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Nickel recovery from low-grade laterites: study of thermal pre-treatments to improve the efficiency of the hydrometallurgical process

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Abstract

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Keywords

laterite; calcination; nickel; pre-treatment; leaching

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Nickel recovery from low-grade laterites: Study of thermal pre-treatments to improve the efficiency of the hydrometallurgical process

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Abstract

The processing of lower-grade laterites to obtain nickel has increased due to the gradual depletion of higher-grade sulphide ore reserves. However, the extraction from laterites has been limited because conventional technologies imply a considerable expense of energy or reagents. In this document, the effect of thermal pre-treatments on a laterite sample is demonstrated to improve nickel leaching under moderate conditions. The influence of agents such as coke, coal and NaCl in the heat treatment was also studied. With the results it is presumed that part of the nickel occluded in the goethite migrates to the iron oxides surface during the heat treatment; this is why the dissolution of nickel is linked to that of iron. The highest extractions (64.7% nickel) were achieved by combining heat treatment and leaching with 1 M H₂SO₄ at ambient conditions. Compared to direct leaching of unpretreated laterite, leaching rates for this metal are increased by 26.5%. The chlorinating calcination and the optimization of the studied variables will be favourable to reach higher metallic extractions.

Keywords: laterite, calcination, nickel, pre-treatment, leaching

1. Introduction

Historically, the nickel industry has played an important role in the socio-economic development of mankind. Its innumerable properties have allowed it to be used in different engineering fields. Currently, sustainable mineral processing has focused on the field of clean energy technologies, electric vehicles, and batteries to store energy. Nickel is one of several metals used today in these types of applications. Therefore, these issues are of interest to researchers worldwide since the increase in demand for these minerals is expected by the year 2050 (108% increase in the case of nickel) [1].

Even though the increase in the demand for metals and mining activity constitutes an opportunity for developing countries with reserves of these minerals, it also represents a challenge. If environmentally safe mining practices are not in place, their negative impact will affect vulnerable communities and the environment.

Annually, nickel mining produces more than 2.5 Mt of metal worldwide [2]. The decrease in

sulphide mineral reserves has led to obtaining nickel from the large laterite reserves, which correspond to 70% of world reserves [3]. However, the production from this mineral is only 45% of the total nickel [4], due to its complex mineralogy, its low grade, and the use high energy consumption. For this reason, pyrometallurgical extraction from laterites was limited by economic factors [5]. But since it is the essential nickel in industry and modern technology, laterites turn out to be an attractive option to obtain it.

Typically, the pyrometallurgical treatment of laterites produce ferronickel or a low-iron matte. This process involves drying, calcining, reducing, smelting, and refining of the ore. This has generated a high environmental footprint due to the high energy requirement, the production and accumulation of tons of slag, and atmospheric emissions. For this reason, is why research is currently focused on new (or modified) hydrometallurgical technologies to improve the economics of the process and reduce the associated environmental impacts [6].

The traditional hydrometallurgical treatment of laterites has been developed through leaching

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involving high or atmospheric pressures (HPAL//AL) in heaps (HL) or by agitation. Researchers have worked on improving these processes to operate at moderate conditions and to allow favourable nickel production. Many of these works seek the substitution of reagents used (acids and bases) and their concentration [7]. The temperature, pressure, and time used in the process have also tried to be optimized [8–11].

Despite the favourable results of obtaining hydrometallurgical nickel, these methods have disadvantages such as:

- Lack of selectivity.
- Little moderate operating conditions.
- Some methods require optimizing aspects such as working times and amounts of solid processed per unit volume.
- Some of the agents used are considered toxic and corrosive, therefore untreated leaching solutions can become an environmental problem.

According to the World Bank [12], in 2050 the treatment (from extraction to final consumption) of strategic metal ores should represent only 6% of greenhouse gas emissions generated by technologies based on fossil fuels. Therefore, the search to find a balance between the reduction of environmental impacts and the profitability of the production process of new processing methodologies is still ongoing. The benefits in terms of technique and nickel recovery percentages must be related to the carbon footprint reduction.

Moderate thermal treatments of minerals have been developed to increase the release of metals based on their structure or mineral matrix modification [13]. Thus, processes with high energy and reagent requirements are avoided.

