

# **Integrated Eco-sustainable System for Alluvial Gold Leaching- Recovery**

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## **Abstract**

An Integrated system for alluvial gold leaching-recovery was design to leach gold from alluvial ore as alternative to mercury and cyanide leaching. NaClO is obtained by NaCl 1 % *in-situ* electrolysis at pH 2 and used as leachate solution. Gold leaching optimization is achieved using a rotary drum reactor fed with the leaching solution, the process takes 6 hours and 95 % of gold recovery is obtained, the remnant gold from the alluvial ore is treated in an stationary reactor with NaClO 200 ppm at pH 2; reaching 99.6 % recovery of the total alluvial gold. This leaching-recovery alluvial system has the potential to replaced mercury and cyanide leaching process.

*Keywords:* leaching, chloride, hipochloride, rotary drum reactor, alluvial gold, alluvial ore.

## **Introduction**

Artisanal gold (alluvial gold) production in Peru is mainly represented by the departments of Puno, Arequipa, Piura and Madre de Dios. In such places, artisanal gold extraction commonly uses mercury for its amalgamation and subsequent elimination by heating, which leads to mercury being found not only in the air, but also in water and soil.<sup>8</sup> High use of mercury in Madre de Dios by formal and informal artisanal mining, is affecting the natural reserves and is causing serious damage to inhabitants health.<sup>9</sup> It is estimated that around 30,000 artisanal miners are in Madre de Dios.<sup>8</sup> In recent decades, research has been conducted to find alternative chemical substances to avoid mercury and / or cyanide used for gold extraction. Thiourea<sup>10</sup>, thiocyanate<sup>11</sup>, ammonia<sup>12</sup> and others<sup>13</sup> can be used as alternatives to cyanide for gold leaching. Additionally, chlorine is a powerful oxidant used for gold leachate during the second half of the century; however, it was replaced for cyanidation process due to the low cost. The environment needs an eco-friendly alternative to gold leaching, for this reason, chlorine and hypochlorite are being used as an interesting non-polluting proposal<sup>1,14,15</sup>.

Publications of recent years, make reference to the use of chloride-hypochlorite for gold leaching in minerals containing pyrite obtaining  $\text{AuCl}_4^-$ <sup>2-4,16</sup>. Hypochlorite produced by electrolysis of sodium chloride has been used to leach gold from the mercury amalgam<sup>5,6</sup>. In this research work an integrated system is designed for the leaching of gold in alluvial ores using sodium hypochlorite in sodium chloride at pH 2 as a leaching solution.

## **2. Experimental part**

### *2.1 Preparation of the sample*

The alluvial ore sample was obtained from the "La Familia" Mining Concession of the lower Puquiri, belonging to Madre de Dios department, Peru. The sample was homogenized by roll and quartering, from which two portions were taken at random, mixed and separated into fractions and storage. The alluvial gold was separated from the alluvial ore sample using a gravimetric table, obtaining alluvial sand as a remnant of the process.

### *2.2. Characterization of the alluvial ore sample*

The mineralogical characterization of the alluvial sand sample was made using a Nikon Trinocular Stereomicroscope SMZ-745T. Additionally, a semiquantitative mineralogical analysis of all present minerals (crystalline phases) was performed by X-ray diffraction with a D8 Advance Tube Co-diffractometer (38kV, 25mA): KAlfa1: 6930.48 eV KAlfa2: 1.7891 Å, Filter: Kbeta: Ni.

The alluvial gold separated by gravimetry was isolated by the Fire Assay technique<sup>17</sup> and quantified with an atomic absorption spectrometer using a Perkin Elmer - Analyst 200 Atomic Absorption Spectrometer with a hollow cathode lamp of Au at  $\lambda = 242.8$  nm. For the analysis of the remaining gold in the alluvial sand, 100 g of sample was weighed, a solid / liquid dilution (S/L) was made in a proportion of 5 to 2. Following this, 300 ppm of NaCN was added and the pH was adjusted to 11 with NaOH 4 mol L<sup>-1</sup>. The solution was stirred for 2 hours and then left to stand for 3 days at room temperature and analyzed by atomic absorption spectrophotometry.

