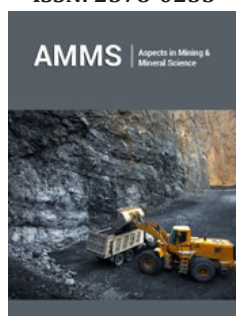


Hermetic Oxidization/Cyanidation Leaching and Elimination of Contaminants from the Calcine for the Recovery of Gold and Silver by Electrocoagulation Technology

ISSN: 2578-0255



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Submission:  January 02, 2025

Published:  January 21, 2025

Volume 13 - Issue 1

How to cite this article: Jose Refugio Parga Torres, Víctor Vásquez Vazquez, Gregorio González Zamarripa, Nelson Oshogwue Etafo, Mario Rodríguez Reyes and Dagoberto Vázquez Obregón. Hermetic Oxidization/Cyanidation Leaching and Elimination of Contaminants from the Calcine for the Recovery of Gold and Silver by Electrocoagulation Technology. *Aspects Min Miner Sci.* 13(1). AMMS. 000804. 2025.

DOI: [10.31031/AMMS.2025.13.000804](https://doi.org/10.31031/AMMS.2025.13.000804)

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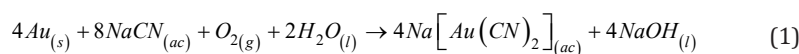
Abstract

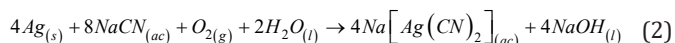
Traditional hydrometallurgical gold and silver extraction involves a 48-hour cyanide process (using Sodium Cyanide) followed by the seven-day Merrill-Crowe cementation and smelting process. This research explores alternative methods. Firstly, a simultaneous oxidation-cyanidation process in a pressurized reactor achieved 95% gold and silver recoveries within 90 minutes. Secondly, for metal recovery, an innovative Electrocoagulation (EC) technology was employed. EC eliminates the need for expensive reagents and effectively treats low-concentration solutions, such as those from heap leaching. The EC reactor demonstrated 98% gold and silver recovery in 10 minutes with 99% current efficiency. This study presents a promising, cost-effective, and environmentally friendly approach to gold and silver recovery.

Keywords: Gold and silver extraction; Cyanidation; Mineralization; Metals; Electrocoagulation technology

Introduction

Most metals, including gold, silver, and platinum, naturally exist as compounds within complex mixtures of sulfides, oxides, carbonates, and silicates [1]. Mineral beneficiation typically involves crushing and grinding the ore to liberate valuable minerals from the surrounding rock (gangue) [2]. Subsequently, these minerals are collected through various methods, including gravity separation, magnetic separation, flotation, or, in the case of oxidized ores, direct cyanidation [3-5]. Cyanidation is generally the most common process for recovering gold and silver minerals. This is due to the high selectivity of free cyanide for both metals and the high stability of the cyanide complexes $Au(CN)_2^-$, $k = 2 \times 10^{38}$ and $Ag(CN)_2^-$, $k = 2 \times 10^{20}$ in solution [6]. Equations 1 and 2 depict the gold and silver dissolution reactions in a cyanide solution with a pH exceeding 11. This alkaline environment is crucial to neutralize acid produced during cyanidation, thereby inhibiting cyanide degradation and the formation of toxic Hydrogen Cyanide (HCN) gas [7].





Cyanide leaching reactions occur with minimal oxygen, and the rate-limiting step shifts from cyanide to oxygen diffusion as cyanide concentration increases. Leaching kinetics are influenced by factors such as grain size, agitation, temperature, pressure,

and the presence of foreign ions, cyanides and carbon. To control impurity levels (zinc, copper, lead), a portion of the leach solution must be regularly discarded while the majority is recycled [8]. The cyanidation process, patented by Mac Arthur et al. involves the dissolution of sulfide minerals in the presence of oxygen and cyanide [9], as illustrated in Figure 1.

