Oxidation alters the mineral surface by forming an adherent layer of hydroxides (Rogers, 1962; Palagi, 1976). The effect of these layers on flotation is variable and the chemistry is poorly understood. Barzyk (Barzyk, 1981) found that heavily oxidized, synthetic chalcocite consumed approximately 100 times as much collector to obtain the same level of flotation as the least-oxidized samples. Sulfide oxidation also produces soluble ions of iron and lead (Senior, 1991), which may interfere with the flotation process (Taggart, 1964). The objective of the Bureau research is to determine the effect of oxidation on the flotation behavior of chalcocite, chalcopyrite and pyrite as a step towards improving technology for mineral beneficiation. This effort was directed primarily toward ores containing oxide or oxidized minerals rather than toward the effects of galvanic interactions occurring during crushing and grinding. Although flotation plants routinely process ores containing weathered minerals (Feltman, 1989; Palagi, 1976), much is unknown about how weathering alters the fundamental chemistry of flotation processes (Chander, 1991; Rogers, 1962).

Flotation is a result of surface chemistry of minerals. Electrochemical analyses of conductive minerals yield extensive information about the mineral surface. AC impedance characterizes the electrical resistivity of the surface layer. Increased impedance indicates formation of nonconductive layers on the surface of conductive sulfide minerals. These nonconductive layers may result from formation of oxides and from adsorption of collector. Cyclic voltammetry is used to study surface layer chemistry (Chander, 1987), including xanthate adsorption on sulfide minerals (Woods, 1985; O'Dell, 1985; Walker, 1984).

Experimental

Mineral Samples

Unoxidized chalcocite and chalcopyrite from Transvaal, South Africa, and pyrite from Mexico were obtained from Ward's Natural Science Establishment, Inc[1]. Compositions of the minerals are reported in Table 1. Mineral identification was confirmed by x-ray diffraction and SEM examination.

Reagents

Electrolyte solution was prepared from reagent-grade $Na_2B_4O_7$ dissolved in 18 MΩ deionized water at a concentration of 0.05 M, producing a solution buffered at pH 9.2. Potassium ethyl xanthate (EX) was purified of oxidation products by dissolving in acetone and crystallizing with ether three times (Harris, 1988). EX solution was prepared daily to ensure minimum decomposition.

Table 1. Chemical analyses of minerals used in this study.

Mineral	Cu (%)	Fe (%)	S (%)
Chalcocite	79.7	0.255	20.1
Chalcopyrite	34.9	27.2	34.8
Pyrite	0.067	44.3	53.1

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[1] Reference to specific trade names does not imply endorsement by the U.S. Bureau of Mines.

Equipment

A modified Hallimond cell as depicted in Fig. 1 was used in this study (Gebhardt, 1991). The cell had a platinum current collector supporting the mineral bed. The reference electrode attached to the cell through a capillary that ended near the top of the particle bed. The counter electrode was constructed of platinum gauze.

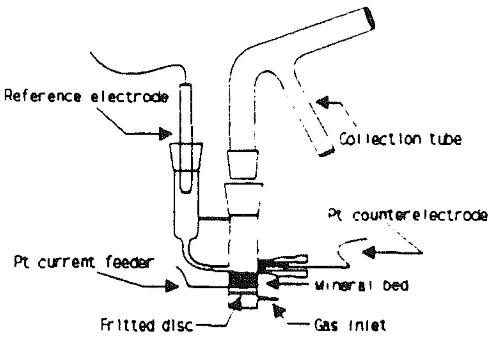

Figure 1. Modified Hallimond microflotation cell.

Impedance measurements were performed using EG&G Princeton Applied Research Corp (PARC) Model 388 Electrochemical Impedance system. The data acquisition and analysis were controlled by computer. Cyclic voltammetry tests were performed using a PARC Model 273 potentiostat/galvanostat linked to a computer.

Procedures

Mineral Preparation:

Minerals for this study were crushed in a mortar and pestle, and dry sieved to 65 x 325 mesh. Oxidized samples were prepared by exposing the sized samples to air at approximately 65° C for predetermined time periods accompanied by daily wetting of the minerals with deionized water to simulate cyclic wetting and drying in natural weathering. After preparation, samples were stored in a vacuum desiccator until used.

