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RECOVERY OF GOLD AND SILVER FROM SLAG FROM THE “LA HERRADURA” UNIT; EFFECT OF ZINC DURING LEACHING IN THE THIOSULFATE-AMMONIA-Cu(II) (TACu) SYSTEM

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Abstract: The gold and silver contained in the smelting slag from the La Herradura unit, in concentrations of 168 g Au/ton and 437 g Ag/ton, are present in a metallic state, mainly as Au-Ag (75%) and Au-Ag-Cu (22%) with spherical or rounded shapes, of the type that causes fusion and solidification. Most of these are associated with zinc silicate particles, mainly as inclusions (12 microns), followed by simple bonds at the edges of the zinc silicate particles. The results of gold dissolution rates in slag in the Thiosulfate-Ammonia-Cu(II) (TACu) system, based on solution analysis via atomic absorption, indicate that these are higher than those obtained in cyanide. The presence of zinc ions (100, 200, and 400 ppm), in accordance with their hard-soft nature, increases the dissolution rate. However, it should be emphasized that the size of metal inclusions and the composition of the slag are factors to be considered in the chemistry of a leaching system, which is characterized by its complexity and stability.

Keywords: Au-Ag smelting slag, TACu(II),

INTRODUCTION

According to the 2023 annual report of the Mexican Chamber of Mining (CAMIMEX), the mining and metallurgical sector in Mexico continues to develop, supplying raw materials to more than 70 productive sectors, which consolidates this sector as one of the most important industries. Mexico continues to be one of the ten largest producers of precious metals and base minerals in the world, which allowed this industry to represent 2.46% of the national GDP and 8.63% of the country's industrial GDP in 2022, thus confirming its importance for national development [1]. Figure 1 shows mining and metallurgical production in 2022, with gold accounting for 30.6% of the total value of this sector's production. Although there is significant production of metals such as copper, silver, zinc, lead, and molybdenum,

among others, the gold industry continues to be of great importance and contribute significantly to the national economy in Mexico.

MAIN PRODUCTS IN THE VALUE OF MINING AND METALLURGICAL PRODUCTION IN 2022 (SHARE %)

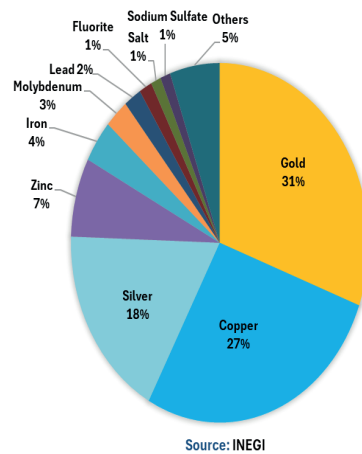


Figure 1.- Main products in mining and metallurgical production in Mexico in 2022 [1].

In 2022, Mexico contributed 3.4% of global gold production (Figure 2a) and 24.2% of global silver production, consolidating its position as the world's largest producer (Figure 2b) [1]. These statistics show the importance of this industry in the country. Although Mexico produces other metals and non-metals such as Cu, Pb, and Zn, among others, the high value of gold and its production in Mexico consolidate the extraction of this metal as one of the main mining and metallurgical activities in the country. In this sense, an important challenge for this industry is the recovery of these values from both their ores and alternative or secondary sources.

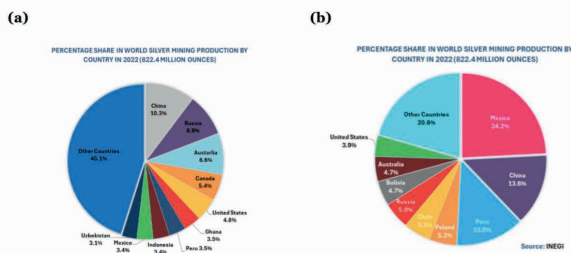


Figure 2. (a) Global and national overview of gold production in 2022 and (b) global and national overview of silver production in 2022 [1].

