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The processing of a low-grade gold refractory sulfide ore by flotation-biooxidation-cyanidation route

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ABSTRACT

By depletion of high-grade and easy-to-treat gold ores and the increase in gold price, the extraction of gold from low-grade and refractory ores, such as sulfide ores has been given attention. Biooxidation is one of the efficient, relatively low-cost and environmentally friendly processes to enhance gold extraction by releasing fine gold encapsulated in sulfide phases. So, in this research, the efficiency of concentrate biooxidation-flotation system in increasing gold recovery from sulfide-refractory low-grade ores was investigated. Biooxidation experiments with mesophilic and moderate thermophilic microorganisms were conducted on the flotation concentrate. Finally, cyanidation experiments were conducted on the flotation concentrate, and biooxidized flotation concentrate. Gold recovery from non-biooxidized flotation concentrate was 63.59%, while it increased to 80.21% after biooxidation with mesophilic microorganisms and to 79.84% after biooxidation with moderate thermophilic microorganisms.

Keywords: *Biooxidation, Concentrate, Microorganism.*

1. Introduction

Currently, biotechnology has attracted much attention for gold recovery from sulfidic refractory gold ores (Natal'ya, Muravyov, & Kondrat'eva, 2010). In refractory gold ores, the gold particles are very fine and may be locked within particle boundaries or the structure of sulfide minerals, such as pyrite and arsenopyrite (Wang et al., 2020). Direct cyanidation method is not effective for extracting gold from refractory sulfide ores; therefore, a pretreatment process, such as roasting, pressure oxidation or biooxidation should be considered to liberate encapsulated gold from the sulfide minerals. Compared to other processes, biooxidation of sulfide refractory gold ores has several advantages, including low investment cost and environmental friendliness (Wu, 2018) (N. V. Fomchenko, Kondrat'eva, & Muravyov, 2016). In this process, metal sulfides are oxidized by iron- and sulfur-oxidizing microorganisms, forming soluble metal sulfates and sulfuric acid. Pyrite and arsenopyrite are well-known minerals that are easily biooxidized (Olson, 1994). During this process, microorganisms are catalytically used for oxidizing pyrite and arsenopyrite to release and expose gold for more processing (N. V. Fomchenko et al., 2016). In general, biooxidation for processing of refractory gold concentrate involves a typical flowsheet that includes crushing, milling, producing concentrate from the flotation process, followed by cyanidation and recovery of gold from biooxidized residues (Miller & Brown, 2016).

The operating temperature of a biooxidation process is very important because each microbial culture has its own optimal temperature for growth and oxidation (Muravyov, 2019). The bioleaching process of sulfide minerals involves two chemical and biological stages (N. Fomchenko et al., 2017). Nouhi et al. (2025) used acid drainage for the biooxidation of a high-grade refractory sulfide gold ore, and gold recovery increased from 73% (without biooxidation) to 99% (in the biooxidized residue). Beiranvand et al. (Beiranvand et al., 2023) investigated the effect of mechanical activation on the oxidation and extraction of gold from a high-grade flotation concentrate using mesophilic and moderate thermophilic microorganisms. Gold recovery from the non-mechanically activated and non-biooxidized concentrate was 83.9%, while after biooxidation this value reached 98.8%. Also, gold recovery from the activated but non-oxidized concentrate was 77.3%, which reached 97.6% after biooxidation.

By decreasing easy-to-treat gold ores, developing technologies and an increase in gold price, the recoverable grade of gold ores has decreased gradually. The development and optimization of biooxidation technology for increasing the recovery of gold from lean grade refractory sulfide wastes have not been previously well investigated. Flotation followed by biooxidation and cyanidation processesis one of the potential routes to treat such low-grade and refractory ores and tailings. So, in this research the ability of this strategy was evaluated to extract gold from the low-grade stockpiles of Mouteh gold mine.

