



A XANES and XRD study of chalcopyrite bioleaching with pyrite



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ABSTRACT

The galvanic effect between chalcopyrite and pyrite was investigated by solution studies, X-ray Diffraction (XRD), and synchrotron X-ray Absorption Near Edge Structure (XANES). The solution studies indicate chalcopyrite leaching efficiency was significantly improved in galvanic assisted leaching with more than 90% of copper being dissolved in 31 days in leaching at both 48 °C and 30 °C. In contrast, the addition of microorganism negatively affected the leaching efficiency at 30 °C. XRD and iron K-edge XANES analysis indicate the presence of microorganisms decreased the chalcopyrite leaching selectivity, likely linked to the increased solution potential. Chalcopyrite was selectively leached in chemical leaching as a result of galvanic effect. On the other hand, dissolution rate of pyrite was fast in the case of bioleaching where the Eh was high.

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1. Introduction

The understanding of dissolution behaviour of chalcopyrite is of significant interest because chalcopyrite is the most abundant copper source but also the most refractory of copper sulfides. Thus, low cost technologies, such as heap and in situ leaching, are the target of the mining industries. The disadvantage of heap and in situ leaching is the slow dissolution rate of chalcopyrite, which is affected by a range of parameters such as temperature, the addition of pyrite and iron ions and silver ions and activated carbon (Li et al., 2013). To enhance the chalcopyrite leaching rate, it is important to understand how the environmental parameters affect chalcopyrite leaching and the mechanisms involved in it.

The effect of pyrite on chalcopyrite leaching is commonly explained as galvanic effect, which results in the preferential leaching of chalcopyrite in a chalcopyrite-pyrite system. Two commercial processes which make use of galvanic interaction are the Galvanox™ process (Dixon et al., 2008; Nazari et al., 2011) and the Geocoat™ process (Petersen and Dixon, 2006).

In an ideal galvanic leaching scenario, the minerals of interest would be selectively leached while the other minerals can be recovered and recycled to the leaching circuit for further processing. However, the leaching selectivity of galvanic system has not been investigated in detail. Mehta and Murr (1983) noted a drop

in solution pH and an increase in solution redox potential during microbial leaching of chalcopyrite and pyrite mixtures. These behaviours may be linked to biooxidation behaviour of bacteria and dissolution of pyrite, though it has not been explicitly mentioned in the paper. The latter is a common observation during pyrite leaching (Schippers et al., 1996). A later study on heap bioleaching of copper-gold concentration using the Geocoat technology by Petersen and Dixon (2006) suggested that significant pyrite dissolution occurred during bioleaching at 30 °C. They attributed the pyrite dissolution to the high solution redox potential. In these studies, the selectivity of chalcopyrite leaching has not been quantified and the link between leaching selectivity and solution properties has not been established to our knowledge.

In a chemical galvanic leaching, 98% or greater copper recoveries have been achieved in as little as 4 h under atmospheric pressure and 80 °C with initial iron salts addition (Dixon et al., 2008). At the same time, the presence of microorganisms can influence the leaching efficiency of chalcopyrite. In a bacterial galvanic leaching, Mehta and Murr (1982) reported an improvement in copper leaching efficiency by a factor of 2.1 when chalcopyrite and pyrite were leached in the presence of microorganisms in an initially iron-free system. However, it is unclear how microorganisms affect the leaching in initially iron-containing medium. Previous studies on pure chalcopyrite indicate chemical leaching with appropriate initial Eh (ferrous/ferric ratio) could be more effective than bioleaching at the same conditions (Córdoba et al., 2008a; Yang et al., 2015). Therefore, it is interesting to know whether

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microorganisms affect chalcopyrite-pyrite galvanic leaching in the same way.

Bioleaching is affected by temperature and microorganisms in use. For instance, leaching thermophiles have better performance over mesophiles, which is mostly a result of increased kinetics of abiological reaction and could also be partially related better iron and sulfur oxidation capacities (Zhu et al., 2011). On the other hand, microbes with similar optimal temperature also have different roles in bioleaching because of their difference in metabolism. For instance researchers found chalcopyrite was oxidized by *A. ferrooxidans* but not by *A. thiooxidans*, which is mainly because of the lack of iron oxidation capacity of *A. thiooxidans* (Lizama and Suzuki, 1991).

The motivation of this work is to study and understand the influence of different microbes and temperatures on galvanic leaching behaviour of chalcopyrite concentrates. Quantitative phase analysis of XRD and XANES were used to understand the leaching selectivity and the leaching products formation.

2. Experimental

2.1. Microorganisms and minerals

Three species of mesophiles were used in this work. *Acidithiobacillus thiooxidans* (*A. thiooxidans*) and *Acidithiobacillus ferrooxidans* (*A. ferrooxidans*) and *Leptospirillum ferrooxidans* (*L. ferrooxidans*). The thermophile mixture are enriched from acid Mine Drainages (AMD) from several chalcopyrite mines in China, which mainly contains *A. caldus*, *L. ferriphilum*, *S. thermosulfidooxidans* and *S. acidophilus* (Zeng et al., 2010). The mesophiles and thermophile were pre-cultured in 9 K media (Silverman and Lundgren, 1959) at 30 and 48 °C, respectively. 10 g/L of chalcopyrite was used as energy source for culturing *A. ferrooxidans*, *L. ferrooxidans* and thermophilic mixture, and 10 g/L sulfur was used as energy source for culturing *A. thiooxidans*. The microorganisms were subcultured for at least three times before use in the leaching experiments.