### *2.3. Gold corrosion studies by cyclic voltammetry*

For electrochemical studies, a commercial gold electrode of 0.08 cm<sup>2</sup> area was used, which was previously treated with concentrated HNO<sub>3</sub> for 5 minutes. Then, it was washed with distilled water, and immersed in a H<sub>2</sub>SO<sub>4</sub>/H<sub>2</sub>O<sub>2</sub> solution 1:1 v/v, finally it was washed with distilled water.

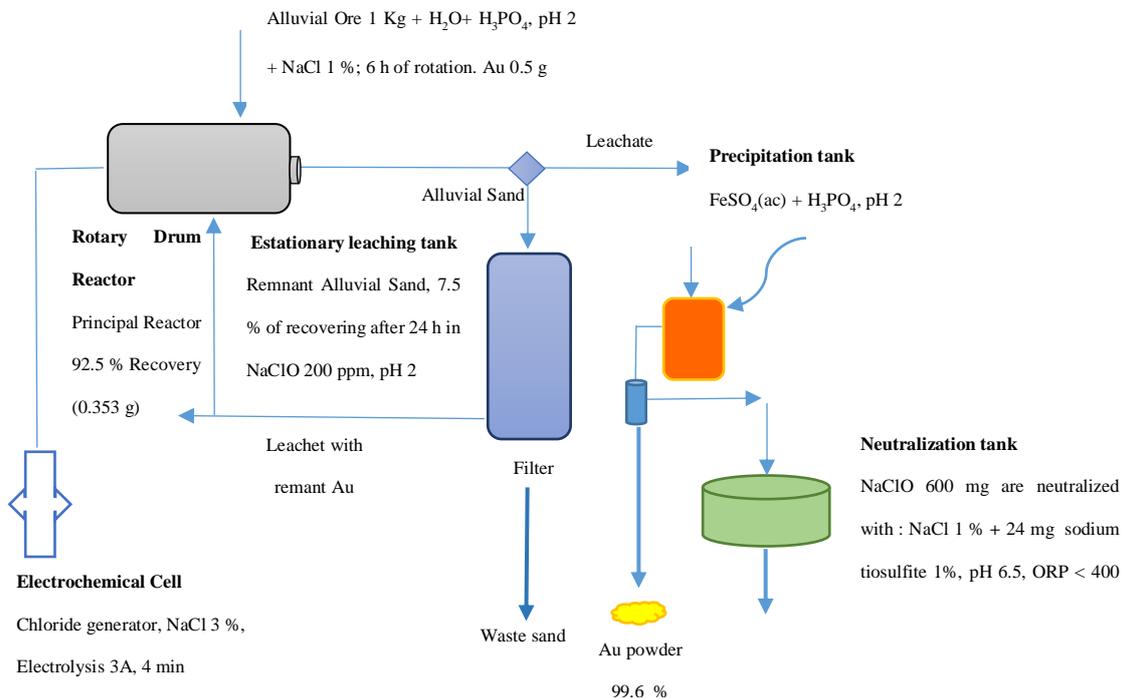
The commercial gold corrosion studies were performed in a three electrode electrochemical cell. A commercial gold electrode was used as the working electrode, a high surface area graphite electrode was used as the auxiliary electrode, and Ag / AgCl as a reference electrode saturated in KCl 3 mol L<sup>-1</sup>. The measurements were made using a Potentiostat-Galvanostat PalmSens. The Na<sub>2</sub>SO<sub>4</sub> 0.1 mol L<sup>-1</sup> was used as supporting electrolyte, the solution was deoxygenated by N<sub>2</sub> bubbling. The measurements were made by cyclic voltammetry in a potential window from 0 to +1.5 V vs Ag / AgCl, at a scanning speed of 100 mV s<sup>-1</sup>. During the measurements, successive additions of NaCl were made from 1,000 ppm to 10,000 ppm. After this, the effect of the pH was evaluated (pH: 2, 3, 4, 5), adjusting the pH with concentrated HCl. Additionally, the effect of hypochlorite in the leaching solution was studied, 10,000 ppm NaCl was used as the supporting electrolyte, 40,000 ppm of NaClO was added and it was evaluated at different pH values (2,3 and 4).