Studies with laterite have revealed that the calcination of the mineral at 600 and 700°C improved the mineralogical and physicochemical properties of the mineral due to the increase in its porosity and its surface area [14]. This thermal activation leads to an increase in nickel extractions for a final 97.52% or 92% with H₂SO₄ [15] or HCl [16]. These routes can be good alternatives, but the leaches are worked at high temperatures and pressures (80°C, 1.8 MPa, and 3 M leach concentration) and in some cases, it has required more than a single leaching reagent [16].

In this investigation, calcinations of a laterite sample were used as leaching pre-treatments to improve nickel recovery. To verify the effect on the morphological modification of the mineral, coal, coke, and NaCl were used in the thermal treatment.

Their function is to generate changes in the mineralogical phases of the sample. The addition of carbon to the process is expected to promote the formation of carbon monoxide and the reduction of nickel and iron oxides. With NaCl it is intended to recover the nickel as well as all the chlorine in the form of soluble compounds during leaching; so, the possible contamination of the air that is produced with the current chlorination processes that use gaseous chlorine is eliminated. Unlike the heat treatments reported by other authors, here the objective is to demonstrate that the use of these additives is a successful strategy for the treatment of low-grade laterites with favourable results in terms of extraction and low environmental impact.

2. Materials and methods

2.1. Mineral

The laterite used in this study is of Colombian origin with a deposit whose location was not provided.

The sample particle size was reduced and classified between ranges of 63, 75 and 150 µm using a ball mill and pulverizer. The particle size distribution was carried out with sieves of the ASTM-E-11 series.

The mineral was characterized thermally, chemically, and mineralogically. Thermal characterization was carried out by Thermogravimetric Analysis (TGA) in the TA Instruments SDT-Q600 equipment. Chemical characterization was performed by chemical digestion with aqua regia (HCl:HNO₃, 3:1). The metallic contents were analyzed by atomic emission spectroscopy with microwave plasma (MP-AES) on the spectrometer Agilent 4210. The mineralogical characterization was carried out by X-ray diffraction in the Panalytical X'pert Pro equipment, which uses Bragg-Brentano geometry with a cobalt tube; samples were analyzed with the ICDD® database (International Center for Diffraction Data).

Calcined and leached solids were analyzed by SEM in a ZEISS EVO MA10 Series equipment Electron Microscope, also analyzed by EDS with an Oxford Instruments microprobe.

2.2. Methods

2.2.1. Mineral heat treatments

The calcinations carried out on the mineral were of the oxidizing, reducing, and chlorinating types. For this, agents such as coal, coke, and NaCl were added to the mineral in an initial proportion of 6%

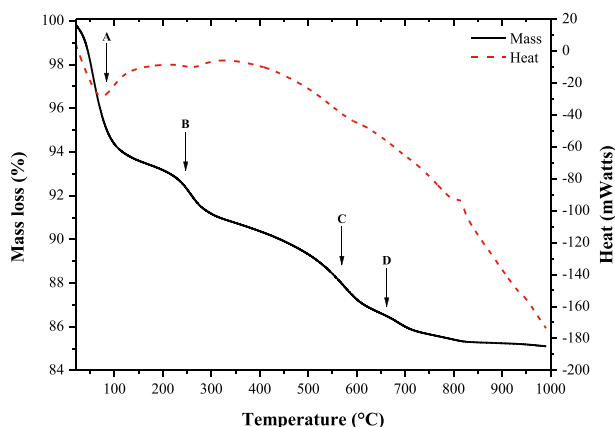


Fig. 1. TGA/DSC of the studied laterite (A: dehydration (~100°C); B: goethite decomposition (~250°C); C: beginning of conversion to Fe₂O₃ (~550°C); D: complete conversion to Fe₂O₃ (~650°C)).

weight by weight (w/w). It was later modified to verify its effect in leaching tests.

The samples were placed in ceramic crucibles and taken to the muffle for heating at 600°C, regulating the ramp (1 h) and heat impregnation of the process (2 h). Test temperature was selected based on the thermogravimetric analysis previously performed on the laterite sample. Higher calcination temperatures impair subsequent leaching [16]. Figure 1 indicates the temperature where the greatest loss of mass occurs.

