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DETOXIFICATION OF EFFLUENTS WITH CYANIDE. APPLICATION ON A CYANIDED GOSSAN ORE.

MASTER FINAL PROJECT

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0. ABSTRACT

The cyanidation of minerals is carried out for recovering their gold and silver content. As a consequence of this process, effluents and solid wastes are generated, which will contain free cyanide as the most toxic form. Cyanides in solution can be transformed to less toxic forms through the application of a degradation processes.

The objective of this work is to study the degradation of cyanide effluents by using sodium hypochlorite (NaClO) or ozone (O₃), determining the best conditions for the degradation of the total cyanide, without generating any other type of contaminants. The optimum conditions will be used in the cyanidation process applied over a Gossan ore during its gold and silver recovery.

Gossan ore of Rio Tinto (Huelva, Spain) is used for the application of the detoxification of effluents with cyanide and using the techniques of characterization to determining the main elements: iron (25.0%) and silicium (21.2%), the amount of gold and silver is 2.00 ppm and 87.4 ppm respectively, and the main mineralogical species: quartz, barite, goethite, hematite, beudantite and jarosite. With the application of techniques such as alkaline chlorination and ozonation it is possible to remove cyanides to achieve percentages higher than 98% leaving less than 40 ppm cyanide in 15 minutes, reducing the effluent toxicity and solid waste. The degradation of cyanide with ozone can generate carbonates that are much friendlier to the environment compared to hypochlorite that forms cyanates. However ozone is very little used in mining because it has some drawbacks: its high price and high consumption.

Keywords: Cyanide, effluents, detoxification, oxidation, hypochlorite, ozone.

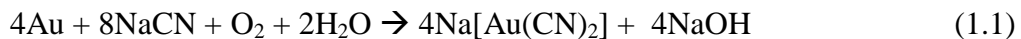
1. INTRODUCTION

The discharge of effluents and solid wastes contaminated with cyanide to the nature without any treatment exceeds the capacity of auto-retrieve receiving bodies or soil, since they alter their characteristic and cause an eminent pollution and major environmental impact. They destroy ecosystems and affect the health of populations. The recovery of gold and silver with the cyanidation process produce effluent and solid waste with a big number of pollutants, specifically cyanide.

Peru is one of the of most important mining countries in the world, with a huge metallurgical potential for copper, gold, silver, iron, lead, and other metals, constituting one of the most important items in the socio-economic structure of this country. While it is true that mining is an activity that produces wealth, it is also true that damage that produces is very serious, especially in what refers to environmental pollution around the world (Vásquez, 2006).

Mining is one of the activities that use a significant amount of cyanide. Since the end of the XIX century, the process of cyanidation is the most important method used in the recovery of gold and silver from ores (Logsdon *et al.*, 2001).

The cyanide process is based on the fact that when the finely divided mineral, containing only a few grams per ton of metal, is treated with a dilute solution of potassium or sodium cyanide, gold and silver are easily dissolved, while other metallic components remain unchanged. The reaction for leaching metallic gold or silver in a diluted cyanide solution is represented by the Elsner equation (Ballester *et al.*, 2000):



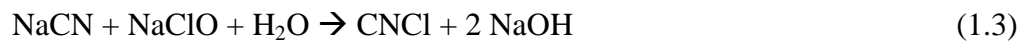
Although, it has been tried to minimize the potential toxic and dangerous power of the cyanide, by using other reagents for silver and gold recovery, such as thiosulfate or thiourea, the best reagent is cyanide, being the most stable, efficient and economically profitable (Logsdon *et al.*, 2001).

After the extraction of gold and silver by the process of cyanidation, in the effluents remain cyanides: the free cyanide, cyanide weakly complexes and cyanide strongly complexes constituting the “total cyanide”. The forms of free cyanide are important because they are considered as the most toxic cyanide. The analysis of the cyanide types is important to ensure the effective choice detoxification method (Moran, 2001). It uses the Standard

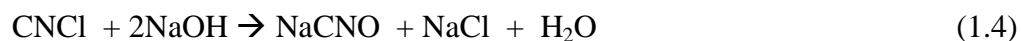
Methods 4500-CN-D; ASTM D-2036-91, as a method to determine the concentrations of CN^- over 5 mg/L present in the sample (Nava *et al.*, 2007).

The gold and silver dissolution rate is believed to be dependent on the concentration of NaCN and the alkalinity of the solution. For this reason, it is necessary to perform a treatment of effluents and solid waste generated in the cyanide process. The cyanide can be oxidized to less toxic cyanate (CNO^-), with the help of, a strong oxidizing agent such as hypochlorite, ozone or hydrogen peroxide, among others.

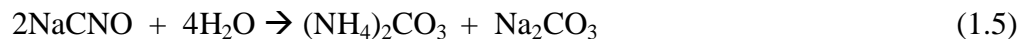
Alkaline chlorination is the oldest and best known process of degradation of cyanide, being a chemical process which consists of the oxidation and degradation of the forms of free cyanide and weak acid dissociable cyanide in an alkaline medium. Chlorine is provided as sodium or calcium hypochlorite (Smith *et al.*, 1996). The degradation of cyanide using hypochlorite consists of oxidation of the cyanide to form a chloride intermediate cyanogen, expressed in the following reaction:



The medium should be maintained at an alkaline pH and the temperature at not lower values than 18°C. The theoretical use of chlorine to oxidize consumption of cyanide to cyanate is 2.73 g of Cl_2 per 1 g CN^- oxidized, but in practice is 3 to 8 g Cl_2 per 1 g CN^- oxidized, since the consumption of chlorine is much higher. The cyanogen chloride intermediate is quickly hydrolyzed to cyanate; through in this reaction cyanide is completely destroyed to form cyanate, according to the following reaction:



The first step of complete oxidation takes about 15 minutes to a pH of 10.5. The second step of the oxidation involves the hydrolysis of cyanate to ammonia and carbonate, in the presence of chlorine or hypochlorite according to the following reaction:



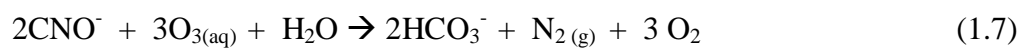
In normal conditions of pressure and temperature, alkaline chlorination removes all forms of cyanide, except complex cyanides of iron and cobalt which are extremely stable. The concentration of complex iron cyanides can be reduced by using high temperature alkaline chlorination. Consequently, alkaline chlorination applications are limited to those wastewater containing insignificant proportions of complex iron cyanides. As the process is conducted with high values of pH, the precipitation of metals in the form of hydroxides also occurs.

On the other hand, ozone (O₃) has been occasionally used as an oxidizing agent for the detoxification of cyanide effluents. Ozone (O₃) is a strong oxidizing agent generated by oxygen. A mixture of ozone and oxygen causes strong oxidizing conditions when it is injected in the form of fine bubbles in an aqueous solution. These conditions manage to dissociate the complex of cyanide (to give free cyanide), even the most stable as iron (II) and (III), although in a slower way (Young *et al.*, 2000). During the process, ozone disintegrates and generates no harmful byproducts. Theoretically, the oxidation of 1 mole of cyanide is fast, consumes 1 mole of ozone and produces 1 mole of cyanate (Young, 2000). The cyanide oxidation process can be described by two main chemical reaction pathways as highlighted below.

The first step (1.6) is the oxidation of cyanide to produce cyanate:



The second reaction (1.7) shows the reaction where cyanate is hydrolyzed and oxidized in the presence of excess ozone to form bicarbonate, molecular nitrogen and oxygen (Álvarez, 2007):



It is necessary to have a rigorous control of pH during the process, since the groups OH⁻ can decompose ozone. The reactions take place in an alkaline medium (pH 10 - 12) with a reaction time between 20 to 30 minutes. Most of the time is required for the second reaction. For the oxidation of cyanide to form cyanate, it is required 1.85 g of ozone per 1 g of CN⁻ and for the oxidation of cyanide to form bicarbonate/molecular nitrogen, 4.6 g ozone per 1 g of CN⁻ are demanded (Ametller, 1992). However, the drawbacks of using ozone are its high price and high consumption.

Gossan ore from Rio Tinto (Huelva, Spain) was used for the application of the detoxification of effluents and solid waste with cyanide (see Figure 1.1). The extraction of Gossan ore in this area has reached approximately $7 \cdot 10^6$ t/yr in the last few years. The recovery of gold and silver by the application of conventional cyanidation was started in 1970 (Viñals *et al.*, 1995).



Figure 1.1: Gossan ore from Rio Tinto (Huelva, Spain)

This work pretends to minimize the concentration of cyanide of the effluents and solid waste generated in the process of leaching of gold and silver, by detecting the optimal experimental conditions when sodium hypochlorite (NaClO) or ozone (O_3) are used. These optimum conditions will be used in the cyanidation process applied over a Gossan ore during its gold and silver recovery.

2. OBJECTIVES

- To detoxify effluents with cyanide by using sodium hypochlorite as oxidant.
- To detoxify effluents with cyanide by using ozone as oxidant.
- To determine the optimal dose of sodium hypochlorite (NaClO) and ozone (O_3), for cyanide removal.
- To apply the optimal conditions over the effluents and solid wastes generated during the cyanidation process applied on Gossan ores for gold and silver recovery.

3. MATERIALS AND EXPERIMENTAL PROCEDURE

3.1. Materials

3.1.1. Sample of Gossan

Gossan or “monteras of iron” are outcrops of rocks that originally contained sulphides and that have been subjected to a process of supergene alteration (changes physical and chemical by the action of agents such as rain, wind, solar action or groundwater). Gossan has a physical appearance of reddish color as a result of the transformations of the original sulphides, mainly rich in iron oxides (López, 1991).