### *2.4. Integrated system for alluvial gold leaching-recovery*

The integrated system for alluvial gold leaching-recovery consists of an electrochemical filter-press cell, which generates chlorine in the form of sodium hypochlorite in situ from sodium chloride electrolysis at pH 2. The electrolyzed solution enters to the rotary drum reactor to leach the alluvial gold, time rotation programmed was 6 hours, after the set

time, the leached solution is filtered and sent to the next stage in which it proceeds to gold precipitation by adding dilute ferrous sulfate in a stoichiometric ratio of 2:1 with respect to gold. After 1 hour of resting solution, the metal is separated by filtering and dried it for storage.

In an internal stage of the lixiviation process, the alluvial sand coming from the rotary drum reactor is separated from the leachate, and it is taken to a stationary leaching tank in which it will finish recovering the remaining unleached gold in the drum, after 24 hours the leachate of the tank stationary arrives to leach the total gold contained in the sand, the solution obtained in this stage can be used to feed the rotary drum reactor electrolyte and process a new load of sand for leaching.



**Figure 1:** Flow diagram of the integrated alluvial gold lixiviation-recovery system

A type H cell with cationic membrane separation was used for in situ sodium hypochlorite generation, commercial Ru-Ir / Ti electrode was used as anode and sodium chloride 3% was used as anolyte. The efficiency was determined by the iodometric method and neutralized with sodium thiosulfite. To determine the efficiency, 40 mL of 2 % NaCl was added to the anodic compartment once the oxidation process was finished. Aliquots of 15 mL of a total of 100 mL were taken. It was placed in a flask and 5 mL of glacial acetic acid and 1 g of KI<sub>(s)</sub> were added. Following this, a titration was performed with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> 0.01 N, previously standardized with K<sub>2</sub>Cr<sub>2</sub>O<sub>7(ac)</sub> 0.1 N.

The theoretical production of active chlorine under the same conditions was determined with the following equation:

$$m_{prod} = \frac{M_{eq} \times I \times t}{96500 \frac{C}{mol}} \quad \text{Eq. 1}$$

The general equation for electrolysis time setting is:

$$t_{horas} = 75.5 \frac{g.de\ cloro}{AxEf} \quad \text{Eq. 2}$$

Once, the electrolyte is produced in the press-type electrochemical cell, it enters to the rotary drum reactor containing the alluvial ore sample, 1 % NaCl and water, the pH was adjusted to 2 with phosphoric acid. To control the consumption of hypochlorite, the oxidation-reduction potential (ORP) instrument is used. The remaining gold, whose kinetics is slower due mainly to the existence of passivating components in the alluvial ore, it is transferred to a stationary leaching vessel, mixed with a solution of 200 ppm of sodium hypochlorite at pH 2 during 24 hours.

The leached gold was recovered by precipitation using ferrous sulphate at pH 2, with a 3:1 Fe / Au ratio, which represents 200 % of the stoichiometric value.

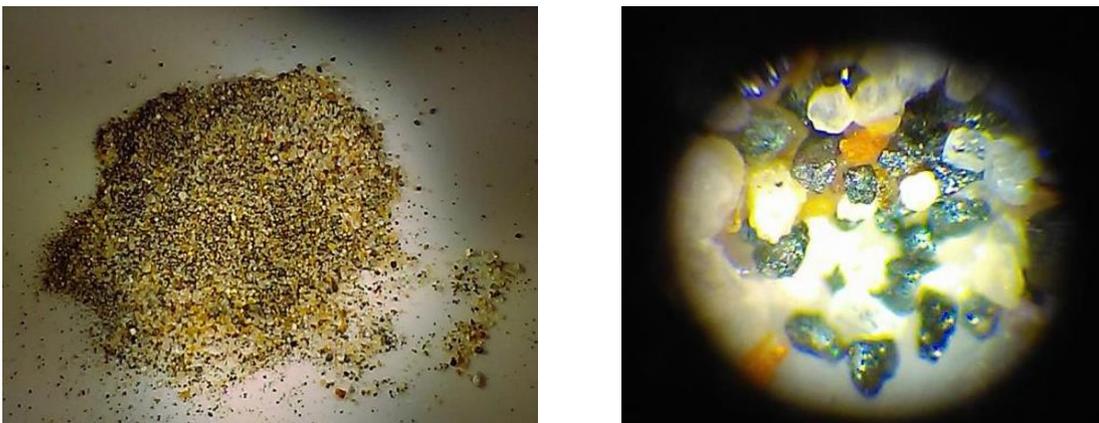
## 2.6. Alluvial gold and alluvial ore leaching tests

