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Comparative study of chemical process and biotechnological process for the removal of bismuth from mining concentrates

Estudio comparativo de un proceso químico y un proceso biotecnológico para la remoción de bismuto de concentrados mineros

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Abstract

In 2018, approximately 17.7 billion metric tons of minerals were produced worldwide, which is of great importance in the industrial sector. However, most of the minerals are bound with metallic elements, which lower the price of the metals of interest. Secondary materials in valuable metal extraction processes can cause various problems in the mining industry, both economic and environmental. This research aimed to evaluate two processes for the reduction of the bismuth concentration in gold and silver mineral concentrates: Bioleaching and co-precipitation with trioxide arsenic; in the first, native microorganisms of the mineral concentrate were analyzed to reduce the concentration of bismuth; in the second, arsenic trioxide was added to form a precipitate that contained bismuth. The results showed that the chemical co-precipitation process with arsenic trioxide can decrease the bismuth concentration to a greater extent compared to the bioleaching process, with a reduction of up to 16% of the initial concentration. Also, a decrease of the concentration of other undesirable elements in the mineral concentrates such as antimony and cadmium was performed, obtaining values of 32% and 11%, respectively. A combination of both techniques could be proposed for a higher reduction of bismuth concentration to achieve greater efficiency.
Keywords: Bioleaching, arsenic coprecipitation, bismuth.

Resumen

En el 2018, aproximadamente 17.7 billones de toneladas métricas de minerales fueron producidas mundialmente, lo cual es de gran importancia en el sector industrial. Sin embargo, la mayoría de los minerales están ligados a elementos metálicos, los cuales disminuyen el precio de los metales de interés. Los materiales secundarios en los procesos de extracción de metales valiosos pueden causar varios problemas en la industria minera, tanto económicos como ambientales. El objetivo de esta investigación fue evaluar dos procesos para la reducción de la concentración de bismuto en concentrados minerales de oro y plata: la biolixiviación y la coprecipitación con trióxido de arsénico; en el primero, se analizaron microorganismos nativos del mineral concentrado para reducir la concentración del bismuto; en el segundo, se añadió trióxido de arsénico para formar un precipitado que contuviera bismuto. Los resultados mostraron que el proceso de coprecipitación química con trióxido de arsénico puede disminuir la concentración de bismuto a mayor grado comparado con el proceso de biolixiviación, con una reducción de hasta el 16% de la concentración inicial. Además, se llevó a cabo una reducción de la concentración de otros elementos no deseables en los concentrados mineros tales como el antimonio y el cadmio, obteniendo valores de 32% y 11%, respectivamente. Una combinación de ambas técnicas podría ser propuesta para una mayor reducción de la concentración de bismuto para alcanzar una mayor eficiencia.
Palabras clave: Biolixiviación, coprecipitación de arsénico, bismuto.

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1 Introduction

Some sulfured minerals contain Au, Ag, and Cu, which represent an alternative source of these high-value metals, however, they are highly refractory and their extraction by conventional processes can carry pollutants that affect their market value (Castells, 2012; Ferrer, 2003; Ha, Kwon, Park, & Mohapatra, 2015; Martínez-Pérez, 2002). The use of alternative procedures, both biological and chemical, may have some advantages; within the biological ones, the economic and environmental benefits are evident, whilst in the chemical extraction processes, the percentage of recovery of metals of interest and the elimination of contaminants are very high (Javad, Kargar, & Nowroozi, 2019).

Two of the most used processes to extract metals from mineral ores are bio-oxidation and bioleaching; Bioleaching is mainly used in minerals such as chalcopyrite (Álvarez, 2019; Noguchi & Okibe, 2020). However, the information of this process has been refined and proposed for the extraction of other metals of interest, as well as for different methods; stirred tank, columns, stacks among others (Srichandan *et al*., 2020). On the other hand, the extraction of various metals from mineral ores through chemical processes is widely used, regularly combining chemical treatment techniques with some mechanical treatments (Ficeriová, Baláž, & Villachica, 2005). Also, some chemical leaching processes use acid solutions to achieve the desired separation of the metals of interest (Ha *et al*., 2015). Bismuth acts as a refractor agent that is currently penalized depending on its concentration in mining concentrates since it shortens the lifespan of casting ovens (Dreisinger, 2019). To reduce Bi concentration, very few treatments are available. However, some authors suggest the use of arsenic for the removal of bismuth through a chemical co-precipitation process (Xiao *et al*., 2012). For this reason, this research aimed to evaluate the removal of bismuth from mineral concentrates through two processes: bioleaching and co-precipitation with arsenic trioxide.