3.1.2. Preparation of NaCN dissolution

To prepare a solution of 2 g/L of sodium cyanide (NaCN) in lime saturated Ca(OH)_2 , was required: Weigh 2 g of sodium cyanide (NaCN) and 5 g of calcium hydroxide- Ca(OH)_2 to 1L of distilled water and place a magnetic stirrer, shake and wait for the non-dissolved lime to precipitate. The pH was 12.43 measured at $T^\circ=24.2^\circ\text{C}$.

It has been considered the concentrations between 2 g/L, 1 g/L and 0.5 g/L of CN^- , for which was used industrially concentrations and depended cyanide process has been performed. Also the cyanide concentration of 2 g/L of CN^- allows us to demonstrate that at high concentrations is achieved degrade the cyanide.

3.2. Techniques for characterization of ores

Table 3.1 shows the main techniques for characterization of minerals.

Table 3.1: Techniques for characterization of ores (Ballester *et al.*, 2000)

Techniques for characterization	Fundament
X-ray fluorescence (XRF)	Overall chemical composition
Emission spectrometry (ICP)	Elemental chemical composition
X-ray diffraction (XRD)	Mineralogical composition
Reflected optical microscope (ROM)	Mineralogical composition, grain size and texture
Scanning electron microscope (SEM)	Punctual chemical composition, grain size and texture

3.2.1. Chemical characterization

3.2.1.1. X-ray fluorescence (XRF)

This technique allows knowing very fast the quantitative elemental chemical composition of the sample Gossan ore. It allows detection of almost all elements of the periodic system in a wide range of concentrations. It is a spectroscopic technique which uses the wavelengths and energy characteristics of the constituents of a sample. It is exposed to a primary X-ray beam to generate a beam secondary fluorescent X-ray, which are characteristic of the elements present (Ballester *et al.*, 2000).

3.2.1.2. Emission spectrometry (ICP)

This technique is complementary with XRF to determine the chemical composition of the sample of Gossan, and also it provides information on the majority, minority and traces elements. In the ICP (Inductively Coupled Plasma), the ionization is performed by an induced high frequency current. It is a technique of inorganic analysis that is able to identify and quantify most of the elements of the periodic table except the

atmospheric. This is a destructive technique because it requires the attack of the sample and analysis of the resulting solution.

3.2.1.3. Gold and Silver Cupellation (Fire assay)

This method is a quantitative determination, in which a metal or metals are separated from impurities by fusion processes, afterwards analyzed by ICP to determine the amount present in the sample. Metals recovered in fire assaying are the metals that emerge from the fusion of the material in an assay furnace, for gold usually at temperatures of about 1,100 °C. The cupellation is centuries old, but it is still the most reliable method for performing assays (to determine the metal content of a sample) of ores that contain precious (noble) metals.

It is mandatory to use a representative sample for testing; in this case it was used 10 g of Gossan ore, for fire assaying ores, fluxes, materials such as borax (12.5 g), sodium carbonate (7.5 g), litharge (5 g) and flour (4g). The amounts of each chemical added to the flux mixture depend upon the elements present in the particular ore, such as the iron oxides. Then, the flux mixture were added to a crucible, placed in an assay furnace, which was pre-heated to 600 °C that gradually increase its temperature until 1,100°C, to melt or "fuse" the contents of the crucible. When the slag cools, it is removed from the mold with a hammer to break away the lighter material, leaving only the lead button. The lead button is placed in a clay crucible called a cupel. The cupel is designed to absorb the lead oxide when heated to the melting point, and they are rated in the grams of lead they will absorb. So, the cupel with the lead buttons are again placed in an assay or cupellation furnace, heated to about 950 °C and the lead is absorbed into the cupel, leaving only a tiny gold and silver bead. Then, the cupel is removed from the furnace, is cooled and the bead is tested to know the amount of silver (after attack with nitric acid 33% and it taken to a volume of 50mL) and gold (after attack with aqua regia to volume of 10mL), and analysis was carried out by ICP.

3.2.2. Mineralogical characterization

3.2.2.1. X-ray diffraction (XRD)

This technique allows identifying the mineralogical phases or mineralogical composition, determining the majority and minority components present in the sample of Gossan ore. Diffraction is a phenomenon of X-ray scattering in which all

the atoms constituting the irradiated material are involved. Through to the periodic arrangement of atoms in a crystal structure, the rays scattered at different atoms. Each substance has its own crystal diffraction spectrum as the positions of the lines depend on the dimensions of the unit cell and the intensities of the type of atoms present and their distribution in the crystal.

3.2.2.2. Reflection optical microscopy (ROM)

It is the most effective method for the location and description of the mineralogical composition, grain size and texture. Most metal ores are opaque and therefore must be studied by optical microscopy reflected light as they can become transparent to light at a given thickness. The information that this technique can provide is the following: Optical properties of minerals, relationship between the constituent minerals, grain size and proportions, associations between mineral balance, petrographic classification of rock and/or mineral and degree of alteration minerals.

3.2.2.3. Scanning electronic microscopy (SEM) and X-ray energy dispersive analysis (EDX)

It is an ideal and essential technique that allows timely observations of chemical composition, grain size, morphological and textural samples of Gossan ore, to increase levels even >20,000X, excellent depth of field, high contrast and high resolution, and with EDX microanalysis the chemical composition of different points can be obtained. With this technique, an amplified image composition is achieved and the resolution is much larger than the optical microscope, which is complementary to the X-ray diffraction (XRD) and Reflection Optical Microscopy (ROM) techniques.

3.2.3. Characterization by Ion Chromatography

Ion chromatography is a method for analysis of anions. It is based on the use of resins (analytical column) ion exchange. When an ionic sample passes through these columns, the ions are separated through to the different retentions suffering interacting with the fixed phase of the analytical columns. Once separated, the sample passes through a detector (electrochemical conductivity) where the obtained signal is recorded respect to the retention time.

3.3. Experimental procedure

3.3.1. Determination of free cyanides

The determination of free cyanides is performed using 2 mL of NaCN dissolution and titration with silver nitrate (AgNO_3) 0.01M in the presence of potassium iodide (KI) as an indicator. The precipitation of yellow silver iodide indicates the end point of the titration (Nava *et al.*, 2007).

3.3.2. Detoxification of liquids containing cyanide (CN^-)

3.3.2.1. Reaction with sodium hypochlorite (NaClO)

Alkaline chlorination is performed to obtain an oxidation process with the degradation of free cyanide in an alkaline medium in normal environmental conditions (ambient temperature and 1 atm pressure).

Figure 3.1 shows the technique of determination for degradation and analysis (section 3.3.1.), for determining the remaining cyanide after sodium hypochlorite is added to the dissolution of cyanides. The particular experimental conditions for each experiment are included in the “Results and Discussion” section.

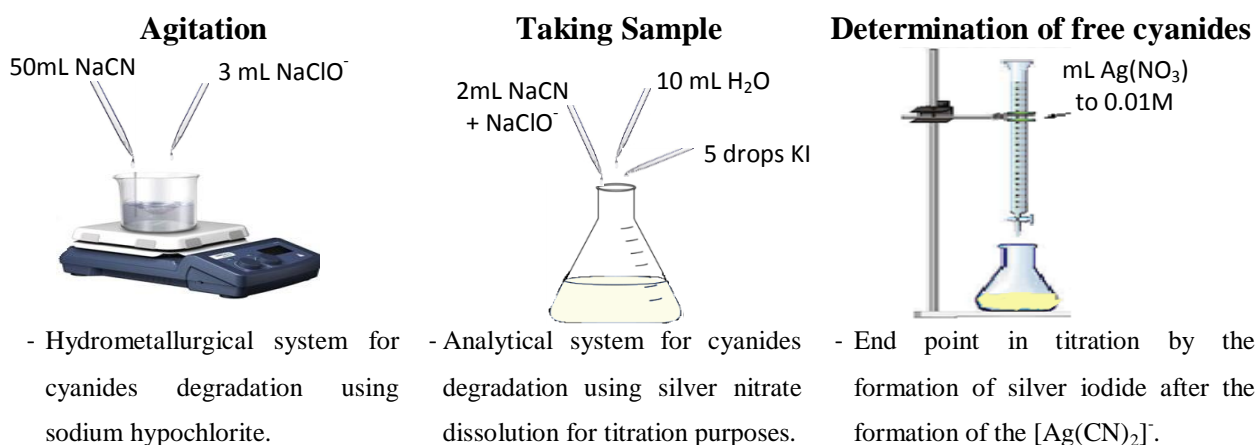


Figure 3.1: Experimental technique for degradation and analysis of cyanides using sodium hypochlorite

The determination of active chlorine (Cl_2) in sodium hypochlorite was also performed. Hypochlorite is an oxidant which liberates chlorine in acidic medium, the amount of chlorine released to acidify a solution of sodium hypochlorite is called active chlorine. The determination involves adding 10 mL of potassium iodide (KI), 2 mL of acetic acid (CH_3OOH) and 1 mL of concentrated sodium hypochlorite. The solution turns yellow due to iodine released. Hypochlorite in the reaction is reduced

to chloride ion. The liberated iodine is titrated with sodium thiosulfate solution ($S_2O_3^{2-}$) 0.1 M. In the titration reaction thiosulfate ion acts as a reducer and is oxidized to tetrathionate ion ($S_4O_6^{2-}$), while iodine is again reduced to iodide ion. The solution gradually loses the yellow color of iodine as the assessment progresses indicating the endpoint. Finally hypochlorite oxidizes iodide ion to iodine equivalent to active chlorine containing sodium hypochlorite. It is shown in the following reactions:



Also, it has been determined that the concentration of active chlorine (Cl_2) in the solution of sodium hypochlorite used is 15.8 mg of active chlorine (Cl_2)/mL.

3.3.2.2. Reaction with ozone (O_3)

A mixture of ozone and oxygen is a strong oxidizing system for the detoxification of cyanide when it is injected in the form of bubbles in an aqueous solution. An ozone generator (Triogen Ozone Systems, Model T₀C-B2) with 10W maximum power, was used in this study under ambient temperature and pressure conditions.