The recovery of metal values (gold/silver) from their ores is increasingly complicated due to their mineralogical complexity, known as “refractoriness,” which refers to those minerals where gold/silver is recovered with low efficiency, less than 80% by conventional methods such as cyanidation or flotation. The latter can be measured in terms of the degree of recovery as high refractoriness (recovery <50%), moderate refractoriness (recovery 50-80%) or low refractoriness (recovery 80-95%). The causes of refractoriness are diverse and focus on the physical or chemical properties of the mineral, where mineralogical association plays an important role. In this sense, gold recovery presents two major challenges in the metallurgical industry: on the one hand, the search for treatments that optimize the recovery of gold-silver from refractory minerals and, on the other hand, the economic recovery of gold from alternative sources, such as electronic scrap, slag, or other materials [2].

Slags are vitreous solid wastes produced in various pyrometallurgical processes and are formed due to the presence of impurities and unwanted elements that are usually found in the gangue associated with the ore of the deposit being mined. These impurities can be oxides, sulfides, silicates, or other compounds [3, 4]. The slags resulting from the metallurgical process for obtaining precious metals in the form of ingots known as “doré” include a series of unit operations such as those shown in Figure 3. Once the mineral ore is leached

with a sodium cyanide solution, the resulting rich solution is stored prior to precipitation of the valuable components. It is first filtered to clarify it (< 10 ppm solids), then fed to a deaeration tower (reducing the oxygen in solution to 0.2-0.8 ppm), its chemical composition is adjusted, and finally it is sent to the precious metal precipitation stage by adding zinc powder [5]. The precipitate is then filtered and sent to smelting, where doré bars and slag are produced [6]. Although these slags are waste products from the metallurgical smelting stage, they are currently of great interest due to their high gold content, which can sometimes reach up to 279 g of Au per ton, as observed in the gold recovery processes at the “La Herradura” unit. This grade is similar to that commonly observed in precious metal concentrates that undergo cyanidation, which is why the recovery of these values from slag is of interest to the metallurgical industry.

On the other hand, the search for alternatives to cyanide leaching of gold has been a controversial and highly evaluated topic in metallurgy. Thiosulfate has been shown to be a possible environmental alternative to cyanide for gold leaching [7-13]. This project proposes to evaluate the recovery of gold from slag from the La Herradura unit using thiosulfate leaching systems, as well as to evaluate the effect of additives such as Zn and thiourea on gold dissolution.

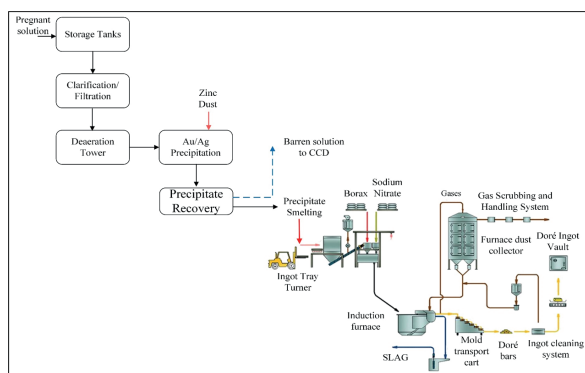


Figure 3. Simplified flow diagram of the precious metal precipitation process with zinc powder for doré production [6, 14, 15].

BACKGROUND

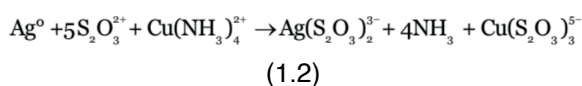
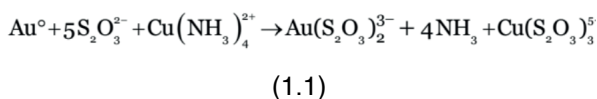
Cyanidation has been the most efficient, simple, and economical method for extracting precious metals. However, in recent years, research has focused on finding alternative solvents and treatment methods, as CN^- is a toxic leaching agent. The use of the thiosulfate-ammonia-copper (TACu) system is an alternative method that has several advantages, including that it is non-toxic and has faster precious metal dissolution rates than those obtained with CN^- . Although its first uses date back to the 1900s, a few years ago it was revived for the treatment of some refractory ores, specifically those associated with or containing cyanide-soluble copper (secondary oxides and sulfides) and those associated with pre-roasting carbonaceous matter [7-13].

