PRESSURE OXIDATION OF REFRACTORY TAILINGS IN AN AUTOCLAVE FOR GOLD RECOVERY OPTIMIZATION

Máximo Cisneros Tejeira [1], Juan M. Santos Bazo[1], Gonzalo A. Azurza Manrique[1], Omar F. Altamirano Puitiza [1], Raúl Minaya Huaman[1], Fiorella V. Güere Salazar [2*], , William C. Santos Tello [3], Eliana Mullisaca Contreras[4]

[1] Universidad Nacional José Faustino Sánchez Carrión, Huacho, Perú.
[2] Universidad Nacional Mayor de San Marcos, Ciudad Universitaria, Lima 15081, Perú
[3] Universidad Continental, Av. Alfredo Mendiola 5210, Los Olivos 15306, Perú
[4] Universidad Nacional de Juliaca , Puno, Perú.
*Corresponding: fgueres@unmsm.edu.pe

Abstract. The treatment of refractory tailings through pressure oxidation in an autoclave is an efficient alternative for improving gold recovery in mining processes. This study evaluated the effect of oxidation pressure and pyrite particle size on gold recovery from refractory tailings using an autoclave system. Experimental tests were conducted under operating conditions of 4.8 PSI and a particle size of 200 mesh. The results showed that, under these conditions, gold recovery reached 96.8%, whereas at lower pressure values (2 PSI), the recovery decreased to 85%. The pressure oxidation process significantly enhanced sulfide oxidation and facilitated subsequent gold extraction.The experimental data demonstrated that the variable with the greatest influence on the process was oxidation pressure, followed by particle size and residence time. Furthermore, the study confirmed that appropriate control of operating variables is essential to optimize oxidation efficiency, reduce processing costs, and maximize gold recovery from refractory ores. These findings highlight the potential application of autoclave technology as a sustainable and effective method for refractory gold processing in the mining industry.

1. Introduction

Refractory tailings, characterized by the presence of sulfides (pyrite and arsenopyrite), encapsulate gold and silver, making their recovery through direct cyanidation difficult. Pressure oxidation in an autoclave (POX) is a more efficient and environmentally friendly alternative to roasting, as it reduces the emission of SO, and gaseous arsenic.

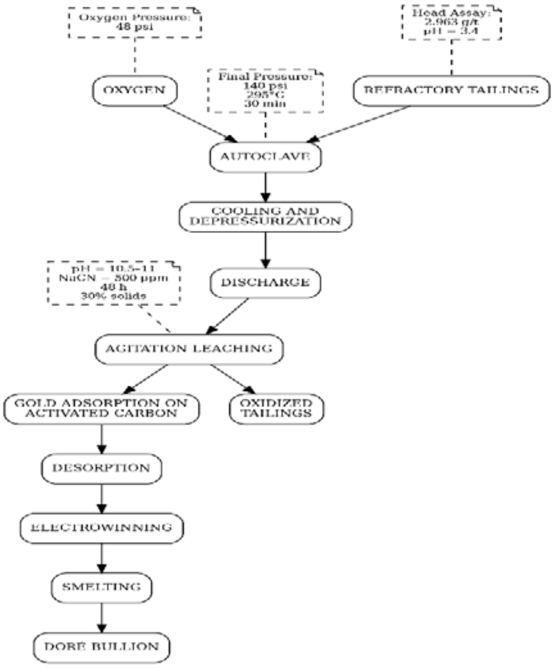


Figure 1. Flow diagram of sulfide oxidation and gold leaching.

2. Materials and Methods

2.1. Materials and Equipment

Materials

- Refractory tailings composed mainly of pyrite and arsenopyrite.
- Stainless-steel vertical autoclave equipped with pressure and temperature control systems.
- Industrial oxygen cylinder fitted with a pressure gauge.
- Mechanical agitator with rotational speed (rpm) control.
- Digital pH meter.
- Burette.

Reagents

- Sodium cyanide (NaCN).
- Potassium iodide (KI).
- Silver nitrate (AgNOƒ).
- Sodium hydroxide (NaOH).

