

# Characterization of gold-bearing tailings by diagnostic leaching for reprocessing by flotation and leaching [Caracterización de relave aurífero mediante lixiviación diagnóstica para reproceso mediante flotación y lixiviación]

Juan Antonio Vega González <sup>a</sup>, Nilthon E. Gutierrez Zavaleta <sup>a</sup>, Jheri A. Quispe Cueva <sup>a</sup>.

<sup>a</sup> Facultad de Ingeniería, Universidad Nacional de Trujillo. Av. Juan Pablo II s/n – Ciudad Universitaria, Trujillo, Perú.

\* [jvega@unitru.edu.pe](mailto:jvega@unitru.edu.pe)

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## Resumen

El propósito de la presente investigación es la evaluación para reprocessar los relaves de Sayapullo, para extraer el oro y la plata que aún posee, para lo cual se realizó un análisis químico inicial, análisis granulométrico y pruebas de flotación del mineral para oro y plata, luego se determinó el porcentaje de La extracción de oro se realizó por diagnóstico de lixiviación oxidativa (DLO).

La DLO consiste en la cuantificación del porcentaje de extracción de oro por cianuración a medida que aumenta el estado de oxidación del mineral. Para ello se realizaron ataques químicos progresivos con agentes oxidantes para cada etapa en el siguiente orden: Carbonato de Sodio ( $\text{Na}_2\text{CO}_3$ ), Ácido Clorhídrico (HCl), Ácido Sulfúrico ( $\text{H}_2\text{SO}_4$ ) y Ácido Nítrico ( $\text{HNO}_3$ ). El contenido de oro en las soluciones se determinó por ICP-OES y en los sólidos por ensayo al fuego.

Se concluye que los relaves de Sayapullo tienen 44,68% de oro libre, 3,47% de oro asociado a sulfatos, 11,64% de oro asociado a carbonatos, 6,13% de oro asociado a sulfuros de Cu-Zn, 31,49% asociado a piritas y arsenopiritas; y 2,60% de oro asociado a silicatos.

**Palabras clave:** Lixiviación, DLO, flotación, relaves

## Abstract

The purpose of the present investigation is the evaluation to reprocess the Sayapullo tailings, to extract the gold and silver that it still has, for which an initial chemical analysis, granulometric analysis and mineral flotation tests for gold and silver were made, then The percentage of gold extraction was performed by diagnosis of oxidative leaching (DLT).

The DLT consists of the quantification of the percentage of gold extraction by cyanidation as the oxidation state of the mineral increases. For this, progressive chemical attacks were carried out with oxidizing agents for each stage in the following order: Sodium Carbonate ( $\text{Na}_2\text{CO}_3$ ), Hydrochloric Acid (HCl), Sulfuric Acid ( $\text{H}_2\text{SO}_4$ ) and Nitric Acid ( $\text{HNO}_3$ ). The gold content in the solutions was determined by ICP-OES and in the solids by fire assay.

It is concluded that the Sayapullo tailings have 44.68% free gold, 3.47% gold associated with sulfates, 11.64% gold associated with carbonates, 6.13% gold associated with Cu-Zn sulfides, 31.49% associated with pyrites and arsenopyrites; and 2.60% gold associated with silicates.

**Keywords:** Leaching, DLT, flotation, tailings.

## 1. Introduction

Extractive mining is based on the extraction of valuable metals contained in ores, these metals can be extracted by different types of metallurgical processes, however, refractory minerals cannot be efficiently processed by any of the known metallurgical processes, In the case of conventional cyanidation of a refractory mineral, it generates a low recovery, causing an excess of cyanide consumption. In addition, it is important to consider that the particle size is a variable that influences the metallurgical processes in the extraction of valuable metals, since it has been proven that the smaller the material to be processed, the greater the extractions. (Misari, 2010).

