



Biohydrometallurgical treatment of old flotation tailings of sulfide ores containing non-ferrous metals and gold



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ABSTRACT

Proposed in the work is mining waste treatment using a three-step biohydrometallurgical process. The process includes: (i) a step of acid leaching (with sulfuric acid solution) to remove highly soluble copper and zinc forms, (ii) a step of leaching with biogenic ferric iron for the oxidation of sulfide minerals containing copper and zinc, as well as for the partial oxidation of gold-bearing pyrite, and (iii) a biooxidation step for a more complete pyrite oxidation, providing the release of gold associated with it. The subject of the research was a sample of pyrite flotation tailings containing copper, 0.26%, zinc, 0.22%, and gold, 0.67 g/t. Investigated was an acid leaching of pyrite tailings under different conditions. The leaching allowed to recover up to 35–36% of copper and zinc after 10 min. Leaching of pyrite tailings with biogenic ferric iron was studied. The highest efficiency shown was for the process carried out at the temperature of 80 °C, the pulp density of 30%, the ferric iron concentration of 30 g/L in the leaching solution, and the process duration of 8 h. As a result, approximately 14% of the pyrite oxidized, and the total zinc and copper recovery increased up to 86% and 64%, respectively. The highest efficiency of tailings treatment was shown for the biooxidation during the 12-day three-step process, which allowed to oxidize 97% of pyrite and to recover 96% of gold by carbon-in-pulp cyanidation. The total copper and zinc recovery during the three-step process was 79% and 96%, respectively. A flowsheet was proposed for the three-step process.

1. Introduction

Associated with the production of large amounts of wastes, including flotation tailings is the treatment of sulfide ores of nonferrous metals (Lèbre et al., 2017). Billions of tons of such wastes have been accumulated in Russia and all over the world for the decades of operation of mining plants. Stockpiled annually are over 15 tons of gold with flotation tailings from mining enterprises of the Ural mining and metallurgical company (UMMC). A total of over 250 tons of gold and hundreds of thousands of tons of copper and zinc have accumulated at the UMMC enterprises. The average content of valuable components in the flotation tailings of the UMMC enterprises is as follows: copper, 0.3%; zinc, 0.5%; gold, 0.8 g/t (Shadrinova et al., 2005). Such wastes can cause problems due to their large amounts and possible impact on local ecosystems (Antonijević et al., 2008; Kefeni et al., 2017). Nowadays, pyrite wastes can be regarded as technogenic raw materials for nonferrous and precious metals. Experience in the utilization of wastes from mining and the metallurgical complex is evident of great opportunities for their efficient use as raw materials for the construction industry (Thomas et al., 2013; Lottermoser, 2011; Mohamed et al., 2002).

The involvement of copper-zinc ore flotation tailings into the production of building materials is limited due to the increased content of toxic metals, as well as the presence of sulfides (Argane et al., 2016). The development of efficient technologies for the treatment of flotation tailings will make it possible to produce additional commercial products and to reduce the negative impact on the environment at waste storage sites.

Spread worldwide is the biooxidation of sulfide ores and concentrates for recovery of gold, copper, nickel, cobalt and other metals (Petersen, 2016; Kaksonen et al., 2014; Brierley and Brierley, 2013; Watling, 2006; Riekkola-Vanhanen, 2013; Morin and d'Hugues, 2007; Brierley and Brierley, 2001; Rawlings et al., 2003). The biooxidation is based on the oxidative activity of acidophilic microorganisms, which allows to release metals from minerals into the solution during sulfide oxidation and to oxidize the sulfur produced (Johnson, 2014). The development of biohydrometallurgical methods for recovery of metals from flotation tailings of sulfide ores has attracted the interest of researchers (Gahan et al., 2012; Ye et al., 2017; Falagán et al., 2017; Hao et al., 2016; Ahmadi et al., 2015; Muravyov et al., 2014; Erüst et al., 2013; Kondrat'eva et al., 2012). Traditional processes of tank

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biooxidation are characterized by the low oxidation rate and the significant retention time of sulfidic ore materials in bioreactors (4–6 days). Approaches based on the separation of the process into a high-temperature chemical step of leaching with biogenic ferric iron and a step of ferric iron bio-regeneration using microorganisms were proposed to intensify sulfide oxidation (Fomchenko and Muravyov, 2017; Muravyov et al., 2015a; Patel et al., 2014; Carranza et al., 2004; Smalley and Davis, 2000). At the same time, conditions for the best process indices can be provided at every step. Recent studies have shown that a preliminary leaching of gold-bearing sulfide concentrates with the biosolution obtained by microbial ferrous iron oxidation promoted an improvement of the biooxidation of sulfide minerals (Fomchenko et al., 2016; Wang et al., 2016; Muravyov et al., 2015b; Liu et al., 2015; Muravyov and Bulaev, 2013; Fomchenko et al., 2010).

The goal of the work was to study processes of recovery of non-ferrous metals and gold from old pyrite flotation tailings of copper-zinc ores. The novelty of the work is an application of a combination of sulfuric acid leaching, leaching with biogenic ferric iron, and biooxidation for separate metal removal from oxidized and sulfidic phases.