Figure 3.2 shows the technique of determination of free cyanide (section 3.3.1.), for determining the remaining cyanide after oxygen-ozone are added to the dissolution of cyanides. The particular experimental conditions for each experiment are included in the “Results and Discussion” section.

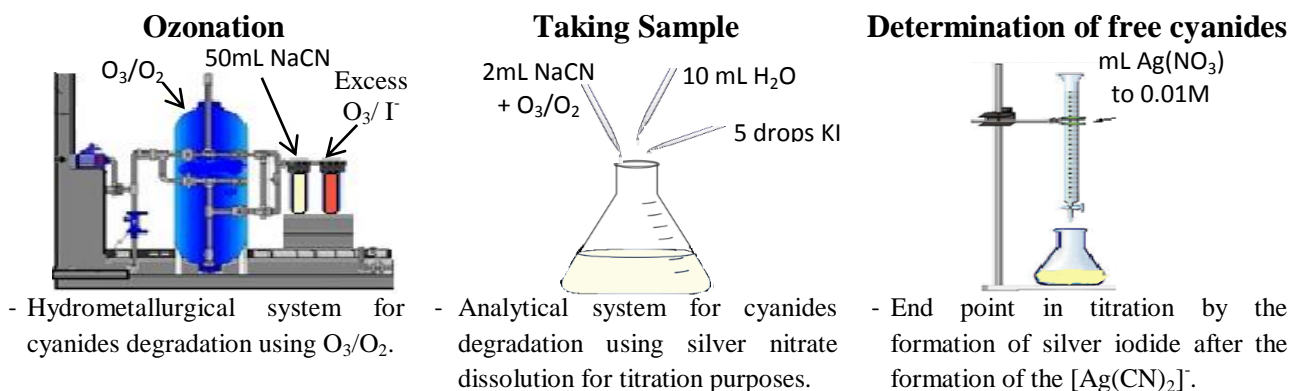


Figure 3.2: Experimental technique for degradation and analysis of cyanides using oxygen-ozone.

3.3.2.3. Ion Chromatography

An ion chromatograph DIONEX DX-300 with electrochemical suppression, electrochemical conductivity detector and the analytical column is AS11-HC of 4x250 mm and the mobile phase of 30 mmolar of NaOH was used. The software for handling the data was CHROMELEON® Version 6.80. The result was used chromatograms where the position of the top indicates the present ion (qualitative) and its area indicates that existing amount of such ion (quantitative). For example, Figure 3.3 show the chromatography of several anionic components.

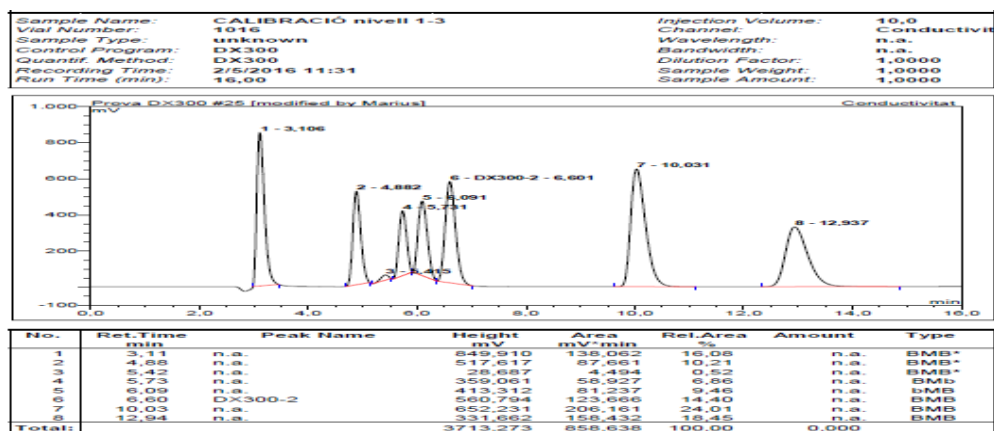


Figure 3.3: Calibration chromatography of several anionic components: Peaks, 1=F⁻; 2=Cl⁻; 3=CO₃²⁻; 4=NO₂⁻; 5=CNO⁻; 6=SO₄²⁻; 7=NO₃⁻ and 8=PO₄³⁻

3.3.3. Leaching of Gossan ore by cyanidation

This technique is developed with a solution of sodium cyanide 2g/L of NaCN, with excess of Ca(OH)₂, to regulate and maintain the solution pH in an alkaline medium. 400 mL of NaCN and 100 g of Gossan ore were used in the test. Cyanidation was carried out by stirring in a reactor, a magnetic stirrer and an external oxygen supply was used. This agitation leaching was carried out for a period of 10 hours, taking sample in 1, 2, 4, 6, 8 and 10 hours. Samples of 10 mL of leachate were taken to be filtered, and to perform the analysis of free cyanide by titration (used 2 mL sample), and analysis by ICP. Over the final residue, chemical analysis of gold and silver by fire assay was carried out.



Figure 3.4: Experimental procedure of leaching of Gossan ore by cyanidation

3.3.4. Gold and silver recovery

The recovery of gold and silver was performed by the Merrill Crowe process, described below.

3.3.4.1. Precipitation of gold and silver from a cyanide solution

After the separation of solid/liquid by sedimentation, in this case only the liquid part was used. Zinc powder cementation for gold and silver recovery is one of the best known contacts reducing process in mineral processing. Merrill-Crowe process is the technique used to separate gold and silver from a cyanide leach solution; it is a cementation using zinc powder. This technique was performed as follows: Taking a cyanide leach solution, adding 1 g of zinc powder and 0.1 g of lead nitrate stirring for 15 minutes, and taking a sample for analysis to verify the recovery of gold and silver with zinc powder.

4. RESULTS AND DISCUSSION

The results of experiments conducted in this work are presented in the following: Characterization of Gossan ore, degradation of cyanide with sodium hypochlorite and ozone, leaching of Gossan ore by cyanidation, detoxification of the effluents and solid waste generated during the cyanidation process and mass balance of the process and was balance referent to the Gold, Silver and CN^- behavior.

4.1. Characterization of Gossan ore

4.1.1. Chemical characterization

Results of chemical characterization are presented in this item:

- **X-ray fluorescence (XRF)** Table 4.1 presents the percentage of the different elements identified in the Gossan sample. The main elements detected are iron (Fe) and silicium (Si) as majority; as minority elements (between 2% and 0.1%) are aluminium (Al), lead (Pb), barium (Ba), arsenic (As), potassium (K), magnesium

(Mg), titanium (Ti), calcium (Ca), antimony (Sb), copper (Cu) and tin (Sn). The elements considered <0.1% weight are trace (Da Cruz, 2016).

Table 4.1: Elements detected in the Gossan ore (FRX) (Da Cruz, 2016)

Element	% Weight	Element	ppm	Element	ppm
Fe	25.0	Na	400	I	57.1
Si	21.2	P	200	La	40.2
Al	1.68	Zr	188	Mn	40.0
Pb	1.60	Bi	173	Rb	27.9
Ba	1.20	Hg	154	Sc	26.3
As	0.700	Zn	150	W	25.4
K	0.442	Cr	110	Mo	24.2
Mg	0.434	V	79.2	Tl	9.30
Ti	0.253	Ce	77.4	Nb	7.20
Ca	0.250	Ag	67.5	Cd	5.30
Sb	0.200	Y	59.8	Br	4.60
Cu	0.100	Sr	58.8	Ta	4.50
Sn	0.100	Se	57.2	Ga	3.00

- **Emission spectrometry (ICP)** Table 4.2 shows the percentage of the majority, minority and trace elements identified in the sample by ICP. The main elements detected are iron (Fe) as majority; as minority elements are lead (Pb), arsenic (As), sulfur (S), barium (Ba), aluminium (Al) and calcium (Ca) and another elements are considered as trace elements (Da Cruz, 2016).

Table 4.2: Elements detected in the Gossan ore by ICP

Element	% Weight	Element	ppm	Element	ppm
Fe	21.6	K	846	Zn	162
Pb	1.57	Si	588	Ti	129
As	0.620	Cu	504	Cd	112
S	0.545	Sb	457	Ag	51.6
Ba	0.396	Mg	324	V	34.0
Al	0.349	Na	219	Sr	17.8
Ca	0.294	Mn	182	Au	9.60

The I.R. (insoluble residue after the attack with water regia) was 50% and contains all the silica (quartz) and some silicates probably including Fe. These are the main difference between results obtained by XRF and ICP.

- **Gold and Silver Cupellation (Fire assay)** Silver was determined by the standard method of cupellation. The amount of silver in Gossan ore is 87.4 g/t (ppm). Gold are also determined by this method but it was a problem during the melting step and the corresponding value is calculated in section 4.3.1.

4.1.2. Mineralogical characterization

The results of Mineralogical techniques of characterization (XRD, ROM and SEM-EDX) are presented.

- **X-ray diffraction (XRD)** Figure 4.1, present the diffractogram of Gossan ore. The majority species identified in Gossan ore is quartz. Minority species detected are: barite, goethite, hematite, beudantite and jarosite (Da Cruz, 2016).

