



Effect of calcium, sulphate and gypsum on copper-activated and non-activated sphalerite surface properties



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ABSTRACT

This work presents results of an experimental study on the depression of sphalerite by the precipitation of calcium sulphate dihydrate (gypsum, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), and the role of the addition of sodium carbonate (Na_2CO_3) in restoring hydrophobicity. Zeta potential, microflotation, contact angle measurement and scanning electron microscopy were used. The zeta potential results show that sphalerite and gypsum were negatively charged over the pH range studied (5–10), and therefore no interaction between the two species was observed; however, when sphalerite was activated with 5 mg/L of Cu at pH 9, the mineral developed a positive charge, thus enabling the electrostatic interaction with gypsum particles which resulted in gypsum covering a significant fraction of the mineral surface. The contact angle measurements show that the simultaneous presence of Ca^{2+} and SO_4^{2-} ions decreased the hydrophobicity that the mineral had developed due to copper activation in the absence of these two species (19.6° and 28.7° , respectively). The presence of 10 mg/L of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ in equilibrium with the saturated solution ($0.0205 \text{ mol/L CaSO}_4$), further decreased the contact angle to 9.7° . In turn, the addition of 1 g/L of Na_2CO_3 and 15 min of conditioning substantially restored the hydrophobicity of the sphalerite by a mechanism consisting of gypsum dissolution and calcium consumption via calcium carbonate precipitation, which disperses in the bulk solution. This last practice resulted in a contact angle of 23.4° .

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1. Introduction

Increasing the use of recycled water in the mineral processing industry is becoming compulsory due to more stringent environmental regulations. These regulations are intended to ensure the preservation of the environment and to encourage the efficient use of water. However, since process water chemistry is completely different to that of fresh water, there is a latent concern regarding the effect of the chemical species present in the recycled water on the recovery of sulphide minerals (Rao and Finch, 1989).

It is recognized that the recycled water used in flotation significantly affects the floatability and separation selectivity of the minerals of interest. Typical contaminants of this water are colloidal matter, heavy metal ions, thiosalts, sulphite, sulphate, chloride, magnesium, calcium, sodium and potassium ions. These species (besides reactive residues such as frothers, collectors and depressants (Biçak et al., 2012)) may act as specific inadvertent mineral depressants: for example, sulphate and calcium ions precipitate as gypsum decreasing the floatability of galena and sphalerite (Grano et al., 1995). Calcium and sulphate ions are two of the most common species found in sulphide ore concentrator process water.

The presence of calcium is due to the use of calcium hydroxide as a pH regulator and the dissolution of calcium bearing minerals (e.g., calcite, dolomite, etc.), when they are present in the ore. In turn, sulphate concentration above the saturation concentration for gypsum precipitation is due to the natural oxidation of sulphide minerals by atmospheric oxygen during grinding, conditioning, flotation and thickening. It is known that under alkaline conditions the sulphide (S^{2-}) is oxidized to sulphate (SO_4^{2-}), the more stable oxidized species, going through polysulphide (Sn^{2-}), thiosulphate ($\text{S}_2\text{O}_3^{2-}$) and sulphite (SO_3^{2-}) (Ralston et al., 2007). Typical process waters have calcium concentrations in the order of 700 mg/L, while sulphate concentration may be at the saturation point (about 1700 mg/L) or greater (when water is at metastable equilibrium) (Grano et al., 1995; Sui et al., 1998; Levay et al., 2001; Deng et al., 2013).

The effect of dissolved ions in the recycled water on mineral floatability has been the subject of many studies. Previous work has demonstrated the depressing effect of Ca^{2+} ions on sphalerite (Sui et al., 2000; Ikumapayi, 2010; Ikumapayi et al., 2012); it was revealed that Ca ions adsorbed on the mineral increasing its superficial charge and significantly decreasing the number of available sites for Cu adsorption during the activation stage. The studies also confirmed the dominant effect of the calcium ions compared to the effect of the sulphate ions, also present in the solution (Ikumapayi,

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2010; Ikumapayi et al., 2012). As well, several studies have reported that the addition of sodium carbonate enhanced the flotation kinetics of galena and sphalerite (Grano et al., 1995; Espinosa et al., 2009), as this reagent causes the dissolution of gypsum (adhered on the mineral surface) and the precipitation of calcium carbonate (which disperses in the bulk solution), thus reducing the concentration of calcium in solution and its adverse effect on the hydrophobicity of sphalerite (Sui et al., 1998).

In this context, the aim of the present study was to investigate the effect of the individual and combined presence of calcium, sulphate and gypsum on the hydrophobic character of copper activated sphalerite. The interactions in the ZnS–CaSO₄–gypsum–Na₂CO₃ system were also studied.

2. Experimental methodology

2.1. Sphalerite characterization

A sphalerite specimen was collected from the Bismark mine of Peñoles, which belongs to the mining district of La Ascención (Chihuahua, Mexico). Elemental analysis of the mineral specimen (by atomic absorption spectroscopy and inductively coupled plasma mass emission) gave the following composition (% by weight): 59.07% Zn, 29.09% S, 0.03% Cu, 5.71% Fe, 0.012% Pb and 6% insoluble. It is worth noting that the sphalerite specimen used is practically free of copper and lead; X-ray diffraction analysis revealed only sphalerite (Powder diffraction file #03-065-0309).