Floatability and AC Impedance Tests:

Floatability tests were performed to determine the concentration of EX needed to attain the full flotation recovery at each level of mineral oxidation Floatability, open circuit potentials (E_{oc}), and AC impedance measurements could be made in a single procedure. All electrochemical potentials are reported with respect to the standard hydrogen electrode (SHE).

The standard procedure was to fill the modified Hallimond cell with borate solution, add 2-3 g of crushed mineral, and add 1 x 10^{-7} M EX. The mineral bed was conditioned for 10 min and floated with air in the absence of frother. Chalcocite and chalcopyrite were floated for 2 min, and pyrite for 10 min for the floatability determinations. These flotation times were chosen from preliminary testing to maximize the sensitivity of the flotation test. The volume of floated mineral was determined by measuring the height of the bed in the collection tube. The floated product then was returned to the bottom of the cell, another aliquot of collector was added, and the flotation test repeated until complete flotation of the mineral was obtained or until EX concentration exceeded 10^{-2} M.

AC impedance characterizes the resistance to charge transfer (R_t) through the surface layer, commonly called the polarization resistance, and diffusion resistance (R_p) to ions moving through the pores of the surface layer. Values for these parameters are calculated from empirical circuit models constructed to fit the AC impedance data (Chander, 1991). Impedance measurements were conducted in air-saturated tetraborate solution following floatability testing. The mineral bed was conditioned for 2-3 h before impedance measurements to reach the steady-state E_{oc} of the mineral bed. An AC signal of 10 mV amplitude over the frequency range of 0.01 to 5 x 10^4 Hz then was applied to the mineral bed at the E_{oc}. The measured impedance spectra were fitted to equivalent circuit models using the Impedance Modeling Software (Boukamp, 1989) to obtain the resistance values.

Cyclic Voltammetry:

Cyclic voltammetry typically is used to observe redox reactions occurring on the surface of conductive solids. The technique has proven useful for studying surface layer chemistry (Chander, 1987) and xanthate adsorption on sulfide minerals (Woods, 1985; O'Dell, 1985; Walker, 1984). Cyclic voltammetry scans were performed using 0.5 g mineral samples in 30 mL of air-saturated, 0.05 M tetraborate solution. The minerals rested on a current collector constructed from platinum gauze. The counter electrode also was made from platinum gauze, and was separated from the mineral bed by a fritted glass disc. A saturated calomel electrode (SCE) served as a reference. The potential range was chosen for each mineral to span the interval between reduction of the mineral to produce H_2S and oxidation to form metal hydroxides (Pang, 1990; Chander, 1987; O'Dell, 1987; Buckley, 1985). The scan rate for these tests was 5 mv/s. Fifteen complete cycles were run for each test.

Xanthate Consumption Study:

EX solution concentrations were measured using a Hewlett Packard 8452A diode-array UV-visible instrument. EX consumption was calculated from ultraviolet absorbance at 302 nm. For consumption testing, 2 g crushed mineral was washed with distilled water to remove readily soluble metals, then conditioned in 20 mL of 5×10^{-5} M EX in tetraborate solution until concentration reached steady state. Supernatant EX solution was then withdrawn and centrifuged for UV analysis.

Results and Discussions

Chalcocite

Flotation and EX Consumption:

Flotation recovery exceeding 80% was observed at the E_{oc} for freshly ground chalcocite in borate solution containing 2×10^{-5} M and greater EX concentrations as shown in Fig. 2. Moderate oxidation of chalcocite (3.5 h at 65° C in moist air) decreased flotation recovery in 2×10^{-5} M EX solution; complete flotation was regained with 5×10^{-5} M EX. Oxidation for one day or longer totally inhibited flotation even in 1×10^{-3} M EX.

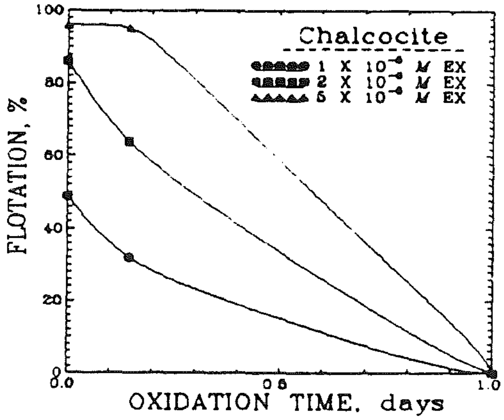

Figure 2. Flotation response of oxidized chalcocite at various EX concentrations.