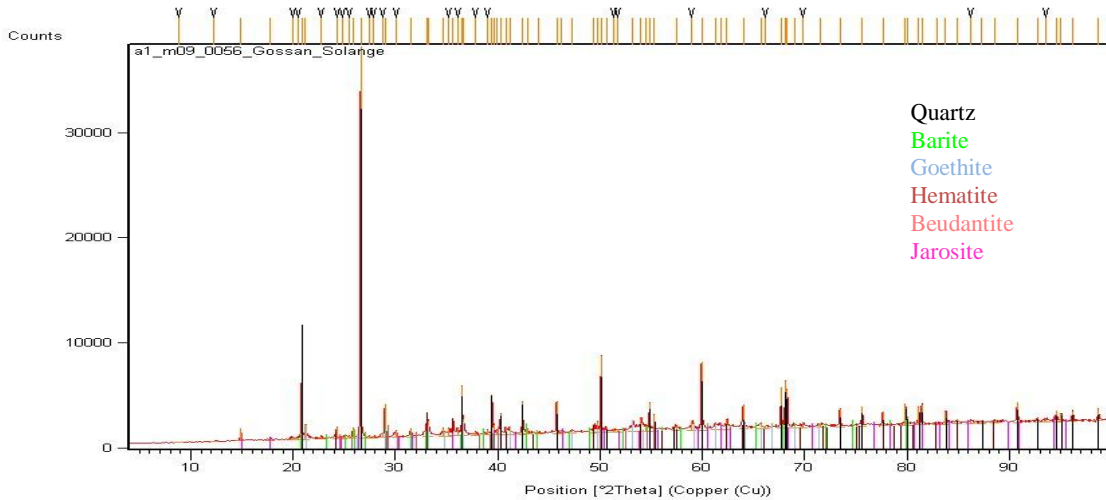


Figure 4.1: X-ray diffraction of Gossan ore

- **Reflection optical microscopy (ROM)** Figure 4.2 shows micrographs of Gossan ore, where different fields of the sample of Gossan can be observed: approximately 9 types of particles with different characteristics were identified. Their particle sizes were from 5 to 400 μm . Identifying each particle type requires electron microscopy (SEM).

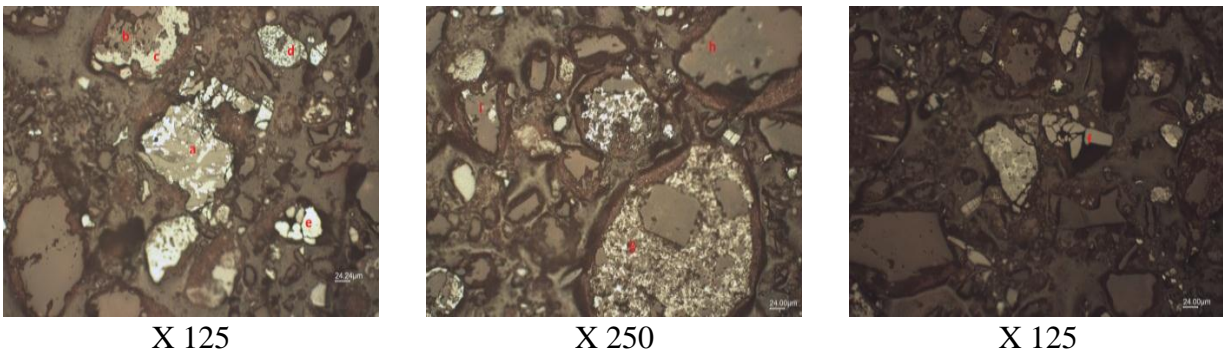
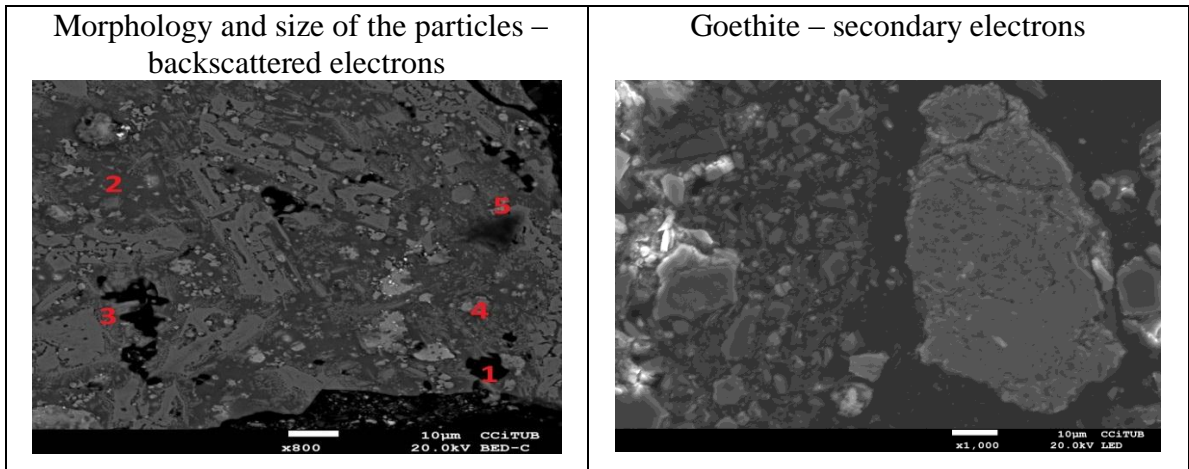
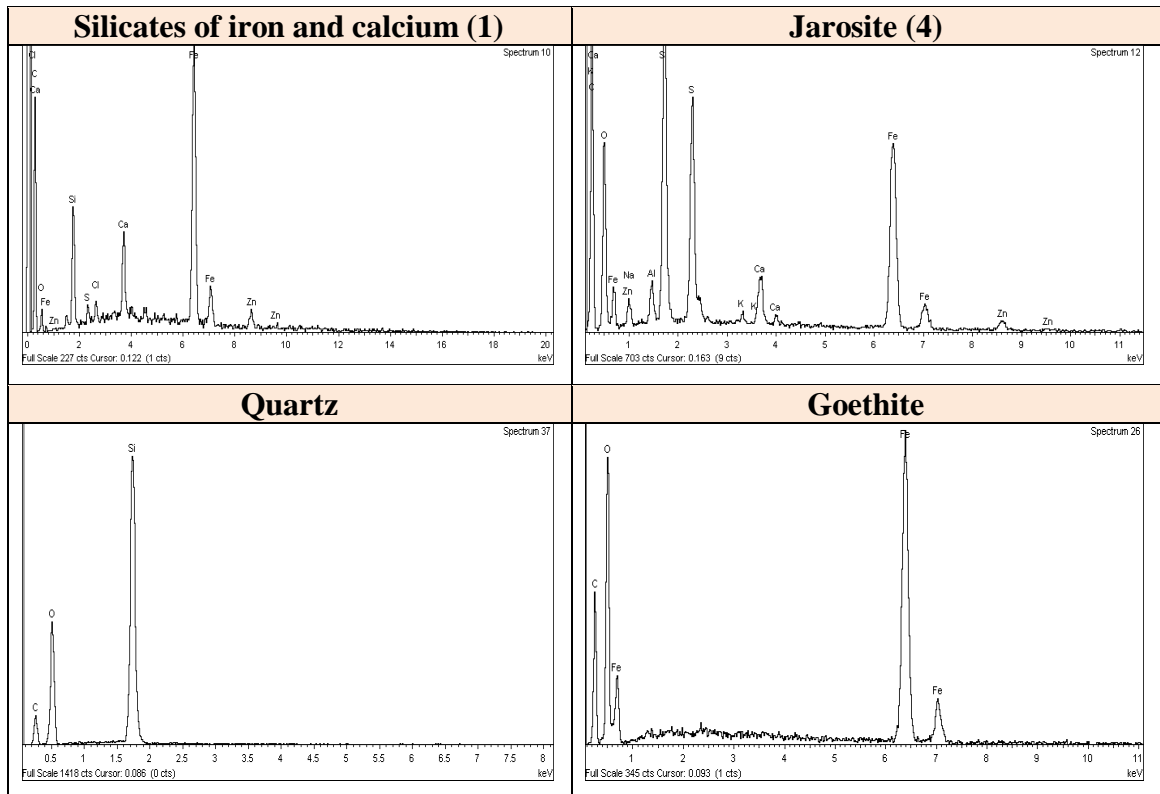


Figure 4.2: Particles observed by reflection optical microscopy

- **Scanning electronic microscopy (SEM) and X-ray energy dispersive analysis (EDX)** the main particles identified by SEM/EDX were quartz, barite, silicates, jarosite and iron oxides. Figure 4.3 shows particles detected by SEM, and Figure 4.4 is EDX diagrams of different chemical species. Results from SEM/EDX confirm the others obtained by techniques such as XRD and optical microscopy.



Figures 4.3: Particles identified by SEM



Figures 4.4: EDX diagrams of different particles

4.2. Study of degradation of cyanide with sodium hypochlorite and ozone

4.2.1. Reaction of cyanide with sodium hypochlorite (NaClO)

A) Table 4.3 and Figure 4.5 show the results obtained in the following experiment carried out at room temperature, with magnetic stirring. A first mL of sodium hypochlorite solution was added to the initial solution of sodium cyanide; after 5 min reaction a sample was taken for cyanides determination. This operation was

carried out until 4 mL of solution of sodium cyanide were added. As can be seen, cyanides concentration decreased: after the addition of these 4 mL, and the cyanide degradation level was higher than 98%.

Table 4.3: Degradation of cyanide using NaClO as oxidant

Agitation time (min)	Vol. Add NaClO (mL)	Equivalent amount of Cl ₂ (mg)	CN ⁻ Remaining concentration (g/L)	CN ⁻ degraded (%)
0	0	0	2.13	0
5	1	15.8	1.61	24
10	2	31.6	0.39	82
15	3	47.4	0.29	87
20	4	63.2	<0.04	>98

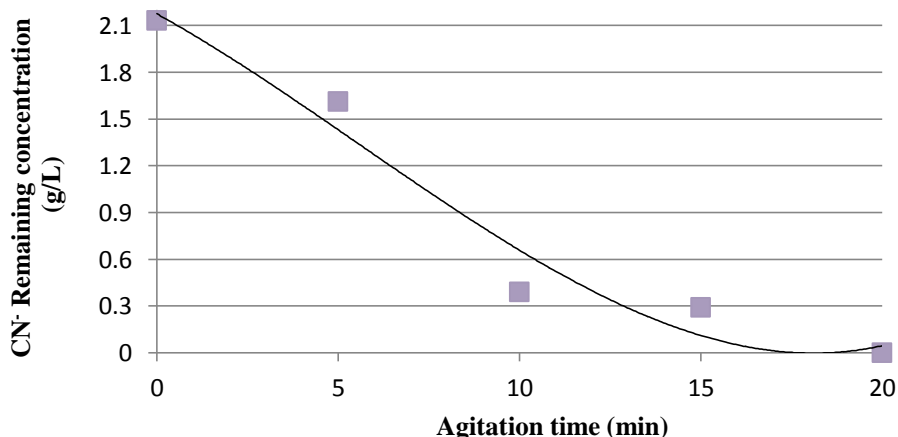


Figure 4.5: Evolution of the degradation of cyanide using NaClO as oxidant.

