Bioleaching: Validation of the Extraction of Precious Metals Through Selective Recovery of Iron, Copper and Zinc

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Abstract: In Ecuador, large gold deposits were discovered in the areas of Nambija and Ponce Enríquez, but they contain metal sulfides that, when exposed to the action of air and water, intervene in a series of physical, chemical and biological phenomena. The oxidation of sulfides to sulfates occurs by the catalytic action of bacteria, in addition to the production of sulfuric acid that dissolves heavy metals such as iron, copper and zinc; a process known as bioleaching. These solutions, with a high level of acidity, are carried away by water currents or runoff, becoming a great contaminant of water and soil of the surrounding sector. Acid mine drains are one of the main problems of environmental pollution; the mining deposits are located in areas of great biodiversity. In these areas there are births of water used for human consumption, agriculture and mining work; the mismanagement of tailings, tailings and sands that are discharged into rivers and streams generate serious environmental damage. The objective of the work is to use selective precipitation to recover iron, copper and zinc from acid leaching solutions produced by bioleaching during the extraction of precious metals at the laboratory level and from acid drainage of natural mine, to comply with environmental regulations regarding the discharge of effluents and reduce the effect of environmental pollution produced by acid mine drains. This investigation presents the conditions for a successful individual recovery of the main base metals contained in a bioleaching solution with high copper, zinc, and iron concentrations by pH-based selective precipitation. Tests were made with standard solutions of known concentrations of copper, iron, lead, and zinc and by titration the concentrations were checked, which allowed to validate the volumetric titration method. The selective precipitation of heavy metals was carried out in three phases using real acid main drainage and bioleaching solutions generated at the laboratory. The first phase in a pH range of 2 to 4 to recover iron; the second phase in a pH range of 4 to 6 to recover copper; and the third phase in a pH range of 6 to 10 to recover zinc. The selective precipitation allowed the heavy metals to be completely removed from the solution or to achieve concentrations below the maximum allowable limit to be discharged to a body of water or public sewer. Validation of SOLBIO 2 and Orenas bioleaching solutions was performed.

Keywords: Bioleaching, Selective Recovery, Effluent Treatment

1. Introduction

Mining is the extractive activity of selectively obtaining rocks, mineral, and fuels from the Earth's crust to work, process, and convert them into goods of economic value [1]. Sustainable mining involves extracting minerals from the Earth's crust by limiting to the maximum the environmental damage they may cause in the area; and when the mine material runs out, carry out activities for its recovery. It should contribute to local development, increase employment, allow the participation of indigenous communities, and contribute to diversifying regional economies thus compensating for the definitive loss of non-renewable material from mining [2].

Ecuador has large gold mining deposits in the areas of Nambija and Ponce Enríquez, in addition to traditional mining activities in areas of Zaruma and Portovelo [3]. From
the exploitation of these deposits, the artisanal and industrial mining activity is carried out in the south of the country, generating large investments, national and foreign, as well as changes in mining legislation [4-6]; see Figure 1.

But any mining or production process generates liquid and solid waste. The main focal stations for acid solutions in mining operations are underground mine drains, runoff in opencast mining and leachates from mining and waste [7]. These acidic solutions, with an important heavy metal content in solution, are called "acid mine drainage" (AMD) [8]. AMD contains heavy metals such as lead, mercury, iron, copper, and zinc, toxic in small concentrations, and tend to bioaccumulation [9].

In mining districts, gold deposits also contain metal sulfides such as pyrite, chalcopyrite, arsenopyrite, galena and sphalerite, and being exposed to the action of air and water are involved in a series of physical, chemical, and biological phenomena. Thus the oxidation of sulfides to sulfates by the catalyst action of bacteria such as Thiobacillus Ferrooxidans, T. Thiooxidans, T. Sulfooxidans, T. Thioparus, among others, occurs in addition to the production of sulfuric acid that dissolves heavy metals such as iron, copper and zinc; so-called bioleaching. These solutions, with a high level of acidity, pH around 2, are dragged by streams of water or runoff, being a powerful pollutant of water and soil [10].

Figure 1. Vein of metal sulphides and precious metals, Bella Rica Mining Cooperative (Ponce Enríquez mining district, Azuay province, Ecuador).

The objective of the research, using selective precipitation, was to recover iron, copper, and zinc from acidic solutions produced by bioleaching during the extraction of precious metals at the laboratory level and natural acid mine drainage, enabling compliance with environmental regulations regarding effluent discharge and reducing the effect of environmental contamination generated by acid mine drains [11-13].