For the alluvial ore leaching tests, the reactor was loaded with 1 L of sodium hypochlorite solution produced in the electrochemical cell, 500.56 mg of alluvial gold was added in 500 g of alluvial sand and the rotating operation was started. During the process samples of the rotary drum reactor were taken every 30 minutes to observe the evolution of the oxidation reduction potential (ORP) of the process. The amount of leached gold is determined by atomic absorption spectrometry. Additionally, an alluvial gold leaching test previously separated from the alluvial sand was carried out, in order to know the influence of the sand on the leaching rate. For this experiment 500 mg of alluvial gold was used.

## 3. Results and discussion

### 3.1 Physicochemical characterization of the alluvial ore

Alluvial ore mineralogical characterization by Trinocular Stereoscopic Loupe is observed in Figure 2B, the mineralogical characteristics of the possible components are observed in Table 1.



**Figure 2:** A) Alluvial ore sample. B) Images of the alluvial ore sample with the Trinocular Stereo microscope

**Table 1:** List of Possible Components in the alluvial ore sample

<b>Component</b>	<b>Color</b>
Quartz (SiO <sub>2</sub> )	White
Magnetite (Fe <sub>3</sub> O <sub>4</sub> )	Brilliant black
Malachite (Cu <sub>2</sub> CO <sub>3</sub> (OH) <sub>2</sub> )	Green
Calcite (CaCO <sub>3</sub> )	White
Sulfur	Yellow
Others	...

The results of the mineralogical analysis by X-ray diffraction, expressed as relative mass percentage (g / g) of the alluvial sand sample are presented in Table 2. Where it can be seen that the grit contains quartz, zircon, ilmenite, hematite and pyrrhotite.

**Table 2:** Result of the Mineralogical Analysis by XRD of the alluvial sand sample

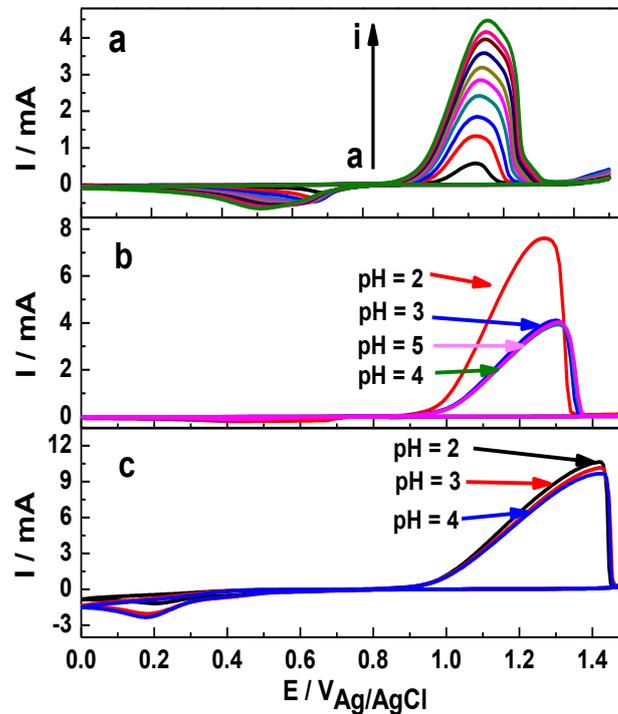
<b>Name of the mineral</b>	<b>General formula</b>	<b>Approximate result (%)</b>
Quartz	SiO <sub>2</sub>	44
Zircon	ZrSiO <sub>4</sub>	39
Ilmenite	FeTiO <sub>3</sub>	14
Hematite	Fe <sub>2</sub> O <sub>3</sub>	1
Pyrrhotite	Fe <sub>1-x</sub> S	1
Phlogopite	KMg <sub>3</sub> (Si <sub>3</sub> Al)O <sub>10</sub> (F,OH) <sub>2</sub>	<L.D

D.L .: Detection Limit

The alluvial gold analysis by the Fire Assay Method shows a gold content of 80.76 %, 98 ppm of Au was found and 0.02% of the residual gold present in the alluvial sand.