Based on results indicated in Table 4.3 and Figure 4.5 leaving the experiment of 3 mL of sodium hypochlorite solution (47.4 g/L active chlorine) during 60 minutes, the degradation level was >99%. Consequently, it is possible to determine the stoichiometry of this reaction. It is necessary 3.5 mole active chlorine per mole of cyanide. This result is in accordance to that obtained by Smith *et al.* (1991).

B) Effect of temperature in the degradation of cyanides

Table 4.4 presents a comparison of the degradation of cyanide at ambient temperature (25°C) and at 84°C with the same development as has been described in the previous experiment (A). Similar percentages of >98 and >99% degradation of cyanide were obtained in both experiments. Figure 4.6 shows the results, also presented in Table 4.4.

Table 4.4: Comparative of the degradation of the CN⁻ with respect to temperature

Temp. (T°)	Agitation time (min)	Vol. Add NaClO (mL)	Equivalent amount of Cl ₂ (mg)	CN ⁻ remaining concentration (g/L)	CN ⁻ degraded %
25	-	-	-	2.13	-
25	5	1	15.8	1.62	24
	10	2	31.6	0.39	82
	15	3	47.4	0.29	86
	20	4	63.2	>0.04	>98
84	5	1	15.8	1.86	13
	10	2	31.6	0.39	82
	15	3	47.4	0.17	92
	20	4	63.2	>0.02	>99

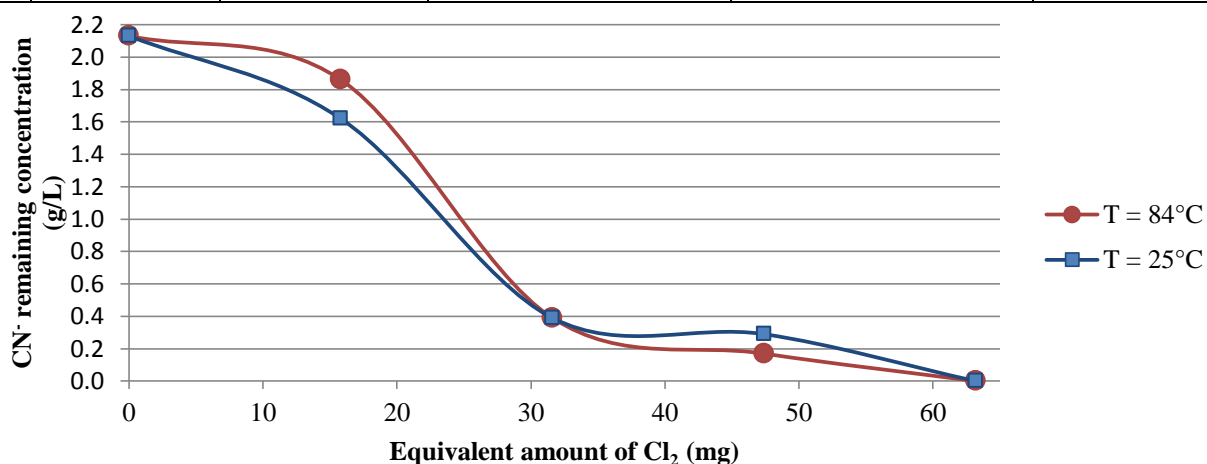


Figure 4.6: Degradation of CN⁻ at temperatures of 25°C and 84°C.

C) Effect of the initial concentration of cyanide in the degradation of CN⁻

Table 4.5, presents a comparison of the degradation of cyanide at an initial concentration of 2.13 and 1.06 g NaCN/L with the same development as has been described in previous experiment (A). Similar percentages of >98 and >99% degradation of cyanide has been obtained in both experiments. Figure 4.7 shows the results presented in table 4.5.

Table 4.5: Effect of concentration on the degradation of cyanide (CN⁻)

Test	Agitation Time (min)	Vol add NaClO (mL)	Equivalent amount of Cl ₂ (mg)	CN ⁻ Remaining Concentration (g/L)	CN ⁻ degraded %
1	0	0	0	2.13	-
	5	4	63.2	0.24	86
	15			0.09	96
	20			<0.04	>98
2	0	0	0	1.06	-
	5	2	31.6	0.09	96
	15			0.06	97
	20			<0.02	>99

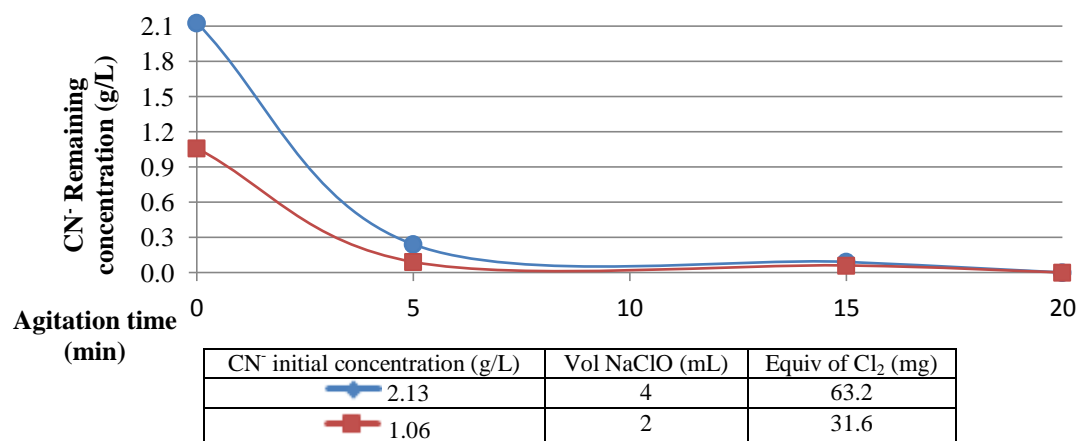


Figure 4.7: Effect of the concentration of cyanides in the degradation of CN⁻ using NaClO

According to the results obtained it seems that the cyanide in solution can be degraded at values of >99% operating at room temperature and with reaction times near to 20 minutes.

4.2.2. Reaction of cyanide with ozone (O₃)

A) Effect of the ozone feeding rate in the degradation of cyanides

Table 4.6 shows the results of degradation of cyanide by ozonation, using different ozone feeding rate. A titration was performed from 5 minutes to 20 minutes to determine the amount of degraded cyanide. The reaction increased by increasing the ozone feeding rate. Between 10–20 minutes cyanide degraded >99% by using 7.80×10^{-4} mole O₃/min in the experimental conditions employed; by using 3.90×10^{-4} mole O₃/min, degradation was between 20–30 minutes and by using 1.95×10^{-4} mole O₃/min degradation was in 30 minutes. Figure 4.8 shows the evolution of the degradation of cyanide with different ozone feeding rate.

Table 4.6: Degradation of cyanide using O₃ as oxidant at different feeding rate

Test	O ₃ concentration (mole/min)	Ozonation time (min)	CN ⁻ remaining concentration (g/L)	% CN ⁻ degraded	O ₃ (moles dosed)
1	7.80×10^{-4}	0	2.16	0	0.0000
		5	0.35	84	0.0039
		10	0.05	98	0.0078
		20	0.03	>99	0.0156
2	3.90×10^{-4}	0	2.16	0	0.0000
		5	0.78	64	0.0020
		10	0.10	95	0.0039
		20	0.05	98	0.0078
		30	0.04	>98	0.0117

Test	O ₃ concentration (mole/min)	Ozonation time (min)	CN ⁻ remaining concentration (g/L)	% CN ⁻ degraded	O ₃ (moles dosed)
3	1.95x10 ⁻⁴	0	2.16	0	0.0000
		5	1.81	16	0.0010
		10	0.69	68	0.0020
		20	0.08	96	0.0039
		30	0.05	98	0.0059
		45	0.04	>98	0.0088

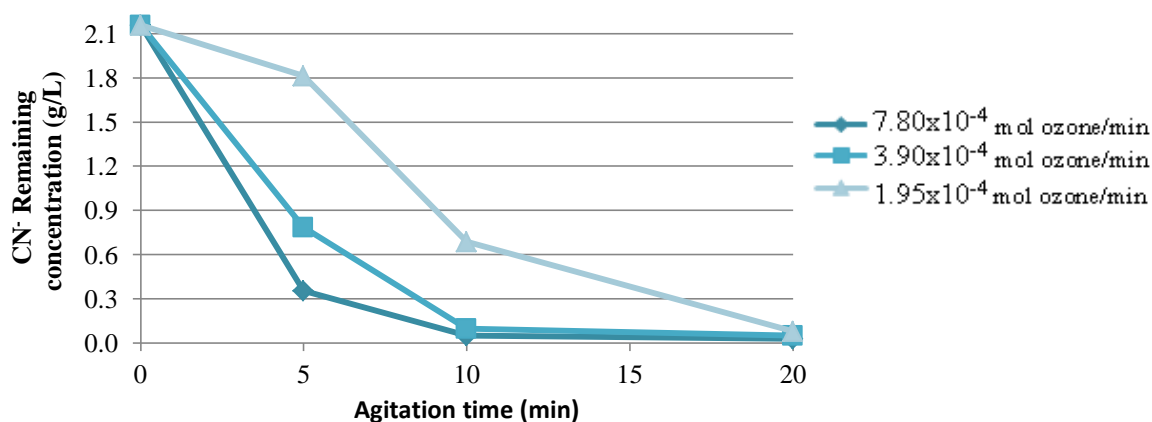


Figure 4.8: Evolution of the degradation of cyanide using O₃ as oxidant at different ozone feeding rate.

