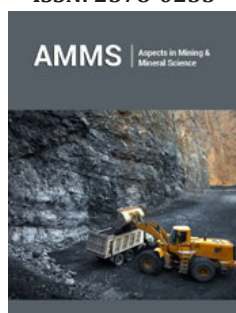


# Pollutants Removal and Electrocoagulation Processes for High Quality Precipitate from Cyanide-Contaminated Solutions

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## Abstract

Cyanidation, a primary gold and silver recovery method since the 1880s, dissolves these metals in alkaline cyanide solutions. Subsequent Merrill-Crowe processing recovers the metals. However, large-scale operations often yield low-quality precipitates due to co-precipitation of copper and lead. This increases zinc dust consumption, smelting flux requirements, crucible lifespan, and lowers Dore quality. In this study, two processes are used to clean the high concentrations of zinc, copper, lead and iron cyanide ions from the barren solutions of the Merrill-Crowe process, to produce in the next step a Dore of high quality (99%). The first step in this process is to induce nucleated precipitation of copper, zinc, lead, and silver in a conical hermetic reactor using sodium sulfide as a reagent precipitator and sulfuric acid for pH control, while also regenerating the NaCN with sodium hydroxide. The results reveal that pH has a considerable effect on zinc and copper cyanide removal efficiency, with the ideal pH range being 8 to 9. At this pH, lead, copper, and zinc cyanide removal efficiency was up to 70%, whereas cyanide linked with copper, zinc, and iron cyanide complexes recovered at 80%. The second step is used for the total elimination of these cyanide complexes and lead ions from the barren solution with an attractive electrocoagulation process. In this hermetic reactor, all the remaining complex metal ions and cyanide recoveries were 99%. Then, the clean solutions passed through the cementation step to give a first-order reaction which was faster when compared to the barren with cyanide complex of copper/zinc. Finally, this treatment procedure was successfully practiced at Minera Labri / William, La Sorpresa and Bacis Maning Company in México.

**Keywords:** Sulphide precipitation; Electrocoagulation; Merrill-crowe; Dore and gold/silver

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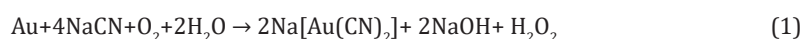
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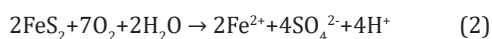
## Introduction

Gold-silver ore cyanidation faces challenges due to high cyanide consumption and difficulties in controlling the leach process, especially when dealing with variable levels of cyanide-soluble copper, zinc, and iron in the ore. Gold dissolution in cyanide solutions is widely recognized as following the Elsner reaction shown in Equation (1).

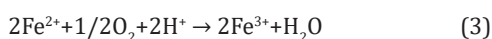


Elsner's equation highlights the critical role of oxygen in gold dissolution. A 4:1 molar ratio of cyanide to oxygen is essential. Maintaining an alkaline pH 10-11 with lime is crucial [1]. Understanding the host minerals of gold and silver is vital for effective cyanidation. Minerals like pyrite react with oxygen, producing ferrous ions, sulfate ions, and elemental sulfur, which can influence the dissolution and liberation of gold and silver [2].

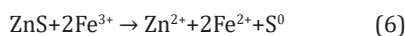
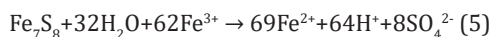
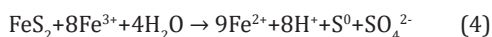
The primary reactions are:



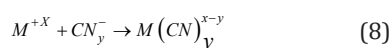
The ferrous ions from reaction (2) are oxidized to ferric ions



Ferric ions primarily contribute to sulfide leaching, also oxidizing ores like pyrite, pyrrhotite, sphalerite, and chalcopyrite:



Effective gold and silver leaching require sufficient free cyanide. Furthermore, many common copper, zinc, and iron minerals are soluble in the leaching solutions used in gold cyanidation [3]. The following chemical equation represents the complex ion formations with the cyanide:



Cyanide forms complexes with various metallic species, particularly transition metals. These complexes exhibit diverse stability and solubility. The Merrill-Crowe process, traditionally used for gold and silver recovery, faces challenges due to

increasingly stringent purity standards for Dore metal. To address this, there's growing interest in developing a novel process to remove copper, zinc, lead, and cadmium cyanide ions from pregnant solutions before cementation [4]. Heavy metal contamination is a significant issue in wastewater from mining operations processing ferrous and non-ferrous metals. To address this, transition metals are commonly removed through precipitation [5]. Industries also employ various unit operations like coagulation, flocculation, ion exchange, and membrane processes. Precipitation, specifically hydroxide, carbonate, and sulfide precipitation, is a common technique, but it's time-consuming and requires complex setups. Each step necessitates separate tanks and multiple pH adjustments with added chemicals like acid, lime, or caustic and polymeric flocculants [6]. Substantial sludge is a byproduct of this process, requiring disposal. Hydroxide precipitation, a simple, cost-effective method with easy pH control, is commonly used [7]. Figure 1 illustrates the solubility of metal hydroxides and sulfides. Both precipitation methods involve reacting metal cations with  $\text{OH}^-$  or  $\text{S}^{2-}$  ions to form insoluble compounds. To optimize gold and silver recovery through zinc cementation, the optimal pH for selective precipitation of interfering metals (like zinc and copper) is crucial. Sulfide precipitation at pH 12 to 8 effectively removes these metals [8].

