

Production of an Iron Electrode Precursor from Black Tailings in the Nickel Industry by Acid Leaching and Chemical Precipitation

Obtención de un precursor de electrodos de hierro a partir de colas negras de la industria del níquel mediante lixiviación ácida y precipitación química

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Abstract

The nickel industry in Moa generates significant amounts of solid waste, known as black tailings, with high iron exceeding 40 %. Recovering this element represents a sustainable alternative for its use as a precursor material of negative electrodes in rechargeable nickel-iron batteries. This study evaluates the feasibility of using these residues as a source of iron by using sulphuric acid leaching for which a factorial experiment was designed to optimize the process. Iron extraction efficiencies between 27 % and 62 % were achieved with acid concentration identified as the most influential factor. The iron-rich liquor was subjected to a precipitation stage by pH adjustment with NaOH and oxygen bubbling, generating a brown solid. XRD analysis identified goethite (FeOOH) as the main phase in the precipitate, with an iron content of 47.13 %. The results show the potential of these residual tailings as precursors for electroactive materials.

Keywords: acid leaching, chemical precipitation, black tailings, goethite, iron electrodes

Resumen

La industria del níquel en Moa genera significativas cantidades de residuos sólidos, conocidos como colas negras, con un elevado contenido de hierro (>40 %). La recuperación de este elemento representa una alternativa sostenible para su uso como material precursor de electrodos negativos en

baterías recargables de níquel-hierro. Este estudio evalúa la viabilidad de utilizar estas colas como fuente de hierro. La síntesis se realizó mediante lixiviación con ácido sulfúrico, para lo cual se diseñó un experimento factorial que optimizó el proceso. Se alcanzaron eficiencias de extracción de hierro entre 27 % y 62 %, siendo la concentración del ácido el factor más influyente. El licor rico en hierro se sometió a una etapa de precipitación por ajuste de pH con NaOH y burbujeo de oxígeno, generando un sólido de color marrón. El análisis por difracción de rayos-X identificó goethita (FeOOH) como fase principal en el precipitado, el cual presentó un contenido de hierro de 47,13 %. Los resultados demuestran el potencial de estas colas residuales como precursores de materiales electroactivos.

Palabras clave: lixiviación ácida, precipitación química, colas negras, goethita, electrodos de hierro

1. INTRODUCTION

The nickel and cobalt producing companies in Holguín province annually generate thousands of tons of solid waste, which are deposited in dams or tailings ponds. These wastes contain various chemical elements, with iron being the most abundant, followed by chromium and other metals, while nickel and cobalt are present in smaller proportions (Ramírez-Pérez, 2002).

Several researchers have proposed initiatives aimed at recovering these elements, with the objective of developing technologies that allow their concentration. This would contribute both to sustainable development and to the generation of new sources of income for the national economy (Sánchez *et al.*, 2007; Palacios-Rodríguez and García-Peña, 2014; Navarro-Brefe, 2017). Specifically, iron extraction has been achieved through various methods, among which atmospheric pressure acid leaching stands out, which has yielded favorable results under experimental conditions (García-Peña *et al.*, 2013; Medina *et al.*, 2006).

In addition to the need to valorize these wastes, the current electro-energy conditions in the country have promoted the creation of an energy transition program, with the aim of fostering the use of new technologies, equipment, systems, and management methods, through the practical execution of research, development, and innovations that favor the increase of energy efficiency and energy conservation in all areas. Thus, comprehensive and sustainable energy development aligns as a fundamental response to the growing global energy need (Rendón and López, 2022; UNIDO, 2023).

The drawbacks arising from the intermittency and variability of renewable energies can be effectively solved by electrochemical energy sources, capable

of transforming chemical energy into electrical energy and vice versa. Therefore, the creation of efficient electrical energy storage systems, such as batteries, is fundamental to increase the use of renewable energies and ensure sustainable progress (Linden and Reddy, 2001). These electrical energy systems can be classified into two categories: batteries and electrochemical capacitors. Depending on their behavior during charge and discharge, they present great potential as energy conversion and storage devices (Pérez-González, 2022; Wang *et al.*, 2016; Zu *et al.*, 2017).