After the treatment, the samples were allowed to cool in the muffle to avoid possible oxidation of the reductive calcination.

2.2.2. Leaching tests

Three series of laboratory scale agitation leaching tests were carried out to analyze the effect of i) granulometry, ii) additives in the calcinations, and iii) % w/w of the most favorable additive. 1 M H₂SO₄ analytical grade (J.T. Baker®) was used as leaching

agent. Compact digital stirrers (Model 50006-03 – COLE-PARMER) with blade-type propellers were used; the agitation was set at 500 rpm and worked with a S/L ratio of 1:20 for 24 h. The experiments were carried out at ambient conditions (16°C and 1022 bar).

The study’s experimental procedure is presented in Fig. 2.

3. Results and discussion

3.1. Sample characterization

The results of the chemical analysis show that the laterite under study is of low grade due to its nickel content (Table 1) while the iron is present in a greater quantity due to the nature of the mineral. Nickel in laterites is generally associated with various mineral compounds, especially iron oxides [17–19]. Therefore, data on iron dissolution behaviors are important to understand the mechanism of nickel leaching. Lead, chromium, copper, and cobalt are little present, and their dissolution is negligible (data not included). Therefore, the leaching behavior of these metals will not be analyzed.

Figure 3 shows the X-ray diffraction pattern of the sample. It is evident that the mineral is composed mainly of iron in the form of goethite FeO(OH) and hematite Fe₂O₃ by magnesium silicates in the form of nepouite Mg₃Si₂O₅(OH)₄ and silica as quartz SiO₂.

Nickel phases are not evident in the spectrum due to its low proportion in the mineral composition, its wide diffusion and complex association both with iron hydroxides and with silicate structures [19].

Table 1. Metallic content of the laterite sample.

Element	Ni	Fe	Mg	Co	Cr	Cu	Pb
Tenor (%)	1.80	12.49	11.04	0.03	0.02	0.03	0.14

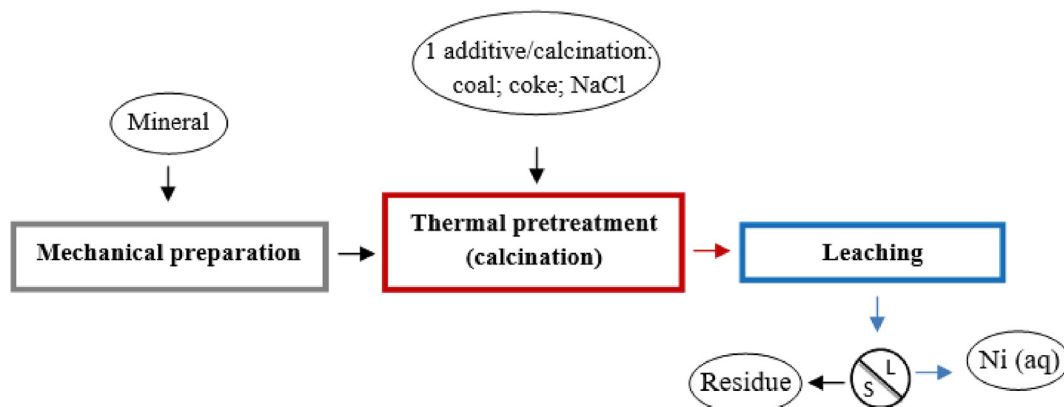


Fig. 2. General study procedure.