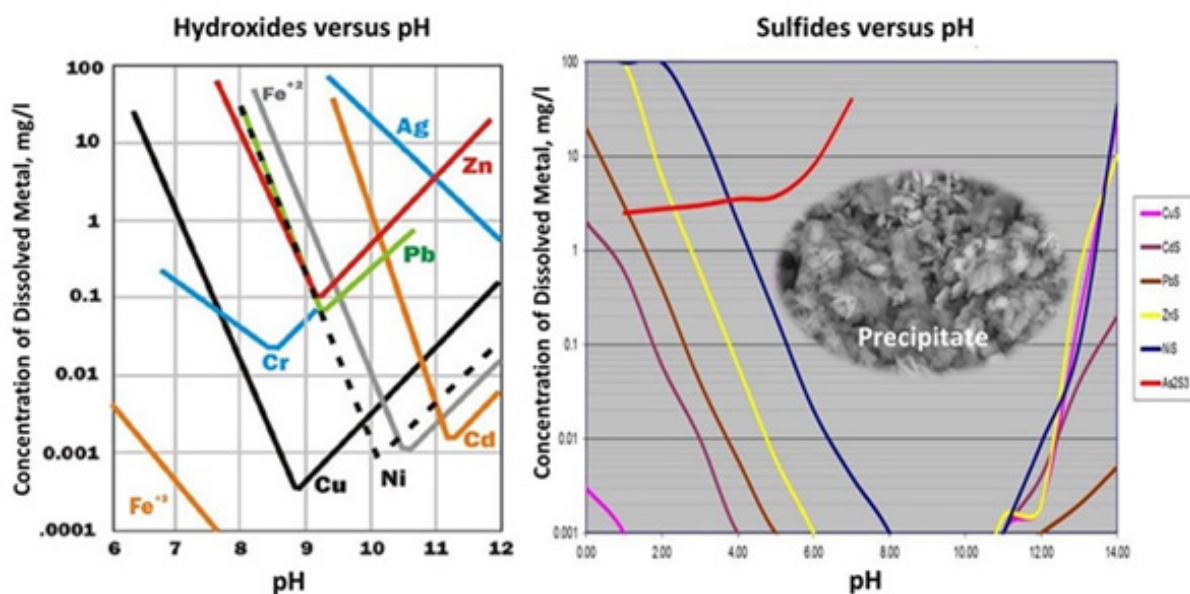


Figure 1: Solubility of metal hydroxide and sulfides.

Sulfide precipitation is a preferred method for producing high-quality Dore due to its lower solubility compared to hydroxide precipitates. During electrochemical processes, metal ions can react with hydroxide ions to form insoluble hydroxides. However, metals like Ba, Ca, Mg, Sr, Na, and K form soluble hydroxides, limiting their removal. Figure 1 illustrates the pH-dependent solubility of various metal hydroxides. As pH increases, metal hydroxides precipitate in a specific order: Cr, Cu, Zn, Ni, and Cd. This order aligns with the decreasing trend of average removal efficiency for these metals,

as shown in Table 1 [9]. The Pb anomaly likely stems from the limitations of solubility charts, which assume pure substances and ideal conditions, unlike wastewater. While metal hydroxides are the primary removal mechanism, trapping in floc or incorporation into iron poly oxyhydroxides also contributes. However, this doesn't fully explain non-removal instances or other potential cellular processes [10]. Metal removal efficiency is influenced by final pH, as discussed in Figure 1, but other factors also play a role.

**Table 1:** The characteristics of the barren solution before and after treatment.

Parameters	Units	Before Treatment
Color	-	Yellow
pH	Units of pH	13.2
Conductivity	μS/cm	125.400
Chlorides	mg/l	90.500
Density	g/ml	1,156
Cyanides	mg/l	10.000
Copper	mg/l	7.085
lead	mg/l	8.86
Zinc	mg/l	8.491
Iron	mg/l	4.774

The 2 steps to convert cyanide to HCN gas: 1) free cyanide: Lowering the pH to about 8.0 converts 99% of free cyanide to HCN gas and 2) Formation of metal-cyanide complexes: A more acidic pH is required to break down these complexes and release HCN gas, and HCN gas is stripped from the barren solutions and then absorbed by an NaOH solution according to reaction (9), [11].



This research aims to explore Electrocoagulation (EC) as a novel, chemical-free method to minimize copper, lead, zinc, and iron in barren solutions from the Merrill Crowe process. EC offers a promising alternative to traditional methods, potentially overcoming their limitations. However, its full potential remains untapped due to technical and economic considerations [12-14].

### The theory of electrocoagulation

Electrocoagulation utilizes sacrificial anodes (aluminum or iron) to generate polyvalent metal cations. These cations neutralize negatively charged contaminants, causing them to coagulate. Simultaneously, electrophoretic forces draw particles toward the electrodes, further accelerating coagulation. The produced electrolysis gases (hydrogen and oxygen) also contribute to flocculation, enhancing the overall removal efficiency [14]. Electrocoagulation (EC) is a process that leverages electrochemical reactions to remove pollutants from wastewater. Metal ions react with hydroxide ions generated at the cathode to form insoluble metal hydroxides. These hydroxides act as coagulants, neutralizing negatively charged colloidal particles and facilitating their agglomeration. The efficiency of EC depends on the preferential oxidation-reduction reactions occurring at the electrodes. Bipolar electrode configurations are commonly used in EC systems due to their simplicity and effectiveness in removing heavy metals. By minimizing the number of electrodes connected to the power source, bipolar electrode arrangements reduce energy consumption and simplify maintenance [12].