2 Materials and methods

Two different processes were implemented to decrease the concentration of bismuth present in mineral concentrates. By re-inoculating native microorganisms, a bioleaching process was developed. On the other hand, a coprecipitation process was implemented by adding arsenic trioxide.

2.1 *Bioleaching*

The experimental design of the bioleaching tests was carried out using Design-Expert software. The numerical independent variables were temperature (X_1) (20 and 40 ºC), pH (X_2) (1 and 7), stirring speed (X_3) (0 and 300 rpm) and pulp density (X_4) (10 and 30%), while the categorical variable was the culture medium (X_5) with two levels (Table 1). The incubation time was four days, with daily monitoring of cell density (only for bioleaching tests), ORP, and pH as responses. A Neubauer darkfield chamber and a Hanna HI 991003 potentiometer with a Hanna HI 1297 titanium electrode were used. Removal of gold, silver, copper, lead, zinc, iron, bismuth, antimony, and cadmium was analyzed at the end of experimentation using the standard chemical analysis protocols and the QEMSCAN Particle Mineral Analysis (PMA). Control treatments were evaluated for each bioleaching experiment, where the same variables were analyzed at the same levels and operating times as in the bioleaching tests, but in the absence of inoculum and using sterile mineral. Each experiment had an average inoculum of 7.41 $\times 10^7$ cells / mL and they were performed in 500 mL Erlenmeyer flasks containing 100 mL of culture medium.

The results were analyzed by response surface superimposition, using multiple quadratic regressions (Equation 1), and analysis of variance (ANOVA) was applied with a maximum level of error probability of 5%.

Table 1. Levels of the independent numerical variables.

	Levels of variation	
Factor	Maximum	Minimum
Temperature (°C)	40	20
pH	7	1
Stirring rate (rpm)	200	0
Pulp density (%)	30	10

Culture medium it is an independent categorical variable (levels of variation are API-BS and Mier).

$$y = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4X_4 + B_5X_5 + B_{11}X_1^2 + B_{22}X_2^2 + B_{33}X_3^2 + B_{44}X_4^2 + B_{55}X_5^2 + B_{12}X_1X_2 + B_{13}X_1X_3 + B_{14}X_1X_4 + B_{15}X_1X_5 + B_{23}X_2X_3 + B_{24}X_2X_4 + B_{25}X_2X_5 + B_{34}X_3X_4 + B_{35}X_3X_5 + B_{45}X_4X_5 \quad (1)$$

2.1.1 Bacterial strains

Samples of concentrates were inoculated using two different culture media: (1) Mier, composed of ammonium sulfate (0.4 g/L), magnesium sulfate heptahydrate (0.5 g/L) and dipotassium phosphate (0.2 g/L) (Mier, Gómez, Ballester, & González, 1994); and, (2) API-BS, which is composed of sodium chloride (10 g/L), yeast extract (1 g/L), magnesium sulfate (0.2 g/L), dipotassium phosphate (0.01 g/L), ferrous ammonium sulfate (0.1 g/L), sodium lactate (5.2 g/L) and ascorbic acid (0.1 g/L) (DIFCO MANUAL, 2016). Microorganisms were cultivated under the specific conditions for each medium: in the Mier culture medium, the pH was adjusted to 1.5; while for the API-BS culture medium, the pH was 7. The temperature was 35 ºC for both culture mediums. These culture mediums were selected for the specificity of the bacteria that were required to grow (sulfate-reducing), due to the presence of sulfate in the mining concentrates (Table 2). A mineralogical assessment was conducted on a copper concentrate sample, which was donated by Avino Silver & Gold Mine Ltd (Table 3), located in Durango, México.

2.1.2 Morphological Identification

Two different staining techniques were performed on the microorganisms: Gram stain and Ziehl-Neelsen stain, thus providing information of their morphology, cell wall composition, and motility (García, 2004; Rawlings, 2005; Rodríguez, Gamboa, Hernández, & García, 2005).

2.2 Coprecipitation with arsenic trioxide

To evaluate the removal of Bi through the coprecipitation with arsenic trioxide, the methodology proposed by Xiao *et al*. (2012) was carried out. Each experiment was performed in 500 mL Erlenmeyer flasks, containing 10% w / v of pulp density dissolved in distilled water.

Table 2. Composition of mining concentrates (Mineralogical assessment).