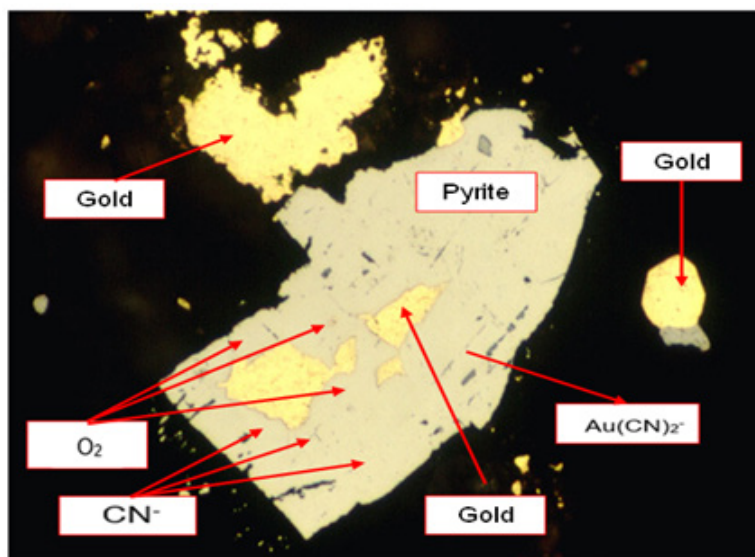


Figure 1: Chemical mechanism of de dissolution of gold in pyrite particle with cyanide.

This analysis suggests that roasting sulfide concentrates to improve gold and silver dissolution through cyanide leaching is problematic due to environmental concerns related to arsenic and SO₂ emissions. This research demonstrates that precious metal recovery exceeding 96% for both gold and silver can be achieved within 90 minutes in a hermetic steel reactor under optimized conditions: low oxygen pressure, low temperature, and high cyanide concentration with 0.5MPa oxygen pressure, 75 °C, and

20g/L sodium cyanide respectively [10-12]. Literature reviews reveal that pressure oxidation preceding cyanidation has been the most widely adopted method for refractory gold treatment in the last 50 years [13]. This conventional approach, utilized globally, involves oxidizing the ore before cyanidation, typically requiring 48 hours (Figure 2). This study innovates by integrating oxidation and cyanidation into a single, 90-minute solar-powered leaching process [12].

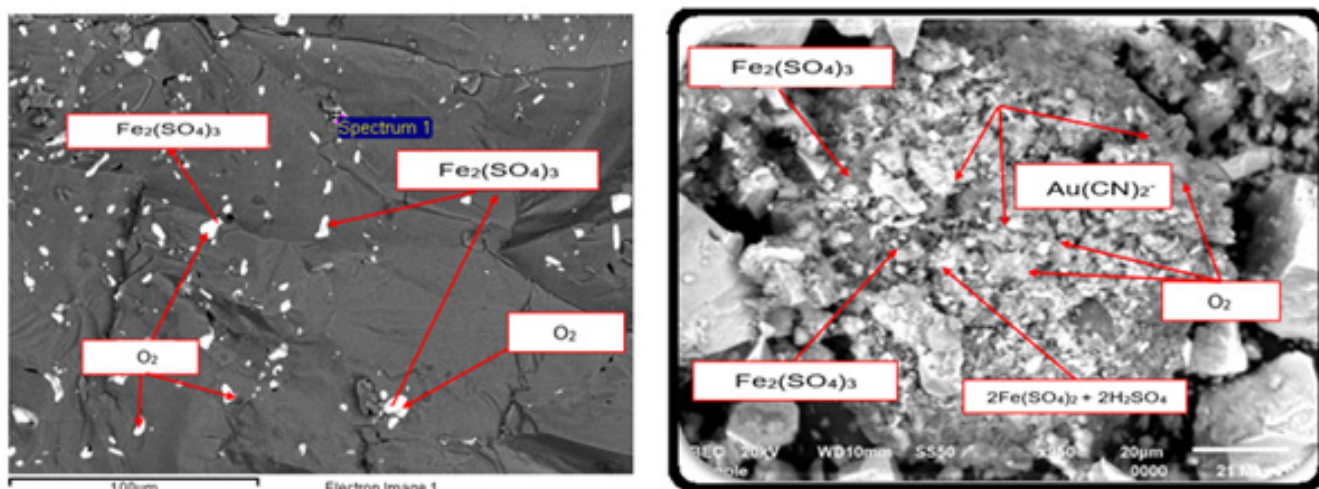


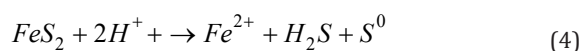
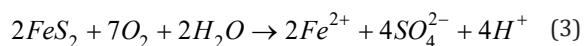
Figure 2: Mechanism of gold and silver dissolution with CN and oxygen injection.