### Cementation theory for recovery precious metals

After removing heavy metals, the clarified pregnant solution containing precious metals is deoxygenated under vacuum. Gold and silver are then precipitated using zinc merrillite. The cementation reaction kinetics for noble metal ions often follow a first-order diffusion process (10), and the reaction rate constant (k)

can be calculated using the standard first-order rate equation [4]:

$$\frac{dc}{dt} = \frac{-kAC}{V} \quad (10)$$

With constant k and area, equation (11) integrates into the first-order expression.

$$\log \frac{C}{C_0} = -\frac{kAT}{2.3V} \quad (11)$$

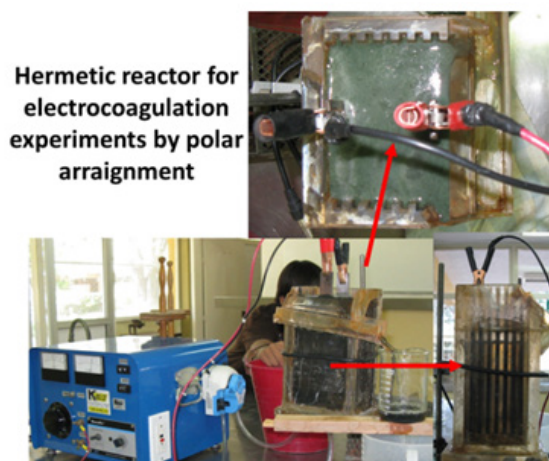
## Materials and Methods

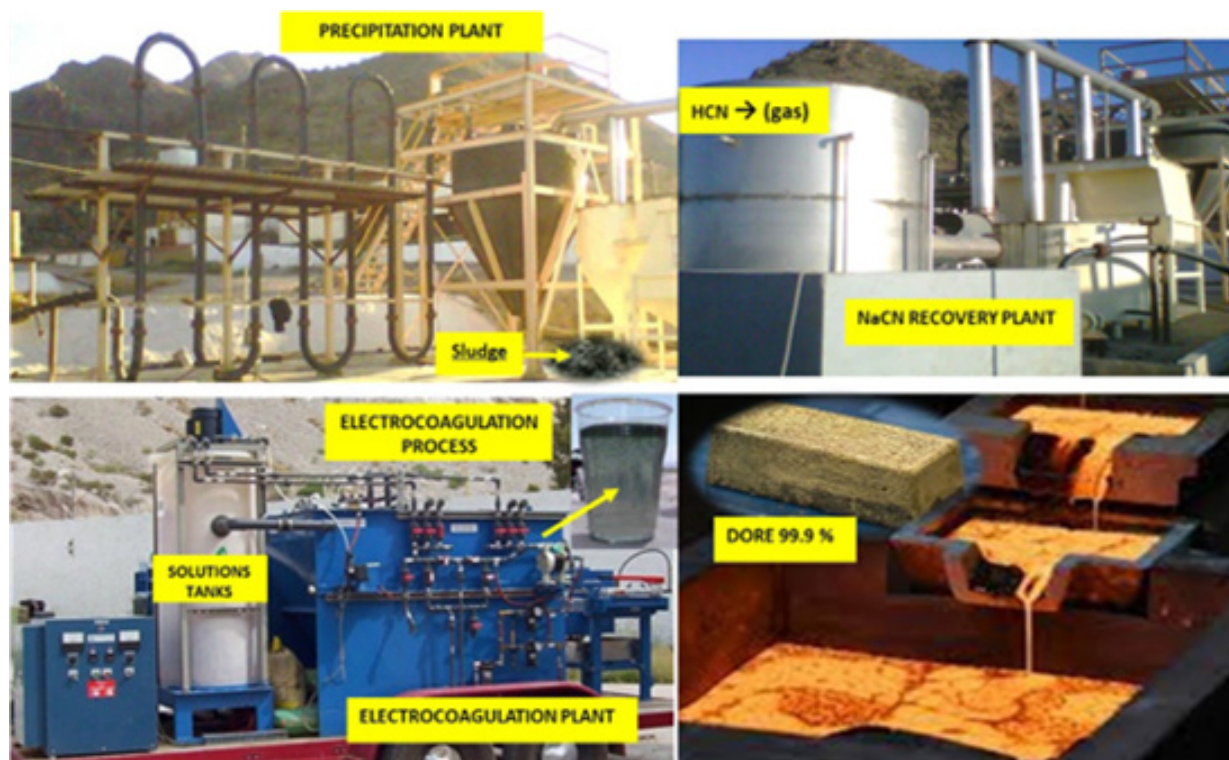
### Pollutants removal by precipitation experiments

The precipitation test consists of 2-4L of different types of graduated cylinders (probetes) and the addition of dilute  $\text{Na}_2\text{S}$ , sulphuric acid at different pH to follow the kinetics of the precipitation reaction and also choose the appropriate pH for the elimination of parts of the calcium sulfate and the heavy metals ions.