2. Material and Methods

2.1. Material

Samples were taken from low-grade refractory sulfide gold ore stockpiles of Mouteh gold mine (Isfahan province, Iran). At first, the sample was homogenized. Crushing was performed in two stages to achieve the desired particle size. To achieve a particle size of -75 µm, the samples were ground using a rod mill with a ratio of 1:8 (1 kg samples: 8 kg rod) after crushing. The representative samples were used to analyze chemical and mineralogical parameters by XRD, XRF, and Fire Assay methods.

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2.2. Flotation

Flotation tests were conducted using a Denver flotation machine with a cell volume of 2200 ml to provide the required concentrate for biooxidation experiments. In flotation tests, 150 g/t potassium amyl xanthate (PAX) was used as the main collector, 30 g/t dithiophosphate as the secondary collector, and 30 g/t methyl isobutyl carbonyl (MIBC) as the frother agent. The tests were carried out with a solid content of 25%. The water used for the experiments was local water from the Mouteh gold mine. After conducting tests, the flotation concentrates and tailings were filtered and dried for gold assay and subsequent experiments.

2.3. Biooxidation experiments

Biooxidation experiments of the concentrate were carried out in 500 ml Erlenmeyer were located in shaking incubator. The volume of pulp was 200 ml that consisted of 10% solid content, 20% bacterial solution, and 155 ml culture media (9K, Norris). The Norris culture media consists of 0.4g $(NH_4)_2SO_4$, 0.5g $MgSO_4$ and 0.4g $Ca(PO_4H_2)_2$ (Silverman & Lundgren, 1959). Culture media (9K, Norris) were prepared with distilled water. The residence time of the test was 28 days. The initial pH of the solution was adjusted to 1.6 using 2M sulfuric acid. Oxidation/reduction potential and pH of the solutions were monitored daily and maintained in the range of 1.6-1.8 using 2M sulfuric acid or lime (20% solid). During the experiment, the evaporated solution was compensated for with distilled water. At the end of the experiment, the contents of each Erlenmeyer were filtered separately, the remaining solution for determination of iron content was maintained, and the remaining solid content was washed and dried for performing the subsequent experiments. After these experiments, the effect of processing water of Mouteh gold mine on biooxidation was studied.

2.4. Cyanidation experiments

Non-biooxidized flotation concentrate and biooxidized concentrate with mesophilic and moderate thermophilic microorganisms were subjected to cyanidation testing for 48 hours for gold recovery. Experiments were conducted with a solid content of 25% and 4 g/l NaCN (NaCN 50%) by the mechanical stirring method. Lime (15% solid content) was used to adjust the pH in the range of 10.5-10.7. At the end of the experiment, the final solution for gold and cyanide analysis and the final solid were washed with hot water, dried, and sent for gold analysis. The initial cyanide concentration was 1410 ppm, which after 48 hours of cyanide leaching, reached 450, 240, and 330 ppm for the flotation concentrate, mesophilic biooxidized concentrate, and moderate thermophilic biooxidized concentrate, respectively.

3. Results and discussion

3.1. Flotation

Flotation experiments were conducted with a solids content of 25% using PAX as the collector and MIBC as the frother reagent, respectively. After conducting flotation tests, the gold content in the initial sample, flotation concentrate, and flotation tailings was determined to be 0.5, 3, and 0.2 g/t, respectively, using fire assay analysis. Gold recovery in these tests was 64%.

The primary feed, concentrate and tailing of flotation tests were characterized. The results of the elemental analysis of the flotation concentrate are shown in table 1.

The XRD patterns of primary samples showed that the main mineral phases were quartz, albite, chlorite and muscovite. The XRD diagram of the flotation concentrate is shown inFigure 1. According to the SEM analysis of flotation concentrate (Figure 2), the irregular particles with sharp edges and varying size are crushed pyrite, resulting from sample preparation. According to the EDS diagram (Figure 3), the peaks related to iron and sulfur are clearly visible, which emphasizes that the main phase in this region could be pyrite.