High cyanide concentrations are necessary to complete reactions with refractory metals (Cu, Zn, Fe). Pressurized oxygen increases dissolved oxygen, driving the reaction forward (Figure 3). Oxygen dissolves readily soluble sulfides under mild oxidative

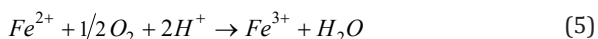
conditions, forming ferric sulfate and leaching gold and argentite from the refractory matrix [6,10,11]. The chemical mechanism for the simultaneous cyanidation and oxidation follows the reaction equation below [10]:



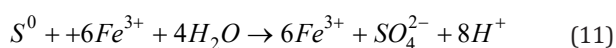
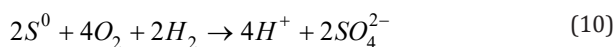
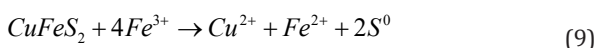
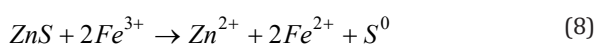
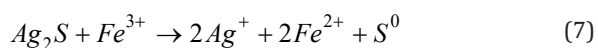
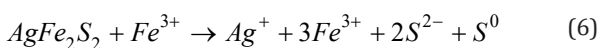
Figure 3: Industrial autoclave processing of silver and gold leached from pyrite and argentopyrite, utilizing solar and wind-generated electricity.



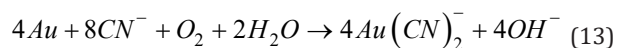
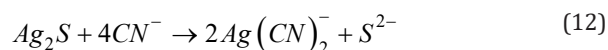
Ferrous produced in equation (3) and (4) are subsequently oxidized to Ferric ions



Ferric ions can oxidize silver sulfide, argentite, pyrite, pyrrhotite, sphalerite, and chalcopyrite.



Equations (12) and (13) depict the formation of a porous, non-protective elemental sulfur layer. This allows cyanide and dissolved oxygen to reach and react with previously inaccessible gold, silver, and electrum.



A Mexican industrial plant recovered 96% of precious metals in 90 minutes using a chemical process under low conditions (60-80 °C, 40-80 psi) in a steel autoclave [10,11]. Prior to this, the only operational plant utilizing simultaneous oxidation/cyanidation employed by ARES Mining Unit in Peru features two cylindrical ILR 5000 reactors. This process intensively dissolves gold and silver using hydrogen peroxide, 30-micron particles, and a high cyanide concentration (16%) within a semi-hermetic environment. The plant processes 18 tons of 98% reground concentrate (less than 74 microns) at 30% solids within the reactor for 50 hours. Table 1 presents the concentrate grade and final tailing chemical analyses after intensive treatment in the ILR 5000 reactors. Data analysis revealed 96% gold and 95% silver dissolution in the concentrate. Below is an image of the two reactors operating in a mining in Peru (Figure 4).



Figure 4: Photograph of the ILR 5000 reactor, with the feed tank and the 20m³ solid/liquid separation tank.

Table 1: Chemical analysis of plant concentrates and tailings.

	Au (Gr/Ton)	Ag (Gr/Ton)	Cu%	Fe%	Pb%	Zn%	Insol.
Concentrate	41	15000	0.51	21.75	7.8	13.42	23.76
Tailing	2	657	0.28	2.64	6.3	14.36	25.80
Recovery	96.37%	95.23%					

Gold and silver mineralization exhibits diverse forms, from free gold pebbles to finely disseminated gold within sulfide minerals, including lattice-bound occurrences. Cyanide-bearing ores may also be present. Consequently, a standardized recovery process is infeasible. Achieving high gold and silver recoveries, producing 99% pure Doré, often necessitates employing multiple processing techniques [11].

The electrocoagulation technology theory

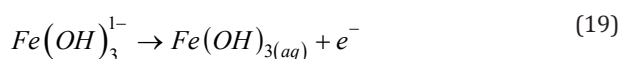
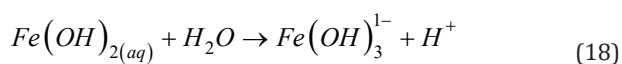
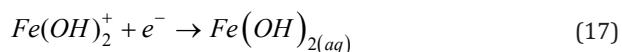
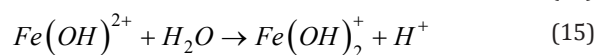
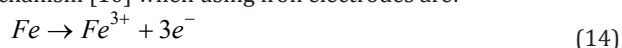
Electrocoagulation (EC) is a promising technique for recovering precious metals like gold and silver from cyanide leach solutions. It involves using sacrificial anodes (usually aluminum or iron) that dissolve into the solution when an electric current is applied. This creates metal ions that react with the cyanide complexes of gold and silver, forming insoluble precipitates that can be easily filtered and collected [14]. The process is considered environmentally friendly as it does not require the addition of chemical reagents and produces minimal waste. Additionally, EC can treat cyanide-containing wastewater, further reducing its environmental impact [15].