Energy storage systems depend largely on the quality of their electrodes, positive and negative. Taking advantage of the amount of iron present in the waste generated by the nickel industry, the work is aimed at obtaining an iron concentrate that can be evaluated as active materials in the negative electrode of nickel-iron batteries. Hence, the objective was to obtain an iron concentrate by acid leaching of black tailings from the carbonate-ammoniacal process and subsequent chemical precipitation by pH adjustment.2.

2. MATERIALS AND METHODS

2.1. Materials

As raw material for the atmospheric pressure leaching tests, tailings from the Caron metallurgical process obtained at the Ni+Co producing plant in Moa were used. The liquors obtained from acid leaching were used for the precipitation of the iron concentrate.

2.2. Reagents

- Sulfuric acid of industrial origin.
- Sodium hydroxide, technical grade (98 % purity) as precipitating agent.

2.3. Sampling and sample preparation

Samples were collected at the outlet of the Ammonia Recovery Plant at the sampling point located at the discharge to the tailings pumping well to the dam. Subsequently, they were dried at 100 °C and homogenized (Figure 1) using the cone and quartering method (Ramírez-Pérez, 2002; Sánchez-Guillén *et al.*, 2014).

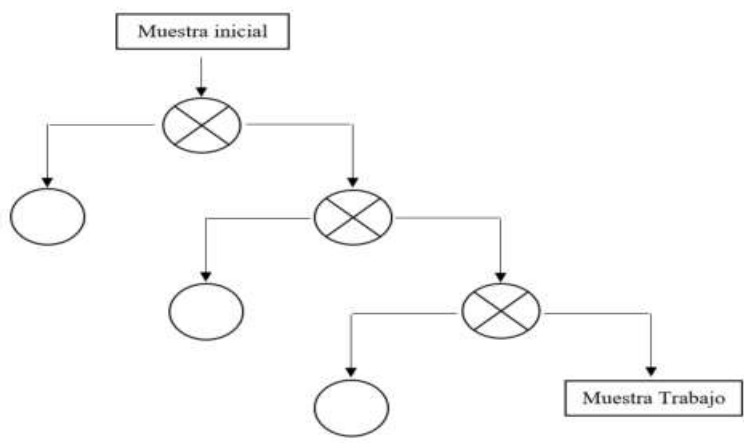


Figure 1. Homogenization and quartering procedure.

2.4. Experiment program

Acid leaching tests with industrial sulfuric acid were carried out to evaluate the influence of different parameters, seeking to obtain a liquor with high iron content. A full factorial experiment with two independent variables (reagent concentration and reaction time) and two replicates was designed, considering the iron extraction percentage as the dependent variable. A full 2² factorial matrix (two factors, two levels) was designed, resulting in four unique experimental conditions (Tables 1 and 2). Each condition was replicated three times, for a total of 12 experiments (Fernández-Bao, 2020).

Table 1. Factorial design matrix

Factors		
Runs	A	B
1	-	-
2	+	-
3	-	+
4	+	+

Table 2. Factors and levels of the factorial design

Factors	Low	High	Levels	Units
Time (A)	40	80	2	minutos
Acid Concentration (B)	5	7	2	mol/L

The leaching efficiency calculation was performed according to formula (1) (Díaz-Bello, 2016; Cisneros-Sánchez *et al.*, 2021):

$$EP_{me} = (m_{Mesi} - m_{mesf}) \cdot \frac{100}{m_{mesf}} \quad (1)$$

Where:

EP_{me} : Leaching efficiency of metallic species, in %

m_{Mesi} : Mass of metallic species in initial solution, in g

m_{mesf} : Mass of metallic species in final solution, in g

Statistical analysis of the results was performed using the program Statgraphics Centurion XV, with which the regression equations were obtained that allowed graphing the behavior of the different variables.

2.5. Experimental setup

The installation used for the execution of the experimental tests of the iron concentration process, by acid leaching, is shown in Figure 2. The experimental system included a 1 L reactor, pH meter, variable speed stirrer, thermostat, and peristaltic pumps with adjustable flow.



Figure 2. Experimental setup for atmospheric pressure leaching with sulfuric acid.