From the experiments included in the table 4.6 it can be deduced that it is necessary 0.0039 mole ozone to react with 0.0022 mole cyanide. Consequently, it is necessary 2moles ozone per 1 mole cyanide. These results are in accordance with those obtained by Ametller (1992). A solid was formed during the oxidation reaction. XRD confirmed that it was calcium carbonate. This fact demonstrates the generation of carbonates during the process.

B) Effect of the initial concentration of cyanide in the degradation of CN⁻

Table 4.7 presents a comparison of the degradation of cyanide at an initial concentration of 2.16, 1.03 and 0.27 g NaCN/L with the same development as has been described in previous experiment (A) but using the same feeding rate of ozone (7.80x10⁻⁴ moles O₃/min). Similar percentages of >98 and >99% degradation of cyanide has been obtained in these experiments and cyanide degraded at concentrations <0.04 g/L; in the 3 experiments the same results were obtained. Figure 4.9 shows the results obtained.

Table 4.7: Degradation of cyanide using O₃ as oxidant with different concentration of cyanide

Test	Ozonation time (min)	CN ⁻ Remaining concentration (g/L)	% CN ⁻ degraded	O ₃ (moles)
1	0	2.16	0	0
	5	0.35	84	0.0039
	10	0.05	98	0.0078
	20	0.03	>99	0.0156
2	0	1.03	0	0
	5	0.18	92	0.0039
	10	0.06	97	0.0078
	20	0.04	>98	0.0156
3	0	0.27	0	0
	5	0.19	91	0.0039
	10	0.08	96	0.0078
	20	0.04	>98	0.0156

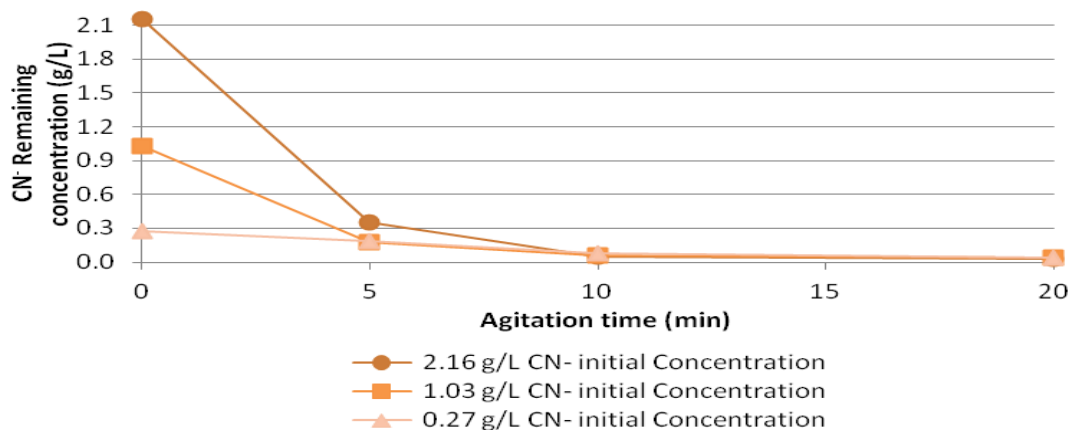


Figure 4.9: Effect of the initial cyanide concentration on its degradation of cyanide using O₃ as oxidant.

According to the results obtained it seems that the cyanide in solution can be degraded at values of >99% operating at room temperature and with reaction times near to 20 minutes for initial cyanide concentrations between 0.27 and 2.16 g/L CN⁻.

4.2.3. Ion Chromatography

Table 4.8 shows the concentrations of anions in the distilled water and sodium cyanide which must be considered to compare with cyanate y carbonate concentrations reached in each test.

Table 4.8: Anionic components of distilled water and sodium cyanide solution

Distilled water					
Components	F ⁻	Cl ⁻	CO ₃ ⁻²	SO ₄ ⁻²	
Concentration (ppm)	0.660	2.16	6.67	1.38	
Sodium cyanide (NaCN), saturated Ca(OH)₂					
Components	Cl ⁻	CO ₃ ⁻²	CNO ⁻	SO ₄ ⁻²	NO ₃ ⁻
Concentration (ppm)	135	1 322	35.0	1.38	84.3

Table 4.9 shows the concentrations of the components formed after the reaction of sodium cyanide 2g/L with 3 mL of sodium hypochlorite over time. Check up on that the reaction completely degraded the cyanide for form cyanate (CNO⁻) in concentrate between 1390 and 1865 ppm. Figure 4.10 shows the cyanate concentration behavior versus time. The diminution of CNO⁻ can be due to the transformation to carbonates. The theoretical amount of cyanate if all of the CN⁻ is oxidized to CNO⁻ is 1800 ppm. Thus, almost all the cyanide is oxidized to cyanate; few amounts may be oxidized to carbonates, but they can not be detected because the peak of CO₃²⁻ appears at the same retention time that ClO⁻ and Cl⁻. Also, the amount of nitrate detected in all the experiments is from the original sodium hypochlorite solution (see Table 4.9).

Table 4.9: Anionic components of the reaction of cyanide with sodium hypochlorite

Sodium hypochlorite - NaClO	
Components	NO ₃ ⁻
Concentration (ppm)	138

Sodium hypochlorite - NaClO (5 min)			
time (day):	0 (5min)	1 day:	7 day:
Components:	Concentration (ppm):		
CNO ⁻	1 507	1 686	1 483
NO ₃ ⁻	131	146	139

Sodium hypochlorite - NaClO (10 min)			
time (day):	0 (10min)	1 day:	7 day:
Components:	Concentration (ppm):		
CNO ⁻	1 489	1 629	1 433
NO ₃ ⁻	130	129	129
Sodium hypochlorite - NaClO (15 min)			
time (day):	0 (15min)	1 day:	7 day:
Components:	Concentration (ppm):		
CNO ⁻	1 448	1 534	1 390
NO ₃ ⁻	130	124	127

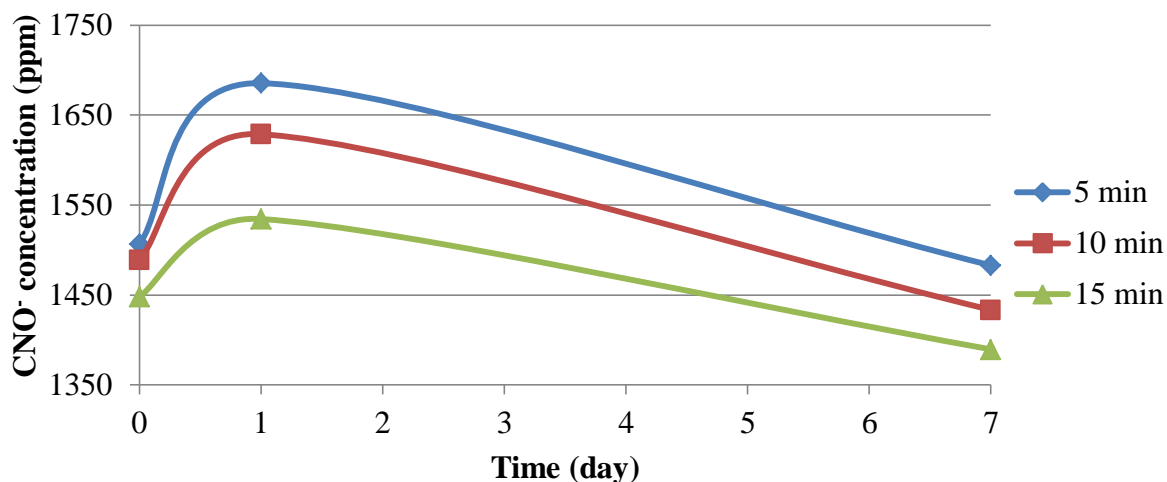


Figure 4.10: Evolution of CNO⁻ versus time

Table 4.10 shows the concentrations of the components formed after the reaction between cyanides and different feeding rate of ozone with 3.90×10^{-4} moles O_3/min and 1.95×10^{-4} moles O_3/min . Cyanides transformed to cyanate. Then, it seems that cyanate partially transforms to carbonates and nitrates. The anions sulfate, chloride and fluoride seem to be present in the original cyanide solution. By mass balance it is possible to determine the percentage of each species. In the case in which 3.90×10^{-4} moles O_3/min ozone was used, 80% cyanide was transformed to cyanate and $\approx 20\%$ was transformed to carbonate and nitrate. These results are for 7 days and for 28 days after treatment with ozone. In the case in which 1.95×10^{-4} moles O_3/min ozone was used, 60% cyanides transformed to cyanate and $\approx 40\%$ in carbonate and nitrate. These results are consistent with the feeding rate of ozone used in each case.

Table 4.10: Anionic components of the reaction of cyanide at different feeding rate ozone

Ozone - 3.90×10^{-4} moles O_3/min		
time (days):	7	28
Components:	Concentration (ppm)	
CO_3^{-2}	818	868
CNO ⁻	1 456	1 488
NO_3^-	793	790
SO_4^{-2}	280	270
Cl ⁻	169	126
F ⁻	68.9	42.8

Ozone - 1.95×10^{-4} moles O_3/min		
time (days):	2	24
Components:	Concentration (ppm)	
CO_3^{-2}	8.00	802
CNO ⁻	969	987
NO_3^-	1 400	1 424
Cl ⁻	268	268
SO_4^{-2}	187	137
F ⁻	52.7	45.4

Table 4.11 shows the retention time for the following ions: cyanate, carbonate, nitrate, sulfate, chloride and fluoride. Figure 4.11 shows two chromatograms where the position of the different components appears; the first chromatogram is representative of degradation with sodium hypochlorite and the second chromatogram is representative of degradation with ozone. The area of the peaks leads to obtain the quantitative amount of each component (it is necessary to use patterns to obtain the corresponding values).