In this study, EX consumption reached a steady state after 10 min conditioning period for all levels of chalcocite oxidation. Flotation recovery and EX consumption on chalcocite in 5×10^{-5} M EX solution, both reported as percentages, are plotted against oxidation time in Fig. 3.

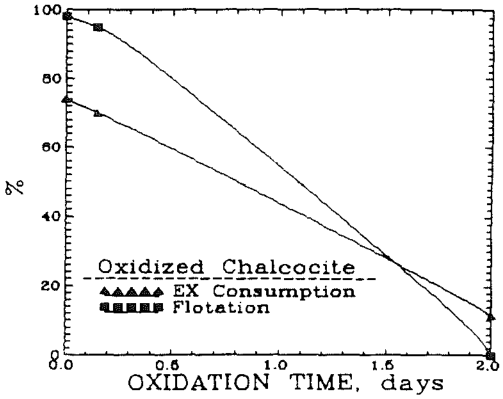

Figure 3. Chalcocite flotation and EX consumption in 5×10^{-5} M EX solution.

Oxidation markedly decreased EX consumption, which correlates well with reduced floatability resulting from oxidation. Oxidation apparently decreased the number of surface sites available for reaction with EX. Chalcocite oxidation in aqueous solutions proceeds through the formation of several intermediate compounds, containing sequentially less copper, before ultimate conversion to copper oxide or hydroxide (Hamilton, 1984). We suggest that the oxidized chalcocite became hydrophilic through a combination of a copper hydroxide layer on the surface and a decreased number of sites for adsorption of the xanthate collector.

AC impedance measurements:

The E_{oc} of freshly ground chalcocite in 2×10^{-5} M EX solution was 0.24 V. The potential became more positive with increased oxidation as reported in Table 2. Significant increases in the calculated R_t and R_p in EX solution were observed following oxidation of the chalcocite. This indicates that a highly resistive layer formed on the chalcocite surface during oxidation, and that the resistance of this layer increased with increasing oxidation period. This increased resistance of the surface layer coincided with the loss of flotation.

Table 2. Calculated parameters for chalcocite in 2×10^{-5} M EX solution.

Oxidation time, d	0	1	27
E_{oc}, V	0.24	0.27	0.38
R_t, kΩ	0.06	37.20	165.10
R_p, Ω	2.00	2.41	25.1
Flotation (%)	95	0	0

Cyclic voltammetry:

No xanthate adsorption or desorption peaks were observed on any of the oxidized chalcocite samples. A typical scan is shown in Fig. 4. This indicates that xanthate adsorption through an electrochemical reaction on the surface is not occurring. Although this does not preclude adsorption through other mechanisms, it agrees with UV data suggesting that oxidation of chalcocite sharply reduces xanthate adsorption.

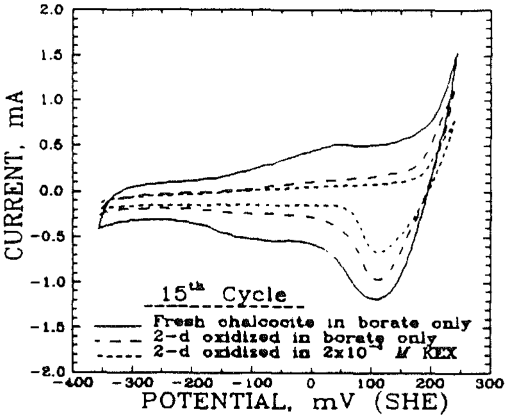

Figure 4. Voltammogram of chalcocite at pH 9.2.

Chalcopyrite

Flotation and EX Consumption:

At the E_{oc}, flotation recovery for freshly ground and moderately (3.5 h) oxidized chalcopyrite was high in 5×10^{-5} M as shown in Fig. 5, and continued high at higher EX concentrations. Oxidation for three days caused complete loss in the floatability which could not restored by the addition of more EX. However, high flotation recovery was regained with 1×10^{-4} M EX solution after 25 d oxidation.