### 3.2. Gold corrosion studies by cyclic voltammetry

Figure 3 shows the cyclic voltammograms for commercial gold, using  $\text{Na}_2\text{SO}_4$   $0.1 \text{ mol L}^{-1}$  as supporting electrolyte, aliquots of NaCl were added to vary the concentration in the range of  $1 \text{ g L}^{-1}$  to  $10 \text{ g L}^{-1}$ . According to the Pourbaix diagram, the  $[\text{AuCl}_4]^-$  is the most stable species of soluble gold chloride at high  $\text{Cl}^-$  concentrations in the pH range of 0-8<sup>18</sup>. The curves of the Figure 3a show the typical behavior for gold corrosion, the corrosion peak is observed at approximately +1.2 V, it is due to the formation of the  $\text{Au}^{3+}$  ion mainly in its complex form  $[\text{AuCl}_4]^-$ . In addition, it is observed that the corrosion current increases with the of NaCl concentration increment.



**Figure 3:** a. Cyclic voltammetry for Au in  $\text{Na}_2\text{SO}_4$   $0.1 \text{ mol L}^{-1}$ . Concentration of NaCl from  $1 \text{ g L}^{-1}$  to  $10 \text{ g L}^{-1}$ .  $v = 100 \text{ mV s}^{-1}$ . b. Cyclic voltammetry for Au

in NaCl 10 g L<sup>-1</sup>, at pH 2, 3, 4 and 5.  $v = 100 \text{ mV s}^{-1}$ . c. Cyclic voltammetry for the Au in NaCl of 10 g L<sup>-1</sup>, NaClO of 40 g L<sup>-1</sup> NaClO 40 g L<sup>-1</sup> at pH 2, 3 and 4.  $100 \text{ mV s}^{-1}$ .

Figure 3b shows the cyclic voltammeteries for gold in NaCl 10 g L<sup>-1</sup> as supporting electrolyte, the measurements were made at different pH between 5 to 2. The chlorine gas in aqueous solutions also acts forming three oxidizing species, depending on the pH ( $\text{Cl}_{2(\text{ac})}$ ), hypochlorous acid (HClO) and hypochlorite ions ( $\text{ClO}^-$ ). These species can be generated by the addition of sodium hypochlorite (NaClO) or calcium hypochlorite ( $\text{Ca}(\text{OCl})_2$ )<sup>19</sup>, these salts are ionized in water, under acidic conditions (pH <7.5) the hypochlorite ion becomes hypochlorous acid. Under very acid conditions (pH <3.5) and in the presence of chloride ions, aqueous chlorine is formed. All chlorine species are powerful oxidants but HClO is the most effective. Therefore, the pH must be maintained in a range of stability of HClO<sup>20</sup>.

Figure 3b shows that at pH 2 the corrosion current highest value is obtained. Figure 3c shows gold cyclic voltammeteries in NaCl 10 g L<sup>-1</sup> / NaClO 40 g L<sup>-1</sup> at different pH values (4, 3, 2). It can be seen that the current corrosion increases when pH is increased.

The equations for gold leaching in  $\text{Cl}_2$  and HClO presence are shown below<sup>21</sup>:



Reaction (1) may correspond to that of Figure 3a where chlorine produced electrolytically in the presence of chloride ions dissolves gold in Au (III) in its complex form  $[\text{AuCl}_4]^-$ , while reaction (2) may correspond to Figures 3b and Figure 3c.

### 3.3. Integrated system for alluvial gold leaching-recovery

The integrated system for gold leaching works with chlorine generated in situ. In this work a commercial Ru-Ir / Ti electrode is used for chlorine generation. The efficiency of the commercial Ru-Ir / Ti electrode is obtained by the iodometry method, using type H cell. The results are shown in Table 3. According to the working conditions, the theoretical production of active chlorine is 661.2 ppm; experimentally a production of 395.8 ppm was obtained, being the efficiency of 59.8 %. According to the reaction stoichiometry, 1 g of Au is leached by 0.535 g of Cl<sub>2</sub>. To generate 0.535 g of Cl<sub>2</sub>, at a current of 10 A, and an efficiency of 59.8%, 4.05 minutes of electrolysis are required. In the rotary drum reactor, a gold leaching of 97.5 % is obtained, 6 hours was determining as reaction time. On average, 7.5 % of undissolved gold remains.