2.6. Methodology used in atmospheric pressure leaching

The criteria for selecting the methodology are based on previous experiences in atmospheric pressure leaching, in which raw materials with characteristics

similar to those proposed were used. Considering that this methodology establishes a series of logical steps to carry out iron leaching (Sánchez *et al.*, 2007; Sánchez and Martínez, 2007), the following was proposed:

- Weigh the black tailing samples.
- Define the factor values according to the factorial design.
- Heat the mixture of sulfuric acid and tailings to the working temperature (80 °C).
- Start the reaction and measure the time once the temperature is reached.
- After the scheduled time, proceed to filter the pulp, wash, and dry the solid.
- Prepare samples of the liquor from leaching, wash water, and the filtered solid for analysis.

2.7. Procedure for precipitation

A 1 L reactor with heating jacket, mechanical, and pneumatic agitation was used. The liquor obtained from leaching was heated to the pre-set temperature for the experiments; the precipitating agent was added until reaching a pH of 4.5 to 5.0. Upon completion, liquid-solid separation was performed, and the precipitate was washed and dried for subsequent chemical and diffractometric analysis. Table 3 shows the operating parameters used.,

Table 3. Precipitation process parameters

Operating Parameters	Conditioning
Temperature	80 °C
pH	4,5 a 5 units
Reaction time	1 hour
Mechanical agitation	Throughout the reaction time

2.8. Analytical techniques used for characterization

Several analytical techniques were used. As part of the chemical characterization of the tailings, qualitative phase analysis was performed. The determination of mineralogical phases was carried out by X-ray diffraction (XRD), using the powder method in a PANalytical X'PERT3 diffractometer with

the following conditions: Gonio scan type in [2 θ] angular range from 4° to 80° with step size of 0.008° with cobalt radiation and nickel filter. The potential difference is 40 kV and current 30 mA. Qualitative phase analysis was performed using the program Crystallographica Search-Match.

The atomic absorption spectrophotometric method is based on the selective absorption of electromagnetic radiation from various regions of the spectrum by the homogeneous system. It was used to determine the chemical composition of the solids and liquors obtained.

The morphologies of the raw materials were determined using a high vacuum scanning electron microscope (SEM) from TESCAN, Model Vega 3, using an electron acceleration voltage of 10 kV.3. RESULTADOS Y DISCUSIÓN

3.1. Chemical composition of black tailings

The result of the chemical analysis of the black tailings (Table 4) shows that iron is the majority element, with maghemite and magnetite phases predominating, which explains the magnetic properties of these tailings. Additionally, other metallic elements were detected in appreciable quantities. These residues can be considered as iron ores, with more than 40 % of this element, representing a potential reserve for various applications. Previous works (Rojas-Purón and Turro-Brefe, 2003; Palacios-Rodríguez and García-Peña, 2014; Fernández *et al.*, 2015) revealed results similar to those of this research.

Table 4. Chemical composition of black tailings (%)

Elements	Ni	Co	Fe	Mg	Mn	Al	Cr	Zn
Content (%)	0.35	0.06	44.59	0.18	0.79	5.39	2.84	0.02

The diffractogram of the black tailings (Figure 3) shows several diffraction peaks. These phases are formed during the roasting-reduction and leaching processes, with magnetite Fe₂O₃ (PDF 7-0322) and maghemite Fe₂O₃ (PDF 19-629) predominating in this case as the main iron-bearing phases (Miranda *et al.*, 2000; Ramírez-Pérez, 2002; Valix and Cheung, 2002; Cabrera *et al.*, 2011; Angulo-Palma *et al.*, 2024).

Physically, it is recognized as a black material, with sub-metallic luster, fine granulometry, and predominantly magnetic (Rojas-Purón and Turro-Brefe, 2003).