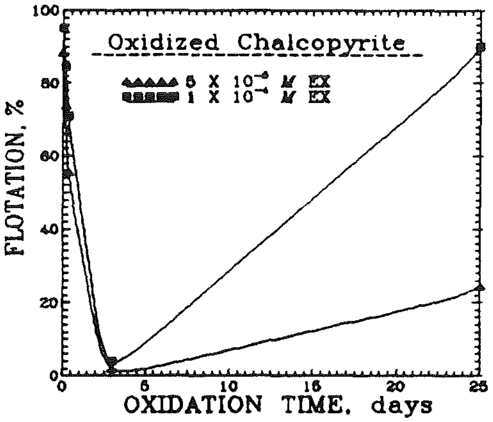

Figure 5. Flotation response of oxidized chalcopyrite at various EX concentrations.

Flotation recovery and EX consumption on chalcopyrite are plotted against oxidation time in Fig. 6. No change in EX consumption was observed for oxidation up to 3.5 h. EX consumption increased with oxidation for longer periods. This increased consumption coincided with the loss of floatability. The higher EX consumption likely resulted from precipitation of EX by metals ions in the solution.

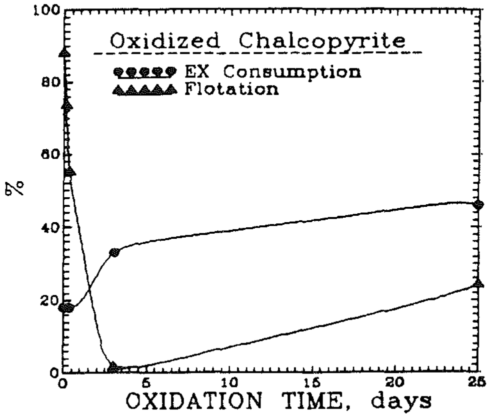

Figure 6. Flotation response and EX consumption of chalcopyrite in $5x10^{-5}$ M EX solution.

AC impedance measurements:

Oxidation of chalcopyrite shifted the E_{oc} in EX solutions in the positive direction as reported in Table 3. The potentials were 0.23 V and higher, which is above the reversible potential for the formation of dixanthogen (Walker, 1986). Thus, dixanthogen formation on the chalcocite surface is presumed to account for the floatability of chalcopyrite.

R_t for chalcopyrite in EX solution increased following two days of oxidation, but decreased after 25 days of oxidation. The decrease in R_p resulting from oxidation of chalcopyrite suggests that the surface layer became more porous during oxidation.

These data demonstrate changes in surface properties as chalcopyrite oxidizes. Oxidation for 2-3 d prevented chalcopyrite flotation and coincided with the formation of a high-resistance, passivating surface layer. Floatability was restored after 25 d oxidation and was accompanied with reduced polarization resistance. The mechanism of chalcopyrite oxidation in aqueous solution is postulated to involve an initial release of iron, followed by a later release of copper and sulfur species (Chander, 1991; Buckley, 1987). Copper and iron may then precipitate on the surface as copper and iron hydroxides. Thus, moderate oxidation of chalcopyrite probably caused the formation of a surface layer containing of a

Table 3. Calculated parameters for chalcopyrite in 5×10^{-5} M EX solution.

Oxidation time d	0	2	25
E_{oc}, V	0.228	0.37	0.31
R_t, kΩ	1.1	248.1	4.4
R_p, Ω	151	37.4	44.6
Flotation (%)	89	2	25

mixture of copper and iron hydroxides and oxysulfides. We suggest that this passivating surface layer is sufficiently hydrophilic to overwhelm the hydrophobicity induced by dixanthogen adsorption.

Further oxidation, however, apparently results in the formation of entirely different surface species. Strong oxidation has been shown to result in formation of elemental sulfur on the surface (Hamilton, 1981; Buckley, 1985). Oxidation for 25 days likely resulted in the formation of hydrophobic sulfur, allowing for improved chalcopyrite flotation compared with that for 2-d oxidized chalcopyrite.

Cyclic Voltammetry:

Voltammetry data for chalcopyrite at pH 9.2 are plotted in Fig. 7 No peaks attributable to xanthate adsorption were observed, which is consistent with data obtained by previous authors (Woods, 1986). Oxidation of chalcopyrite for two days passivated the mineral surface as seen from the reduced currents above 200 mV. Oxidation likewise suppressed the -550 mV peak on the reduction scan, which is attributed to the reduction of Fe(III) hydroxides on the mineral surface. Xanthate appeared to stabilize Fe(II) species in that the observed Fe(II) oxidation potential increased on the anodic scan in the presence of xanthate. Xanthate also enhanced slightly the observed current for the reduction of Fe(III) species on the cathodic sweep.