Table 1. The XRF analysis of the flotation concentrate.

element	SiO_2	BaO	CaO	Fe_2O_3	K_2O	MgO	MnO	P	S	TiO_2
Content (%)	54.60	<0.05	1.69	13.65	1.64	2.13	<0.05	<0.05	8.60	0.73

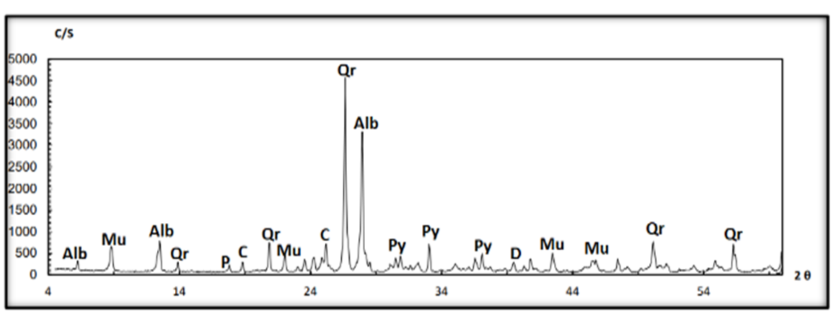


Figure 1. The XRD diagram of the flotation concentrate (Alb: Albite, Mu: Muscovite, Qr: Quartz, P: Potassium Feldspar, C: Chlorite, Py: Pyrite, and D: Dolomite).

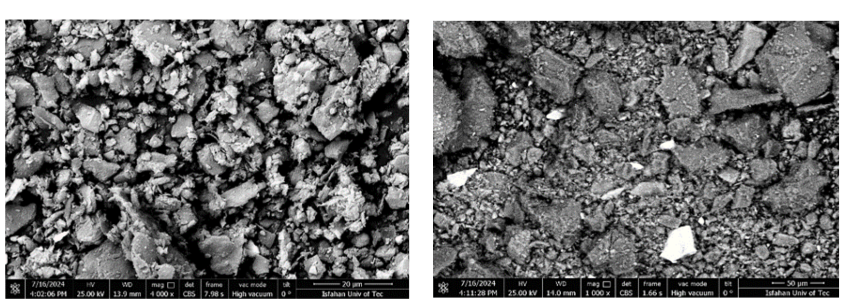


Figure 2. The Scanning electron microscope (SEM) analysis of flotation concentrate.

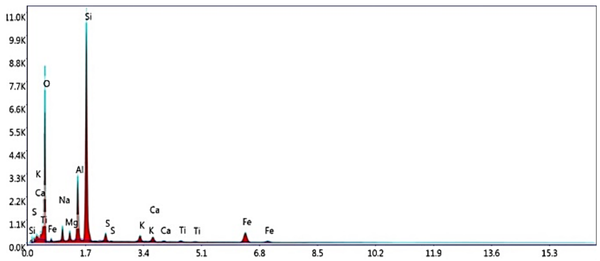


Figure 3. The EDS analysis of flotation concentrate.

3.2. Biooxidation

Concentrate biooxidation experiments were carried out with mesophilic and moderate thermophilic microorganisms and culture media prepared with distilled water. As can be seen from Figure 4(a), For biooxidation with mesophilic microorganisms in Norris culture media, the pH initially increased from 1.5 to 2.4 within the first day, then decreased to 1.7-1.8 after 16 days. The redox potential increased from about 408 mV in first day to 526 mV on the 9th day. The redox potential at the start of the experiment was higher than its value on the first day, which is due to the presence of ferric ions in the bacterial solution. On the first day of the process, as a result of dissolution, ferric iron ions are reduced to ferrous ions, which reduces the redox potential of the solution. After one day, the redox potential of the solution began to increase. The initial total iron concentration in sulfide flotation concentrate before biooxidation was 13.65% (w/w), which after biooxidation with mesophilic microorganisms in Norris culture media, reached 1.613 g/l on day 14 and 1.838 g/l on day 28.