The mechanism of this innovative process includes

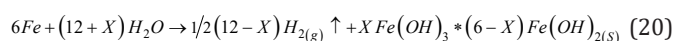
- A. Electrode dissolution:** When an electric current is applied, the sacrificial anodes dissolve, releasing metal ions (Fe^{2+} or Al^{3+}) into the solution.
- B. Hydroxide formation:** These metal ions react with hydroxide ions (OH^-) generated at the cathode, forming metal hydroxide precipitates.

- C. Metal precipitation:** Gold and silver ions in the solution are attracted to the negatively charged metal hydroxide flocs and get adsorbed onto their surface.
- D. Floc growth and settling:** The flocs grow larger, capturing more metal ions, eventually settling to the tank's bottom.
- E. Metal recovery:** The settled sludge containing the recovered gold and silver can be further processed to extract the pure metals.

The chemical reactions that have been proposed to describe EC mechanism [10] when using iron electrodes are:



Overall reaction.



During Electrochemical Processes (EC), the experimental medium typically experiences a pH increase. This leads to the

formation of Green Rust $[x\text{Fe}(\text{OH})_3 \cdot (6-x) \text{Fe}(\text{OH})_2 (\text{s})]$, which remains suspended in the aqueous phase as a gelatinous material. This suspension can effectively remove gold and silver from cyanide-rich solutions through complexation or by attracting magnetic

nanoparticles via electrostatic forces, followed by coagulation and flotation. To enhance the EC process, bipolar electrodes are commonly employed [8], as illustrated in Figure 5 for a reactor with iron electrodes [16,17].

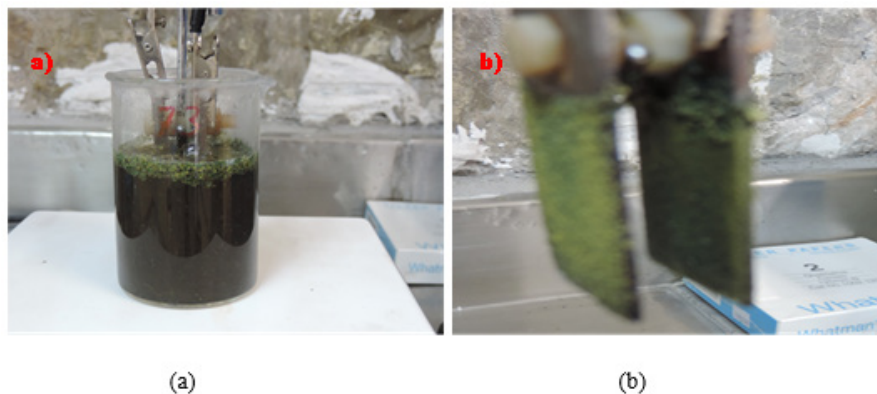


Figure 5: a) Generation of green rust in the iron electrodes b) hydroxide magnetic material attached to the iron electrodes.

Experimental Methods and Materials

The chemical analysis

Gold, silver, zinc, iron, copper, and arsenic concentrations were determined by ICP-AES. Free cyanide was measured directly by titration, while total cyanide was determined by titration after distillation.

The pressure leaching experiments

Pressure leaching experiments were performed in a 4L Parr autoclave equipped with an impeller, thermowell, pressure gauge, and gas inlet/outlet lines. Samples, ground to 84% passing 40 μm , were leached in aqueous slurries. Experimental parameters, including NaCN concentration, lixiviation time, temperature, particle size, pH, and pulp density, were varied to investigate their influence on leaching behavior [10].

Variables analysis for the dissolution of gold and silver

One kilogram of argenopyrite concentrate was ground in a ball

mill for 3 hours at a 60% solids pulp density. The ground product was then subjected to pressure oxidative cyanidation in a 1-gallon Parr reactor for 90 minutes at 60 °C and a 25% solids concentration.