### Electrocoagulation experiments

The electrocoagulation system included a flow-through cell, an electrode assembly, a feed pump, and a power source. Figure 2 shows a picture of the electrocoagulation reactor. The EC reactor contained five parallel carbon steel electrode plates (11.0cm×11.4cm) spaced 6mm apart to generate four parallel cells. The cell has an internal volume of about 1200ml, and a variable transformer was employed to control the current and potential. Table 1 shows the essays of electrolyte solutions to trite. The conductivity was increased with 1g NaCl per liter (Fisher, 99.8% A.C.S. Certified, lot #995007), and the pH was adjusted as needed with a ~0.13M NaOH solution made with NaOH pellets (EM, 97%, lot #36349739). Before each trial, the pH and conductivity of each solution were measured [15]. The solution was pumped through the cell at a flow rate of approximately 525ml/min, and after treating 2.0 to 4.0 liters of electrolyte solution, the run was terminated, and the slurry pH and conductivity were determined. Finally, the set equipment or prototype for precipitation and electrocoagulation experiments was escalated up to use in the mining industry in order to solve the production of high-quality Dore, in the following (Figure 3), shows pictures of equipment designed for real-life application processes and has been patented for industrial application in IMPI Mexico [15-18].

**Figure 2:** Hermetic reactor EC.



**Figure 3:** Total system for complete elimination of the cyanide complex and recovery or regeneration of sodium cyanide using acid /basic precipitation and electrocoagulation process.

### Suspended particle cementation experiments

The batch reactor experiments were conducted in four-necked, one-liter glass reaction cells using a constant temperature bath. A condenser, stirrer, nitrogen dispersion tube, and sample instrument were put into the reactor via the lid's aperture. To keep the atmosphere oxygen-free, pure nitrogen was pumped through the solution using a dispersion tube before and during each experiment.

### Characterization

It is difficult to distinguish between the iron oxide and oxyhydroxide species using a single analytical method. In this investigation, powder X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) were employed to analyze the solid products generated at steel electrodes during electrocoagulation. Electrometry was used to evaluate the liquid product (0.01 pH, 0.5 conductivity, 20mg/L DQO, 0.05mg/L Cl, 0.4mg/L  $\text{NH}_3\text{-N}$ , 0.002mg/L CN) at the CONDORCHEM ENVITECH MOT-LAB in Premia de Dalt (Barcelona).

### The analysis of cyanide concentration

Procedure: The batch cementation tests were conducted in a 1000cm<sup>3</sup> Pyrex reactor. The silver-loaded solution was initially adjusted to pH 11 using sodium hydroxide and the necessary cyanide concentration for each test. An inert gas ( $\text{N}_2$ ) was then bubbled for 15 minutes to reduce the  $\text{O}_2$  content from 7.2 ppm to 0.1 ppm, which is the acceptable level for precipitation experiments. Once the  $\text{O}_2$  level had been decreased, zinc powder was applied. At the time of addition, the silver cementation time was recorded.

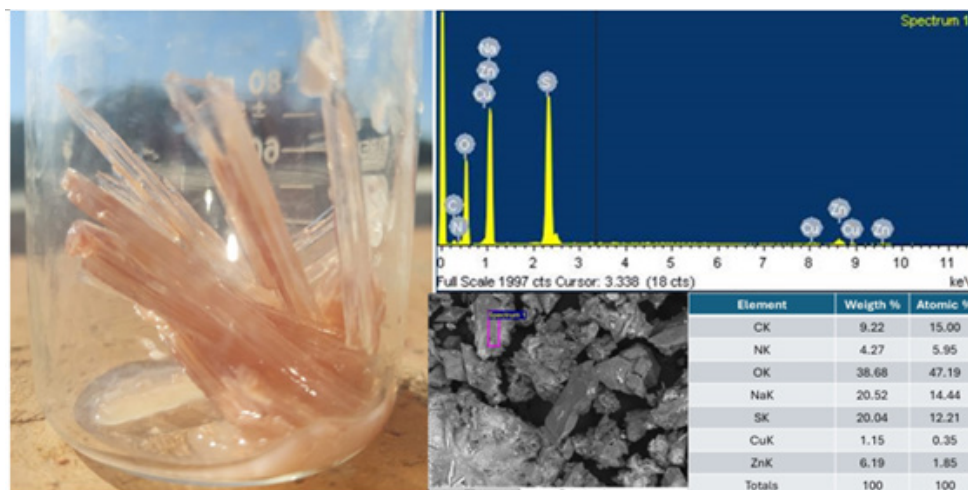
Each test required monitoring the content of the pregnant silver solution at various sampling times.

### Result and Discussions

The EC products contain ferrimagnetic materials such as magnetite or maghaemite ( $\gamma\text{-Fe}_2\text{O}_3$ ) and possibly oxyhydroxides evenly distributed throughout the sludge network. The process appears to offer the potential of reducing the concentration of heavy metals in slurries at an economic cost because the magnetically loaded adsorbent can be easily recovered from aqueous slurry by using conventional methods of magnetic separation such as the magnetic drum separator. Once the magnetic particles (loaded adsorbent) have been recovered, the adsorbed species may be released by treating the adsorbent with a suitable solution or solvent, regenerating the adsorbent, and allowing for recycling and reuse.

#### Step 1: The precipitation results

The high cyanide concentration, necessary to prevent metal precipitation and dissolve gold and silver, resulted in a high pH and increased zinc dust waste due to altered chemical equilibrium. Inefficient cyanide removal by the EC process necessitated the development of an intermediate electrochemical step. High cyanide levels led to rapid electrode saturation with zinc and copper during electrolysis, requiring high voltages and amperages. Anionic flocculants in the cyanidation solution hindered electrochemical processes. The low temperatures (<10 °C) induced cyanide-metal complex crystallization, blocking pipes and hindering operations. SEM analysis confirmed the presence of zinc, copper, sodium, and sulfur in these crystals (Figure 4).