On the other hand, in the old extractive metallurgical processes, due to ignorance or lack of adequate technology, the tailings were discharged with grades that could now be considered important for their extraction. The most widely used technique for gold extraction by hydrometallurgical is cyanidation. (Misari, 2010)

One way to characterize and determine the refractoriness of gold and in which minerals gold is associated or encapsulated is diagnostic leaching (DLT), and together with scanning electron microscopy (SEM), which complements the characterization of minerals or tailings (Celep & Serbest, 2015). The complementary and very useful techniques in the characterization of minerals, is through the use of software that helps in the identification of minerals such as QUESCAM (Mohammad et al., 2017).

In this investigation, chemical analysis was carried out using ICP-OES of the tailings obtained from Sayapullo, a granulometric analysis test to determine the percentage through mesh # 200 (75  $\mu$ m), then with the data obtained, mineral flotation tests were carried out to determine the extraction of gold, with the concentrate obtained from the flotation tests, the characterization was carried out by diagnostic leaching (DLT), previously the concentrate was tested by grinding to obtain the granulometry of 90% through mesh #400 (38  $\mu$ m), with the in order to evaluate the extraction of gold and determine in which mineralogical species it is found in the highest percentage.

## 2. Materials and Methods

### a. Tailings sampling

The original gold tailings are from the district of Sayapullo, department of La Libertad, the sample corresponds to 200 kilos of tailings that was sampled by carrying out 04 test pits with an area of 1 m<sup>2</sup> and a depth of 1 m, this sample was transferred to a laboratory where it was dried at 105 °C for 24 hours in a Schemin oven, then the entire sample was homogenized by the cone method using a shovel, followed by cracking using a Jones cracker, until representative samples were obtained for the metallurgical tests and trials.



Figure 1. Sampling of Relavera Sayapullo's tailings

**b. Chemical analysis of tailings**

A 500 g sample was used for the chemical analysis of the elements silver, copper, lead, zinc, iron, arsenic and antimony, using an inductively coupled plasma optical emission spectrometer (ICP – OES), Teledyne brand, model Prody XP with dual view. For the gold analysis, a 500 g sample was used and it was carried out by means of the fire test. For the determination of the acidity of the mineral, it was carried out using the Hanna brand multiparameter pH and temperature meter, 100 g of sample was used with 200 ml of distilled water, and a magnetic stirrer where the sample was mixed with the water for 30 minutes, then the pH was measured. (Vega et al, 2021a)

**c. mineral flotation**

Mineral flotation tests were carried out, using anionic collectors due to the presence of iron sulfides, as well as MIBC foaming agent. 1 kg of ore was used with a ratio of L/S: 2/1, dosing 15 g/MT of potassium amyl xanthate anionic collector, 10 g/MT of Aero 404 dithiophosphate and 30 g/MT of Methyl Isobutyl Carbinol (MIBC). The concentrate was dried by weight and analyzed chemical for gold and silver by fire assay. Then the concentrate obtained was subjected to diagnostic leaching. (Teague, Swaminathan & Deventer, 1998).

**d. Diagnostic Leaching (DLT)**

Diagnostic leaching was developed in order to determine the extraction of free gold, or that is released after being oxidized the mineralogical species that may contain it, through the use of acids, bases and roasting in the case of carbonaceous minerals. (Lorenzen, 1995).

The DLT stages used for the Sayapululo tailings are described below.

**d.1 Cyanide Washing and Cyanidation**

A mixture of two reagents in solution was prepared, adding 0.02 g/L of NaOH (extra pure) Loba Chemie brand and 0.1 g NaCN (analytical agent) Panreac brand, the prepared solution was used after each leaching with acids.

A flash leach (QLT) was done with a liquid/solid ratio of 2/1. NaOH was added to obtain a pH of 10-11, then NaCN (analytical agent) was added to obtain a concentration of 5.0 g/L, and leaching was carried out under normal conditions, that is, at room temperature (20 °C) and a pressure atmosphere. At the end of the leaching, the pH was measured with a Hanna brand multiparameter pH and temperature meter. To measure the remaining cyanide, it was done by volumetry with AgNO<sub>3</sub> as a titrant and 5-(4-Dimethylaminobenzylidene)-rhodanine as an indicator. The pulp was filtered, the filtered solution was analyzed for gold, then the solid was washed with distilled water and filtered. The filtered solid was dried and weighed ready for the second diagnostic leaching stage (DLT). This step is repeated throughout the DLT process between each oxidative acid leach.