The Electrocoagulation (EC) experiments

Electrocoagulation (EC) experiments were conducted in a 400ml beaker using two 6cm x 3cm carbon steel electrodes spaced 5mm apart. A universal AC/DC adapter provided the power source. pH was measured with a VWR scientific 8005pH meter. Gold and silver adsorption onto iron species was investigated using pregnant cyanide solutions, with analysis by ICP-AES. Solution conductivity was adjusted to 1g/L NaCl. To characterize the formed iron species, X-Ray Diffraction (XRD) and Scanning Electron Microscopy with Energy Dispersive X-ray spectroscopy (SEM/EDX) were employed. Solutions and solids were separated by filtration. The EC sludge was dried and characterized. Figure 6 illustrates the experimental setup, including current and voltage measurement. All experiments were conducted in triplicate, with the average value reported [18].

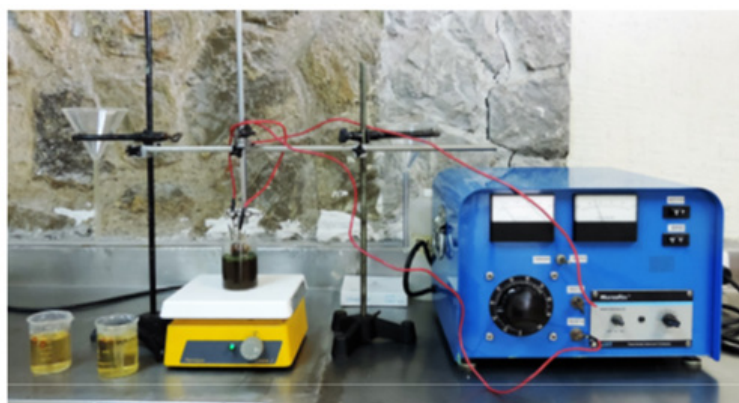


Figure 6: Experimental setup of the reactor, amperemeter and electric agitate stove.

Mineral samples from different parts of the silver concentrate were prepared for mineralogical analysis. Elemental composition was determined by Atomic Absorption Spectroscopy. X-ray diffraction was employed to identify mineral species. Scanning Electron Microscopy with Energy Dispersive X-ray spectroscopy (SEM-EDX) was used to investigate the morphology and distribution of gold and silver within the concentrate particles.

Results and Discussion

Chemical analysis of the concentrate to be leached

The representative concentrate head sample was analyzed by two laboratories to determine its elemental composition in the concentrate, the results show in Table 2. The primary elements identified were gold and silver, predominantly associated with pyrite, argentopyrite, galena, and chalcopyrite.

Table 2: The mineral composition obtained from the concentrate.

Laboratory	Au (gr/ton)	Ag (kg/ton)	% Pb	% Zn	% Cu	% Fe	% As
ERSA GLOBAL	86.0	4.723	1.70	2.18	0.83	24.33	0.13
TECNM-ITS	84.8	4.677	1.65	2.17	0.36	24.90	0.12

X-Ray study of the sample

To determine the mineralogical composition of the ore samples, a representative sample was prepared and analyzed using Oxford X-ray diffraction equipment. The identified minerals include P-Pyrite (FeS₂), G-Galena (PbS), E-Sphalerite (ZnS), Si-Quartz, Al-Alumina, C-Chalcopyrite, A-Argentite, and AP-Arsenopyrite

(FeAsS). Silver-bearing minerals are: Argentite, Galena, and Pyrite. Gold-bearing minerals are: Pyrite and Chalcopyrite. Gold and silver-bearing minerals (electrum) are: Pyrite, Quartz, and Argentite. Gangue minerals consist of SiO₂, Al₂O₃, and CaCO₃ (gray-colored material). The following section (Figure 7) presents the X-ray diffraction analysis of the concentrate.

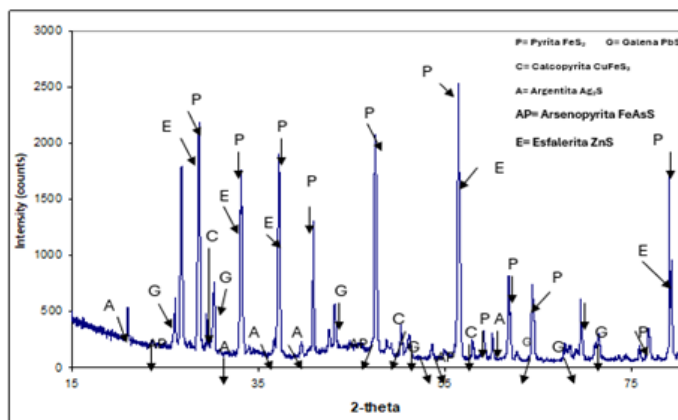


Figure 7: X-ray diffractogram of the argentopyrite concentrate.