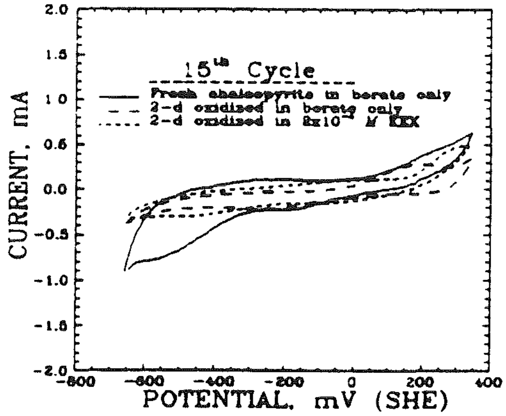

Figure 7. Voltammogram of chalcopyrite at pH 9.2.

Pyrite

Flotation and EX Consumption:

Freshly ground pyrite floated less readily than did the copper minerals. EX concentration in excess of 1×10^{-5} M was required even for partial flotation. Flotation increased with increasing EX concentration as shown in Fig. 8 and reached 98% in 1×10^{-3} M EX solution.

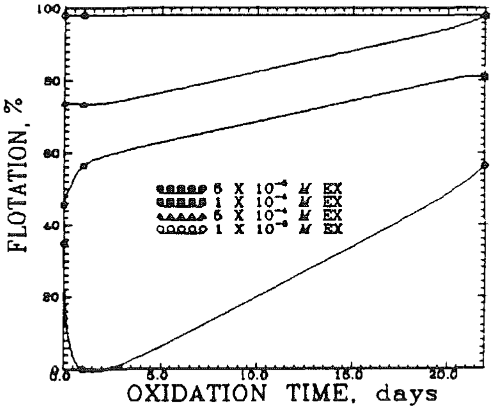

Figure 8. Flotation response of oxidized pyrite at various EX concentrations.

No flotation of 1-d oxidized pyrite was observed in 5×10^{-5} M EX, but floatability improved with increased EX concentration as shown in Fig. 8. Oxidation for 22 days, however, greatly enhanced pyrite flotation. EX consumption shown in Fig. 9 correlated well with flotation results: both decreased following one day of oxidation but increased following 22 day of oxidation.

The UV spectra showed the presence of perxanthate in EX solutions contacting oxidized pyrite (Pomianowski, 1963). Perxanthate was not detected in solutions with the other oxidized minerals. Perxanthate does not contribute to flotation.

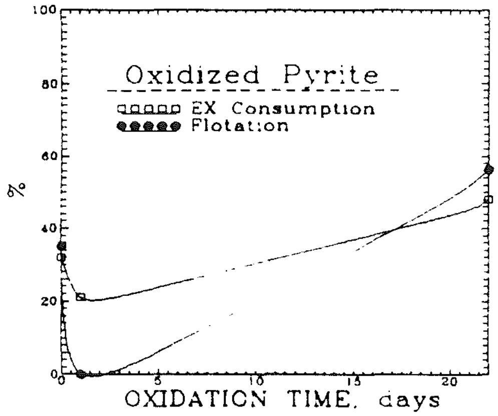

Figure 9. Flotation and EX consumption of pyrite in $5x10^{-5}$ M EX solution.

AC Impedance Measurements:

The potentials for pyrite in EX solution reported in Table 4 are sufficiently oxidizing to suggest that dixanthogen was responsible for pyrite flotation (Woods, 1986; Persson, 1991). R_t for 1-d oxidized pyrite was larger than that for freshly ground pyrite, but decreased after 22 days of oxidation. R_p was smaller for 1-d and 22-d oxidized pyrite in EX solution than for freshly ground pyrite, indicating an increase in porosity of the product layer as a result of oxidation.

Data in Table 5 show that higher concentrations of EX greatly reduce R_t for 1-d oxidized pyrite. This indicates a strong interaction between xanthate and the oxidation layer on the mineral surface.