**d.2 Leaching with soda ash**

If the ore has minerals of calcium sulfate or sodium sulfate, these compounds and some other calcium compound containing the ore are solubilized with a prepared aqueous solution of sodium carbonate, the aqueous leaching solution is prepared with a concentration of 60 g/t of sodium carbonate (analytical agent), L/S ratio of 10/1, is boiled for 2 hours, then the sample is filtered, washed and the remaining solids are mixed in a 0.1 M HCl solution at a L/S ratio of 2:1, leach at pH 1 until acid consumption is negligible.

**d.3 Oxidizing Leaching with Hydrochloric Acid**

Of HCl (analytical agent), it was prepared at 32% by weight with distilled water, the leaching was carried out with a L/S ratio of 3/1, at an average temperature of 60 °C, it was mixed in the magnetic stirrer for 4 hours. The sample was then washed and filtered with distilled water before leaching with sodium cyanide. The final mass of the sample (dry sample before cyanidation) was recorded,

and the calculation was made to determine the weight loss due to dissolution, which could be calcite, dolomite, galena, pyrrhotite and hematite. Then the solid and dry residues are leached with sodium cyanide to extract gold similar to step (d.1).

#### d.4 Oxidizing Leaching with Sulfuric Acid

Water was added to the solids from the previous stage in order to obtain a Liquid/solid ratio of 1/1. H<sub>2</sub>SO<sub>4</sub> (analytical agent), 48% v/v, was then added, mixed with the solids for 24 hours at 80 °C, adding 5 kg/MT of hydrogen peroxide, until the potential was above 500 mV (vs SCE). During the leaching time, it was verified that the potential remained above 500 mV (± 50 mV) and the pH measurement was below 2.

It is recommended at this stage to have a stored solution of sulfuric acid at a pH of 1 and label it as "acid wash solution". After leaching the solids were filtered, the solids were mixed with the acid wash solution at a liquid/solid ratio of 2/1 and then filtered again. The solid was then washed with distilled water and filtered. The filtered solids were weighed to determine the weight lost by this stage, this is done before cyanidation.

#### d.5 Nitric Acid Leaching

Nitric acid leaching will oxidize remaining sulfides such as pyrite, marcasite, and arsenopyrite. Concentrated nitric acid is added to a beaker, where the solids from the previous stage are, working with a liquid/solid ratio of 10/1. It was boiled for 6 hours until no nitrous gases were produced. It is necessary to take into account that if the brown fumes remain after 6 hours, continue boiling for 2 or 3 more hours, adding more nitric acid if necessary. The solids were then filtered, followed by rewashing with distilled water to form a 2/1 liquid/solid ratio pulp, and then filtered.

Given the high acidity, washing is carried out up to three times. It is recommended to analyze the filtered solutions for gold by ICP-OES, to consider if there was any leaching of gold due to the formation of aqua regia, this can be due to the presence of chlorides in the distilled water. The weight was recorded and the solids were leached with sodium cyanide.

After this stage, the gold that remains in the solids, that is, what was not leached in the previous stages, is probably associated with silicates or carbon (Vega et al., 2021b).

### 3. Results

The acidity of the mineral was determined, obtaining a pH equal to 2.6, being a highly acid generator, this due to the presence of iron (10.5%), which, being in contact with the air and rainfall, generates the oxidation of sulfides. of iron, forming acidic waters.

The relative density, was carried out by the fiola method, 2.70 g/cm<sup>3</sup> was obtained. Being a value very close to the quartz material, this also corroborates given that it has a significant presence of silicates.

Table 1 shows the chemical analysis of the tailings from Sayapullo, where it is observed that it contains a high percentage of iron (10.5%), gold (2.55 g/t) and silver (72.45 g/t).