Scanning electron microscope (SEM) studies

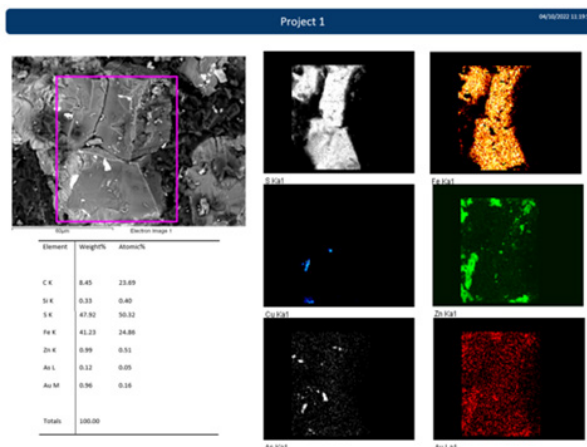


Figure 8: SEM Image mapping of the concentrate.

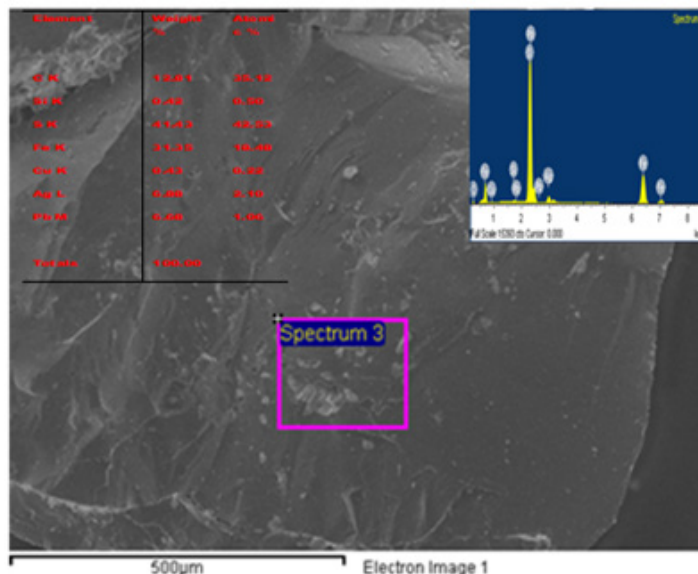


Figure 9: The SEM image with EDAX results of the concentrate..

SEM mapping revealed a uniform distribution of gold and silver within the argentopyrite matrix. Impurities of zinc, iron, and copper were also observed in the matrix of the ore. The EDAX results are presented in Figure 8. Furthermore, the SEM image in the following (Figure 9), also exhibited a similar behavior. The EDAX analysis confirmed that some pyrite grains contain gold and silver. Additionally, galena exhibited silver content, and argentite particles were observed interspersed within pyrite.

Pressure leaching results

The results of the variable analysis: Three recognition tests were conducted using only cyanide. Utilizing previously determined optimal parameters (10,11), an autoclave was employed to generate pregnant leach solution for subsequent gold and silver recovery experiments via Electrocoagulation technology. Table 3 presents the studied and reported variable parameters [10,11].

Table 3: Optimization of the pressure oxidizing cyanidation of the concentrate in a single stage.

Stage of this Research	Variables to Analysis
Analysis of Cyanide Consumption.	10, 20, 30, 40 and 50Kg/Ton of NaCN
Analysis of the Cyanidation Time.	20, 30, 45, 60, 75 and 90 minutes
Analysis of the pH Effect.	9.5, 10, 10.5, 11.0pH
Analysis of the Effect of the % of Solids in Pressure Cyanidation.	15, 20, 25 and 30% of Solids
Analysis of the Temperature Effect.	40, 50, 60, 70 and 80 degrees Celsius
Analysis of the Pressure Effect.	40,50, 60 and 80lbs/inch
Effect of the particle size or particle size, (-200 Mesh).	70, 80, 100% or 40-10microns in diameter

