



# Impact of gypsum supersaturated water on the uptake of copper and xanthate on sphalerite



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## ABSTRACT

Gypsum supersaturated process water has been shown to have an adverse effect on the flotation of sphalerite minerals. This study probes the mechanism of such effect by determining the uptake of copper and xanthate on sphalerite in gypsum supersaturated water using zeta potential distribution measurement, atomic absorption spectroscopy, UV–visible spectroscopy and quartz crystal microbalance with dissipation. Our results indicate that the supersaturation of water by gypsum retards the adsorption of copper and hence the sequence xanthate adsorption on sphalerite. The retardation of copper and xanthate adsorption on sphalerite is also observed by the addition of 800 ppm calcium. The adsorption of calcium is identified to compete with copper species for the reactive surface sites of the sphalerite, resulting in the reduction in copper and xanthate uptake and hence flotation recovery of sphalerite.

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## 1. Introduction

In mineral flotation plants it is a common practice to treat the tailings water and recycle back to the flotation process. The recycle of water can change the quality of process water by accumulating and building up the dissolved ions and flotation chemical additives. Certain ions and residual flotation chemicals in the recycle water could have either positive or negative effect on the flotation process depending on the nature and concentrations of chemicals and mineral systems. Zinc ions from the recycle water, for instance, are beneficial for selective separation of galena from sphalerite in the lead–zinc flotation separation. On the other hand, some ions and chemical additives accumulated in the recycle water can be detrimental to flotation separation (Brown, 2003; Johnson, 2003; Levay et al., 2001; Muzenda, 2010; Schumann et al., 2003). In sulphide mineral flotation operations that practice tailings water recycle, for instance, the process water with the recycle water from the tailings treatment could be often saturated or supersaturated with gypsum. The concentrations of the calcium and sulphate ions up to as high as 800 ppm and 2800 ppm, respectively, are not uncommon, exceeding the solubility limit of gypsum which is 2400 ppm in water at room temperature. The high concentration of calcium and sulphate ions in the process water not only causes scaling problems but also affects the mineral flotation separation.

Problems in flotation recovery and selectivity have been reported in the flotation of galena, sphalerite and chalcocopyrite in the presence of high concentrations of calcium or calcium and sulphate ions (Grano et al., 1997; Lascelles et al., 2003; Liu et al., 1993; Liu and Zhang, 2000; Moignard et al., 1977; Nesset et al., 1998).

It has been speculated that gypsum might precipitate out from the gypsum saturated or supersaturated solution and coat on the mineral surface, which might be responsible for the reduced flotation recovery and selectivity. However, our previous study on silica and sphalerite minerals in the gypsum supersaturated solution system suggested the absence of gypsum coating on mineral surfaces. Despite the presence of needle-shaped gypsum precipitates in the gypsum supersaturated solution system, the gypsum precipitates neither grew on sphalerite mineral surface nor heterocoagulated with sphalerite minerals under the typical flotation chemistry conditions. Our current study focuses on the impact of gypsum supersaturated recycle water on the interactions between flotation reagents and sphalerite mineral.

It is well known that sphalerite mineral responses poorly to thiol collectors due to the relative low stability of zinc-xanthate complexes as compared to other heavy metal xanthates (King, 1982; Rao, 2004). The flotation of sphalerite mineral with short chain thiol collectors (xanthates) is commonly accomplished by activation with other heavy metal ions, usually copper. The activation of sphalerite with copper has been extensively studied for many years in a number of laboratories (Chandra and Gerson, 2009; Finkelstein, 1997; Fuerstenau, 1980; Kartio et al., 1998; Kivitova et al., 1987; Lascelles et al., 2001; Laskowski et al., 1997; Maust and Richardson, 1976; Patrick et al., 1999; Plichon

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and Rollat, 1990; Ralston and Healy, 1980a,b; Sui et al., 1999; Teng et al., 2012 Wark and Wark, 1936). Many activation mechanisms have been proposed depending on the activation pH where activation occurs. It is generally accepted that under acidic conditions, the activation is through an ion exchange process with copper replacing surface zinc ions leading to copper sulphide-like surfaces. At neutral and alkaline pH, however, a variety of mechanisms have been proposed with the involvement of species from copper hydrolysis. The activation process of sphalerite with copper involves two stages (Rao, 2004). At the initial stage, the uptake of copper is rapid, which is a weak function of the copper concentration in solution and is controlled by solution diffusion. Once the uptake of copper exceeds three monolayers, the diffusion of copper in the solid state follows the parabolic law. The activation of sphalerite minerals with copper could be affected by surface charge, contaminants in process water or on the mineral surface (Chandra and Gerson, 2009; Harmer et al., 2008). The gypsum supersaturation in recycle water might have effect on the copper activation of sphalerite and hence the adsorption of xanthate. Calcium ions have been suggested to specifically interact with ZnS surface (Duran et al., 1995) and the exchange rate with activating ions has been suspected to be reduced in the presence of calcium (Moignard et al., 1977). However, others reported no impact of calcium and/or magnesium ions on the uptake of copper and xanthate by sphalerite (Lascelles et al., 2003).

In order to avoid the depression action of gypsum supersaturation in recycle water on sphalerite flotation, it is important to understand the impact of gypsum supersaturation in recycle water on the interactions between flotation reagents (copper and xanthate) and sphalerite mineral. In this study, we used a gypsum supersaturated solution to represent the gypsum supersaturated recycle water. The calcium and sulphate concentrations of the gypsum supersaturated solution are similar to those in the recycle water. The purpose of this study is to learn a better understanding of the impact of gypsum supersaturation in recycle water on sphalerite flotation.