2.2. Experimental Procedure

2.2.1. Sample Preparation for Metallurgical Tests

The refractory gold tailings used in this study were obtained from a pyrite- and arsenopyrite-bearing mining area located in the Conga Community, Cajamarca, Peru.

* Corresponding author: fguere@continental.edu.pe

The material served as the feed sample for all metallurgical tests.

Table 1. Weight of refractory tailings used for metallurgical testing.

No.	Sample	Weight (kg)
1	Tailings	8

2.2.2. Head Grade of the Tailings

Table 2. Head grade of the refractory tailings.

Sample	Element	Grade (g/t)
Tailings	Au	2.963

2.2.3. Autoclave Oxidation of the Refractory Tailings

Pressure oxidation experiments were carried out using industrial oxygen with a purity of 99.5%. The oxidation tests were conducted in a laboratory-scale autoclave reactor equipped with an electrically heated jacket. Oxygen was injected into the reactor through a dedicated gas inlet line connected to the autoclave.

Homogenized samples were used throughout all experiments. Each test commenced upon oxygen injection into the reactor. Prior to each experiment, the oxygen feed pressure was adjusted considering the sum of the water vapor pressure at the operating temperature and the desired oxygen overpressure.

Operating Conditions for Pressure Oxidation Tests

For each oxidation test, 1 kg of refractory tailings with a gold grade of 2.963 g/t, moisture content of 5.8%, and an initial pH of 3.4 was used.

Table 3. Operating conditions for pressure oxidation tests.

Test No.	O, Pressure (psi)	Final Pressure (psi)	Temperature (°C)	Time (min)
01	30	65	285	15
02	48	65	285	30
03	30	140	285	30
04	48	140	285	15
05	30	65	295	30
06	48	65	295	15
07	30	140	295	15
08	48	140	295	30

2.2.4. Cyanide Leaching Tests

Operating Conditions for Leaching Experiments

Agitated cyanide leaching tests were performed using the dried oxidation residues obtained from the autoclave treatment. Sodium cyanide was employed as the leaching reagent at a concentration of 500 ppm. The pH was maintained between 10.5 and 11.0 using sodium hydroxide, and the pulp density was adjusted to 30 wt.% solids. All leaching experiments were conducted under continuous agitation for 48 h.

3. Results and Discussion

3.1. Results

3.1.1. Cyanide Leaching Tests

The results obtained from the cyanide leaching experiments performed on the refractory gold tailings are summarized in Table 4. The pregnant leach solutions obtained after 48 h of leaching were analyzed for gold content.

Table 4. Gold concentration in pregnant leach solutions.

Sample ID	Element	Result	Unit	Time
M001	Au	0.469	mg/L	48 h
M002	Au	0.533	mg/L	48 h
M003	Au	0.813	mg/L	48 h
M004	Au	0.724	mg/L	48 h
M005	Au	0.635	mg/L	48 h
M006	Au	0.698	mg/L	48 h
M007	Au	0.838	mg/L	48 h
M008	Au	0.940	mg/L	48 h

As shown in Table 4, the highest gold concentration in the pregnant solution was obtained for sample M008, reaching 0.940 mg Au/L after 48 h of leaching under a sodium cyanide concentration of 500 ppm.

3.1.2. Metallurgical Balance of the Leaching Process

Table 5. Metallurgical balance of gold recovery during cyanide leaching.

No.	Ore Weight (g)	Head Grade (g Au/t)	Au Content (mg)	Solution Grade (mg Au/L)	Au Recovered (mg)	Tailings Grade (g Au/t)	Au in Tailings (mg)	Recovery (%)
1	942.0	2.69	2.791	0.469	1.032	1.693	1.758	37
2	942.0	2.69	2.791	0.533	1.172	1.559	1.618	42
3	943.0	2.69	2.793	0.813	1.788	0.967	1.006	64
4	940.3	2.69	2.785	0.724	1.588	1.156	1.198	57
5	941.0	2.69	2.787	0.635	1.394	1.344	1.394	50
6	942.0	2.69	2.791	0.698	1.535	1.209	1.256	55
7	943.7	2.69	2.795	0.838	1.845	0.913	0.950	66