**Figure 4:** a) The untreated cyanides with its b) EDX analysis and c) Scanning Electrode Microscope (SEM) micrographs.

To enhance Electrocoagulation efficiency, excess flocculants, and the concentration of zinc, copper, and iron cyanides must be removed, Furthermore, pH adjustment is necessary. A 15-liter barren solution was prepared at pH 13 and acidified with 30cm<sup>3</sup> of H<sub>2</sub>SO<sub>4</sub> per liter. The chemical analysis of this adjusted solution is presented below: The initial treatment is to add sulfuric acid and sodium sulphide to the different pH from each experiment with results shown in Table 2. This white solid precipitate can be seen in the following photographs and with the SEM you can see the microparticles with their respective EDX chemical assay.

**Table 2:** The head of the barren solution before treatment.

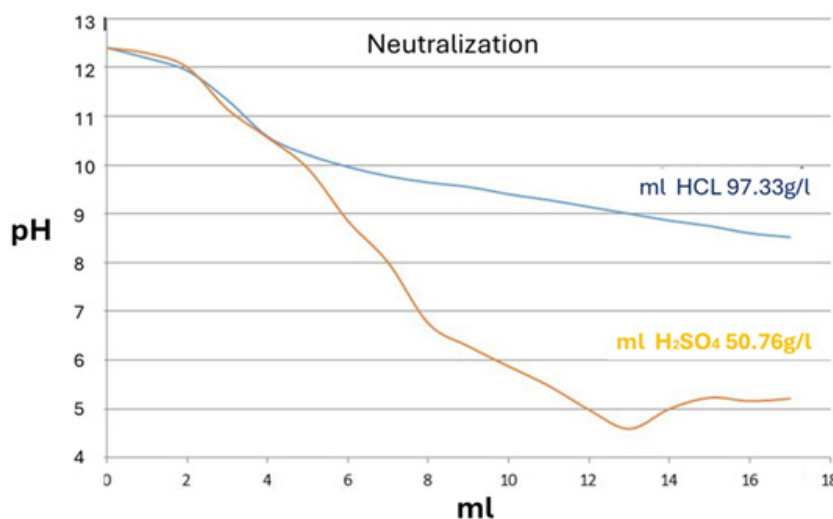
	ppm (mg/l)					
	Ag	Au	Cu	Zn	Pb	Fe
pH 13	0.49	0.020	12500	32313	4.50	907

The initial pH value of the rich solution is determined, and it is then adjusted to various pH values using hydrochloric acid and sulphuric acid (see equations 12-14). The following images show the white solid precipitate, and the SEM shows the microparticles

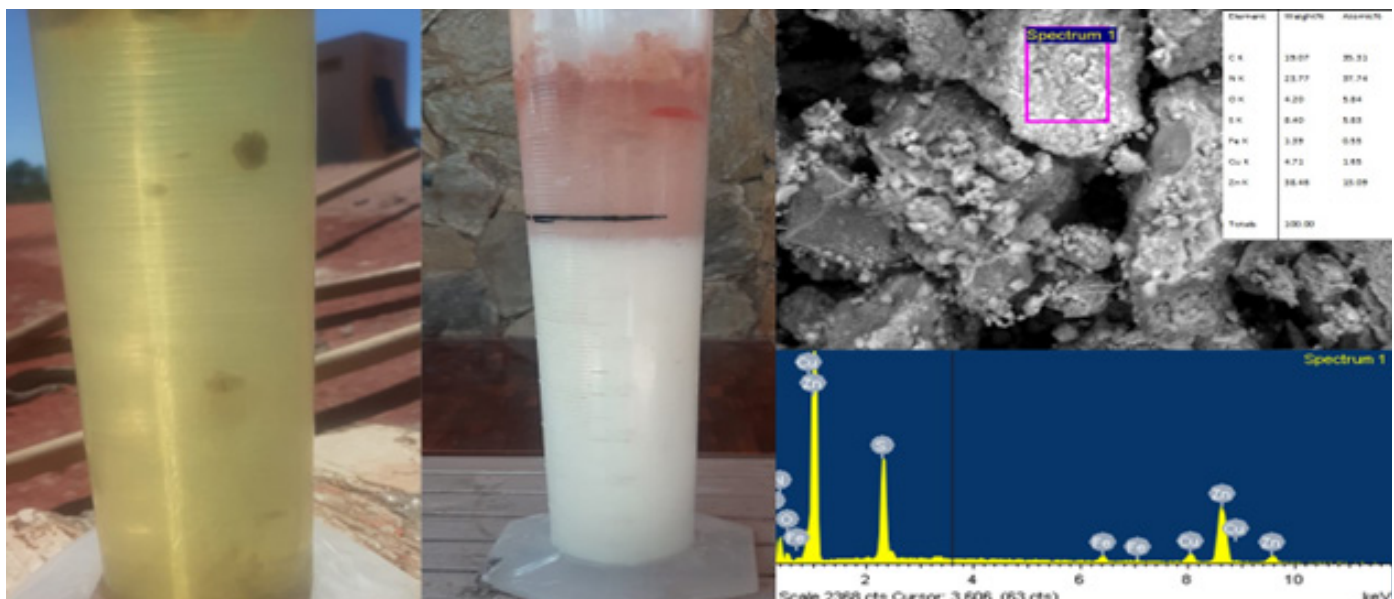
with their specific chemical analysis. Figure 5 shows that sulphuric acid is better and cheaper than Hydrochloric acid in terms of neutralization behavior in the barren industrial solution. The sulphuric acid was observed to decrease the pH and become more acidic in the barren industrial solution. The experimental set up was presented in Figure 6 and Table 3 showing the elemental composition as the pH decreases from 12 to 8. The sulphuric acid was observed to decrease the pH and become more acidic in the barren industrial solution. The result is presented in Figure 5 and Table 3 shows the barren solutions in ppm.