The research is a continuation of [13]. The results obtained from the selective extraction of iron, copper, and zinc, in bioleaching solutions replicated to those of [13], allowed to validate the proposed experimental procedure from the confirmation of the reproducibility of the results.

Like in [13], the results were obtained using volumetric analysis methods for the determination of Fe$^{2+}$, Fe$^{3+}$, Total Fe, SO$_4^{2-}$, Cu$^{2+}$ and Zn$^{2+}$ for field use [14]. Bioleaching solutions with similar characteristics to natural mine acid drainage were prepared for selective precipitation testing of heavy metals in solutions [13, 15]. Pure metal hydroxides were precipitated from natural bioleaching solutions generated under controlled processes in the laboratory, with potential industrial application.

2. Methodology

The methods for processing minerals are specific, depend on the physical and chemical characteristics of the mineral to be treated, in addition to the location and geological characteristics of the mine and the quantity of the mineral to be processed [16]. Prior to the processing of the ore, it goes through previous phases such as: treatment and storage, crushing, grinding, sorting, and concentration.

The concentration aims to produce a high-grade concentrate in small feeding fractions in order to produce a cheaper treatment or also to reject the portion of ore that does not contain valuable metal to reduce the volume of the ore that passes to the next process. In addition, it can be used to reject a portion of the ore that could impair the next process in the extraction of valuable metal. Concentration processes can be gravimetric concentration, flotation, amalgamation, electrostatic separation, and magnetic separation [17]; see Figure 2.

Figure 2. Floating foam containing precious metal, process used in the Sominur Company (Machala, El Oro province, Ecuador).

2.1. Bioleaching

Obtaining precious metal compounds from the catalysis of microorganisms that act on the dissolution of certain mineral ores, for recovery and purification. During the process, the microbial community for its survival uses the mineral as fuel, performing useful work that generates the release of metals and heat [18].

In 1954 Bryner experimented with acidophilic bacteria of the genus Thiobacillus that oxidize pyrite and copper sulfides. Some research has led to a variety of bacteria capable of withstanding extreme acidity conditions and high concentrations of heavy metals. Based on strict environmental regulations in several countries, bioleaching is considered an excellent alternative to processing refractory
ores where strongly oxidizing processes are required, being a viable option because of its low level of pollution and minimal energy expenditure.

Thiobacillus ferrooxidans is currently used to obtain copper, iron, zinc, and arsenic, among others.

On a commercial scale, the remediation of materials contaminated with heavy metals by bioleaching is carried out by irrigation on batteries and by agitated tank. The first method consists of percolation of leaching solution through crushed or concentrated minerals; and the second method consists of the continuous operation of agitated reactors with a high aeration rate [19].

### 2.2. Treatment of Solutions Generated During Bioleaching

One researched process to recover heavy metals from effluents produced in metal extraction is the recovery of iron as jarosite, MFe$_3$(SO$_4$)$_2$(OH)$_6$, leaching solutions, according to reaction (1); temperature-dependent process, pH and contact time. The iron is removed by precipitation of hydroxide by adding lime or limestone to increase the pH to approximately 3 [11, 20, 21]:

\[
3\text{Fe}_2\text{SO}_4 + 6\text{H}_2\text{O} \rightarrow 2\text{MFe}_3(\text{SO}_4)_2(\text{OH})_6 + 6\text{H}_2\text{SO}_4 \quad (1)
\]

M represents any of the ions: Na$^+$, NH$_4^+$, H$_3$O$^+$, Li$^+$, $\frac{1}{2}$Pb$^{2+}$.

Jarosite precipitation produces iron precipitates with relatively good characteristics of sedimentation, filtering and washing. However, the weakness of this process is the co-precipitation of several metal ions (Cu, Zn, Co, Ni, Mn, In, Ga, Ge, Al) in the solution. Co-precipitation increases as the pH of the solution increases and decreases if the iron concentration increases [11, 12, 20, 21].

Biological mechanisms and chemical removal combined with biologicals have been studied for the removal of acid drainage iron from mine. These treatment systems help lower chemical costs and improve the characteristics of sludge produced for better final disposal. Treatments with microorganisms, capable of oxidizing iron, include aeration gaps, agitated tanks, packed bioreactors, and biological rotary connectors. Precipitation of iron as jarosite in biological reactors occurs at pH between 2.5 and 3.5 and its kinetics increases as the iron concentration in the treated sample is higher [21].