No.	Ore Weight (g)	Head Grade (g Au/t)	Au Content (mg)	Solution Grade (mg Au/L)	Au Recovered (mg)	Tailings Grade (g Au/t)	Au in Tailings (mg)	Recovery (%)
8	942.6	2.69	2.792	0.940	2.066	0.698	0.726	74

The metallurgical balance indicates that gold recovery increased substantially following pressure oxidation pretreatment. The highest recovery was achieved in Test 8, reaching 74%, whereas the lowest recovery corresponded to Test 1 with 37%. These results demonstrate the effectiveness of autoclave oxidation in enhancing gold accessibility during subsequent cyanide leaching.

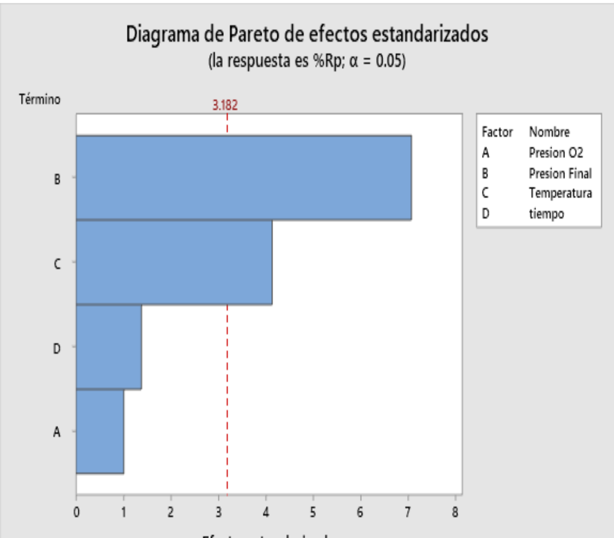
3.1.3. Analysis of Variance (ANOVA) and Pareto Chart

Table 6. Analysis of variance (ANOVA) for gold recovery as a function of oxygen pressure, final pressure, temperature, and retention time.

Source	DF	Adj SS	Adj MS	F-value	p-value
Model	4	1037.50	259.38	17.54	0.020
Linear	4	1037.50	259.38	17.54	0.020
Oxygen Pressure	1	15.13	15.13	1.02	0.386
Final Pressure	1	741.13	741.13	50.10	0.006
Temperature	1	253.13	253.13	17.11	0.026
Time	1	28.12	28.12	1.90	0.262
Error	3	44.38	14.79	—	—
Total	7	1081.88	—	—	—

The ANOVA results presented in Table 6 indicate that final pressure and temperature significantly affected gold recovery, with p-values of 0.006 and 0.026, respectively ($p < 0.05$). In contrast, oxygen pressure ($p = 0.386$) and retention time ($p = 0.262$) did not show statistically significant effects on the response variable.

Figure 2. Pareto chart of the standardized effects of oxygen pressure, final pressure, temperature, and retention time on gold recovery.



According to the Pareto chart, factors B (final pressure) and C (temperature) exceeded the significance threshold of 3.182, indicating a statistically significant influence on gold recovery. In contrast, factors A (oxygen pressure) and D (retention time) remained below the threshold value and therefore were not statistically significant within the experimental range evaluated.

3.2. Discussion

The experimental results demonstrated that pressure oxidation efficiency strongly depended on the operating conditions applied during the autoclave treatment. Under the optimum conditions of 295 °C, a final pressure of 140 psi, and an oxygen pressure of 48 psi, oxidation was completed within approximately 30 min, resulting in the highest subsequent gold recovery. In contrast, experiments performed at temperatures below 285 °C combined with a final pressure of 65 psi and an oxygen pressure of 30 psi exhibited slower oxidation kinetics and only partial sulfide conversion after 15 min of treatment. These findings indicate that elevated temperature and pressure favor sulfide decomposition and improve the liberation of encapsulated gold particles.