Table 1. Chemical analysis of tailings from Sayapullo ICP - OES

Sample	Au (g/t)	Ag (g/t)	Cu %	Pb %	Zn %	As %	Sb %	Fe %
Sayapullo tailings	2.55	72.45	0.04	0.03	0.35	0.18	0.07	10.5

In Table 2, there is the result of the granulometric analysis of the Sayapullo tailings, where it is observed that it has 27.40% accumulated throughput, which is a coarse material for gold leaching, however, for flotation tests it can be carried out, for characterization tests it is very important to determine the particle size and the quantification of gold. (Celep, Alp & Deveci, 2008).

Table 2. Granulometric analysis of Sayapullo tailings

N° Mesh	Opening (um)	retained weight (g)	retained weight (%)	Accumulated retained weight (%)	Accumulated passing weight (%)
50	300	80.66	16.26	16.26	83.74
70	212	54.48	10.98	27.24	72.76
100	150	96.27	19.41	46.65	53.35
120	125	45.18	9.11	55.76	44.24
170	90	69.53	14.02	69.78	30.22
200	75	13.99	2.82	72.6	27.4
270	53	46.46	9.37	81.96	18.04
325	45	66.89	13.48	95.45	4.55
tray		22.58	4.55	100	
	Total	496.04			

Table 3 shows the flotation metallurgical balance for gold and silver of the Sayapullo tailings, where a high recovery of Ag (72.83%), gold (50.47%) and with a grade of 215.32 g/t and 4.75 g/t respectively.

Table 3. Flotation metallurgical balance of tailings minerals from Sayapullo

Products	weight (kg)	Chemicals laws		Metal content		Recovery		Ratio
		Au, g/t	Ag, g/t	Au, mg	Ag, mg	Au (%)	Ag (%)	
Cab. tested	1	2.55	72.45	2.55	72.45	100	100	
Conc. 1	0.266	4.75	215.32	1.264	57.297	50.47	72.83	3.8
Relave	0.734	1.69	29.13	1.24	21.379	49.53	27.17	
Cab. calculated	1	2.5	78.68	2.504	78.675	100	100	

Table 4 shows what is obtained by means of diagnostic leaching (DLT) carried out on the flotation concentrate, carried out in 6 stages. The average free gold extraction percentage was 44.68%, with this result it is determined that the gold in gold tailings can only be recovered up to that limit, that is, free gold, while the remaining fraction of gold in the sample is encapsulated in silicates, pyrite, copper sulfides, etc. This indicates that a pretreatment is necessary to eliminate the matrix that encapsulates the gold. Calculations were made by gold analysis of the pregnant solution obtained after each sodium cyanide leaching step. To determine the percentage of gold extraction, the metallic content of the initial ore (tailings) was used as a base of 100%. Then the accumulated gold extraction was obtained by successive stage.

Table 4. Percentage of gold extraction by stages and cumulative calculated based on the rich solution after DLT

Treatment stage	Au extraction per step (%)	Cumulative Au Extraction (%)
free gold	44.68	44.68
Gold in sulfates	3.47	48.15
Gold in dolomite, calcite and pyrrhotite	11.64	59.79
Gold in Cu-Zn sulfides, weak pyrite and sphalerite	6.13	65.91
Gold in pyrite, marcasite and arsenopyrite	31.49	97.4
gold in silicates	2.6	100

In Figure 2, the weight losses due to oxidative leaching are presented with the different lixivants used for each stage, where it is observed that 85% of mass loss occurs in the oxidation with nitric acid, and corroborates with the high extraction of gold at that stage.