2.2. The contact angle technique (flat surfaces)

In the present work, the contact angle technique used was based on the use of a mineral specimen ground and polished according to the procedure and recommendations of Wark and Cox (1934), and recently reported by Chau (2009), which basically consists of polishing the specimen under controlled conditions making use of deionized and deoxygenated water, giving fairly reproducible measurements. Work performed by Dávila-Pulido (2010) has found that sphalerite recovery and contact angle are directly related (% Recovery = 1.26 × Contact angle (degrees), R² = 0.99), according to results obtained in microflotation experiments using –106/+75 μm particles; thus, the contact angle measured with the methodology and technique used in this work is a fair representation of the phenomena of contact and adhesion between the air bubbles and the hydrophobized sphalerite particles.

2.3. Sphalerite specimen preparation

In the contact angle experiments the sphalerite specimen was mounted in metallographic resin; the specimen was wet ground and polished with silicon carbide sandpaper (80–800#) making use of deionized and deoxygenated water. It is worth mentioning that polishing was carried out prior to each test with the aim of refreshing the mineral surface. After specimen preparation, chemical conditioning was carried out under the different conditions which approximately reproduce those encountered in industrial flotation circuits (see Table 1). All the tests were conducted at 30 °C. Following conditioning, the specimen was transferred to a Plexiglas box containing 200 mL of an aqueous solution at pH 9 and constant ionic strength (10^{–3} mol/L NaNO₃). The specimen was contacted with an air bubble of ca. 0.75 mm diameter, which was generated with a digital syringe (Hamilton); when the contact angle was established, a photograph of the three-phase contact was taken with a digital camera equipped with a 65 mm macro lens allowing 5× magnifications (e.g., 4.33 × 2.88 mm images). The image was interpreted with image analysis software (Image-Pro 5.1) to obtain the contact angle (see Dávila-Pulido and Uribe-Salas, 2011). The experiments were performed in triplicate and the average of the contact angle is reported; the error bars correspond to the 99% confidence interval of a Student's *t*-distribution. The sodium nitrate, sodium hydroxide and hydrochloric acid were reagent-grade chemicals (Sigma–Aldrich).

2.4. Zeta potential

Zeta potential measurements were performed using a Pen Kem Lazer Zee Meter model 501, which allows the direct reading of the zeta potential that is calculated from the electrophoretic mobility of the particles in the suspension applying the Smoluchowski equation. To evaluate the zeta potential of sphalerite, a 1 g mineral fragment was dry-ground in an agate mortar and then suspended in the aqueous solution of interest (e.g., Cu(II), NaNO₃) that had been previously deoxygenated by bubbling N₂. The suspensions were prepared with a constant ionic strength aqueous solution (10^{–3} mol/L NaNO₃) and 1 g/L of ground mineral. In the case of gypsum (Sigma–Aldrich), around 0.02 g was suspended in 100 mL of a saturated supernatant solution (which was in equilibrium with solid gypsum); pH was controlled with a 0.01 mol/L of NaOH or HCl solution. In both cases, measurements were performed in duplicate and the average value of the measurements is reported together with the error bars of a 95% confidence interval of a Student's *t*-distribution.

Table 1

Contact angle of sphalerite with air bubble in the presence of the different species of interest.

Test no.	Stage I (15 min)					Stage II (5 min)				Contact angle (°) ^a
	Cu(II) (mg/L)	Ca (mg/L)	SO ₄ ^{2–} (mg/L)	CaSO ₄ ·2H ₂ O (mg/L)	Na ₂ CO ₃ (g/L)	Na ₂ CO ₃ (g/L)	NaNO ₃ (M)	SIPX (M)	Ca (mg/L)	
1	50	–	–	–	–	–	10 ^{–3}	–	–	28.7 ± 3.1
2	50	882	–	–	–	–	10 ^{–3}	–	–	28.0 ± 0.6
3	50	–	1987	–	–	–	10 ^{–3}	–	–	25.3 ± 1.4
4	50	882	1987	–	–	–	10 ^{–3}	–	–	19.6 ± 0.7
5	50	882	1987	10	–	–	10 ^{–3}	–	–	9.7 ± 4.2
6	50	882	1987	10	1	–	10 ^{–3}	–	–	13.6 ± 2.3
7	50	–	–	–	2	–	10 ^{–3}	–	–	14.9 ± 2.5
8	50	882	1987	10	–	1	10 ^{–3}	–	–	19.0 ± 3.1
9	50	–	–	–	–	–	10 ^{–3}	10 ^{–4}	–	54.5 ± 2.1
10	50	–	–	–	–	1	10 ^{–3}	10 ^{–4}	882	53.4 ± 1.8
11	50	882	1987	10	–	–	10 ^{–3}	10 ^{–4}	–	40.5 ± 2.4

Note: The contact angle was measured in the solution used in the Stage II of conditioning.

^a The contact angle measurements were performed in triplicate and the average value is reported, also the value that delimits a confidence interval of 99% (Student's *t*-distribution).

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