The metallurgical balances revealed a significant increase in gold recovery after pressure oxidation pretreatment. Test 8 achieved the highest recovery (74%), whereas Test 1 showed the lowest recovery (37%). This behavior confirms that adequate oxidation conditions improve the accessibility of cyanide to gold-bearing minerals and increase extraction efficiency.

The analysis of variance (ANOVA) demonstrated that final pressure and temperature were the most influential variables affecting gold recovery. Final pressure significantly enhanced oxygen availability within the slurry, accelerating oxidation reactions, while temperature increased reaction kinetics and sulfide conversion efficiency. Conversely, oxygen pressure and retention time exhibited lower statistical significance, suggesting that these parameters may be maintained within fixed operating ranges without considerably affecting process performance.

The Pareto chart supported the ANOVA results by identifying final pressure and temperature as the dominant variables governing gold recovery. Similar observations have been reported in previous studies, where pressure oxidation efficiency was strongly associated with operating pressure and temperature conditions.

These findings are in agreement with those reported by Soto-Uribe et al. [1], who demonstrated that pressure oxidation significantly improves gold extraction from refractory sulfide concentrates. Likewise, Gudyanga et al. [2] reported that oxidative pretreatment under acidic conditions increases mineral liberation and enhances subsequent cyanidation efficiency. Furthermore, Lemos et al. [3] concluded that pressure oxidation represents one of the most effective technologies for processing refractory gold ores because of its rapid kinetics and improved metallurgical performance.

Compared with conventional roasting, pressure oxidation offers important environmental advantages, including lower atmospheric emissions and reduced formation of hazardous compounds. According to Soto-Uribe et al. [1], pressure oxidation minimizes sulfur dioxide generation while maintaining high oxidation efficiencies. Therefore, the application of autoclave technology represents a sustainable alternative for refractory gold processing.

From an industrial perspective, the results obtained in this study demonstrate that pressure oxidation in an autoclave can substantially improve gold recovery while optimizing operating conditions. Consequently, this technology has considerable potential for increasing the economic viability of refractory ore processing and promoting more sustainable mining practices.

4. Conclusions

1. Pressure oxidation (POX) pretreatment in an autoclave proved to be a more efficient and environmentally safer alternative than conventional roasting for the treatment of refractory gold tailings.
2. The optimum operating conditions identified in this study were 295 °C, a final pressure of 140 psi, an oxygen pressure of 48 psi, and a retention time of 30 min.
3. Final pressure and temperature were the most significant variables influencing gold recovery, whereas oxygen pressure and retention time exhibited comparatively lower statistical significance.
4. Precise control of the operating variables is essential to maximize sulfide oxidation efficiency and enhance subsequent gold recovery during cyanide leaching.

Acknowledgements

The authors express their sincere gratitude to the Universidad Nacional José Faustino Sánchez Carrión for the academic support provided for the development of this research. The authors also thank all co-authors for their valuable collaboration, commitment, and scientific contributions throughout the execution of the study.

References

[1] Soto-Uribe, J.C., Valenzuela-García, J.L., Salazar-Campoy, M.M., Parga-Torres, J.R., Tiburcio-Munive, G., Encinas-Romero, M.A., Vázquez-Vázquez, V.M. (2023) Gold Extraction from a Refractory Sulfide Concentrate by Simultaneous Pressure Leaching/Oxidation. Minerals, 13(1), 116. https://doi.org/10.3390/min13010116

[2] Gudyanga, F., et al. (2025) Pretreatment and Extraction of Gold from Refractory Gold Ore in Acidic Conditions. Minerals, 15(4), 340.

[3] Lemos, F.A.N., et al. (2025) Recovery of Gold from Refractory Ore Employing Pressure Oxidation. REM – International Engineering Journal, 78(1).

[4] Marsden, J., House, C. (2006). The Chemistry of Gold Extraction. 2nd Edition. Society for Mining, Metallurgy, and Exploration (SME), Littleton, Colorado, USA.