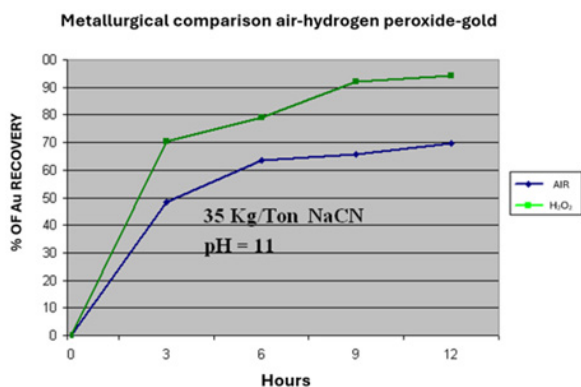


Figure 10: The metallurgical comparison of air-hydrogen peroxide-gold.

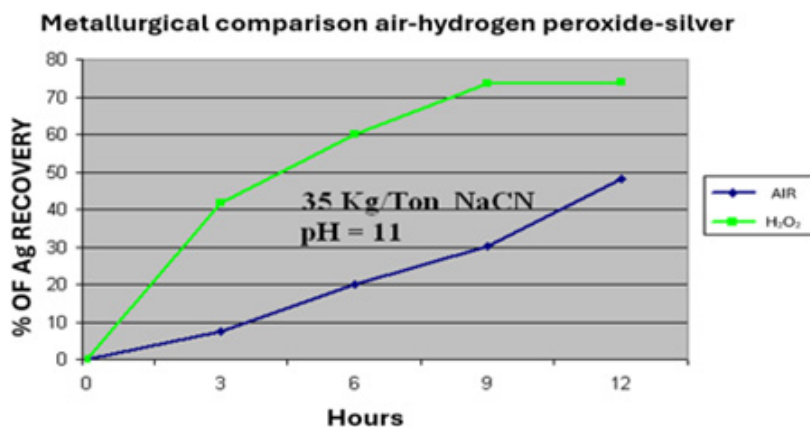


Figure 11: The metallurgical comparison of air-hydrogen peroxide-silver.

Preliminary results of the cyanidation of the ore reground concentrate for 180 minutes are shown below; and by analyzing these results, it is determined that by using hydrogen peroxide as an oxidant for the cyanidation process, the dissolution of gold and silver increases by 20%, compared to traditional cyanidation that uses air. This results in Figure 10, demonstrate that oxygen is very important for the dissolution of gold (Figure 11).

As determined in the figures, using H₂O₂ as an oxidant for the cyanidation process increases the dissolution of gold and silver by 20% compared to traditional cyanidation using air. Then in order to have pregnant rich solution for the EC test, we used the best variables determined for the dissolution simultaneous of gold and silver for the argentopyrite concentrate describe on references [10,11] and the conditions for the leaching test are show in the following Table 4. Because now we have different concentrate is necessary to do new tests in order to determine the best concentration of cyanide

for have leaching dissolution of gold and silver more than 95 % for both. The results for this variation of cyanide concentrate are shown on Table 5. Also, we used small diameter of particles below 20 microns. By analyses these results, the best dissolution for gold and silver was 50 kilograms per ton of concentrate. These results demonstrate that gold and silver dissolution reached 96% and 94%, respectively, when using particle sizes below 20 microns and a pressure oxidative cyanidation time of 90 minutes or less.

Table 4: Conditions for the pressure/temperature leaching test.

Conditions for the Leaching Test	
Temperature=50 °C	Ca1=16kg/ton of concentrate
Pressure=50Lb/inch ²	PbO=2gr/ton of concentrate
Time=60 minutes	R.P.M.=600
% of Solids=25, pH=11.0	NaCN=variable

Table 5: Results of the variation of the cyanide concentration.

Variation of CN Concentration	% Au Recovery	% Ag Recovery	CN Consumption kg/Ton	Lime Consumption kg/Ton
35	96	90	16	7
40	96.5	91	18	8
45	97	92	20	8
50	96	94	22	9

Electrocoagulation results

Electrocoagulation (EC) experiments were conducted at room temperature using pregnant-rich solution from the autoclave and the variables used are shown in Table 6. Each EC test was conducted using these variables, and the results are presented in Table 6. The result of the electrocoagulation experiment showed 98.3% and 94.7% recovery of Au and Ag respectively after 10 minutes of residence time as shown in Table 7. Simultaneous adsorption of copper, lead, zinc, and iron onto the iron sludge was observed after the electrocoagulation experiment. These results, presented in the table inside the SEM image indicate moderate selectivity of the EC

process towards copper, lead, zinc, and iron ions. Figure 12 shows an SEM image of the sludge product at pH9.

