



Two step meso-acidophilic bioleaching of chalcopyrite containing ball mill spillage and removal of the surface passivation layer



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HIGHLIGHTS

- ▶ Two-step bioleaching method for chalcopyrite yielded higher copper recoveries.
- ▶ Carbonate based washed solution effectively removed the surface passivation layer.
- ▶ Analytical studies indicated further bioleaching of jarosite layer devoid residues.

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ABSTRACT

Meso-acidophilic bacterial leaching of ball mill spillage (containing chalcopyrite >80%) was carried out in an innovative two-step bioleaching method. The major drawback of meso-acidophilic bioleaching limiting industrial application is the passivation phenomenon over the ore surfaces in iron–sulfur rich environments. In the present study, we present a novel wash solution that efficiently removed the passivation layer. FTIR characterization of the bioleached sample indicated that the residues could be further leached to recover extra copper after wash solution application. XRD study indicated accumulation of sulfates (SO_4^-) of Na, K, Fe and oxy hydroxides of iron [$\text{FeO}(\text{OH})$] in the form of jarosite outlining the passivation layer. SEM, FESEM-EDS studies indicated severe corrosion effects of the wash solution on the passivation layer. Two step bioleaching of the ore sample yielded 32.6% copper in 68 days in the first interlude and post wash solution application yielded 10.8% additional copper.

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1. Introduction

Chalcopyrite (CuFeS_2), the economically important primary copper sulfide mineral accounts for about 70% of total copper resources in the world (Panda et al., 2012b and references therein). Concern has been raised particularly for processing of low-grade chalcopyrite due to the depletion of high-grade resources, its availability in abundance, metal content and several environmental issues while dumping. Over the years, the process of bacterial assisted leaching of metals (bioleaching) has developed as a strong alternative to hydro or pyro-metallurgical processing of low-grade ores (Qiu et al., 2011 and references therein). A number of acidophilic chemolithotrophic microorganisms either bacteria or archaea execute bioleaching as potential agents for metal dissolution.

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Amongst all the reported bioleaching or bio-mining microorganisms, *Acidithiobacillus ferrooxidans* (*A. ferrooxidans*) a meso-acidophilic γ -proteobacteria is the best-studied industrial microorganism (Bonney and Holmes, 2011; Rawlings, 2005).

Mixed thermo-acidophilic bacterial consortium has shown hopeful results towards improved copper dissolution (mostly concentrates) (Liang et al., 2012; Zeng et al., 2010; Zhou et al., 2009). However, certain disadvantages like greater sensitivity of their cell wall to higher pulp densities, lower metal tolerance capacity and energy intensive (thermal) process as compared to meso-acidophiles have limited its industrial application (Gomez et al., 1999). In contrast, a heap bioleaching setup (best suited for meso-acidophiles and processing of low-grade chalcopyrite) is favored by most of the industries today which operates under atmospheric conditions (Panda et al., 2012b and references therein). Consequently, the application of this technology to industrial sectors seems to be more beneficial for bioleaching of low-grade chalcopyrite in a simple and economic way (Panda et al., 2012c; Pradhan et al., 2008).

Chalcopyrite is well known to passivate at high solution potentials i.e. >600 mV vs. Ag/AgCl in presence of meso-acidophiles (Petersen and Dixon, 2002) which offers a major drawback to the bioleaching process. The formation of passivation layer (composed mainly of insoluble iron precipitates, elemental sulfur and jarosites) is very problematic as it slows down the rate of chalcopyrite dissolution by preventing the direct attack of bacteria (contact or direct mechanism) and bacterial generated Fe(III) (non-contact or indirect mechanism) to the mineral surface (Sand et al., 2001; Tributsch, 2001). It is assumed that this layer is less reactive than chalcopyrite and reduces the flow of electrons to and from the ore thereby slowing down the metal dissolution (Pradhan et al., 2008).

A number of studies have reported the formation of the passivation layer and several methods were proposed for improving chalcopyrite dissolution (Hu et al., 2002; Klauber et al., 2001; Run-lan et al., 2011; Smith et al., 2006; Zhu et al., 2011). Somewhat surprisingly very little or no work has been attempted towards overcoming the passivation problem which is prominent in all those cases. In our previous study on low grade chalcopyrite bioleaching in heaps, the cumulative rate of copper dissolution was largely affected by the passivation problem (Panda et al., 2012b). If a suitable method can be available for removal of this passivation layer, then heap bioleaching with improved performance can crop up as a full-blown technology in industrial sectors.