Minerals	Chemical formula
Acanth / Argentite	Ag_2S
Pyrargyrite/ Stephanite	Ag_3SbS / Ag_5SbS_4
Stromeyerite	$AgCuS$
Freibergite	$Ag_6Cu_4Fe_2Sb_4S_{13}$
Ourayite	$Pb_4Ag_3Bi_5S_{13}$
Chalcopyrite	$CuFeS_2$
Chalcocite / Covellite	Cu_2S
Bornite	Cu_5FeS_4
Tetrahedrite	$Cu_6[Cu_4(Fe,Zn)_2]Sb_4S_{13}$
Sphalerite	ZnS
Galena	PbS
Bismuthinite/ Bismuth	Bi_2S_3 / Bi
Canizzarite	$Pb_4Bi_6S_{13}$
Hodrushite/ Wittichenite	$Cu_8Bi_{12}S_{22}/Cu_3BiS_3$
Cd - Sulphide	CdS
Pyrite	FeS_2
Arsenopyrite	$FeAsS$
Iron Oxides	$Fe_2+Fe_{23}+O_4/Fe_2O_3$
Quartz	SiO_2
K - Feldspars	$KAlSi_3O_8$
Chlorite/ Muscovite	$(Mg,Fe,Al)_6(Si, Al)_4O_{10}(OH)_2$
Rutile/ Anatase	TiO_2
Calcite	$CaCO_3$

Table 3. Initial chemical analysis of the copper mineral.

Minerals	Bioleaching	Coprecipitation
	Contents (wt. %)	
Au	15.883	17.2
Ag	2573.283	2531.6
Cu	19.105	18.87
Pb	2.683	2.46
Zn	2.928	3.07
Fe	20.98	21.57
Bi	1.908	1.939
Sb	0.105	0.103
Cd	0.052	0.051

Table 4. Levels of the D-Optimal experimental design for the coprecipitation with arsenic trioxide.

Factor	Levels of variation	
	Maximum	Minimum
Temperature (°C)	40	20
pH (-)	7	1
Stirring rate (rpm)	200	0
Pulp density (%)	30	10

As_2O_3 (Sigma - Aldrich) was added according to a D- Optimal experimental design (Table 4) (1.2 and 3 g/L). Bi removal was evaluated for all treatments as a response.

To analyze the minerals in the concentrate the standard chemical analysis protocols and the QEMSCAN Particle Mineral Analysis (PMA) were performed on the unsized sample of the copper concentrate.

3 Results and discussions

3.1 Bacterial Strain

The strains from the Mier culture medium showed to have activity only during 4 days after inoculation (Figure 1), while those that were cultivated in the API medium had their highest activity between the ninth and thirteenth day, which can be observed in Figure 2, with a maximum cell density of 4.25×10^6 and 8.28×10^7 cells / mL, respectively. All the bacterial strains of the two-culture media showed the morphology of gram-negative bacilli, so it can be deduced that they have one or more polar flagella, which gives them mobility. This type of bacteria has a more complex cell wall with an outer membrane that confers structure, a periplasmic space of a peptidoglycan layer, and an inner cytoplasmic membrane (García, 2004; Rodríguez *et al*., 2005).

3.2 Bioleaching

The results of the bioleaching tests were analyzed using multiple quadratic regressions. The resulting equation for the removal of bismuth is given by Equation 2,

$$RBi = 2.67 + 0.383A - 1.14E + 0.69AE \qquad (2)$$

where A is the temperature, E is the culture medium and AE is the interaction, the value of r^2 for this is 0.414.

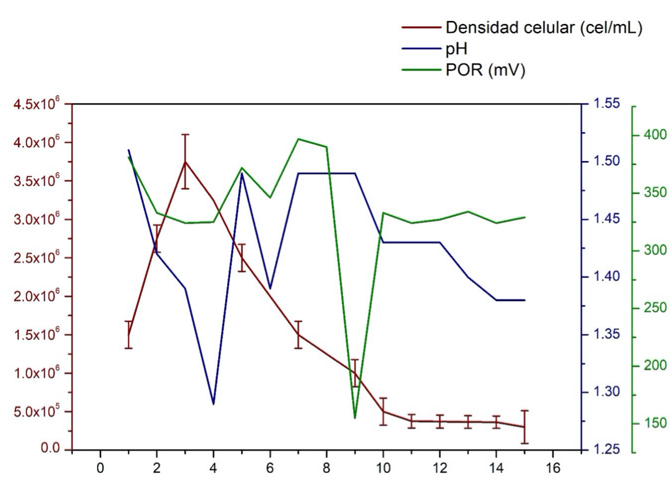

Fig. 1. Microbial kinetics of microorganisms in the Mier culture medium (Cell density in cherry color, the potential for oxide reduction in green color, and pH in blue color).