For biooxidation with mesophilic microorganisms in 9K culture media (Figure 4(b)), the pH increased from 1.6 to about 2 within 1 day, and decreased to 1.6-1.7 after 16 days. The redox potential was 424 mV on the first day of the experiment which increased to 600 mV on the 14th day. In this study, it was found that the lag phase of mesophilic microorganisms in 9K culture media was longer than their lag phase in Norris culture media. The total iron concentration after biooxidation with mesophilic microorganisms in 9K culture media, reached 1.849 g/l on day 14 and 2.103 g/l on day 28.

Biooxidation experiments of flotation concentrate were conducted using moderate thermophilic microorganisms in two culture media, Norris and 9K, under similar conditions. For biooxidation with moderate thermophilic microorganisms in Norris's culture media, as can be seen from Figure 4(c), the pH increased from 1.6 to about 2 after 1 day. Then after 16 days, it decreased to 1.6-1.7. The redox potential was 442 mV on the first day of the experiment, and began to increase on the 3rd day of the experiment. This increase continued until the 14th day, reaching to 520 mV. The total iron concentration after biooxidation with moderate thermophilic microorganisms in Norris culture media, reached 1.798 g/l on day 14 and 2.008 g/l on day 28.

To investigate the effect of 9K culture media on the biooxidation with moderate thermophilic microorganisms, the biooxidation experiment was performed simultaneously and under the same conditions as in Norris medium. As can be seen from Figure 4(d), the redox potential of the experiment on the first day was 434 mV, which increased to 585 mV by the 11th day. On the 3rd day of the experiment, the redox potential of the solution began to increase, and this increase continued until the 11th day. The pH increased from 1.6-2.2 after 1 day, and decreased to 1.5-1.6 after 16 days. By comparing the effect of culture media type on biooxidation of flotation concentrate with moderate thermophilic microorganisms, it was found that by conducting experiments in 9K culture media, microorganisms had greater growth and activity than in Norris culture media, but their lag phase was the same in both types of culture medium. The total iron concentration after biooxidation with moderate thermophilic microorganisms in Norris culture media, reached 2.116 g/l on day 14 and 2.241 g/l on day 28.

To investigate the effect of water type on concentrate biooxidation, experiments were carried out with mesophilic and moderate thermophilic microorganisms and culture media prepared with processing water of Mouteh gold mine. During the biooxidation with mesophilic microorganisms in Norris culture medium prepared with mine water (Figure 5(a)), the pH increased from 2 to 2.5 within 1 day. Its value decreased to 1.5-1.6 after 9 days. The redox potential was 395 mV on the first day of the experiment. Its value increased to 589 mV after 16 days. The total iron concentration after biooxidation with mesophilic microorganisms in Norris culture media, reached 3.326 g/l on day 14 and 4.767 g/l on day 28.

