



Cooperative bioleaching of chalcopyrite and silver-bearing tailing by mixed moderately thermophilic culture: An emphasis on the chalcopyrite dissolution with XPS and electrochemical analysis



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ABSTRACT

In this work, the interactions between one sample of silver-bearing tailing (223 g/t silver) and chalcopyrite during bioleaching by mixed moderately thermophilic culture were investigated. Bioleaching results showed that copper can be almost totally extracted from chalcopyrite as the result of addition of the silver-bearing tailing, and silver (Ag) extraction can be significantly improved with the addition of chalcopyrite. Hence, cooperative bioleaching process of chalcopyrite and the chosen silver-bearing tailing was feasible. Ag mainly occurred as silver sulfate (Ag_2SO_4), and further work of enhancing the Ag extraction and its recovery is currently in progress. The catalytic effect of the silver-bearing tailing on chalcopyrite dissolution was investigated mainly with X-ray photoelectron spectroscopy (XPS) and electrochemical analysis. Results proved that the presence of the silver-bearing tailing enhanced the oxidation rate of chalcopyrite and also eliminated the passivation effect of polysulfide, thus resulting in an extremely high copper extraction.

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1. Introduction

Bio-hydrometallurgy is considered as one of the most important mineral processing techniques in the future as it is simple, low cost and eco-friendly. Chalcopyrite is the most abundant and widespread copper-bearing mineral in the world, accounting for about 70% of the total copper resources (Pradhan et al., 2008; Li et al., 2013). However, chalcopyrite is recalcitrant to both chemical leaching and bio-hydrometallurgy mainly due to its low dissolution kinetics (Klauber, 2008; Li et al., 2013; Watling, 2013). Many efforts have been made to enhance the bioleaching process of chalcopyrite, and it has been reported that the bioleaching process of chalcopyrite can be accelerated by silver ion (Ag^+) due to the catalytic effect (Blázquez et al., 1999; Gomez et al., 1999; Sato et al., 2000; Yuehua et al., 2002; Wang et al., 2004; Cancho et al., 2007; Feng et al., 2013). However, the industrial application of silver-catalyzed technique is limited because of the high price of silver and it is technically difficult to recover the silver used for producing the catalytic effect. In addition, the mechanism of Ag^+

catalysis of chalcopyrite is complicated and is still in debate. The main suggested mechanisms can be classified into 3 groups. The first one was direct catalysis model, in which Ag^+ was considered to react with the chalcopyrite surface and then catalyze its dissolution (Miller and Portillo, 1979; Miller et al., 1981). The second one was called indirect catalysis model. In this model, chalcopyrite was initially reduced to chalcocite (Cu_2S) and hydrogen sulfide (H_2S), Ag^+ was considered to react with H_2S to form silver sulfide precipitate and cause the further formation of Cu_2S , thus promoting the chalcopyrite dissolution (Hiroyoshi et al., 2002). The third model mainly attributed the catalytic effect to the increase of electrical conductivity of chalcopyrite passive layer due to the addition of Ag^+ (Price and Warren, 1986; Nazari et al., 2012).

In fact, silver-bearing species, mainly including Ag, Ag_2S and Ag_2O , are commonly distributed in many kinds of sulfide ores. Silver-bearing ores with low grade of silver are difficult and uneconomic to be utilized. Hence, these silver-bearing minerals are abandoned as unvalued waste most of the time when the main metals were extracted and the abandoned tailings can also cause serious environmental pollution.

Therefore, in the present work, a sample of typical silver-bearing tailings was selected and the interactions between the silver-bearing tailing and chalcopyrite during bioleaching process were studied. And this work is potentially useful to provide

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an economic and effective technique to recover valuable metals from this kind of silver-bearing tailing and chalcopyrite.

2. Materials and methods

2.1. Samples

The pure chalcopyrite sample was achieved from the geological museum of Guangxi Province of China. X-ray diffraction (XRD) analysis showed that it was of extremely high purity (Fig. 1(a)). The chemical analysis showed that it contained 34.46% Cu, 31.53% Fe and 33.12% S (wt%). The silver-bearing tailing sample was zinc-leached residue obtained from one smelting plant in Chenzhou, Hunan Province, China. Chemical element analysis is shown in Table 1, which indicates that the main chemical elements of the tailings were Zn and Fe, and the content of silver was 223 g/t. Analysis of XRD (Fig. 1(b)) shows that the main mineralogical components of the silver-bearing tailing were zinc ferrite, sphalerite and aluminosilicate. Ore samples were ground and sieved to minus 0.074 mm before used for leaching experiments. All chemicals used were of analytical grade in the present work.

2.2. Bioleaching experiments

Moderately thermophilic culture containing *Acidithiobacillus caldus* (*A. caldus*) and *Leptospirillum ferriphilum* (*L. ferriphilum*) were obtained from the Key Lab of Bio-hydrometallurgy of Ministry of Education, Central South University, Changsha, China. *A. caldus* were sub-cultured into the basic culture medium supplemented with 10 g/L sulfur as the energy source. *L. ferriphilum* were sub-cultured into the basic culture medium supplemented with 44.7 g/L ferrous sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) as the energy source. The basic culture medium was consisted of $(\text{NH}_4)_2\text{SO}_4$ (3.0 g/L), $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ (0.5 g/L), K_2HPO_4 (0.5 g/L), KCl (0.1 g/L), $\text{Ca}(\text{NO}_3)_2$ (0.01 g/L). When microorganisms previously grew to their exponential growth phase with cell concentration of higher than 1.0×10^7 cells/mL, *A. caldus* and *L. ferriphilum* were mixed to be a mixed culture. 10 mL of the obtained mixed culture were inoculated into a 250-mL shake flask containing 90 mL of sterilized culture medium, 1 g of chalcopyrite and certain amount of silver-bearing tailing (0.5, 1.0, 1.5, 2.0, 3.0, 4.0 g). The shake flasks were placed into an orbital shaker at 170 rpm and 45 °C, pH was