Solubility product (Ks). It is the product of molar concentrations of dissolved ions. Ks is the balance constant for the balance that is established between a solute or solid and its ions in a saturated dissolution. For a substance to precipitate, the concentration of the concretion of the dissolved ions elevated to their respective powers (stochiometric coefficients) must be greater than the value of the solubility product [22].

Precipitation of hydroxides. Hydroxide precipitates depending on the concentration of OH ions. The more soluble hydroxide is, the more OH ion concentration necessary to exceed your solubility product; that is, the lower the pH value must be for precipitation to begin [23].

The solubility product allows to calculate the pH value at which the precipitation of a hydroxide begins and ends. For copper hydroxide, iron hydroxide and zinc hydroxide, after dissociating, Ks and hydroxide concentration [OH] [13] are calculated:

\[
K_s = [\text{Cu}^{2+}] [\text{OH}]^2 = 2.2 \times 10^{-20}, [\text{OH}] = \sqrt[2]{\frac{K_s}{[\text{Cu}^{2+}]}} \quad (2)
\]

\[
K_s = [\text{Fe}^{3+}] [\text{OH}]^3 = 4.0 \times 10^{-38}, [\text{OH}] = \sqrt[3]{\frac{K_s}{[\text{Fe}^{3+}]}} \quad (3)
\]

\[
K_s = [\text{Zn}^{2+}] [\text{OH}]^2 = 1.8 \times 10^{-14}, [\text{OH}] = \sqrt[2]{\frac{K_s}{[\text{Zn}^{2+}]}} \quad (4)
\]

### 2.3. Experimental Procedure

Volumetric methods were validated as analytical methods for the determination of Fe$^{3+}$, Fe$^{2+}$, total Fe, SO$_4^{2-}$, Cu$^{2+}$ and Zn$^{2+}$ [13, 14], and tests were performed to show that there is no interference between the ions to be analyzed.

Samples of gold ore and acid mine drainage (AMD) were collected in the mining district of Ponce Enriquez, Azuay Province [13, 19]. In the area are located stacks of material accumulated during years that, due to the high temperatures and constant rains, generate large amounts of acid drainage of mine.

Bioleaching tests were performed on four tanks with agitation and continuous aeration for six months [13]. The tanks contained natural mine acid drainage, K9 nutrient medium, gold mineral, and sulfuric acid to keep a pH low. Table 1 characteristics of nutrient medium K9 [13, 24]. In addition, panela and ethanol were added to the first tank. During the six months of testing, periodic analyses of Fe$^{3+}$, Fe$^{2+}$, total Fe, SO$_4^{2-}$, Cu$^{2+}$ and Zn$^{2+}$ were performed.

The methods for characterizing the samples were: method for the determination of Fe$^{3+}$ in solution, method for determining SO$_4^{2-}$ in solution and atomic absorption, with hydrochloric acid matrix [13, 19]. The characterized samples were:

Bioleaching solution SOLBIO 2: product of bioleaching tests carried out in the laboratory. To generate the solution was used natural mine acid drainage, K9 nutrient medium, hydrogen peroxide, mineral of the Bella Rica Mining Cooperative (Ponce Enriquez mining district) and sulfuric acid to maintain a pH of 2 [13].

Orenas bioleaching solution: the piles of material accumulated in the company Orenas, mining district of Ponce Enriquez, exposed to rain, humidity, and high temperatures, generate leachates of acid solutions. To leachate samples of acid solutions, after adding nutrients to stimulate bacterial generation, they are used for bioleaching testing [13].

Bioleaching solutions SOLBIO 2 and Orenas, prepared with natural mine acid drainage and gold mineral, were subjected to selective precipitation of iron, copper, and zinc [13]. Each test followed a logical order for the sequential separation (in stages) of heavy metals and, by means of an atomic absorption method, for both filtration and precipitates, partial and final, recovery percentages were determined.
Test 1 Like the First Test in [13]. 250 ml of SOLBIO 2 bioleaching solution was taken, 25 ml of NaOH 1M was added to have a pH variation of 2.46 to 3.58. Under these conditions precipitate one was obtained and 10 ml of H_2O_2 was added to obtain the precipitate two. The presence of H_2O_2 caused the pH of the solution to go down, allowing a third precipitation to be performed by varying the pH from 2.63 to 3.65 by the addition of 46.5 ml of NaOH 1M. Finally, the three precipitates were leaked and analyzed together. After this first stage of the process, an iron removal of 99.31%, copper of 20.52% and zinc of 9.45%.

The filtered solution entered a new precipitation process by adding 11.5 ml of NaOH 1M resulting in a pH variation of 4.45 to 6. The solution was leaked, and the precipitate was analyzed. In this second stage of the process iron was removed by 100%, copper by 95.87% and zinc by 28.86%.