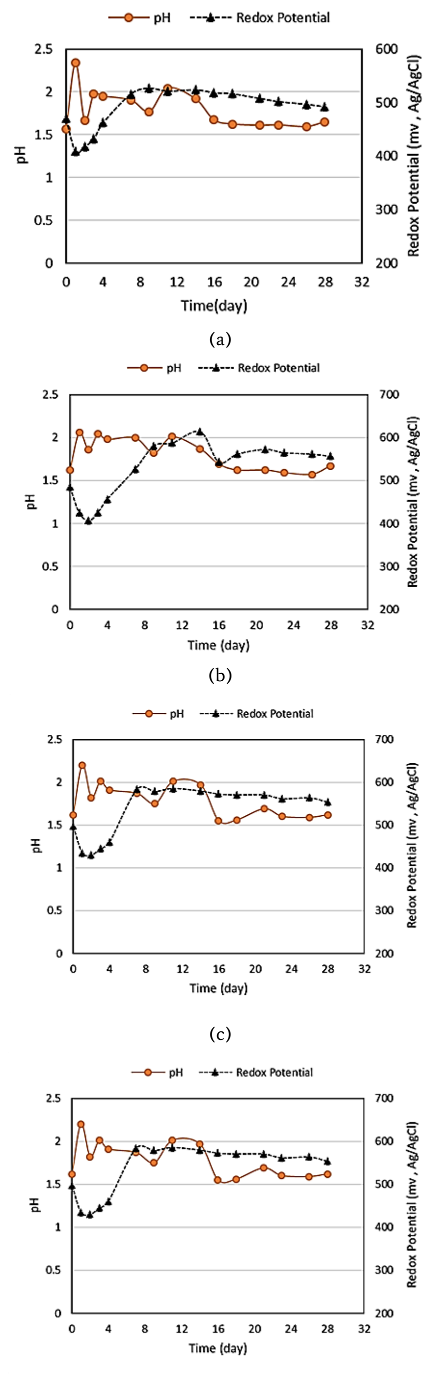


Figure 4. The pH and ORP of microorganisms in Norris and 9K culture media prepared with distilled water in biooxidation of flotation concentrate as a function of time.

For biooxidation with mesophilic microorganisms in 9K culture media, as can be seen from Figure 5(b), the redox potential increased from about 397 mV on the first day to about 602 mV on the 14th day. On the 2nd day of the experiment, the redox potential began to increase, this increase continued until the 14th day. The pH increased from about 1.5 to 2.1 after 1 day, and decreased to 1.5-1.6 after 9 days. The total iron concentration after biooxidation with mesophilic microorganisms in 9K culture media, reached 4.213 g/l on day 14 and 5.554 g/l on day 28.

The biooxidation experiments of flotation concentrate were also repeated with moderate thermophilic microorganisms to investigate the type of water used in the preparation of the culture media. During biooxidation with moderate thermophilic microorganisms in Norris media (Figure 5 (c)), the pH increased from 1.5 to 1.7 within 1 day. Its value decreased to 1.5-1.6 after 9 days. The redox potential was 404 mV on the first day. On the 2nd day of the experiment, the redox potential began to increase, this increase continued until the last day of the experiment (day 28) and increased to 603 mV. The total iron concentration after biooxidation with moderate microorganisms in Norris culture media, reached 2.882 g/l on day 14 and 3.548 g/l on day 28.

For biooxidation with moderate thermophilic microorganisms in 9K culture media, as can be seen from Figure 5 (d), the redox potential was 389 mV on the first day of the experiment. Its value increased to 579 mV after 12 days. The pH increased from 1.7 to about 2.7 after 1 day, and decreased to 1.6-1.7 after 9 days. The total iron concentration after biooxidation with moderate thermophilic microorganisms in 9K culture media, reached 3.326 g/l on day 14 and 4.626 g/l on day 28.

3.3. Cyanidation

After biooxidation experiments, the washed solid residue was subjected to cyanidation experiments. Experiments were conducted on non-biooxidized and biooxidized flotation concentrates with mesophilic and moderate thermophilic microorganisms. For further investigation, the biooxidized concentrate with mesophilic and moderate thermophilic microorganisms was cyanidated separately. Gold recovery from non-biooxidized flotation concentrate was 63.59%, while this value reached 80.21% after biooxidation with mesophilic microorganisms and 79.84% after biooxidation with moderate thermophilic microorganisms. As can be seen, biooxidation increased the gold recovery from the concentrate.

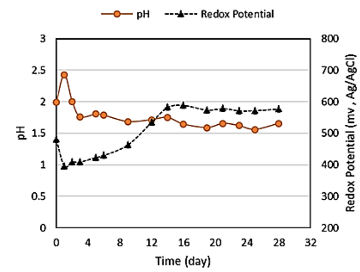
In the biooxidation of flotation concentrate using mesophilic and moderate thermophilic microorganisms, microorganisms grew and were more active in the 9K culture media prepared with mine water; also, the rate of iron dissolution was higher using mesophiles, so gold recovery from biooxidized concentrate was higher with mesophiles, which indicates the better performance of mesophiles in this research. Janson investigated solid culture media for the isolation and enumeration of acidophilic bacteria, so observed that the growth of mesophilic bacteria was supported by the solid culture media that included ferrous iron (Johnson, 1995). Given the low gold recovery in the flotation experiments conducted, it is suggested that researchers conduct flotation experiments under optimal conditions and then perform the other steps. Figure 6 shows the used flowsheet for this research.