**Table 4:** Ru-Ir/Ti efficiency by iodometric method.

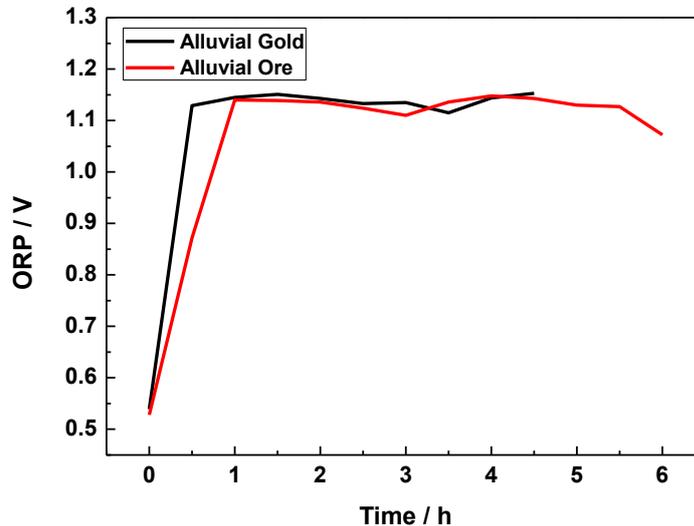
Electrode	Sample volume / (mL)	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 0.01 N / (mL)	Free chlorine / (ppm)	free Chlorine <sub>average</sub> / (ppm) %	Efficiency / %
Ru-Ir/Ti	15	17.8	398.8	395.8	59.8
	15	17.7	396.6		
	15	17.5	392.1		

The unleached gold is treated in a stationary alluvial mineral leaching container, it takes 24 hours to leach 100% of the gold with a solution of NaClO 200 ppm at pH 2. The potential was controlled by the oxidation-reduction potential (ORP) instrument, at a pH 2 the potential is maintained at a value higher than 1000 mV, the decrease in value is interpreted as the total consumption of the NaClO and the need to add a second dose to complete the leaching. The variation of hypochlorite consumption can be associated to

the nature of the sand and clays that accompany the gold, so the need to control the pH and ORP to regulate it throughout the process must be considered.

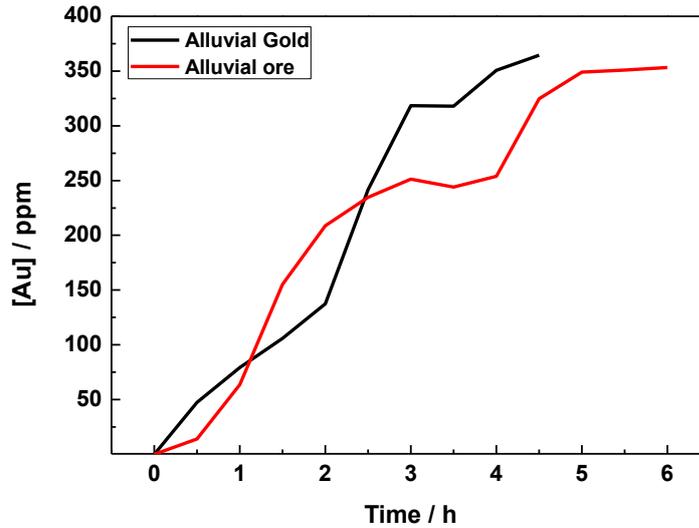
### 3.3.1. Alluvial gold and alluvial ore leaching tests

The results of the oxidation reduction potential (ORP) evolution during alluvial gold leaching in the rotary drum can be seen in figure 5. In the absence of NaClO a potential of 539 mV is obtained (time 0), the potential is rises to 1129 mV when NaClO is added and continues stable during 4.50 hours of rotation. Figure 4 shows the evolution curve. In the case of alluvial ore throughout the process, the ORP remained at values above 1000 mV, ensuring the necessary amount of hypochlorite for leaching. Comparing the alluvial gold with the alluvial ore (Figure 4), it can be seen that it is necessary more time in contact of the leaching solution with the sample containing the alluvial sand in comparison to the alluvial gold.