The thiosulfate leaching system ($\text{S}_2\text{O}_3^{2-}$) consists of a slightly alkaline aqueous solution containing copper and free ammonia. The main advantages of this system are: (a) lower cost than cyanide (USD 3,000/ton of NaCN, USD 400/ton of $\text{Na}_2\text{S}_2\text{O}_3$); (b) the copper used as a “catalyst” may be present in some Cu-Au oxide ores, and (c) low toxicity, in addition to forming stable complexes with gold ($\text{Log}K_{\text{Au}(\text{CN})_2^-} = 38.3$, $\text{Log}K_{\text{Au}(\text{S}_2\text{O}_3)_2^-} = 28.7$).

In contrast to these and other advantages not mentioned, the chemistry of the thiosulfate-ammonia-copper (TACu) system is relatively complicated, requiring the search for and maintenance of adequate concentrations of its constituents in order to achieve potential (Eh) and pH conditions that allow for good precious metal extraction and low reagent consumption [16]. Copper and ammonia in the leaching system form copper tetramine $\text{Cu}(\text{NH}_3)_4^{2+}$, which favors the dissolution rate of gold (18 to 20 times that of cyanide). However, its excess can cause the oxidation-degradation of thiosulfate and, therefore, the loss of gold-silver to the solid. Excess free ammonia in the solution can lead to the dissolution of

undesirable impurities, which often cause excessive oxidation of thiosulfate [17]. Conversely, its absence can cause passivation of gold due to the accumulation of sulfur coatings on the surface of the gold particles, the latter resulting from the decomposition of thiosulfate [18].

The dissolution reactions of metallic gold and silver in the TACu leaching system, in addition to other possible reactions, are as follows [19, 20]:



The apparent simplicity of the mechanism summarized by reactions (1.1) and (1.2) in the TACu system is extremely complex. The large number of equilibria and chemical species involved in the system requires that the working conditions be controlled due to the decomposition of the former into polysulfates, so an alkaline solution must be used to prevent this decomposition at acidic pH. The Eh-pH diagram for the S-H₂O system, shown in Figure 4 and reported in the literature by several authors, reveals the narrow stable zone of thiosulfate and its decomposition into a wide variety of polithionates (trithionate $\text{S}_3\text{O}_6^{2-}$, tetrathionate $\text{S}_4\text{O}_6^{2-}$, pentathionate $\text{S}_5\text{O}_6^{2-}$) [7, 21].

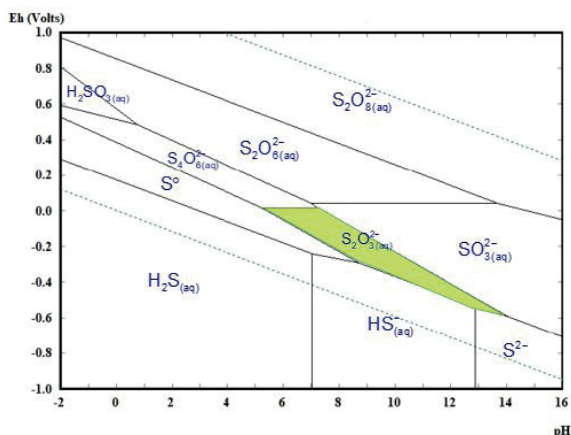


Figure 4. E_h -pH diagram of the S- H_2O (0.10 M) system[21] .

Despite this duality, the dissolution rates shown by the TACu system on precious metals, together with the high softness of S^{2-} , make it a promising candidate for recovering metals contained in smelting slag from heap leaching and dynamic leaching operations at the La Herradura unit[14, 15] .

EXPERIMENTAL METHODOLOGY

In this section, we summarize the experimental system, the materials and reagents used, and the testing procedure.

REAGENTS AND STUDY SLAGS

All reagents used were analytical grade for quantitative chemical analysis. Some of them are also available in technical grade, but these are only used in general practices.

AU-AG PRECIPITATE SMELTING SLAGS

PHYSICAL CHARACTERIZATION

The study material consisted of approximately one kilogram of dry-ground smelting slag with a particle size of $P_{80} = 138$ microns, resulting from the smelting of precious metal precipitates from the operations of the La Herradura mining and metallurgical unit. Figure 5 shows the particle size distribution of a portion of the ground slag.

CHEMICAL AND MICROSCOPIC CHARACTERIZATION

Table 2 summarizes the chemical analysis of a representative portion of these slags, which is the average of two determinations.