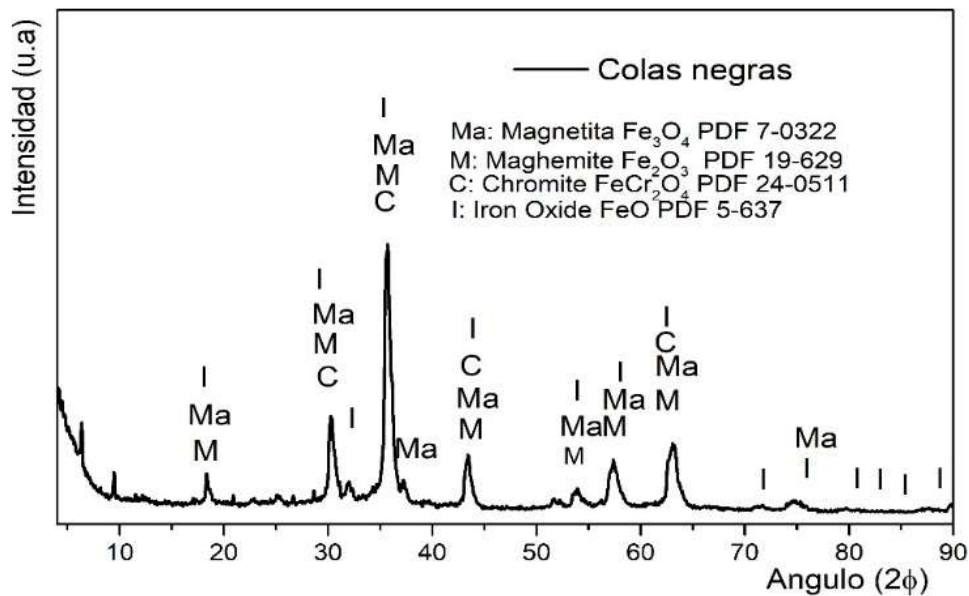


Figure 3. Diffractogram of black tailing samples.

Diffraction peaks that may correspond to secondary phases such as chromite FeCrO_4 (PDF 24-0512), Mg-chromite $((\text{Mg},\text{Fe})\text{Cr}_2\text{O}_3)$ (PDF 24-0512), mixed oxides of magnesium, iron, chromium, and aluminum $(\text{Fe},\text{Mg})(\text{Cr},\text{Al})_2\text{O}_4$ (PDF-9-353), and fayalite $(\text{Fe},\text{Mg})_2(\text{SiO}_4)$ are also observed. These results corroborate those obtained by other authors (Rojas-Purón and Turro-Breffe, 2003; Rojas-Purón, 2003; Medina *et al.*, 2008; Cabrera *et al.*, 2011).

3.2. Scanning Electron Microscopy (SEM)

Considering the SEM images of the black tailings (Figure 4), it can be seen that these residues present particle sizes below $1\ \mu\text{m}$, in addition to the predominance of rounded and elongated particles, which can be observed throughout the region of the images.

Aggregates of particles that tend to group together forming large conglomerates are also observed; this occurs as a result of the reduction process to which the lateritic ore is subjected during the industrial process, as reported by other authors (Medina *et al.*, 2008).

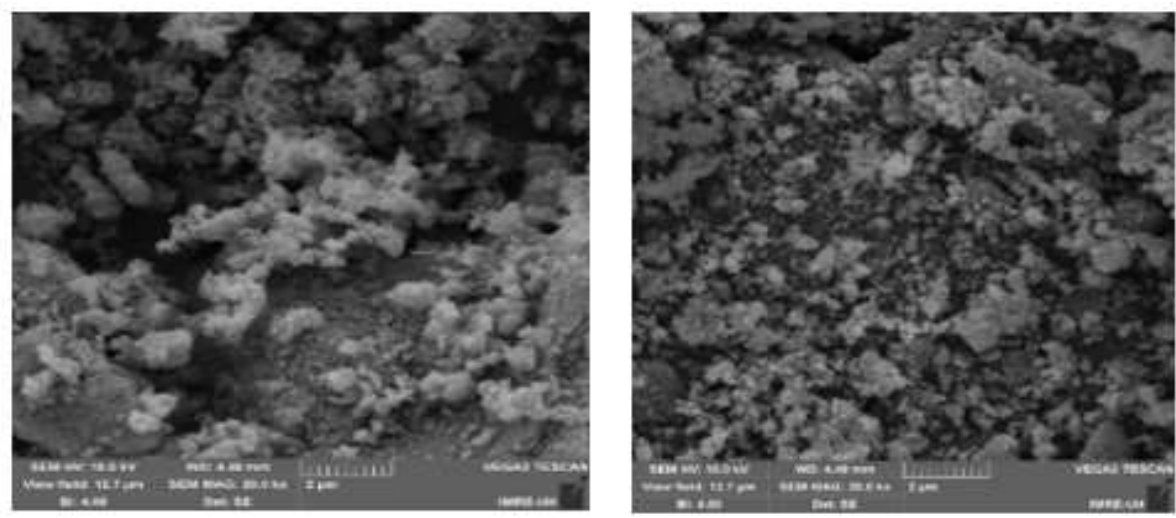


Figure 4. SEM images of black tailings.