The third stage was to vary the pH of the filtered solution from 7.58 to 9.01 by adding 2.5 ml of NaOH 1M, to obtain a final precipitate, and leave the solution partially free of heavy metals. This last step allowed the recovery of 62.21% copper and 99.19% zinc. Figure 3 shows the composition of the precipitates obtained in each selective precipitation stage.

Using this process meets the maximum allowable discharge limits for the public sewer system, a freshwater body, and a seawater body. With the results of this validation test, it will be analyzed on each of the precipitates obtained at each stage to know the contribution of each precipitation process in the recovery of copper, iron, and zinc.

Test 2 Like the Sixth test in [13]. 250 ml of Orenas bioleaching solution was taken, added 150 ml of NaOH 1M obtaining a pH variation of 2.4 to 3.08, and directly added 10 ml of H_2O_2 to obtain the first precipitate. The solution was leaked, and the precipitate was analyzed. Realized this first part of the process was obtained an iron removal of 71.01%, copper 9.63% and zinc of 12.54%.

The presence of H_2O_2 caused the pH of the solution to go down, allowing a second precipitation to be made by varying the pH from 3 to 3.52 by the addition of 18.3 ml of NaOH 1M. The solution was leaked, and the precipitate was analyzed. This part of the process allowed the iron to be removed by 81.59%, copper by 2.84% and zinc by 1.72%. However, the filtrate had a highly cloudy red color, demonstrating the presence of iron in solution; therefore, it was decided to use a flocculant to improve the removal of heavy metals. 1 g/l of flocculant was added to the filtered solution and allowed to shake for half an hour. The solution was filtered, and the filtrate was analyzed. By means of mass balance, the mass composition of the metals present in the precipitate was determined and it was obtained that coagulation allowed the removal of iron by 87.05%, copper by 0% and zinc by 15.21%.

The filtered solution was subjected to a second stage of precipitation when 35.9 ml of NaOH 1M was added, resulting in a pH variation of 3.98 to 6.28. The solution was leaked, and the precipitate was analyzed. The process allowed iron to be removed by 96.16%, copper by 99.26% and zinc by 95.59%.

The third stage was to vary the pH of the filtered solution from 5.7 to 10 by adding 1.7 ml of NaOH 1M, to obtain a final precipitate and leave the solution free of heavy metals. The solution was leaked, and the precipitate was analyzed. The latter process made it possible to recover iron by 57.14%, copper by 100% and zinc by 81.15%. Figure 3 shows the composition of the precipitates obtained at each stage of selective precipitation.

Using this process meets the maximum allowable discharge limits for the public sewer system, a freshwater body, and a seawater body.

3. Results and Discussion

It was verified that it is possible to determine the presence of copper, iron, lead, and zinc in a solution without interference between the ions to be analyzed. Metal solutions were prepared with lead acetate and copper, iron, and zinc sulfates.

In Table 2 [13] results of the characterization of the samples of bioleaching solutions. The characteristics of the solution generated by a controlled bioleaching process in the laboratory are like those of the natural bioleaching solution, Orenas.

Formalized the analyses, iron was determined to be the predominant component of the solutions, but it was found as Fe^{2+} and Fe^{3+}. As the precipitation pH of Fe^{2+} is very close to the precipitation pH of Cu^{2+}, an oxidant was used to move from Fe^{2+} to Fe^{3+} and allow the precipitation range between copper and iron to be wider.

In Figure 3 sequential scheme (per stage) of selective precipitation to recover iron, copper, and zinc; percentages of precipitates per stage are included for the two laboratory tests.

In Table 3 the percentages of removal (solid phase recovery) of iron, copper, and zinc at each of the stages for selective precipitation tests that were applied to SOLBIO 2 and Orenas bioleaching solutions, as validation mechanisms for tests developed in [13]. The table also presents the percentages of composition of precipitate (iron, copper, and zinc).
Table 2. Features of the Solutions.

| Solution (Bioleaching) | pH initial | Volume (l) | Sulfates (g/l) | Fe$^{2+}$ (mg/l) | Fe$^{3+}$ (mg/l) | Total Fe (mg/l) | Copper (mg/l) | Zinc (mg/l) |
|------------------------|------------|------------|----------------|-----------------|----------------|----------------|---------------|-------------|
| SOLBIO 2               | 2.54       | 14         | 53.89          | 1072.13         | 9323.9         | 10396          | 108.4         | 381.8       |
| Orenas                 | 1.92       | 14         | 60.98          | 55.84           | 12660.2        | 12716          | 108.8         | 464.0       |