Description Sample	Assay (g/Ton)	
	Au	Ag
Slag from the La Herradura smelter	167.8	43

Table 2. Chemical analysis of smelting slag from precipitates.

The overall appearance at low magnifications reveals that the sample consists mainly of zinc silicate particles. Figure 6 shows the SEM characterization, where you can see a general view of the sample with Zn silicate particles in semi-homogeneous sizes, with particles ranging from $112.5 \mu m$ to $12.5 \mu m$.

The textural characteristics of these alloy-type species can be summarized as follows: in general, all particles of these species are spherical or rounded in shape, of the type that causes fusion and solidification. The most of these are associated with zinc silicate particles, mainly as inclusions followed by simple bonds at the edges of the zinc silicate particles. The largest sizes correspond to the Au-Ag alloy, which reaches up to $20 \mu m$, followed by the Au-Ag-Cu alloy; in both alloys, the proportions of Au and Ag vary widely, while Cu remains a minor element. The Pb-Ag alloy is minor compared to those mentioned above, with Pb being the dominant element in this alloy. Sulfoselenides with silver are very rare. Table 3 summarizes the textural characteristics of the slags.

Figure 7 shows the micrograph of the Au-Ag alloy present in the slag included in a zinc silicate particle, with sizes of $5 \mu m$ and free particles of sizes of $11.7 \mu m$ surrounded by si magnification; and (c) EDS spectrum of the Au-Ag particles [22].

NAME	FORMULA	P.A., P.M.	Brand	Purity
Sodium thiosulfate	$\text{Na}_2 \text{S}_2 \text{O}_3$	248.17	Karal	99.50
Ammonium sulfate	$(\text{NH}_4)_2 \text{SO}_4$	132.15	Monterrey	Analytical grade
Copper sulfate	$\text{CuSO}_4 \cdot 5\text{H}_2 \text{O}$	249.68	Monterrey	98.5
Zinc sulfate	$\text{ZnSO}_4 \cdot 7\text{H}_2 \text{O}$	287.58	Fermont	98
Sodium cyanide	NaCN	49.0	Monterrey	95.0
Lead nitrate	PbNO_3	331.2	Golden Bell	98
Ammonium hydroxide	$\text{NH}_4 (\text{OH})$	35.06	Fermont	ACS

Table 1. Reagents used and their characteristics.

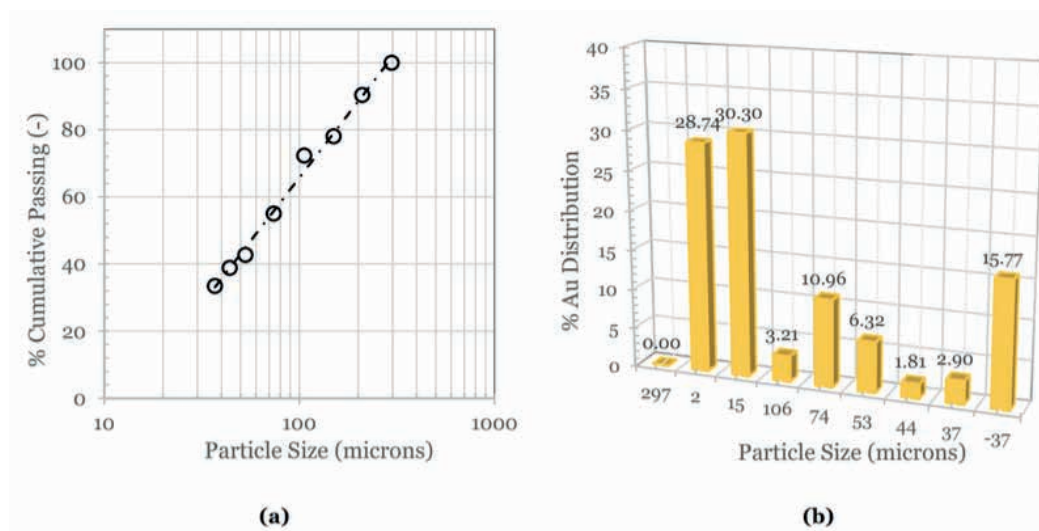


Figure 5. (a) Particle size distribution of ground slag, $P_{80} = 150 \mu\text{m}$, and (b) % Au distribution in each size fraction.