Table 4.11: Retention time (min) for different anions

Components:	F ⁻	Cl ⁻	CO ₃ ⁻²	CNO ⁻	SO ₄ ⁻²	NO ₃ ⁻
Retention Time (min)	3.10	4.56	5.37	6.10	6.57	9.72

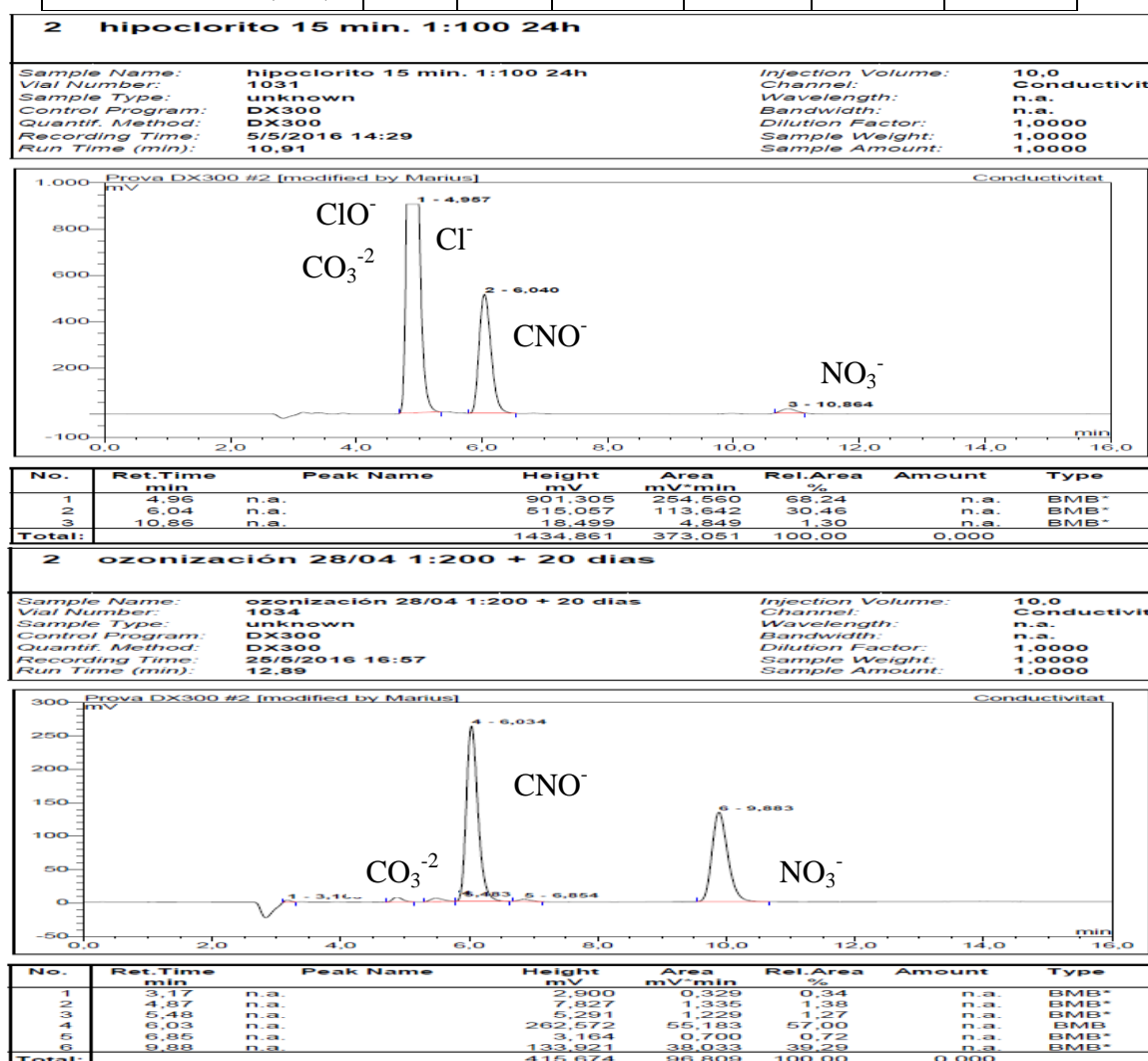


Figure 4.11: Chromatograms of the reaction of cyanide with sodium hypochlorite and ozone.

4.3. Leaching of Gossan ore by cyanidation

4.3.1. Experiment of cyanidation

The experimental procedures are included in section 3.3.3. The result for analysis includes the determination of the degradation of cyanide in cyanidation process over time and analysis to determine the gold and silver recovery.

A) Determination of the degradation of cyanide over time

Table 4.12 shows the evolution of cyanide concentration in this test. The cyanide concentration decreases after 10 hours of stirring to a concentration of 1.57 g/L CN^- . Additionally, the final concentration of cyanide was 1.47 g/L CN^- after recovering gold and silver with Zn precipitation. Figure 4.12 shows the results presented in Table 4.12.

Table 4.12: Degradation of cyanide in leaching by cyanidation

Time (Hours)	CN^- remaining concentration (g/L)	% CN^- undegraded
0	2.16	100
1	2.11	97.7
2	2.01	93.2
4	1.96	90.9
6	1.76	81.8
8	1.62	75.0
10	1.57	72.7
After gold and silver recovery	1.47	68.1

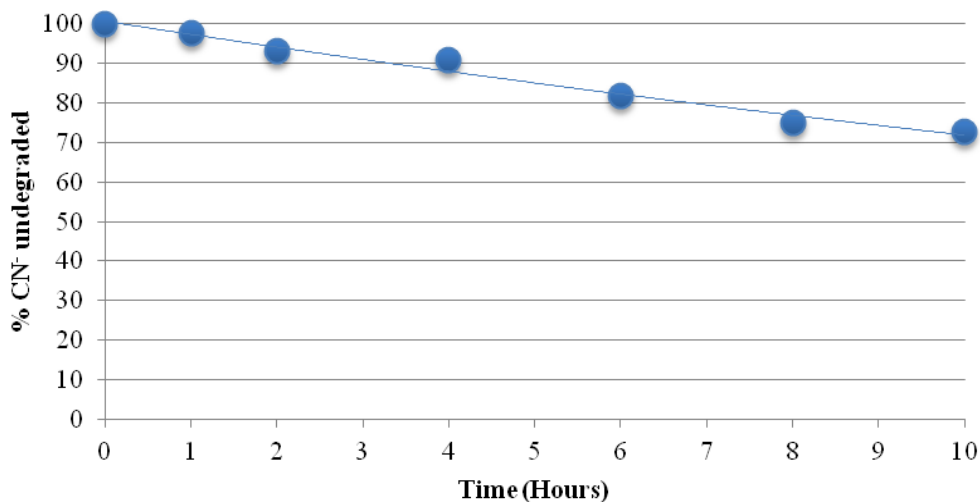


Figure 4.12: Degradation of cyanide in leaching by cyanidation

B) Gold and silver extraction over time

Table 4.13 and Figure 4.13 shows gold and silver dissolved respect to its content in Gossan ore and the extraction versus time. The silver extraction was completed between 4 and 8 hours reaction. The level of extraction was 51.3 ppm of silver and 1.60 ppm of gold contained in Gossan ore dissolution. Original Gossan ore has 87.4 g/t (ppm) of silver and 2.00 g/t (ppm) of gold.

Table 4.13: Gold and silver extraction in cyanidation process

Time (hours)	Ag dissolver (ppm)	Ag (%)	Au dissolver (ppm)	Au (%)
0	0	0	0	0
1	39.7	45.4	0.840	42.0
2	62.4	71.4	1.16	58.0
4	63.7	72.9	2.16*	108*
6	64.1	73.4	1.32	66.0
8	65.2	74.6	1.12	56.0
10	63.1	72.2	1.44	72.0
24	55.9	63.9	0.960	48.0
Before gold and silver recovery	51.3	58.7	1.92	96.0

*Anomalous value.

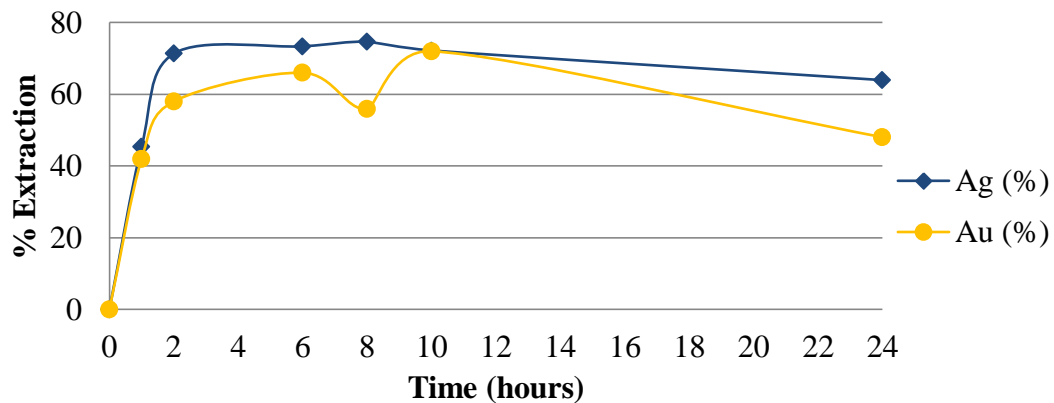


Figure 4.13: Gold and silver extraction curve in cyanidation process

4.3.2. Gold and silver recovery

After separating by settling, the liquid was stirred with zinc powder (1g) and lead nitrate (0.1g). The lead nitrate activates the zinc surface previously to the noble metals

precipitation. Table 4.14 shows the gold, silver and copper are easily precipitated from the solution. Zn passes to the solution obtaining there 277 ppm Zn.