Little or no work has been attempted towards bioleaching of chalcopyrite containing ball mill spillages. In the present study, ball mill spillage of Malanjkhand copper project (MCP), India were bioleached in an innovative two step bioleaching method using BACTERIAL Film Oxidation (BACFOX) bioreactor technology (Panda et al., 2012b; Rao et al., 2008; Sukla et al., 2009). In addition, the study was also motivated to find a suitable solution to the passivation problem and allow improved performance of meso-acidophilic bioleaching (application to heap leaching technology) without involving a thermal setup. For this reason, we used a novel wash solution for removal of the passivation layer formed during mixed meso-acidophilic bioleaching of low-grade chalcopyrite. Furthermore, the bioleached residues and removed passivation layer products were analyzed and characterized using several analytical methods such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Scanning electron microscope (SEM) and Field emission scanning electron microscope (FE-SEM) coupled with Energy dispersive X-ray spectroscopy (EDS). Additionally, the mechanism for the action of wash solution towards removal of passivation layer is also proposed.

2. Methods

2.1. Ore sample

Ball mill spillage samples containing mostly chalcopyrite were obtained from the ball mill-grinding unit of MCP, India. Due to its poor metal content (0.26% Cu, 1.96% Fe, 0.48% S, 91.81% acid insoluble), it is considered as a waste for the industry and further dumped. Since, the cutoff grade for the concentrators at MCP is 0.45% Cu; this waste cannot be utilized through the present conventional methods adopted by MCP for metal recovery. Therefore, bioleaching was favored for the recovery of copper. Mineralogical characterization of the ball mill spillage has been reported in our previous work (Panda et al., 2012a).

2.2. Microorganism and culture conditions

In view of the fact that mixed acidophilic bacterial consortium is much efficient for bioleaching than a pure culture (Fu et al., 2008), a laboratory stock culture of mixed meso-acidophilic

microbial consortium consisting predominantly *A. ferrooxidans* along with *L. ferrooxidans* and *A. thiooxidans* enriched from Malanjkhand mine water samples was used as inoculum for the present experiment. The standard medium for growth of meso-acidophilic bacteria is 9K⁺ media containing (NH₄)₂SO₄ – 3 g/L, KH₂PO₄ – 0.5 g/L, MgSO₄·7H₂O – 0.5 g/L, KCl – 0.1 g/L and FeSO₄ – 44.2 g/L (Silverman and Lundgren, 1959). Repeated sub-culturing was done to ensure activation of the microbial strains. Tolerance to heavy metals is a unique biotechnological feature of these microorganisms. Therefore, the strains were gradually adapted to Cu (1 g/L) prior to bioleaching in columns (Rao et al., 2008). The adapted strain (Iron oxidation rate: 600 kg/m³) was then transferred to the BACFOX bioreactor and used during the entire leaching period.

2.3. Leaching setup and the two step bioleaching method

Column bioleaching is considered as the simulation of heap leaching (Dopson et al., 2008). For this reason, bioleaching of MCP ball mill spillage was carried out using percolation columns. The samples were initially crushed to obtain different particle sizes (range: –10 + 6 mm) and then loaded onto two different columns: one for the test experiment and other for the control. The columns had the following dimensions: height – 130 cm; internal diameter – 7.4 cm and operated at 10 kg scale under ambient temperature conditions (25–35 °C). Two BACFOX bioreactors containing bacterial lixiviant (composition: 0.2 g/L Fe(II), 5 g/L Fe(III), redox potential 680 mV, bacterial count of 7.2×10^7 cells/mL, pH – 1.8) were placed at the bottom of each column. Bacterial lixiviant from the BACFOX bioreactor was supplied through a sprinkler (at top of the columns) using a metering pump and the effluent solution was recirculated.

The advantages of BACFOX bioreactor with successful testing and implementation during a heap bioleaching operation has been previously reported (Panda et al., 2012b; Rao et al., 2008). Given that, the dissolution of CuFeS₂ demands increased oxidative activity by acidophilic microorganisms and optimal Fe(III) concentration (Sand et al., 2001; Tributsch, 2001); air sparging (compressed O₂ supply) was ensured initially to make the bacterial population active and provide the necessary ferric ion. Fe(II) iron (9 g/L) supplemented during the initial stages of bacterial activation was sufficient for the bacterial population to derive its energy and supply the necessary oxidizing Fe(III) ions.

For the control set, bioleaching experiments continued until no further copper dissolution was possible. In contrast, bioleaching was carried out in two steps for the test experiment. The first step involves the conventional bioleaching process to notice the copper recovery patterns and other microbial parameters (discussed in Section 3.1). In order to remove the surface passivation layer formed during meso-acidophilic bioleaching an innovative wash solution was applied to the percolation column of the test experiment (discussed in the next section). The resulting effect of wash solution application over the ore surfaces lead to the development of a second step/stage bioleaching (discussed in Section 3.2). The second step bioleaching involved the application of bacterial lixiviant to the leached residues of the first step to notice any further copper recovery after wash solution application. The results of the two-step bioleaching method in case of the test experiment were compared with that of the control. Total copper recovery was calculated cumulating the individual copper recoveries during the two steps of bioleaching.

2.4. Removal of the passivation layer: development of the innovative washing solution

In order to overcome the universal problem of passivation, we have developed a wash solution prepared by the chemical

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