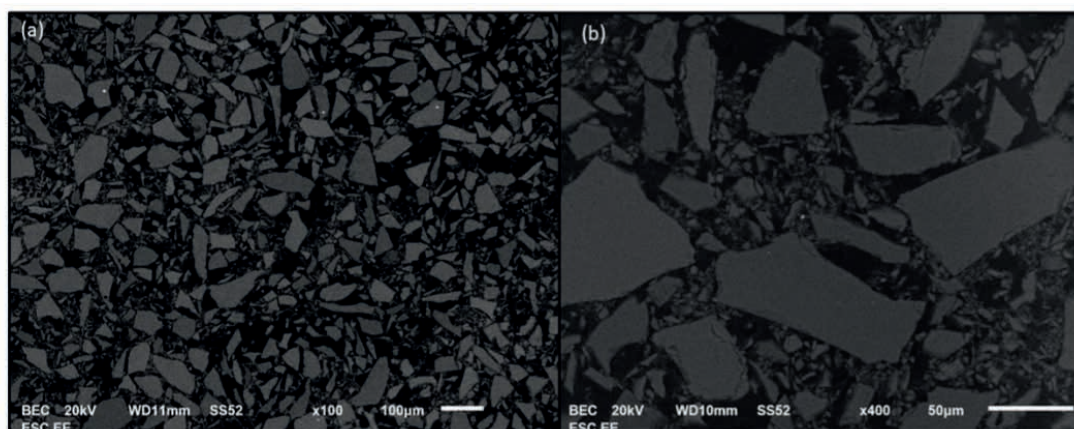


Figure 6. SEM of the slag, (a) 100X magnification and (b) 400X magnification.

Association	Type of bond	Proportion	Size (μm)	
			Average	Variation
Textural characteristics of the Au-Ag alloy (75%)				
Au-Ag	Free	3	12	10
Au-Ag with zinc silicate	Inclusions on edges	45	5	2-8
		25	8	3-12
Textural characteristics of the Au-Ag-Cu alloy (22%)				
Au-Ag-Cu	Free	2	8	6
Au-Ag-Cu with zinc silicate	Inclusions on edges	50	2	<1-5
		30	6	3-8
Textural characteristics of the Pb-Ag (2%) alloy				
Pb-Ag with zinc silicate	Inclusions At edges	9	1	<1-2
		10	1	<1-2

Table 3. Textural characteristics of particles of these species with gold and/or silver [22].

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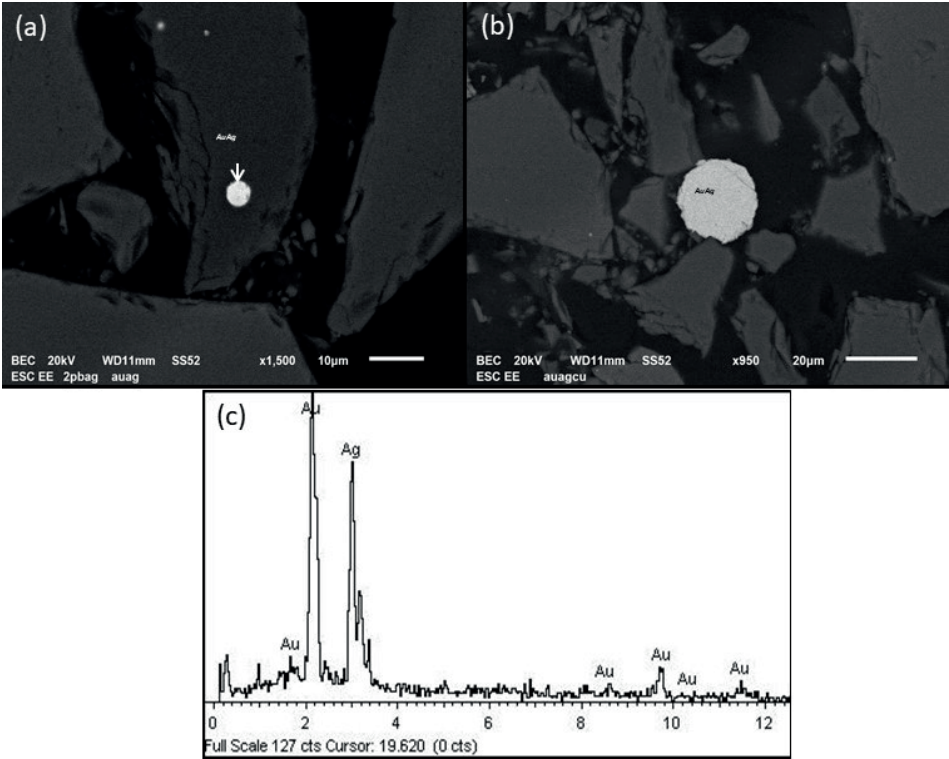


