

Critical copper concentration in sphalerite flotation: Effect of temperature and collector



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ABSTRACT

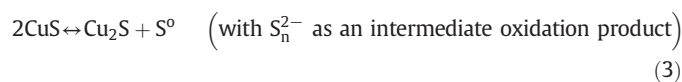
Sphalerite flotation and separation from other sulphide minerals are strongly dependent on pulp solution conditions such as pH and copper concentration but also temperature. The surface of sphalerite needs to be activated with copper ions to enable collector adsorption and therefore sphalerite flotation. It was found that the collectorless flotation recovery and rate of sphalerite increased with increasing copper concentration up to a maximum value before decreasing with further copper addition independently of temperature. This increase in recovery is attributed to the copper-activation of sphalerite with formation of the hydrophobic species of polysulphide/elemental sulphur. Several complementary solution and surface analytical studies have shown that the maximum recovery corresponds to the maximum number of surface zinc ions that can be replaced with copper ions (copper-activation mechanism). The decrease in recovery is not the result of further adsorption of copper hydroxide in molecular form at the sphalerite surface but it is attributed to copper hydroxide precipitation covering the hydrophobic surface species. Addition of xanthate collector restores the flotation recovery and rate of copper-activated sphalerite. It was also found that the flotation recovery and rate of copper-activated sphalerite at pH 10.5 are reduced when temperature is below 12 °C in the absence or presence of collector, which is in good agreement with observations made at several flotation plants that zinc recovery is on average less during winter than in summer. Possible causes for this decrease in sphalerite flotation at lower temperatures are discussed.

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1. Introduction

Sphalerite is a valuable mineral for zinc and has been one of the most studied sulphide minerals in relation to its flotation (Finkelstein and Allison, 1976; Finkelstein, 1997). Recovery of sphalerite from ores is quite a complex process; generally its flotation needs to be first depressed in copper and lead circuit at neutral to mildly alkaline pH values before it can be floated at high alkaline pH values for its separation from pyrite. Moreover, sphalerite is the only one valuable sulphide mineral which reacts poorly with thiol collectors such as xanthate, hence the need to activate its surface with addition of copper ions which act as a bridge and facilitate collector adsorption (Finkelstein and Allison, 1976). Although copper is the most common activator, a few studies have shown that other metal ions such as lead ions (Rey and Formanek, 1960; Houot and Raveneau, 1992) and ferrous ions (Zhang et al., 1992; Brienne et al., 1994) can also activate sphalerite flotation. These metal ion activators may also originate from the dissolution of sulphide minerals present in the ore, which can result in inadvertent sphalerite activation and therefore may reduce the copper and lead grade.

The sphalerite activation process with copper has been extensively studied over several decades (Finkelstein and Allison, 1976; Ralston and Healy, 1980a, 1980b; Ralston et al., 1981; Finkelstein, 1997; Laskowski et al., 1997; Chandra and Gerson, 2009). The process involves several steps; after adsorption of copper ions at low pH values or copper hydroxide at high pH values (Eq. (1)) on the sphalerite surface, the copper ions are known to exchange with zinc ions in the sphalerite lattice (Eq. (2)) due to the higher affinity of the former than the latter for sulphide ions (Ralston and Healy, 1980a, 1980b; Ralston et al., 1981; Finkelstein, 1997; Kakovskii, 1957; Weast, 1976). The cupric sulphide so formed is then reduced to the more stable cuprous sulphide together with the oxidation of sulphide to polysulphide, S_n^{2-} or elemental sulphur, S^0 (Ralston et al., 1981; Buckley et al., 1989a) according to Eq. (3).



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The driving force for copper adsorption is sought to be electrostatic between the positively charged copper species and the negatively charged sphalerite surface. The ion exchange for the uptake of copper and release of zinc into solution (Eq. (2)) is an approximate 1:1 exchange at the sphalerite surface and can proceed deeper from the top surface (Ralston and Healy, 1980b; Ralston et al., 1981; Pugh and Tjus, 1987).

The amount of copper required for sphalerite activation and maximum flotation performance will differ depending on the minerals present in the ore, their surface area and the pulp solution conditions. In particular, it has been shown that there is a critical copper concentration beyond which copper starts to act as a depressant of sphalerite flotation at alkaline pH values (Fornasiero and Ralston, 2006). Also, a change in pulp solution conditions such as its temperature has been found to affect sphalerite flotation performance (Albrecht and Fornasiero, 2011); indeed, several plants in Canada and Sweden (Fornasiero, personal communications, 2008) have observed lower and higher zinc recoveries in winter and summer months, respectively. There is only a small number of studies in the literature on the effect of temperature on flotation and currently little understanding of the mechanisms by which water temperature alters mineral flotation performance (Hukki, 1973; O'Connor et al., 1984; Lin, 1989; O'Connor and Mills, 1990; Lazarov et al., 1994; Alexandrova and Nishkov, 1995; Albrecht and Fornasiero, 2011). For example, changes in temperature may affect flotation performance through a change in mineral surface chemistry/hydrophobicity (due to mineral surface dissolution/oxidation; metal ion/hydroxide and reagent adsorption) and/or change in hydrodynamics (pulp viscosity; gas solubility; bubble size and velocity; froth stability; particle–bubble interactions).

In this study, the copper-activation process in alkaline pH conditions is revisited, especially focussing on the critical copper concentration required to produce maximum sphalerite flotation performance at different pulp temperatures in the absence and presence of a collector. For this, a range of complementary experimental techniques is used which results are critically analysed and compared. In particular, the results of a particle–bubble detachment experiment have clearly shown the different roles of the copper hydroxide species in sphalerite flotation.