3.3. Results of the chemical analysis of leaching liquors

The average concentration values of the main elements at the end of the process, according to the described experiment design, are shown in Table 5 and graphed in Figure 5, to highlight the preponderance of iron in the liquors.

Table 5. Average concentration of metals in the liquors (g/L)

Experimental Conditions	Ni	Co	Fe	Mn	Zn	Mg	Na	Ca
T: 80; C(x):7	0.190	0.033	12.005	0.006	0.437	4.815	0.473	0.014
T: 40; C(x):7	0.200	0.036	16.875	0.007	0.563	4.820	0.514	0.015
T: 40; C(x):5	0.382	0.070	32.255	0.007	0.514	2.943	0.266	0.017
T: 80; C(x):5	0.262	0.049	31.623	0.007	0.415	2.557	0.241	0.017

T: tiempo, min; C: Concentración del ácido sulfúrico, mol/L

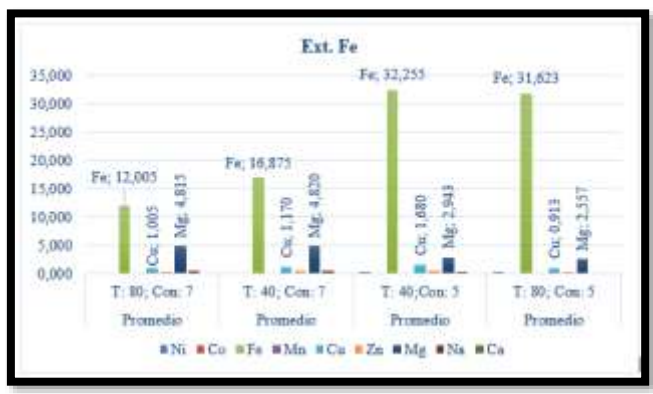


Figure 5. Average concentration values (g/L) of iron and other metals in the liquors.

3.4. Evaluation of the experiment design

Table 6 shows the results of the trials scheduled by the experimental design. while Table 7 shows the average results of iron extraction for each test performed using the experiment design.

Table 6. Iron extraction percentages for each run

Run	(A) Time (min)	(B) Acid Conc. (mol/L)	Iron Extraction (%)
1	40	5	52.20
2	80	5	69.49
3	40	7	86.00
4	40	5	67.93
5	80	7	92.59
6	80	5	71.46
7	40	7	89.91
8	80	7	91.91
9	40	7	87.96
10	40	5	66.67
11	80	5	66.50
12	80	7	93.26

Table 7. Average iron extraction efficiency by acid leaching

Test Conditions	Extraction Efficiency (%Fe)
T: 80; C(x): 7	61.2
T: 40; C(x): 7	45.1
T: 40; C(x): 5	27.7
T: 80; C(x): 5	43.0

Below are the Pareto chart and the main effects plot from the evaluation of the data obtained from the experiment design (Figure 6).