**Table 3:** Chemical analysis of the barren solutions in ppm.

	Ag	Au	Cu	Zn	Pb	Fe
pH 12	0.86	0.029	11345	29970	3.94	363
pH 11	0.76	0.029	10644	28878	7.36	339
pH 10	0.68	0.028	10126	17551	6.43	336
pH 9	0.31	0.028	9887	16899	4.93	338
pH 8	0.33	0.027	8818	1054	4.61	147

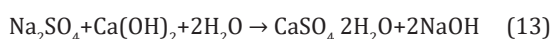


**Figure 5:** The neutralization reaction with the barren industrial solution using Hydrochloric and Sulphuric acid.



**Figure 6:** a) The effect of the first treatment at reduced pH b) Scanning Electronic Microscope (SEM) micrograph c) The EDX.

The precipitated solid was mainly hydrated lime (calcium sulfate) with mostly zinc, copper, and iron sulfate complexes. The reaction mechanism is shown in equations 12 to 14:

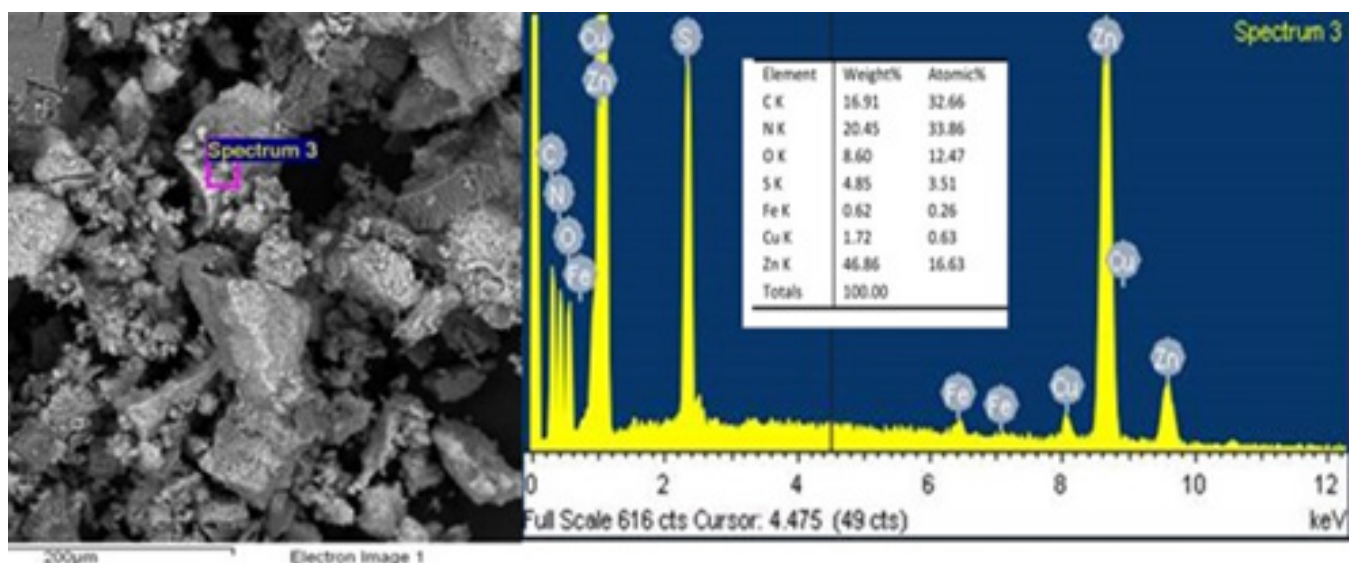


where M=Zn, Cu and Fe

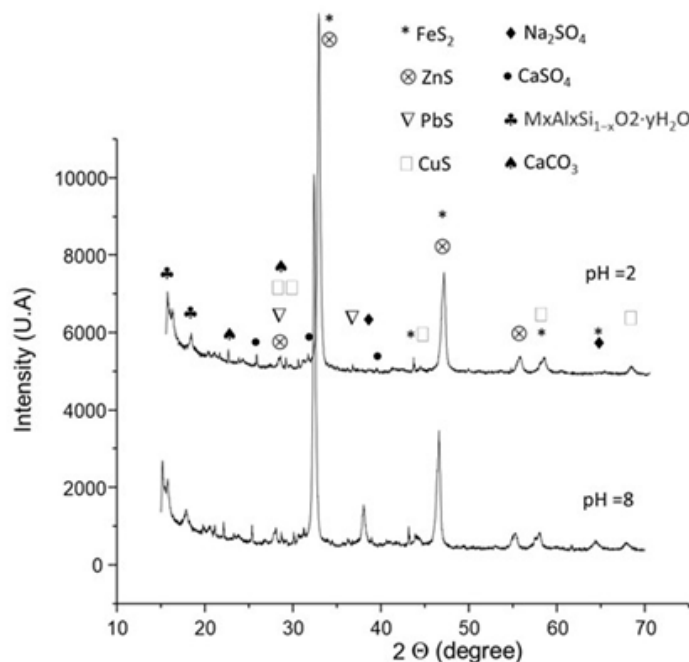
**Step 2: The electrocoagulation results**