Figure 7. SEM of Ag-Au particles present in the slag: (a) 1500x magnification; (b) 950x magnification; and (c) EDS spectrum of the Au-Ag particles [22].

TEST PROCEDURE

Slag leaching tests were carried out in both the cyanide and TACu systems. The setup used for these tests consisted of a 400 mL baffled reactor, agitation was provided by a Caframo, to which a 5 cm diameter turbine-type propeller shaft was attached, and the test agitation speed was set at 400 min⁻¹. Figure 8 illustrates the test setup.

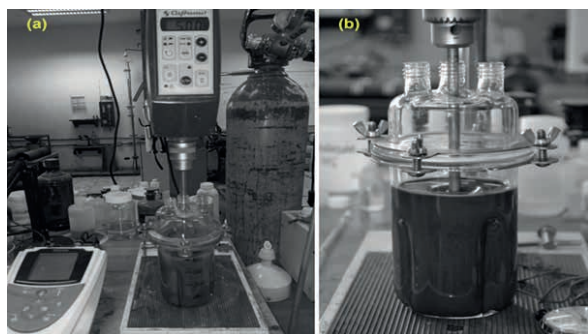


Figure 8. System for leaching kinetics tests: (a) 400 mL baffled reactor, head, and propeller shaft, and (b) details of the baffled reactor.

For each test, the leaching solution to be used was first prepared by placing 300 mL of distilled water in a half-liter beaker, weighing the mass of the test thiosulfate, and adding it to the water. then the milliliters of ammonium hydroxide were taken and also added to the thiosulfate solution. Finally, the copper sulfate was weighed, measured in a volumetric flask, and stirred to homogenize. From this last solution, 400 mL were taken and transferred to the baffled reactor. Next, 40 grams of slag sample were weighed, added to the reactor, and stirring and timing were started.

The test was monitored by taking samples of the test solution every 10 minutes during the first half hour and then every 15 minutes until a total of two hours of testing was completed. For each sample, the agitation was stopped, the slag solids were allowed to settle for five minutes, five milliliters were taken, and immediately centrifuged at 3000 min⁻¹. The centrifuged solution was transferred in its

entirety to a 20 mL vial, from which, for the purposes of chemical analysis, one milliliter was taken and diluted in a vial with 9 milliliters of distilled water.

At the end of each test, the sample solutions were analyzed by atomic absorption on a Perkin-Elmer AAnalyst 200. First, gold was determined on concentrated test samples. To do this, the gold lamp was turned on and allowed to heat for 15 minutes, then the absorbances of the standards (1-5, 7.5, 10, 15, and 20 mg Au/Ton) were read and recorded, and finally the absorbance was read in the test samples. The gold concentrations in the problem samples were obtained from the standard calibration curve. The silver determinations were performed following the same procedure, except that the analytes read were the diluted ones.

RESULTS

The first test results shown are from the leaching of slag in the cyanide system, which is considered the reference system, characterized by moderate dissolution rates of around 3.25 mg/cm²/h (equivalent to a penetration of 1.68 microns/h), as well as in the TACu system, which, as mentioned, can reach speeds 18 to 20 times higher than this [23]. The gold recovery versus time results in these two systems are summarized in Figure 9.

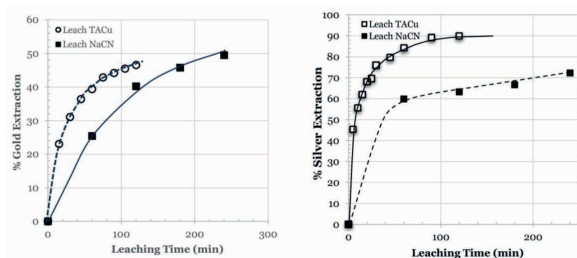


Figure 9. Precious metal recoveries from slag in cyanide leaching systems (0.060 M NaCN and pH=11) and thiosulfate-ammonia-copper systems: (a) gold and (b) silver.