## 2. Experimental

### 2.1. Materials

The sphalerite mineral sample was obtained from Ward's Natural Science Establishment. The sample was crushed to  $-4.75\ \mu\text{m}$  and dry ground to  $-45\ \mu\text{m}$ . Sodium hydroxide and hydrochloric acid from Fisher Scientific were used for the pH adjustment. Calcium sulphate dihydrate from Fisher Scientific and calcium sulphate hemihydrate from US Gypsum Corporation were used as supplied. Calcium chloride (Fisher Scientific) was used to prepare calcium solution. Copper sulphate pentahydrate from Fisher Scientific was used as activator for sphalerite. Sodium isopropyl xanthate (SIPX:  $(\text{CH}_3)_2\text{-CH}_2\text{-O-CS}_2\text{Na}$ ) from Prospec Chemicals, Canada, was used as collector. The commercial SIPX was purified by dissolving it in warm acetone ( $40\ ^\circ\text{C}$ ), followed by recrystallization and washing with acetone for three times. The purified SIPX was stored in a deep freezer at  $-20\ ^\circ\text{C}$ . Deionized water (DI water) from a Millipore Elix 5 purification system was used throughout this study.

### 2.2. Preparation of suspensions and solutions

Suspensions of sphalerite and gypsum were prepared for zeta potential distribution measurements. A sphalerite stock suspension was prepared by grinding 1 g of fine sphalerite ( $-45\ \mu\text{m}$ ) in a mortar with a few drops of water for 15 min and then diluting the ground sample with 50 mL of DI water. The gypsum suspension was prepared by adding 1 g of calcium sulphate dihydrate ( $\text{CaSO}_4$

$\cdot 2\text{H}_2\text{O}$ ) particles into 100 mL gypsum supersaturated solution. Fresh batches of sphalerite and gypsum suspensions were prepared for each testing.

Laboratory gypsum supersaturated solution was prepared using calcium sulphate hemihydrate, following procedures described elsewhere. The calcium and sulphate concentrations of the gypsum supersaturated solution were determined to be 845 ppm and 2100 ppm, respectively. A  $10^{-2}\ \text{M}$  KCl solution was prepared and used as a supporting electrolyte solution for the zeta potential distribution measurements. A 800 ppm calcium solution was prepared by dissolving calcium chloride in DI water. Stock solutions of  $10^{-2}\ \text{M}$  copper sulphate pentahydrate and  $10^{-2}\ \text{M}$  SIPX were prepared in DI water. The stock solutions were diluted with other prepared solutions such as DI water,  $10^{-2}\ \text{M}$  KCl, 800 ppm calcium, gypsum supersaturated solution, to make  $1.25 \times 10^{-5}\ \text{M}$  copper and  $1.25 \times 10^{-5}\ \text{M}$  SIPX concentration. The solutions were prepared freshly for each set of experiments. The pH of the solutions was adjusted to 6.5.

### 2.3. Zeta potential distribution measurement

Zeta potential distributions were measured using a SEPHY/CAD Zetaphorometer III equipped with a rectangular quartz electrophoresis cell. Suspensions of sphalerite were analyzed in a supporting electrolyte solution ( $10^{-2}\ \text{M}$  KCl), 800 ppm calcium solution, and gypsum supersaturated solution in the presence of  $1.25 \times 10^{-5}\ \text{M}$  copper and/or SIPX.

For each zeta potential distribution measurement, the stock suspension was diluted to an optimal concentration of 0.01–0.1% so that between 20 and 150 particles could be tracked by imaging analysis software during a mobility measurement. At least five mobility measurements in each direction were recorded for each sample. All zeta potential distributions were measured at pH 6.5 and room temperature.

### 2.4. Copper and SIPX adsorption

AAS (VARIAN 220FS) and UV-Vis-NIR Spectrophotometer (Shimadzu UV-3600) were used to determine the solution concentration of copper and SIPX, respectively. The sphalerite mineral samples (1 g) were dispersed in desired solutions (100 mL) for 30 min prior to conditioning in  $1.25 \times 10^{-5}\ \text{M}$  copper solutions for 5 min. The solids were collected using vacuum filtration and transferred immediately to 100 mL of desired solutions containing  $1.25 \times 10^{-5}\ \text{M}$  SIPX and conditioned for another 5 min. The supernatant was collected after settling of the solids. The concentration of copper in filtrate and the concentration of SIPX in the supernatant were determined from which the uptake of copper and SIPX by sphalerite was determined.

### 2.5. Quartz crystal microbalance with dissipation (QCM-D) measurement

The adsorption kinetics of copper and SIPX on sphalerite was studied using a QCM-D (Q-Sense AB, Göteborg, Sweden). The QCM-D measures the adsorption mass ( $\Delta m$ ) in terms of a negative shift in resonance frequency ( $\Delta f$ ) of a quartz crystal. For homogeneous, rigid and thin adsorbed layers, changes in mass ( $\Delta m$ ) on the quartz surface are related to changes in frequency ( $\Delta f$ ) of the crystal through the Sauerbrey relationship (Sauerbrey, 1959):

$$\Delta m = -\frac{C \cdot \Delta f}{n} \quad (1)$$

where  $C$  is the mass sensitivity constant equal to  $17.7\ \text{ng cm}^{-2}\ \text{Hz}^{-1}$  when  $f = 5\ \text{MHz}$ ,  $n$  is the overtone ( $n = 3, 5, 7, \text{ or } 9$ ) of the resonance frequency of the applied voltage across the electrodes.

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