maintained at 1.7 with sulfuric acid, and water lost through evaporation was supplemented periodically with deionized water. During the bioleaching process, the cupric ions concentrations were monitored by inductively coupled plasma-atomic emission spectrometer (ICP-AES) (America Baird Co. PS-6), the redox potentials of leaching solution were monitored by a Pt electrode with reference to a Ag/AgCl electrode (3.0 M KCl) (BPP-922), and the mineralogical compositions of solid samples were examined by X-ray diffraction (XRD) (DX-2700). Cell density was determined on a blood counting chamber with a bright field microscope (CX3, Olympus Corporation, Tokyo, Japan).

2.3. Electrochemical experiments

The carbon paste electroactive electrode (CPEE) was used as the working electrode, which contained 0.7 g of minerals (minus 0.038 mm), 0.1 g of solid paraffin and 0.2 g of graphite. A conventional three-electrode system consisting of a working electrode, two wired graphite rods as counter electrode and a Ag/AgCl (3.0 M KCl) electrode as reference electrode was used for electrochemical measurements (Princeton Model 283 Potentiostat, EG&G of Princeton Applied Research). The basic electrolyte was sterile culture medium whose pH was 1.7. Cyclic voltammetry tests were carried out at a sweep rate of 20 mV/s and the potentiostatic tests were all performed for the duration of 240 s. All the electrochemical measurements were carried out in N_2 atmosphere.

2.4. X-ray photoelectron spectroscopy (XPS) experiments

The samples leached for different days were filtered and rinsed with deionized water, then transferred to a vacuum drying oven (DZF-6050) to prevent further oxidation. Afterwards, dry samples were transferred to the spectrometer in an argon atmosphere before detection. X-ray photoelectron spectroscopy (XPS) detection was conducted with the model ESCALAB 250Xi. Spectra were recorded at constant pass energy of 20 eV and 0.1 eV/step using Al K_{α} X-ray source. Binding energies were referred to the C 1s level at 284.6 eV. XPS peaks were fitted by Thermo Advantage 5.52 software. The S 2p peaks occurred as doublets with S 2p_{3/2} and S 2p_{1/2} because of the spin-orbit splitting, and they were fitted using a 2:1 peak area ratio and 1.2 eV splitting for S 2p_{3/2} and S 2p_{1/2}. The Shirley method was used to obtain the background of spectra,

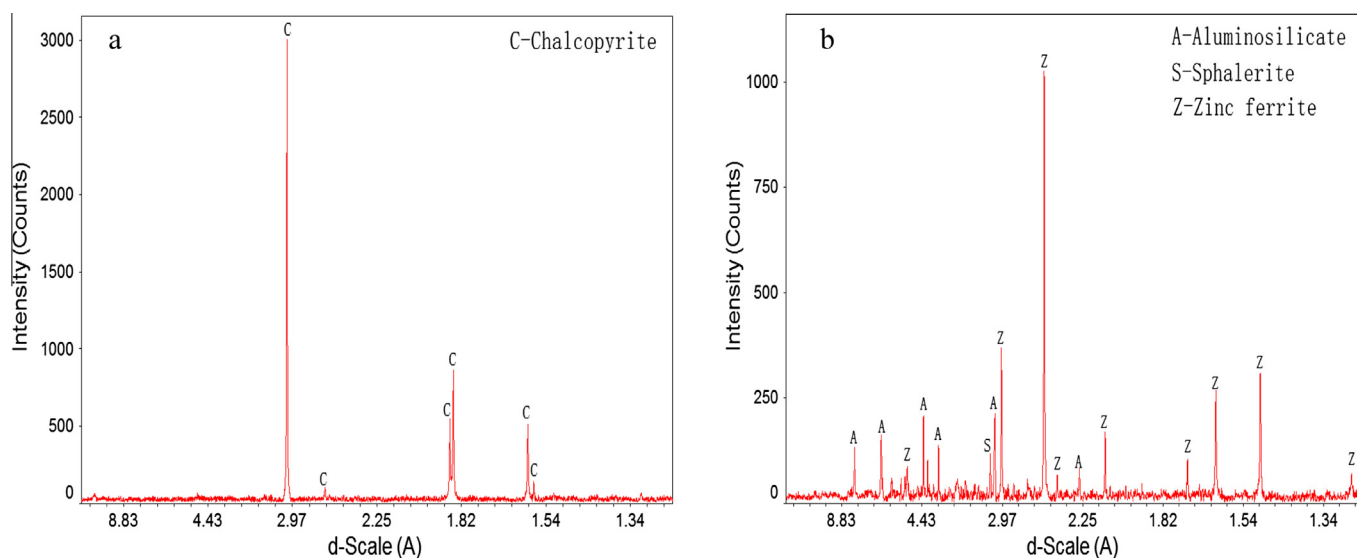


Fig. 1. X-ray diffraction (XRD) analysis of raw samples: (a) chalcopyrite, (b) silver-bearing tailing.

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