Once these preliminary tests had been carried out, the effect of adding certain ions to the TACu leaching system was evaluated. Previous studies on a rotating silver disc and a flotation concentrate from the local mining district showed that the addition of ions such as lead, calcium, and zinc in low concentrations can catalyze the dissolution rate of silver. However, it should be noted that, due to the complexity of the system, the rates observed depend on the concentrations of thiosulfate, ammonia, copper, and pH, i.e., on the chemistry of the solution [24, 25].

In terms of the ABDB principle, according to which cations and anions combine preferentially, i.e., hard with hard and soft with soft. Thus, if $S^{(2-)}$ is a soft anion, the resulting compounds with soft cations are expected to be characterized by high mass action constants, i.e., they are more stable, e.g., with Au(I), $Au(S_2O_3)_2^{3-}$ should be a highly stable compound. In this case, the high softness of $S^{(2-)}$ accounts for, or in a way explains, the high stability constants of Au(I) with ligands such as $HS^{(-)}$ and SO_3^{2-} , among others [26].

Although there are countless ions with soft characteristics, in this study, due to time constraints and the electrochemical characterization performed, only tests were carried out with zinc, adding or maintaining concentrations of 100, 200, and 400 mg/L. The results of these tests, as well as the reference (without adding zinc), are summarized in Figure 10.

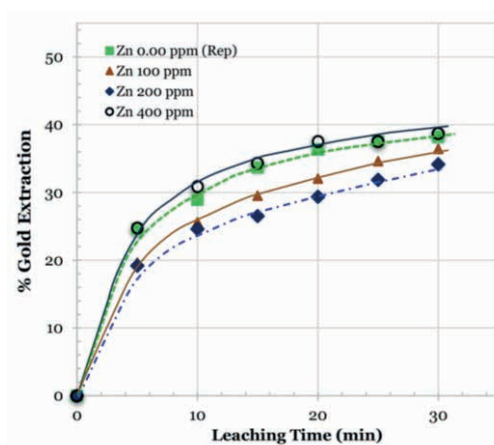


Figure 10. Gold recoveries from slag in the TACu system: 0.10 M, 0.40 M NH_4 , 0.005 M Cu, and 0-400 ppm Zn.

CONCLUSIONS

The partial study reported here has focused on determining important characteristics of the dissolution of gold contained in smelting slag in the thiosulfate-ammonia-copper system, as well as familiarizing ourselves with the behavior of the system in terms of its stability and the effect of adding zinc (a soft ion) as a catalyst for the dissolution rate of precious metals. Most of the effort and time was devoted to defining a reference framework in order to subsequently determine the dissolution rates in the solvent system of interest. In conclusion, we can summarize the following: compared to cyanide, the dissolution rates in the TACu system are higher. Below 200 ppm of zinc, there does not appear to be a catalytic effect on the rate.

The textural characterization of the metal species in the slag reveals that nearly 99% of the precious metals are found in alloys, mainly Ag-Au (75%) and Au-Ag-Cu (22%). Of these, approximately 45% are present as inclusions smaller than 5 microns embedded in silicate phases, which hinders their direct exposure to the leaching agent. This microdispersion, combined with a silver:gold ratio of 2.6:1, results in slow dissolution kinetics.

From an electrochemical point of view, tests carried out with graphite paste electrodes—prepared from a gravimetric concentrate of slag with high gold and silver grades, and maintaining the aforementioned Ag: Au ratio—showed no signs of oxidation in the TACu(II) system. This behavior indicates that the oxidizing potential of the system is insufficient to oxidize the metal alloy, especially under the passivation conditions imposed by the presence of excess silver.

These results suggest that, in order to achieve efficient recovery of precious metals from this type of slag, it will be necessary to optimize the leaching oxidizing system. This could include the use of stronger oxidizing agents or conditions that favor the destabilization of the

alloys, as well as prior mechanical activation strategies that expose the metal inclusions contained in the silicate matrix.

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