4. Conclusion

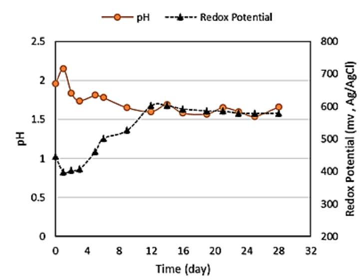
The efficiency of the flotation-biooxidation-cyanidation route to extract gold from the low-grade stockpiles of Mouteh gold mine was investigated and the following results were obtained: Flotation experiments were conducted to prepare the required concentrate for the biooxidation process. The gold grade for the primary feed, concentrate, and flotation tailings were 0.5, 3, and 0.2, respectively. Gold recovery in flotation experiments was also 64%.

Results showed that both mesophillic and moderately thermophillic microorganisms had a good ability to oxidize pyrite at various cultures (nutrient media and water types) especially in local water and 9K nutrient medium. Gold recovery by cyanidation from the non-biooxidized flotation concentrate was 63.59%, which increased to 80.21% and 79.84% after biooxidation with mesophilic and moderate thermophilic microorganisms, respectively. This increase was related to the breakdown the sulfide network as a result of biooxidation.

It was found that flotation followed by biooxidation and conventional cyanidation could be an suitable processing route to extract gold from low grade and refractory gold ores and mine wastes.



(a)



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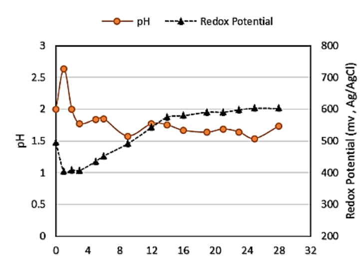
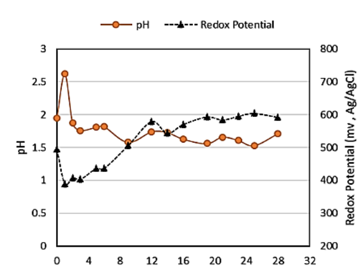


(c)



(d)

Figure 5. The pH and ORP of microorganisms in Norris and 9K culture media prepared with local mine water in biooxidation of flotation concentrate as a function of time.

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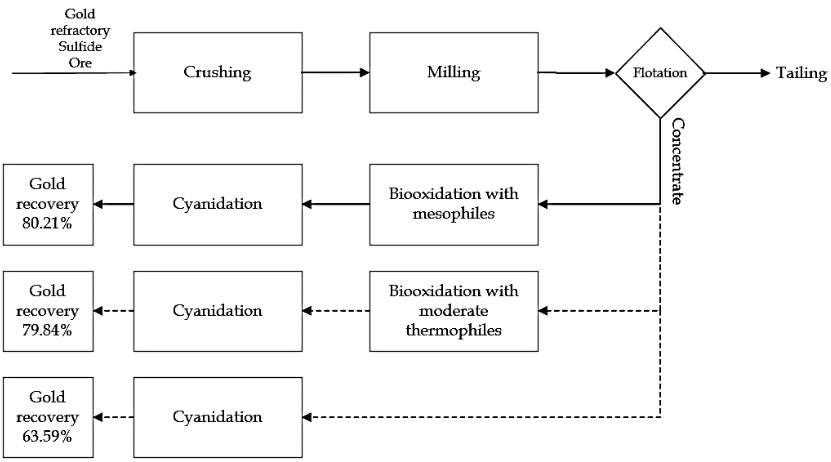

Figure 6. The flowsheet of flotation-biooxidation-cyanidation of gold refractory sulfide ore.

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