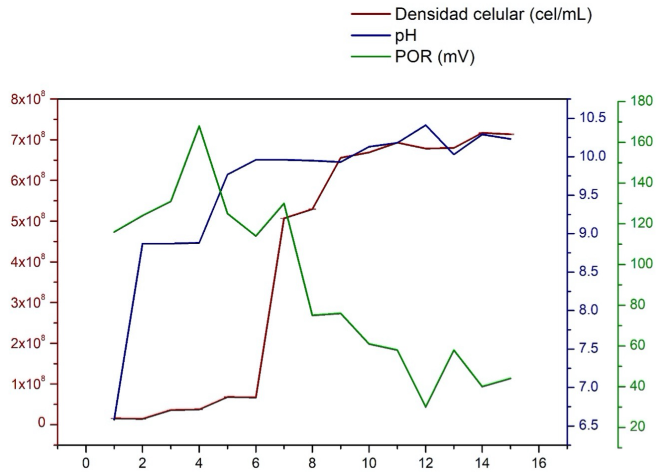

Fig. 2. Microbial kinetics of the microorganisms in the API-BS culture medium (Cell density in cherry color, the potential for oxide reduction in green, and pH in blue color).

Table 5. Biochemical test responses.

	RAu	**RAg**	**RCu**	**RPb**	**RZn** %	**RFe**	**RBi**	**RSb**	**RCd**	**REDOX** mV	**pH**	**Celular density** cel/mL
A	0	2.49	0.514	0	3.09	0	1.49	6.25	37.037	283	0.99	9.92×10^7
B	2.469	1.999	0	6.693	5.05	0	3.872	3.296	11.538	161	7.23	4.92×10^8
C	0	0	10.825	11.188	7.065	11.818	0	13.533	32.692	303	1.23	3.52×10^7
D	10.117	2.95	0	13.986	2.606	0	6.086	35.338	13.461	202	6.3	6.8×10^7
A'	15.227	4.003	2.983	0	0	0	5.848	0	35.185	286	0.99	1.004×10^8
B'	0	1.521	1.955	3.448	6.397	0	4.034	8.791	7.692	164	7.19	4.576×10^8
C'	0	0	4.571	0	4.663	5.012	0	0	32.692	294	1.34	3×10^7
D'	10.117	0	0	6.293	0	0	0	33.834	3.846	193	6.74	6.64×10^7

A: API medium, 0 rpm, 10% pulp, 20 ºC, 1 pH; B: API medium, 200 rpm, 30% pulp, 40 ºC, 7 pH; C: Mier Medium, 0 rpm, 10% pulp, 20 ºC, 1 pH; D: Mier Medium, 200 rpm, 30% pulp, 40 ºC, 7 pH. (All experiment had an average initial inoculum of 7.41×10^7 cells/mL).

Table 6. Control test responses (without inoculum).

	RAu	**RAg**	**RCu**	**RPb**	**RZn %**	**RFe**	**RBi**	**RSb**	**RCd**	**REDOX mV**	**pH**
QA	0	0.167	5.967	0	0	2.422	0	0	31.481	286	1.07
QB	4.938	0	0	0	1.01	0	3.005	13.186	3.486	170	7.05
QC	4.044	0	0	8.041	0	0	4.205	38.345	28.846	296	1.28
QD	0	0	0	11.188	0	0	4.205	29.323	11.538	210	5.88
QA'	0	5.755	10.03	0	0	1.884	17.431	0	33.333	283	1.2
QB'	7.407	0	0	0	0.336	3.47	0	0	5.769	171	6.91
QC'	0	0	0	10.839	0	0	0	42.105	26.923	293	1.28
QD'	0	0	0	11.188	0	0.22	10.143	36.09	1.923	214	6.22

QA: API medium, 0 rpm, 10% pulp, 20 ºC, 1 pH; QB: API medium, 200 rpm, 30% pulp, 40 ºC, 7 pH; QC: Mier Medium, 0 rpm, 10% pulp, 20 ºC, 1 pH; QD: Mier Medium, 200 rpm, 30% pulp, 40 ºC, 7 pH.