After the liquor samples have cooled, they are transported to the electrocoagulation apparatus with iron electrodes and the factors

found for the properties of these barren solutions. Electrochemical electrocoagulation is used in an experimental attempt to eliminate cyanide completely. Then, the characterization of the dry solid was done using the Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD) where the precipitates are mostly zinc, copper, and iron. The SEM results of the Electrocoagulation process showed the irregular structure in the micrograph and the sulfur of zinc is the principal solid in the solid at pH of 8 by the Energy Dispersive X-ray Spectroscopy (EDX) (Figure 7). Also, the results of X-rays of the solid product showed that the species precipitated are: FeS<sub>2</sub>, ZnS, CuS, Na<sub>2</sub>SO<sub>4</sub>, and CaSO<sub>4</sub> (Figure 8).



**Figure 7:** Scanning Electron Microscope (SEM) micrograph and Energy Dispersive X-ray Spectroscopy (EDX) of the precipitate at pH of 8.



**Figure 8:** The X-ray diffraction patterns of the solid products obtained by electrocoagulation.

The conditions and results of the first and second EC treatment phases are shown below in Tables 4 & 5:

**Table 4:** The study variable of the electrocoagulation process.

pH 8 Initial				
Time	Voltage (current)	Current (A)	pH E1	pH E2
0	14	7	8.0	10
3	14	7	9.2	10.4
6	14	7	9.7	10.9
10	14	7	10.0	11.4

**Table 5:** The chemical assay of the Barren solution before and two-stage treatment of electrocoagulation.

Barren Li ppm (mg/l)						
	Ag	Au	Cu	Zn	Pb	Fe
pH 8 initial	0.13	0.007	8818	1054	1.61	17
Stage 1 EC	0	0	2900	292	0.23	274
Stage 2 EC	0	0	24	13	0.10	433

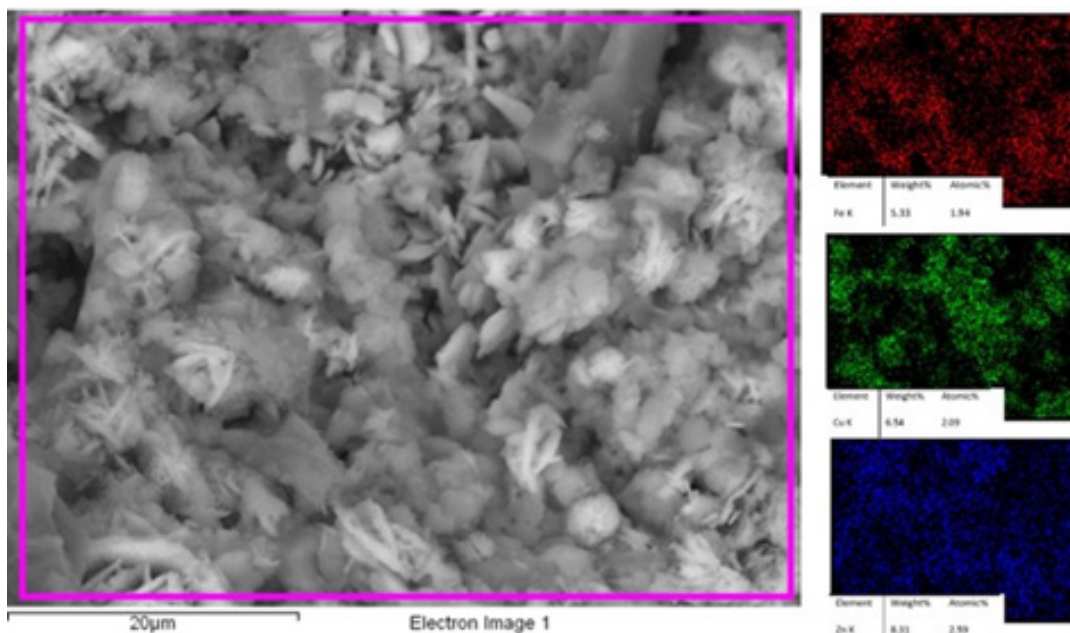
At the base, the result of the chemical analyses of these two treatment stages, we observed the reduced copper, zinc, and lead

cyanide content by 99.4 %, 99.2 %, and 99.5%, respectively. The solution of the second stage was further recycled at pH for the second treatment to eliminate Cu, Zn, and Pb. The average sodium cyanide recovery was 85% and recycled to the initial leaching process of dissolution of gold and silver. The obtained sludge is dried in a Vacuum-dried treatment, then the samples were analyzed using SEM-EDX to determine chemical composition, morphology, and major species formed, which are summarized in Table 6.