2. Materials and methods

2.1. The subject of research

Mining residues of copper-zinc sulfidic ores of the Urals (Gay deposit, Orenburg Oblast, Russia), i.e., old flotation tailings from the concentrator, were used for the research.

Shown in Table 1 is the chemical and mineralogical composition of samples of old flotation tailings from the mining plant. Copper and zinc were both in sulfides (chalcopyrite and sphalerite) and in oxidized minerals. Major non-metallic minerals were quartz and gypsum. The granulometric composition of the sample was determined using a laser analyzer of the particle size and shape Analysette 22 COMPACT (FRITSCH, Germany). Shown in Table 2 is the data.

Table 1

The chemical and mineralogical composition of the tailings.

	Content (wt.%)
<i>Elements</i>	
S _{tot}	17.8
S ^D	1.3
S _s	14.5
S _{so4}	2.0
Fe _{tot}	17.4
Fe _s	11.4
Ca	1.51
Mg	0.67
Al	3.48
Si	20.0
Cu _{tot}	0.26
Cu _s	0.13
Zn	0.22
Au	0.67 g/t
Ag	7.06 g/t
<i>Minerals</i>	
Pyrite (FeS ₂)	26
Chalcopyrite (CuFeS ₂)	0.4
Sphalerite (ZnS)	< 0.3
Gypsum (CaSO ₄ ·2H ₂ O)	13
Jarosite (KFe ₃ (SO ₄) ₂ (OH) ₆)	1
Muscovite (K ₂ Al ₂ (AlSi ₃ O ₁₀)(OH,F) ₂)	4
Plagioclase (NaAlSi ₃ O ₈)	2
Clinochlore ((K,Mg) ₆ (SiAl) ₄ O ₁₀ (F,OH) ₂)	5
Quartz (SiO ₂)	41

The indices s and so4 indicate the content of the element in sulfidic or sulfate minerals, respectively.

Table 2

The granulometric composition of the tailings.

Size fraction (μm)	Content (wt.%)
+100	0
–100 + 80	0.2
–80 + 60	1.9
–60 + 40	7.5
–40 + 20	18.1
–20 + 10	16.2
–10 + 5	18.8
–5 + 1	32.3
–1	5.0

2.2. Acid leaching

Acid leaching was conducted in a reactor that contained 1.0 L of pulp. The stirring rate was 650 rpm, and the temperature was maintained by heat exchangers connected to a thermostat Julabo ED 5 (Germany). The leaching was conducted under batch conditions. The solid phase was loaded and mixed with an H₂SO₄-containing solution (solution of analytical grade H₂SO₄ in distilled water). The liquid was preheated to the desired temperature. During the study of the multiple acid leaching of the pyrite tailings, the pulp was centrifuged at 5000g for 5 min, and the solid phase was fed back to the reactor.

2.3. Generation of ferric iron and the ferric leaching of the tailings

A mesophilic enrichment culture obtained from the old pyrite tailings containing *Acidithiobacillus ferrooxidans*, *Leptospirillum* spp., *Sulfobacillus* spp., and *Ferroplasma* spp. was used as an inoculum to obtain a ferric iron-containing culture liquid for the subsequent ferric leaching of the tailings. The biooxidation of ferrous iron (FeSO₄·7H₂O) was conducted in a 5.0L vessel that contained 4.0 L of the liquid held at 30 °C with an aeration rate of 4 L/min. The liquid phase consisted of a 9 K medium (Silverman and Lundgren, 1959) and 10% (v/v) inoculum. The final Fe³⁺ concentration was 20–30 g/L with pH 1.5. This liquid (the microbial cells were not separated) was used as a lixiviant for the ferric leaching.

The parameters for leaching with the biogenic ferric iron of the tailings were selected based on the previous studies (Fomchenko and Muravyov, 2017; Fomchenko et al., 2010). Ferric leaching was conducted in a reactor with working volume 1.0 L. The stirring rate was 650 rpm, and the temperature was maintained by heat exchangers connected to a thermostat Julabo ED 5 (Germany). The leaching with biogenic ferric iron was conducted under batch conditions. The solid phase was loaded and mixed with the biologically generated Fe³⁺-containing solution. The liquid was preheated to the desired temperature. The pH of the pulp was continuously monitored, and H₂SO₄ or Ca(OH)₂ were added when necessary to maintain the pH value. The duration of ferric leaching was four h. During the study of the multiple ferric leaching of the pyrite tailings, the pulp was centrifuged at 5000g for 5 min, and the solid phase was reloaded to the reactor.

2.4. Biooxidation experiments

The investigation of the biooxidation of the tailings was conducted using two series of experiments: (a) a two-step process (acid leaching + biooxidation) and (b) a three-step process (acid leaching + leaching with biogenic ferric iron + biooxidation). Experiments on the biooxidation of the old flotation tailings were conducted in two reactors that were connected in series under continuous conditions with intermittent feeding and with effluent removal. Biooxidation was conducted in bioreactors that contained 1.0 L of pulp. A salt solution of the 9 K medium without Fe²⁺ was used as the liquid phase. A mesophilic microbial consortium containing *At. caldus* INMI-

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