**Figure 4:** Analysis of ORP vs. days number. Black line: Alluvial Gold; Red line: Alluvial ore. During the leaching in the rotary drum reactor.

During the reaction, aliquots were extracted from the solution and analyzed by atomic absorption, the curve of Au ppm leached as a function of time can be seen in figure 5. In the case of gold leached from alluvial gold the analysis of the law of the Gold employee was 76.31 %. From the observed in the curve it can be deduced that up to 4 hours of rotation the leaching presents a linear range, reaching up to 91.7% of the initial gold total amount. After 4 hours of rotation the slope changes drastically, reducing the leaching rate. The total amount of gold leached after 4.50 hours is 95.47 %. The gold leaching efficiency analysis of the alluvial ore after 6 hours of rotation was 92.6 % (Figure 5). In the tests carried out, it can be concluded that at the time of rotation it is not possible to recover 100 % of the gold contained in the alluvial sand, according to the behavior of the curve it can be observed that at higher values of recovery of 90 %, the Leaching kinetic speed changes drastically decreasing the leaching rate, so it becomes necessary to treat in an additional step the alluvial sand with the remnant gold. An additional stage was carried out, the alluvial sand that was discharged from the rotary drum reactor was placed in a container with NaClO solution under the same conditions of pH and NaCl concentrations used in the reactor. The ORP value was higher than 1000 mV, the analysis performed at 24 hours of rest has a content of 380 ppm of gold, therefore in the process, including this last stage of stationary leaching, and the total recovery of gold from the alluvial mineral was 99.6 %

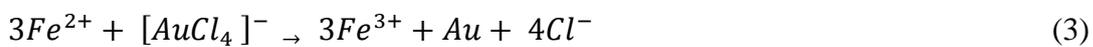


**Figure 5:** Gold concentration curve in the leachate

The leached solution is separated by filtration using filtration by gravity. In a second stage, the filtered solution is conditioned to pH 1.2 by adding phosphoric acid. The ferrous solution is prepared by dissolving iron sulphate in a 3/1 ratio to the gold content, resulting from the experimental value found for maximum precipitation efficiency. The gold is dried and weighed.

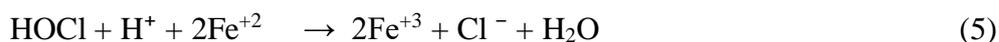
The instrument of ORP and pH meter are used to control the precipitation process at < 320 mV and pH 1.2.

The reaction involved in the process is:



and the stoichiometric ratio is: 1.41 g  $FeSO_4 \cdot 7H_2O$  / 1 g Au

However, the leached solution still contains a remnant of hypochlorous acid evidenced by the high ORP value. In this case the iron sulphate acts as a reducer according to the following reaction.



Therefore, it is acceptable that a greater amount of ferrous sulphate is required to achieve the gold total precipitation in the leached solution. In principle, 100% excess is used. The gold law obtained was 99.91%

#### **4. Conclusion**

The importance of recovery the total gold amount from alluvial ore without using mercury or cyanide is a necessity for miner artisanal and to decrease the impact environment. As an alternative, NaClO can be used as leachate, NaClO 10 000 ppm in NaCl 1 % at pH 2 is the optimal concentration for commercial gold according to corrosion studies by cyclic voltammetry. For the remnant gold in the alluvial ore, an integrated system for alluvial gold leaching-recovery was used to achieve 99.6 % of Au leaching at the end of the process. The proposed system could be used as an environmental friendly alternative to gold cyanidation for extracting Au from low-grade ore.

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#### **Declarations**

##### **a) Availability of data and materials**

The datasets during and/or analysed during the current study available from the corresponding author on reasonable request.

##### **b) Competing interests**

The authors declare that they have no competing interests

**c) Funding**

The authors declare that this research was financed with funds from the FONDECYT-CONCYTEC contract No. 209-2015, covering expenses for materials, equipment, analysis, interpretation and writing of the manuscript

**d) Authors' contributions**

UQ, carried out the experimental work and data treatment, A.B contributed to the discussion of results and writing the manuscript and ALRT directed the entire research project, contributed with the conceptual idea and the integration of the processes. All authors read and approved the final manuscript.

**e) Acknowledgements**

Not applicable

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