**Table 6:** Final sludge composition (ppm).

	Ag	Au	Cu	Zn	Pb	Fe
pH 10 initial Stage 2 EC	0	0	24	13	0.10	433

The micrograph of Scanning Electron Microscopy (SEM) in Figure 9, after the treatment at pH 10 was observed that the solid sludge has 5.33% Fe, 6.54% Cu, and 8.31% Zn. This has great potential in semiconductor applications due to the high magnetic properties detected [19]. The Scanning Electronic Microscopy and X-ray diffraction, techniques demonstrate that the formed species are of magnetic type, like lepidocrocite and magnetite, and amorphous iron oxyhydroxide which adsorbed copper, zinc, and lead particles on his surface due to the electrostatic attraction between them.

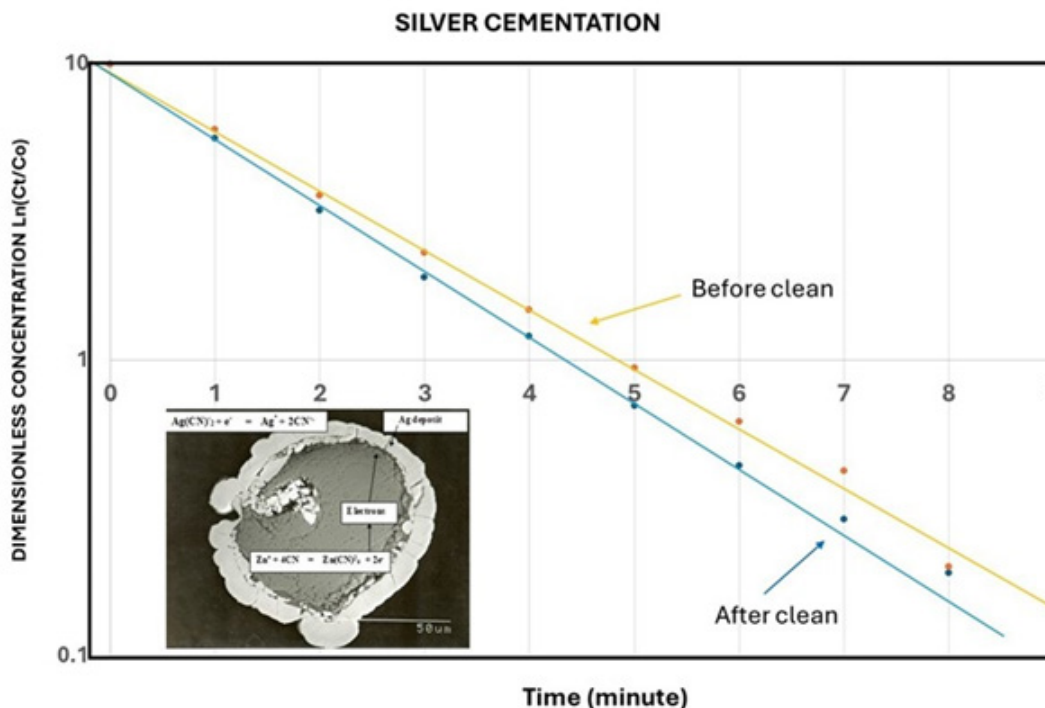


**Figure 9:** The scanning electron microscope showing the complete map of one area of the sludge.

**Step 3: The cementation results**

To assess the efficacy of removing cyanide contaminants from copper, zinc, and lead in barren solutions, we conducted a cementation test using silver cyanide and merrillite zinc (Table 7).

Our findings indicate that silver cementation is significantly faster with clean solutions and adheres to a mass transfer mechanism (Figure 10).



**Figure 10:** First-order reaction kinetics plot for silver cementation for the clean and contaminated solution of cyanide complex.



**Table 7:** Kinetics of silver cementation with zinc powder from a rich solution, where the circumstances are as follows:  $\text{NaCN}=10^{-2}\text{M}$ ,  $\text{pH}=11$ , and 100 milligrams of merrillite zinc per liter of solution.

Time (mins)	Ag, ppm (Clean Solution)	Ag, ppm (Contaminated Solution)
0	10.00	10.00
1	5.60	6.00
2	3.20	3.60
3	3.90	2.30
4	1.20	1.48
5	0.70	0.94
6	0.44	0.62
7	0.29	0.42
8	0.19	0.20

## Conclusion

In this study, we have successfully reported the use of precipitation and electrocoagulation processes for the removal of Cu, Zn, Pb, and Fe. The precipitation reaction involves the use of sodium sulfide and sulphuric acid to control the pH from 13 from the barren solution to 8. From the barren solutions, all cyanide complex metals 99.5% were removed in the experimental EC hermetic reactor, and it was achieved in two stages of EC treatment with a total time of 20 minutes or less with a current efficiency of 99.7%. In this research work a series of tests have shown that the correct selection of the parameters (pH, type of regulator pH, retention time, type of electrode, applied voltage, applied current, etc.) for the Precipitation and EC process, these variables change from one system to another system and the operation of the EC reactor has to be modified accordingly to fit each individual application. Also, the results of this study suggest that EC produces magnetic particles of magnetite, lepidocrocite and amorphous iron oxyhydroxide species that can be used to recover zinc and copper.

The X-ray diffraction and scanning Electronic Microscopy techniques demonstrate that the formed species are of magnetic type, like lepidocrocite and magnetite which adsorbed the copper and zinc particles on their surface due to the electrostatic attraction between both metals. Finally, the main advantage is that the nanomagnetic particles that are found in solid products can be recycled and sold; Therefore, it does not generate toxic waste. In addition, kinetic analysis of the cementation process by using the clean solution shows that the silver cementation process is a first-order reaction.

## Acknowledgment

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