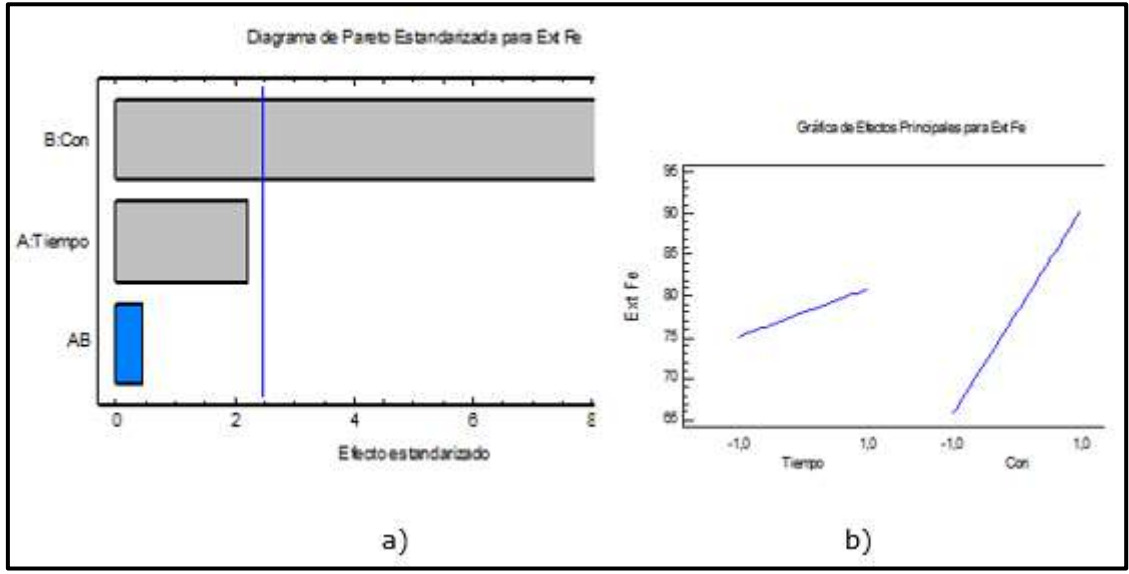


Figure 6. a) Pareto chart; b) Main effects plot.

Equation (2) is the adjusted model:

$$Ext\ Fe = 78.0175 + 12.254 * Con\ reac + 2.855 * tiempo \quad (2)$$

The R^2 statistic indicates that the adjusted model explains 91.34 % of the variability in iron extraction. According to the regression equation and the Pareto diagram from the analysis of the experiment design applied to the acid leaching of black tailings. it is observed that the variable sulfuric acid concentration positively influences the iron extraction percentage during tailing leaching to a greater extent than the time variable.

3.5. Evaluation of solid residues from black tailing leaching

As a result of the experiment design run, acidic liquors with a pH of approximately 1.5 units and a solid residue were obtained, which were characterized from a physicochemical point of view. After leaching. more than 60 % iron extraction is achieved. Figure 7 shows the XRD results of the solid residues after leaching.

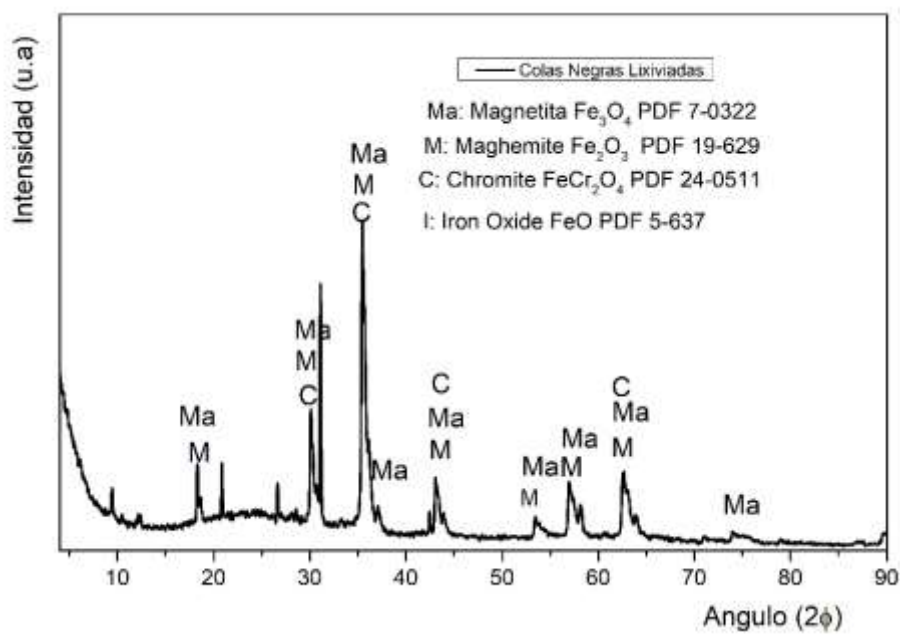


Figure 7. Diffractograms of the solid residue from sulfuric acid leaching of black tailings.