2. Material and methods

All chemical reagents used were of analytical grade and all solutions were prepared using high purity Milli-Q water (conductivity of less than $0.5 \mu\text{S cm}^{-1}$ and pH 5.5 at 20°C) produced by reverse osmosis followed by two stages of mixed bed ion exchange, two stages of activated carbon treatment and finally filtration through a $0.22 \mu\text{m}$ membrane.

The sphalerite mineral sample was from the Elmwood Mine (Carthage, Tennessee, USA). Samples with obvious impurities were discarded. Bulk analysis of the sphalerite samples was conducted by the Australia Mineral Development Laboratories (AMDEL) and the results are shown in Table 1.

For each experiment, the desired amount of mineral was freshly wet ground by hand using a ceramic mortar and pestle in KNO_3 ($2 \times 10^{-3} \text{M}$) solution at pH 10.5 (except at pH 4.0 for the zinc to copper exchange experiment) and immediately transferred to a water jacketed, closed conditioning vessel where the pH of the solution was controlled with the addition of small amount of concentrated nitric acid or potassium hydroxide solutions. Copper nitrate, when required, was added at the beginning of the conditioning period of 20 min. Samples with

Table 1
Chemical analysis of the sphalerite mineral sample (Elmwood mine, Carthage, Tennessee, USA).

Elements	Zn	S	Cu	Fe	Pb	Si	Ca	Mg	Mn
Mass %	67.30	31.30	0.17	0.18	0.13	0.10	0.07	0.02	0.01

particle sizes of $38\text{--}75 \mu\text{m}$ and $3\text{--}50 \mu\text{m}$ were selected for the flotation experiment, and zeta potential and zinc to copper exchange experiments, respectively. A Malvern Mastersizer 2000 was used to determine the size and surface area (assuming particles are spherical with a smooth surface) of the particles.

A Malvern Zeta Sizer Nano (Model 2CA) apparatus was used for the zeta potential measurements. At least ten particle mobility measurements were performed at each pH value and the average mobility was converted to zeta potential using the Smoluchowski equation. After mineral conditioning (0.25g ZnS in 0.4dm^3 solution), stirring was stopped momentarily to allow the larger particles to settle out. A 10cm^3 sample of the mineral dispersion (particles $<10 \mu\text{m}$) was transferred into a vertically mounted cell using a glass pipette. The particle mobility was measured from pH 10.0 to pH 4.0 and back to pH 10.0, allowing 5 min equilibration time for the mineral dispersion at each new pH value before measurement.

An electro-acoustic technique (Holtham and Cheng, 1991; Xu et al., 2011) was used to measure the critical amplitude of vibration (at constant frequency of 50 Hz) required to detach a particle from a bubble in solution. At the end of the conditioning period, one $350 \mu\text{m}$ sphalerite particle (in 0.1dm^3 solution) was attached to a 2 mm bubble generated at the end of the capillary tube connected to the membrane of a loudspeaker. The average critical detachment amplitude was determined by conducting the test for five randomly chosen particles with ten consecutive measurements for each particle.

For the measurements of copper and zinc in solution, the mineral dispersion (0.6g ZnS in 0.12dm^3 pH 4.0 solution) was filtered at the end of the conditioning period and the filtrate was acidified before measurement of the zinc and copper concentrations by inductively coupled plasma-optical emission spectroscopy.

A Kratos Axis Ultra X-ray photoelectron spectrometer (XPS) with an Al $\text{K}\alpha$ monochromated X-ray source (1486.8eV) operated at 130 W was used to measure the species present on the sphalerite surface. All measurements were acquired at a take-off angle normal to the surface. Samples were mounted on a conducting copper tape and placed on the sample holder which was introduced immediately in the fore-vacuum of the XPS spectrometer. The binding energy scale was calibrated with the $\text{C}_{(1s)}$ peak at 284.6eV (Buckley et al., 1989b). The XPS spectra were curve fitted using the Casa XPS program.

The flotation experiments were conducted in a 0.3dm^3 IMN flotation machine (Gliwice, Poland) at a pulp agitation speed of 2200 rpm and gas flow rate of $1.0 \text{dm}^3 \text{min}^{-1}$. Temperature was maintained to within $\pm 3^\circ\text{C}$ via a thermostatically controlled water bath which housed the flotation cell. The solution (pH = 10.5; $\text{KNO}_3 = 2 \times 10^{-3} \text{M}$) used for mineral grinding and conditioning was prepared at the desired temperature. Collector, sodium ethyl xanthate ($5.8 \times 10^{-6} \text{M}$), and frother, DowFroth 250 ($5 \times 10^{-6} \text{M}$), were added to the mineral dispersion (2.5g ZnS in 0.3dm^3 solution) during conditioning 2 min and 1 min before the start of flotation, respectively. Concentrates were collected at 0.5, 2, 4 and 8 min. The flotation rate constant, k , and the maximum flotation recovery, R_{max} , were calculated from the recoveries versus time data using the first order rate equation, $R(t) = R_{\text{max}}(1 - \exp(-kt))$.

3. Results and discussion

3.1. Copper species in solution

Fig. 1 shows the results of the calculations of the predominant copper species as a function of pH and copper concentration at temperatures of 5, 25 and 35°C . The concentration of copper species were calculated from $\log K$ values for the hydrolysis of Cu^{2+} to $\text{Cu}(\text{OH})^+$, $\text{Cu}(\text{OH})_{2,\text{aqueous}}$, $\text{Cu}(\text{OH})_3^-$ and $\text{Cu}(\text{OH})_{2,\text{solid}}$, species ($\log K$ values were in turn calculated from ΔG values at each temperature and corresponding ΔH and ΔS values for each hydrolysis reaction sourced from Garrels (1960)). Cu^{2+} and $\text{Cu}(\text{OH})_3^-$ are the predominant copper species in acidic and strongly alkaline solutions, respectively, while copper

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