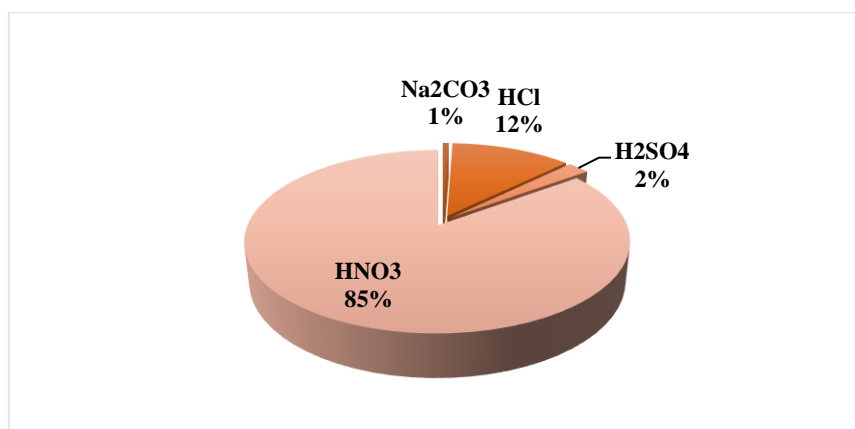


Figure 2. Percent weight loss by stages after DLT

Figure 3 shows the results of the leaching tests carried out after each chemical attack. It is observed that, in the leaching after the attack with HCl and HNO<sub>3</sub>, including free gold, 11.64% and 31.49% extraction of gold are obtained gold respectively, with a total of 97.40% of total gold extracted, the difference remains in the silicate matrix. (Lorenzen & Tumilty, 1992).

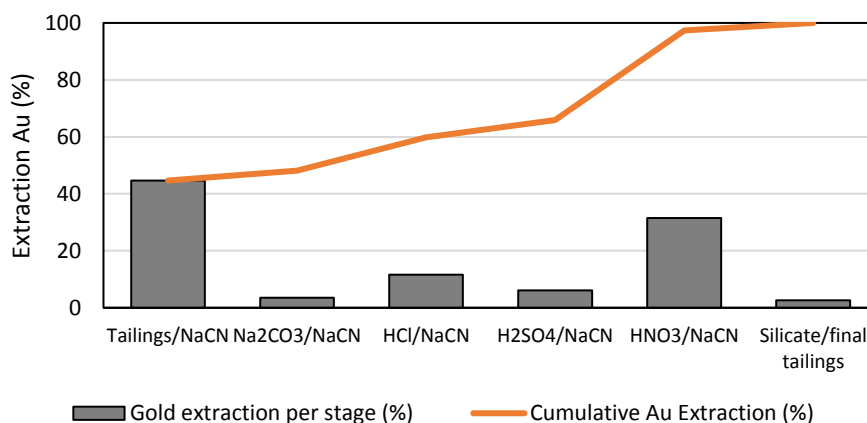


Figure 3. Gold extraction (%), by stages and accumulated based on the pregnant solution of diagnostic leaching (DLT)

#### 4. Conclusions

The tailings from Sayapullo present metallic elements of interest (Au, Ag), to carry out reprocessing for gold and silver that are associated with pyrite minerals. The acidity of the tailings has a pH of 2.6, being very acidic, the relative density is 2.70 g/cm<sup>3</sup>.

The granulometry of the Sayapullo tailings, presents a low percentage through the 200 mesh, (27.40%), for which it is required to do regrinding for leaching, in this case, gold and silver associated minerals were floated 72.83% and 50.47% gold, with a concentration ratio of 3.8.

The concentrate obtained by having a decrease in weight by the ratio of 3.8, and an increase in gold and silver grades, is regrinded to obtain 90% through 400 mesh.

Finally, with the reground concentrate, diagnostic leaching (DLT) is performed, where 44.68% gold extraction was obtained by free gold cyanidation, then the following high gold extraction was obtained in the oxidation stage with HNO<sub>3</sub> (31.49%).

The Sayapullo tailings present a good opportunity to reprocess, given the high prices of gold and silver in the international market, and also to make an adequate tailings deposit to avoid the generation of acidic waters.

It is recommended to do research with the tailings from the new process, since in the flotation carried out a high presence of iron (pyrite) is observed in the concentrate, so the tailings could be used in construction materials.

#### 5. Acknowledgments

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