Chalcopyrite and pyrite were obtained from Mt Lyell, Australia and North Dakota, USA, respectively. ICP studies indicate the chalcopyrite contained 33.7% Cu, 30.8 Fe% and 34.2% S, while the pyrite contained 45% Fe, 51% S, 0.47% Pb, 0.2% Zn, 0.18% Cu. (Tan and Chen, 2012). Both chalcopyrite and pyrite were ground into –200 mesh before use.

2.2. Leaching studies

2.2.1. Batch leaching

Leaching tests were carried out in 250 ml Erlenmeyer flasks. 150 ml of 9 K basic salt media (Silverman and Lundgren, 1959) containing 2.25 g/l Fe²⁺ as FeSO₄·7H₂O and 0.75 g/l Fe³⁺ as Fe₂(SO₄)₃·xH₂O (*x* is close to 7 based on calculation from the percentage of iron) was added in each flask. The amount of chalcopyrite and pyrite used in this study were 1.2 g and 3.6 g, respectively. The minerals were leached with one of three bacterial cultures: (1) pure *L. ferrooxidans*, (2) a mixed culture of *A. ferrooxidans*, *L. ferrooxidans* and *A. thiooxidans* with even initial proportion, and (3) thermophilic mixture. The initial bacteria concentration was 1.0 × 10⁷ cells/mL. The initial pH was adjusted to 1.8 and maintained between 1.7–1.9 by adding sulfuric acid or potassium hydroxide. The flasks were agitated at 170 rpm and kept at 30 °C for experiment with mesophiles and 48 °C for that with thermophiles. Chemical leaching, which refers to flasks with an absence of added microorganisms (abiotic leaching), was performed for comparison. Two flasks that contained chalcopyrite but not pyrite were also leached bacterially with mixed culture and chemically at 30 °C as control, respectively. All flasks received

the addition of ferrous sulfate and ferric sulfate. The solid samples were withdrawn at appropriate time interval and then washed with pH 1.8 sulfuric acid three times before further XRD and XANES tests.

2.3. Solution studies

Solution samples were periodically taken from flasks. The Solution tests were conducted by monitoring pH value, metal ion concentration (Fe²⁺, Fe_{tot} and Cu²⁺) and redox potentials. Eh was measured by a platinum electrode with Ag/AgCl reference electrode; total copper and iron were measured using inductively coupled plasma atomic emission spectrometry (ICP–OES) and ferrous concentration was determined by the 1,10-phenanthroline method (Tamura et al., 1974).

2.4. XRD study

Powder XRD data were collected in Bragg–Brentano geometry on finely ground samples using a Phillips X'Pert diffractometer fitted with a Co long-fine-focus tube operated at 40 kV and 40 mA, and a curved graphite post-diffraction monochromator. The beam path was defined using 1° divergence, 0.3 mm receiving and 1° scatter slits. Data were collected over the range 5 < 2θ < 80° in steps of 0.02° 2θ. The relative proportions of the crystalline phases in each sample was determined using Rietveld-based quantitative phase analysis (QPA) using TOPAS (Version 4.2.) (Bruker, 2009).

2.5. Fe K-edge XANES study

X-ray absorption spectra were recorded at X-ray Absorption Spectroscopy (XAS) beamline of Australian synchrotron. The current of the storage ring was operated in top-up mode with a beam current approximately 200 mA. The XAS beamline is a Wiggler beamline equipped with Si(111) monochromator, with an energy resolution ($\Delta E/E$) of 1.5 × 10⁻⁴. The beam size was 0.25 × 0.25 mm (fully focused) at the sample. The sample chamber was filled with argon to protect the sample from being oxidized when exposed to X-ray.

Data was acquired in fluorescence mode with a Passivated Implanted Planar Silicon (PIPS) detector. The near edge part of the Fe K-edge X-ray absorption spectra that has been used in this paper was collected using a step of 0.25 eV and Dwell time of 1 s in appropriate energy range. The spectra were then calibrated according to the spectra of Fe foil, and normalized according to the pre-edge and post-edge lines. Linear Combination (LC) fitting was then carried out with standard compounds to retrieve the composition of leached samples. All the calibration, normalization and fitting were processed in Athena software, ifeffit package (Newville, 2001).

3. Results and discussion

3.1. Solution studies

The change of copper concentration as a function of time during leaching of chalcopyrite concentrates with or without pyrite is shown in Fig. 1. The results show that copper leaching efficiency was obviously improved in galvanic assisted leaching. In chemical galvanic leaching, about 2.5 g/L copper was released in 31 days, which indicated more than 90% of the copper was leached out. Compared to the chemical leaching, the galvanic leaching rate with microorganisms at 30 °C was slower. In bioleaching at 48 °C, the impairing of galvanic effect by microorganisms was not seen as

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