Table 3. Percentages of recovery (% R) in the solid phase and percentages of the composition (% C) of the precipitate.

| Tests | Description AMD | SOLBIO 2 Bioleaching | Orenas Bioleaching |
|-------|-----------------|-----------------------|--------------------|
|       | Stages          | 1                     | 2                  | 3                  | 1                 | 2             | 3               |
|       | Solid phase recovery (% R) | 99.31 | 100 | 0 | 87.05 | 96.16 | 57.14 |
|       | Cu              | 20.52                 | 95.87 | 62.21 | 0 | 99.26 | 100 |
|       | Zn              | 9.45                  | 28.86 | 99.19 | 15.21 | 95.59 | 81.15 |
|       | Precipitation composition (% C) | Fe | 99.40 | 24.88 | 0 | 98.91 | 16.10 |
|       | Cu              | 0.23                  | 34.51 | 1.09 | 0.10 | 19.31 | 9.32 |
|       | Zn              | 0.37                  | 40.61 | 98.91 | 0.99 | 64.59 | 83.90 |

Table 4. Percentages of recovery (% R) in the solid phase and percentages of the composition (% C) of the precipitate in [13].

| Tests | Description AMD | SOLBIO 2 Bioleaching | Orenas Bioleaching |
|-------|-----------------|-----------------------|--------------------|
|       | Stages          | 1                     | 2                  | 3                  | 1                 | 2             | 3               |
|       | Solid phase recovery (% R) | 99.76 | 100 | 0 | 73.90 | 96.11 | 63.33 |
|       | Cu              | 24.83                 | 96.51 | 100 | 0 | 92.25 | 36.67 |
|       | Zn              | 10.99                 | 18.56 | 99.95 | 9.72 | 94.97 | 100 |
|       | Precipitation composition (% C) | Fe | 99.3 | 11.84 | 0 | 99.12 | 24.41 |
|       | Cu              | 0.27                  | 49.9 | 2.62 | 0.15 | 15.14 | 2.86 |
|       | Zn              | 0.43                  | 38.26 | 97.38 | 0.72 | 60.45 | 89.73 |

Comparing the percentages of removal (recovery in the solid phase) of iron, copper, and zinc at each of the stages for the two selective precipitation tests that were applied to the SOLBIO 2 and Orenas bioleaching solutions, as a validation mechanism with two of the tests developed in [13], see Table 4, the reproducibility of the results is confirmed. Similarly, the reproducibility of the results in the precipitate composition percentages (iron, copper, and zinc) obtained in the two tests developed here with the precipitate composition percentages in two of the tests reported in [13].

4. Conclusions

The validation of the methods of analysis allowed to demonstrate that the presence of copper, iron, lead, and zinc in solution can be determined by means of simple methods of analysis. The methods used are feasible to use in the field since only glass material and chemical reagents are needed.

All the selective tests carried out allowed the heavy metals to be completely eliminated from the solution or left at concentrations lower than the maximum permissible limit so that they can be discharged to a body of water or to the public sewage system. In this way it was proved that the variation of pH is an effective, easy to use and method, inexpensive feasible to be used in the purification of waters that have been polluted with heavy metals.

Bioleaching solutions were prepared with similar characteristics to the acid drainage of natural mines to carry out the tests of selective precipitation of heavy metals in solution. Pure metal hydroxides were precipitated from natural acid main drainage and form the bioleaching solutions generated under controlled processes in the laboratory.

Results show that the process proposed in the present
study for iron, copper, and zinc recovery has a potential industrial application.

5. Recommendations

The problems encountered due to the co-precipitation of metals could be due to adsorption between ions. To avoid co-precipitation it is recommended to carry out tests with different types of coagulants and flocculants to improve the selectivity of precipitation.

The solutions treated in the research were free of heavy metals; however, the sulfate contents before performing the purification processes were quite high, exceeding the permitted discharge limits. With the process used, sulfates were not treated, therefore, it is recommended to carry out studies that allow to lower the levels of sulfates to a minimum.

Bioleaching has advantages and disadvantages with respect to methods such as roasting or chemical leaching. The advantages of bioleaching are the low capital and energy costs, in addition to the great flexibility to be used in situ and to be environmental friendly. Another great advantage is that due to the growth of Thiobacillus thiooxidans, the pH of the leached gradually decreases allowing a selective oxidation of sulfides. Therefore, it is recommended to employ this proposal for industrial gold release processes associated with metal sulfides.

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