Table 4.14: Precipitation of metals with Zn powder

Elements	Before precipitation (ppm)	After precipitation (ppm)
Ag	10.9	<0.500
Au	1.22	<0.100
Cu	5.98	4.16
Fe	1.24	0.630
Zn	0.320	277

4.4. Detoxification of the effluents and solid waste generated during the cyanidation process

4.4.1. Detoxification of cyanide with sodium hypochlorite (NaClO)

Figure 3.1 shows the technique of determination of free cyanide (indicated in section 3.3.1.), for determining the remaining cyanide after sodium hypochlorite addition to the dissolution of cyanides at room temperature.

A. Detoxification of effluents

Table 4.15 shows the results obtained after the addition of sodium hypochlorite obtaining a cyanide degradation of 99%.

Table 4.15: Detoxification of effluents using NaClO as oxidant

Agitation time (min)	Vol. Add NaClO (mL)	Equivalent amount of Cl ₂ (mg)	CN ⁻ Remaining concentration (g/L)	CN ⁻ degraded (%)
0	0	0	1.47	0
15	2.20	34.7	0.03	99

B. Detoxification of the solid waste

The final concentration of cyanide in the pulp was 1.26 g/L CN⁻. Table 4.16 shows that cyanides concentration decreased after the addition of 1.80 mL of NaClO in 15 minutes agitation. The cyanide degradation level was higher than 98%. This decontaminated solid carry out to slag heap.

Table 4.16: Detoxification of solid waste using NaClO as oxidant

Agitation time (min)	Vol. Add NaClO (mL)	Equivalent amount of Cl ₂ (mg)	CN ⁻ Remaining concentration (g/L)	CN ⁻ degraded (%)
0	0	0	1.26	0
15	1.80	28.4	0.04	>98

4.4.2. Detoxification of cyanide with ozone (O₃)

Figure 3.2 shows the technique of determination of free cyanide (indicated in section 3.3.1.), for determining the remaining cyanide after ozone was added to the dissolution of cyanides.

A. Detoxification of effluents

Table 4.17 shows the results obtained after the addition of ozone, obtaining a cyanide degradation of > 99%.

Table 4.17: Detoxification of effluents using O₃ as oxidant

Ozonation Time (min)	CN ⁻ remaining concentration (g/L)	CN ⁻ degraded (%)	O ₃ (moles)
0	1.47	0	0
15	0.03	>99	0.0117

B. Detoxification of the solid waste

The final concentration of cyanide in the pulp was 1.26 g/L CN⁻. Table 4.18 shows that cyanides concentration decreased after 15 minutes, using an amount of 0.0117mole O₃. The cyanide degradation was >99%. This decontaminated solid can carry out to slag heap.

Table 4.18: Detoxification of solid waste using O₃ as oxidant

Ozonation Time (min)	CN ⁻ remaining concentration (g/L)	CN ⁻ degraded (%)	O ₃ (moles)
0	1.26	0	0
15	0.03	>99	0.0117

4.5. Mass Balance of the process and was balance referent to the Gold, Silver and CN⁻ behavior.

Figure 4.14 shows a flow sheet of the developed process including a mass balance related with gold, silver and cyanides, applied to a Gossan ore from Rio Tinto (Huelva, Spain). According to this, the recovery of gold and silver was obtained the extraction was of 60% silver and 75% gold, also the cyanides remaining in the circuit can be degraded to a level higher than 99%.

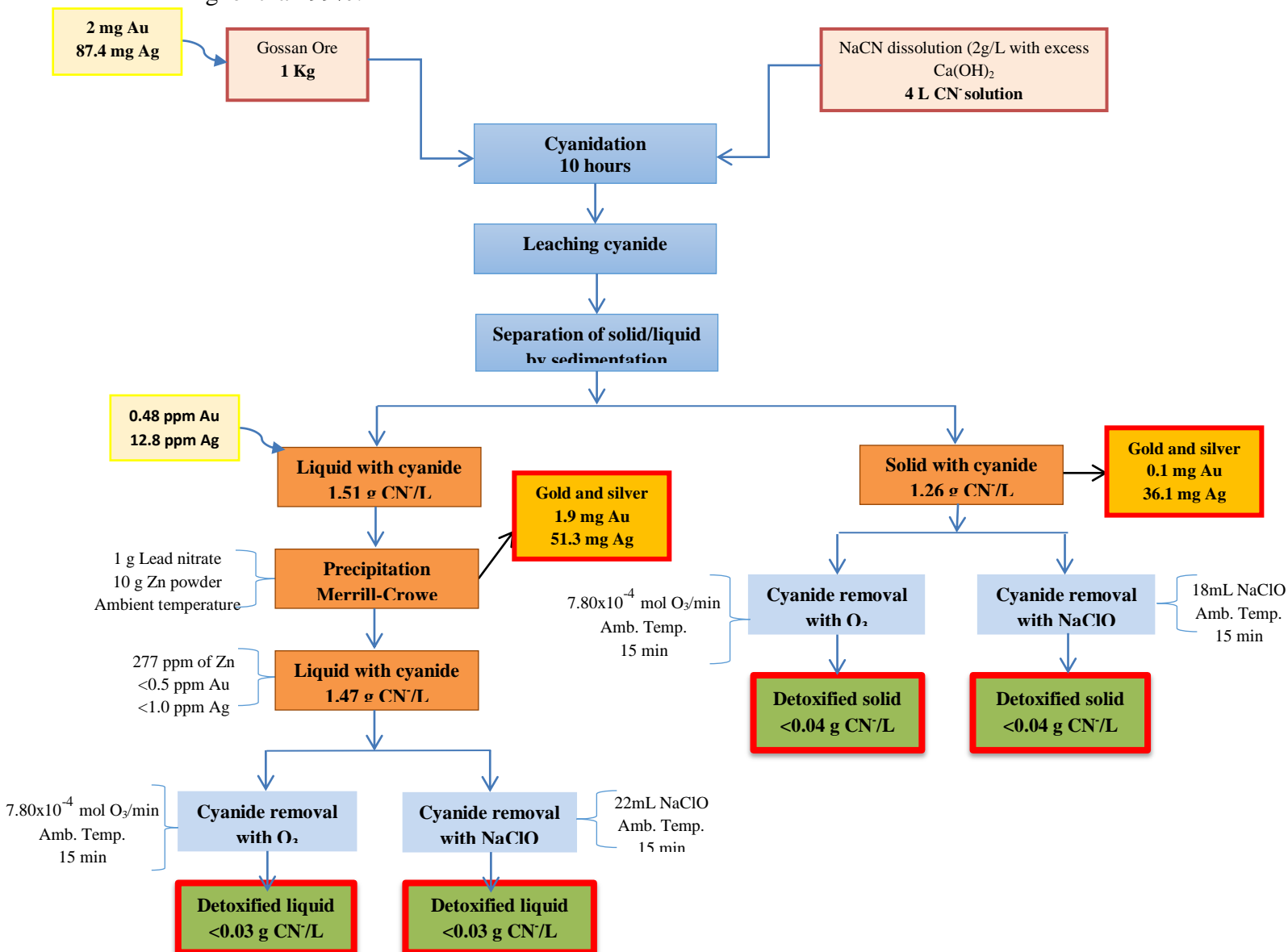


Figure 4.14: Mass Balance of the process and was balance referent to the Gold, Silver and CN⁻ behavior.

5. CONCLUSIONS

- The main elements in Gossan ore are iron and silicium as majority; minority elements are aluminium, lead, barium, arsenic, potassium, magnesium, titanium, calcium, antimony, copper and tin. The elements considered <0.1% weight are trace. Original Gossan ore has 87.4 g/t (ppm) of silver and 2.00 g/t (ppm) of gold.
- The main mineralogical species in Gossan ore are quartz, barite, goethite, hematite, beudantite and jarosite. MOR and SEM-EDX confirms the presence of these species.
- The cyanide (2 g/L of NaCN) is completely degraded using 3 mL of sodium hypochlorite solution (47.4g/L active chlorine) at room temperature. The detoxification of cyanides is higher than 99%, obtaining concentrations <40 ppm (<0.04g/L) of cyanide in 15 minutes, forming cyanates, less toxic than cyanides.
- The stoichiometry of reaction between active chlorine and cyanide indicates that it is necessary 3.5 mole active chlorine per mole of cyanide.
- The cyanide (2 g/L of NaCN) is completely degraded using ozone-oxygen (7.80×10^{-4} moles O_3 /min) at room temperature. The detoxification of cyanides is higher than 99%, obtaining concentrations <30 ppm (<0.03g/L) of cyanide in approximately 15 minutes ozone feeding rate, forming cyanates, carbonates and nitrates.
- The stoichiometry of reaction between ozone and cyanide indicates that it is necessary 2 mole ozone per mole of cyanide.
- The alkaline chlorination and ozonation are techniques to degrade cyanides acting as strong oxidants, reducing effluent toxicity and solid waste.
- Cyanidation of gossan ores leads to obtain extraction levels of 51.3 ppm of silver and 1.60 ppm of gold. The extraction was of 60% silver and 75% gold.
- The oxidation processes by using sodium hypochlorite or ozone-oxygen to the effluents and wastes generated during the Gossan ore cyanidation, leads to obtain degradation levels of 99%, leaving amounts of cyanide less than 0.030 g/L.

6. SUGGESTIONS

- Ozone and hypochlorite emerges as an interesting treatment of effluents of cyanidation option. It is necessary to make an economic study to compare the processes that most currently used in mining.
- To do a complementary study of the formation of nitrate.

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