The XRD results indicate that no significant changes occurred in the mineralogical phases. with the diffractometric pattern corresponding mainly to maghemite and magnetite patterns. coinciding with that reported by other authors (Cabrera *et al.*, 2011; Rojas-Purón and Turro-Brefe. 2003; Medina *et al.* 2008).

3.6. Precipitation of iron concentrate from the liquors resulting from leaching

As a product of precipitation, under the established experimental conditions. a brown-colored precipitate with a high percentage of iron in its composition is obtained (Table 8).

Table 8. Average chemical composition of the precipitated iron concentrate (%)

Elements (%)								
Ni	Co	Fe	Mn	Cu	Zn	Mg	Na	Ca
0.599	0.111	47.13	0.519	0.0087	0.012	3.120	0.009	0.019

The diffractogram in Figure 8 indicates the formation of a crystalline phase, with defined diffraction peaks, which when compared with the patterns recorded in the database, shows a similarity with the crystallographic phase of goethite FeOOH (PDF 2-272) (Yang *et al.*, 2021; El-Issmaeli *et al.*, 2023).

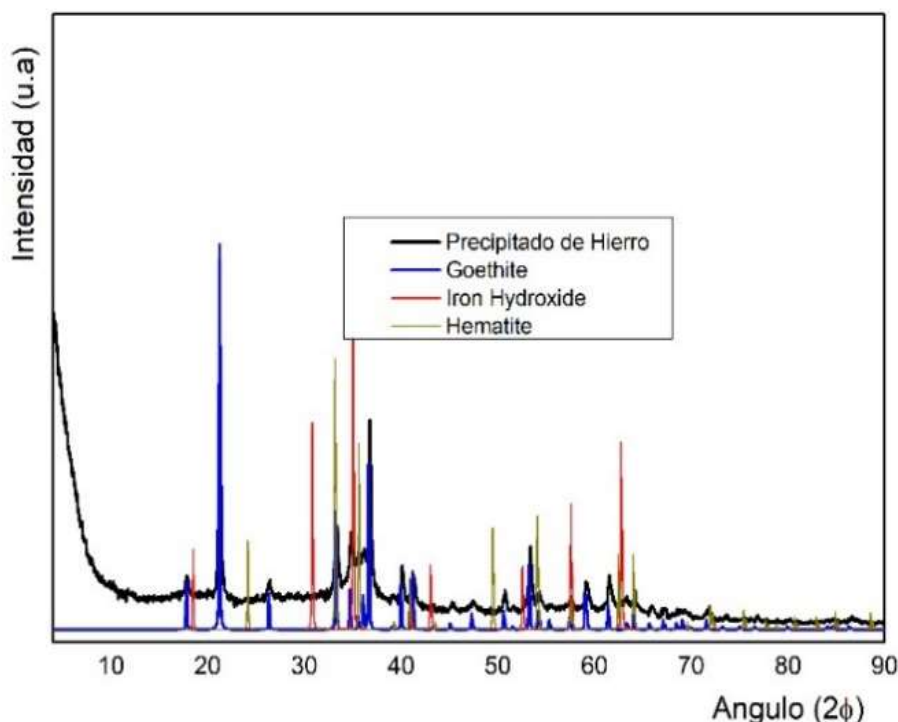


Figure 8. Diffractogram of the solid obtained after precipitation of the product liquor.

4. CONCLUSIONS

- The technical feasibility of extracting iron (up to 62 % efficiency) from black tailings (~44 % Fe) by acid leaching with H_2SO_4 is demonstrated, in which acid concentration is the most significant operating factor.
- As a result of the synthesis of iron hydroxide, from the liquor obtained from the acid leaching of black tailings, a solid is obtained, where the main phase is goethite (FeOOH), with an iron content of 47.13 %; validating its potential as a precursor material for electrochemical applications.

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Additional Information

Conflict of interest

The authors declare that they have no competing financial interests or personal relationships that could have influenced the work presented in this article.

Author contributions

DAGT: experimental part, interpretation of results and approval of the final version. EDA: experiments, interpretation of results, original drafting, bibliographic references and approval

of the final version. ELN: experiments, interpretation of results, original drafting, revision and approval of the final version. YHP: revision, correction and approval of the final version.

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Received: 10/12/2025

Accepted: 11/01/2025