Table 6: summarizes the experimental variables.

Parameter	Value
pH	9
Time	10 minutes
Current	15 Amperes
Voltage	40 voltage
Space	8mm

Table 7: EC test results at different treatment times with an initial pH of 9.

Time of Treatment (min)		mg/l						% Recovery					
		Au	Ag	Cu	Pb	Zn	Fe	Au	Ag	Cu	Pb	Zn	Fe
Initial	pH	1.78	3.60	13818	9.16	18229	2092.30						
2	9	0.96	2.57	13853	8.98	18173	2160.67	44.94	28.61	5.59	-5.77	0.49	3.95
4	9.5	0.51	1.28	14228	7.98	18238	2038.00	71.34	64.44	3.03	6.01	0.13	9.4
6	10.0	0.32	0.93	14176	8.16	18219	2294.00	82.02	74.16	3.39	3.89	0.23	-1.98
8	10.5	0.21	0.42	14568	7.82	18245	2440.00	88.2	88.33	0.72	7.89	0.09	-8.47
10	11	0.03	0.19	14323	8.21	18175	2385.00	98.31	94.72	2.39	3.3	0.47	-6.03

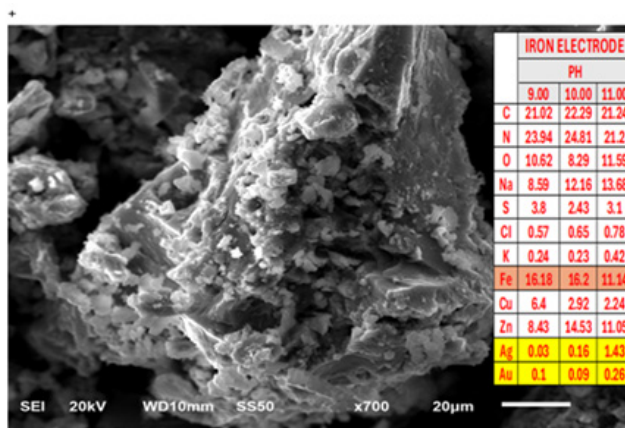


Figure 12: SEM image of the sludge, revealing the selective recovery of gold and silver alongside impurities of copper, zinc, and iron.

The primary constituents of the sludge are magnetite, goethite, and lepidocrocite-magnetic materials that encapsulate gold and silver. The accompanying EDX analysis provides elemental composition data within the image. The EC sludge contains gold, silver, and copper. Sulfoxide extraction may be a viable method for recovering these precious metals [19].

Conclusion

Table 8: Optimal conditions.

Optimal Conditions	
Concentrate Re grind=less than 20microns	NaCN=50kg/ton of concentrate
Temperature=50 °C	Lime=16kg/ton of concentrate
Pressure=50Lb/inch ²	PbO=20gr/ton of concentrate
Time=60 minutes	R.P.M.=600
% Solids=25, pH=11.0	Particle=20 microns

X-ray diffraction and Scanning Electron Microscopy analyses indicate that silver is primarily associated with argentopyrite, while gold is associated with pyrite. Gold and silver values are closely associated with argentopyrite, pyrite, galena, sphalerite, and chalcopyrite. Fine grinding is necessary to liberate gold and silver particles, enhancing their dissolution in the cyanide-oxygen solution. Pressure oxidative cyanidation tests in the autoclave are recommended with a 25% solids content. These lower solids concentration results in a less viscous pulp, facilitating oxygen

diffusion and improving the reaction efficiency of gold and silver with cyanide.” The following parameters were determined to be optimal and are show on Table 8.”

Under these conditions, Single Stage Pressure Oxidizing Cyanidation is feasible in a stainless-steel reactor, offering a cost advantage over titanium reactors. Utilizing a particle size predominantly below 20 microns and a cyanidation time of 90 minutes or less, gold and silver dissolution reached 97% and 94%, respectively. The electrocoagulation technology showed an efficient recovery for gold and silver while eliminating the Cu, Zn, and Fe present.

Acknowledgment

The authors acknowledge the TNM-Institute of Technology of Saltillo, CONAHCYT for their support while carrying out this research.

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