Janetski (Janetski, 1977) identified iron hydroxide on pyrite surfaces. We suggest that the iron hydroxide layer is hydrophilic and exerts a depressing effect on pyrite flotation. It also has been proposed (Persson, 1991) that oxidation products of the disulfide ion in pyrite promote the formation of dixanthogen. Following 22-d oxidation, a sufficient amount of oxysulfides may form on the surface to facilitate dixanthogen formation, thus raising pyrite floatability.

Cyclic voltammetry:

Previous voltammetric investigations of fresh pyrite in xanthate solutions have not reported detectable adsorption peaks; no investigations of oxidized pyrite have been found. No peaks attributable to electrochemical adsorption of xanthate on pyrite were observed in any voltammograms of fresh or oxidized pyrite in this study. However, xanthate significantly reduced the electrochemical currents observed during voltammetry even of oxidized pyrite as seen in Fig. 10. Thus, strong adsorption of

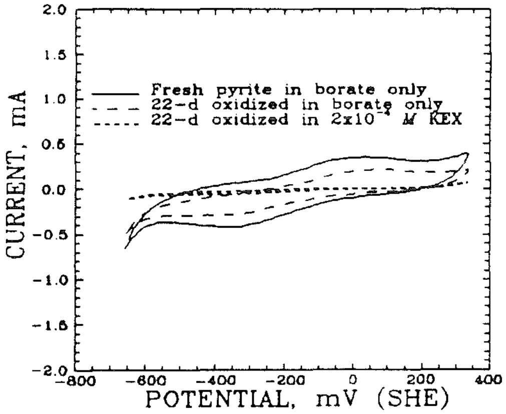

Figure 10. Voltammogram of pyrite at pH 9.2.

Table 4. Calculated parameters for pyrite in 5 X 10^{-5} M EX solution.

Oxidation time, d	0	1	22
E_{oc}, V	0.15	0.21	0.27
R_t, kΩ	5.6	122.8	24.8
R_p, kΩ	3.9	0.07	0.08
Flotation, %	35	0	56

Table 5. Pyrite behavior in 5 X 10^{-5} and 5 X 10^{-4} M EX solution.

KEX Concentration (M)	1 d oxidation	
	R_t, kΩ	Flotation response (%)
5 X 10^{-5}	122.8	33.0
5 X 10^{-4}	23.5	73.5

xanthate by oxidized pyrite is indicated by impedance measurements, voltammetry, and UV spectroscopy.

Comparisons Among Minerals

The data in Table 6 show significant increases for R_t and R_p of the fresh minerals in EX solution as the iron content of the mineral increases. This would suggest that formation of iron hydroxides is a major contributor to the observed surface resistances.

Summary

The effects of oxidation on the flotation behavior of chalcocite, chalcopyrite and pyrite in pH 9.2 buffer solution have been studied. Moderate oxidation (3.5 h for chalcocite and chalcopyrite; 1 d for pyrite) inhibits flotation. A higher concentration of EX collector can partially offset the effects of oxidation for these minerals. Oxidation for 1-3 d inhibited flotation of chalcocite and chalcopyrite even after the addition of a large amount (1×10^{-3} M) of collector. Heavily oxidized (27 d) chalcocite remained unfloatable. However, longer oxidation of pyrite and chalcopyrite restored the floatability. In the case of chalcopyrite, this is believed to result from the formation of hydrophobic sulfur on the mineral surface as a result of oxidation. In the case of pyrite, the restored flotation may result from oxidized disulfide species reacting to cause formation of dixanthogen.

AC impedance studies showed that the surface chemistry of these minerals in EX solutions changed with the degree of oxidation. Depression of oxidized chalcocite, chalcopyrite, and pyrite correlated with the formation of a high-resistance layer, which passivated the minerals. Restored floatability for heavily oxidized chalcopyrite and pyrite was accompanied by a breakdown of passivity.

There was a good correlation between decreased EX consumption and reduced floatability of oxidized chalcocite and oxidized pyrite. A small increase in EX consumption for 2 d oxidized chalcopyrite may be related to precipitation of metal xanthate compounds.

Table 6. Electrochemical parameters of the fresh minerals in EX solution.

	EX M	Fe %	R_t kΩ	R_p kΩ	E_{oc} mV
Chalcocite	2×10^{-5}	0.26	0.06	0.002	240
Chalcopyrite	5×10^{-5}	27.2	1.1	0.151	228
Pyrite	5×10^{-5}	44.3	5.6	3.90	150

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