However, for this response of interest, there was no term in the model that was significant (if $p > 0.05$ no term in the model is significant and $p = 0.499$). A removal of 4.266% of bismuth was obtained (Table 5), this may be because the main objective of bioleaching is to oxidize sulfured compounds, thus exposing unwanted elements and facilitating their removal (Álvarez, 2019). However, in this procedure, a reduction occurred due to the lack of a more oxidizing environment that did not allow the bioconversion of the metal of interest.

Table 7. Arsenic Trioxide test responses.

	RAu	RAg	RCu	RPb	RZn %	RFe	RBi	RSb	RCd
1	10.465	7.931	1.907	6.097	14.657	0	9.437	36.893	11.764
2	13.953	0	4.769	10.975	13.029	0	11.346	29.126	11.764
3	0	0	5.405	8.943	1.302	0	16.039	25.242	7.843
4	10.465	7.931	4.769	6.097	14.657	0	9.437	36.893	11.764
5	13.953	0	4.769	10.975	13.029	0	11.346	29.126	11.764
6	0	0.15	6.465	6.097	5.537	0	12.429	36.893	5.882
7	0	0.15	6.465	6.097	5.537	0	12.429	36.893	5.882
8	6.976	0	13.142	9.756	19.869	2.874	11.964	31.067	15.686
9	6.976	0	13.142	9.756	19.869	2.874	11.964	31.067	15.686
10	0	0	5.405	8.943	1.302	0	16.039	25.242	7.843

1 to 10: arsenic trioxide concentrations: 1,2,1,3,3,1,1,3,3,2 g / L, respectively.

Despite not having a substantial decrease in Bi concentration, other unwanted elements such as antimony and cadmium had a decrease of 14.434 and 21.768%, respectively (Table 5). Sb and Cd are highly polluting heavy metals (Dreisinger, 2019).

For the tests that were carried out in the absence of inoculum, they were analyzed like the previous ones using multiple quadratic regressions, obtaining Equation 3 for the removal of bismuth.

$$RBi = 4.87 - 0.54A - 0.24E + 3.07AE \tag{3}$$

where A is the temperature, E the culture medium, and AE is the interaction. Obtained r^2 was 0.299. No term of the model was significant (if $p > 0.05$ no term in the model is significant and $p = 0.6639$). Average removal of Bi of 4.874% was obtained (Table 6), this may be due to the various reactions of the acids with the mineral, as well as NaOH in the case of the alkaline experiments. For these experiments, decreases of 31.810 and 17.957% were obtained for elements such as antimony and cadmium, respectively (Table 6).

Comparing the results in the removal percentage between the experiments carried out with and without inoculum, it can be deduced that the decrease in bismuth concentration is due to chemical reactions between the mineral and the acids present in the medium.

3.3 Coprecipitation with arsenic trioxide

The results of the coprecipitation with As_2O_3 were analyzed by multiple quadratic regressions. The resulting equation for the removal of bismuth is given by Equation 4.

$$RBi = 16.04 - 0.59A - 4.74A^2 \tag{4}$$

where A is the concentration of As_2O_3 and this is the significant term (if $p > 0.05$ indicates model terms are significant, in this case, A^2 are significant because $p = 0.0017$) for this model and the value of r^2=0.414. Removal of 12.243% of Bi was obtained, despite not being a very high percentage, it triplicates the obtained value using the biological method. Furthermore, the removal of other unwanted metals also increased to 31.844 and 10.588% for Sb and Cd, respectively (Table 7). The reduction of the bismuth content by this method was higher (12.243%) compared to the biological method (4.266%).

It was expected that the removal of Bi would not achieve the concentration levels obtained by Xiao *et al*. (2012), since the mineral analyzed in that research was mainly composed of oxides in an aqueous solution, while in the present study, the mineral was mainly composed of sulfides and it was a dry powdered mineral.

Finally, Dreisinger (2019) reported that the penalty in the commercialization of copper with bismuth as a polluting metal was $10 per ton at a 0.1% proportion. Taking this into account and since bismuth concentration was reduced by 16% in the mining concentrates, this would allow commercializing the mineral with a higher profit.

Conclusions

Coprecipitation with arsenic trioxide is superior compared to bioleaching regarding bismuth removal (12.243 vs 4.266%) from gold and silver concentrates. In addition to the decrease in the concentration of bismuth, it was possible to decrease up to 31.844 and 10.588% in the concentration of antimony and cadmium, which are also undesirable metals in the mineral concentrate, because they decrease the market price. Based on this research, further research is recommended to explore a complementary process involving both bioleaching and coprecipitation with arsenic trioxide to achieve higher bismuth removal.

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