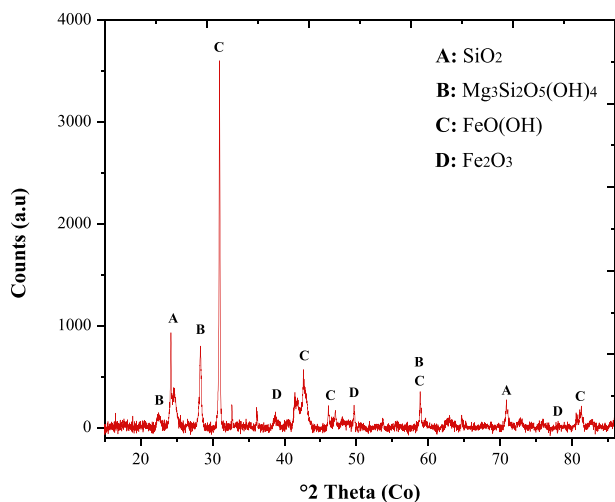


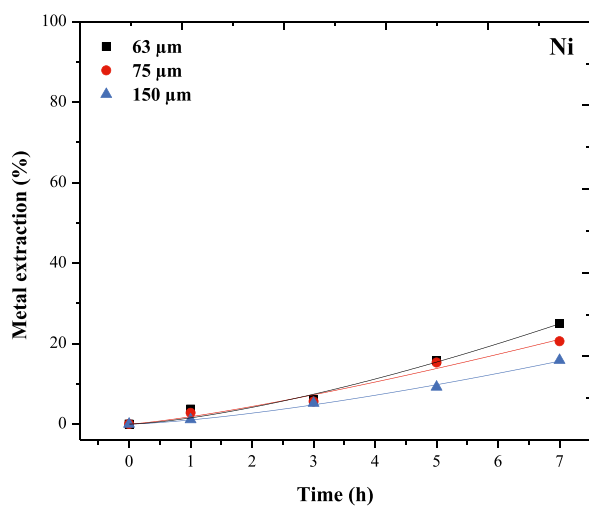
Fig. 3. XRD pattern of the Colombian laterite sample.

Chemically, in the Mg₃Si₂O₅(OH)₄ species, a gradual substitution of the magnesium cation by a nickel cation can occur, allowing the formation of the different minerals of the phyllosilicate series [20]. Being encapsulated in this refractory species, nickel dissolution becomes a challenging task.

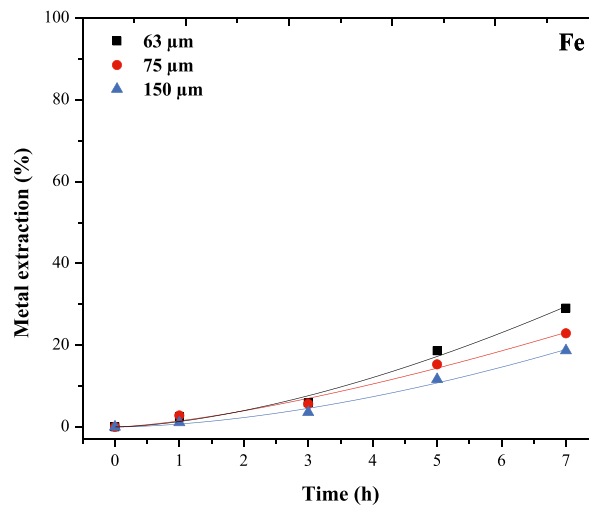
3.2. Particle size effect

The three particle sizes obtained in the granulometric classification were tested. The maximum nickel extraction was 45.9%, obtained from the 63 μm size, followed by 42.2% obtained with 75 μm sizes (Fig. 4).

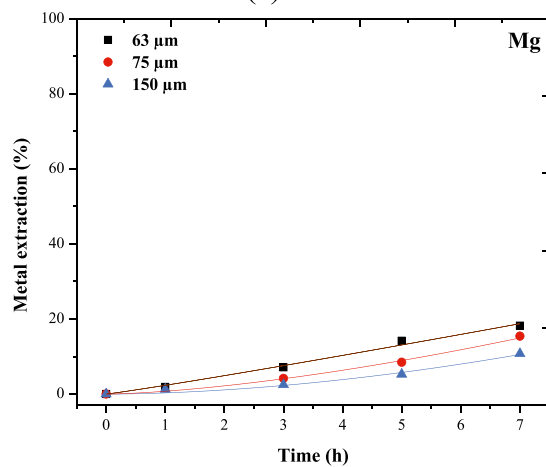
Nickel is easily leached from finer than coarse particle sizes. The contact surface between the reagent and the mineral increases when to work with



(a)



(b)



(c)

Fig. 4. Effect of particle size on the leaching of nickel (a), iron (b), and magnesium (c). Conditions: H₂SO₄; 1 M; 500 rpm; 50 g/L; 7 h.

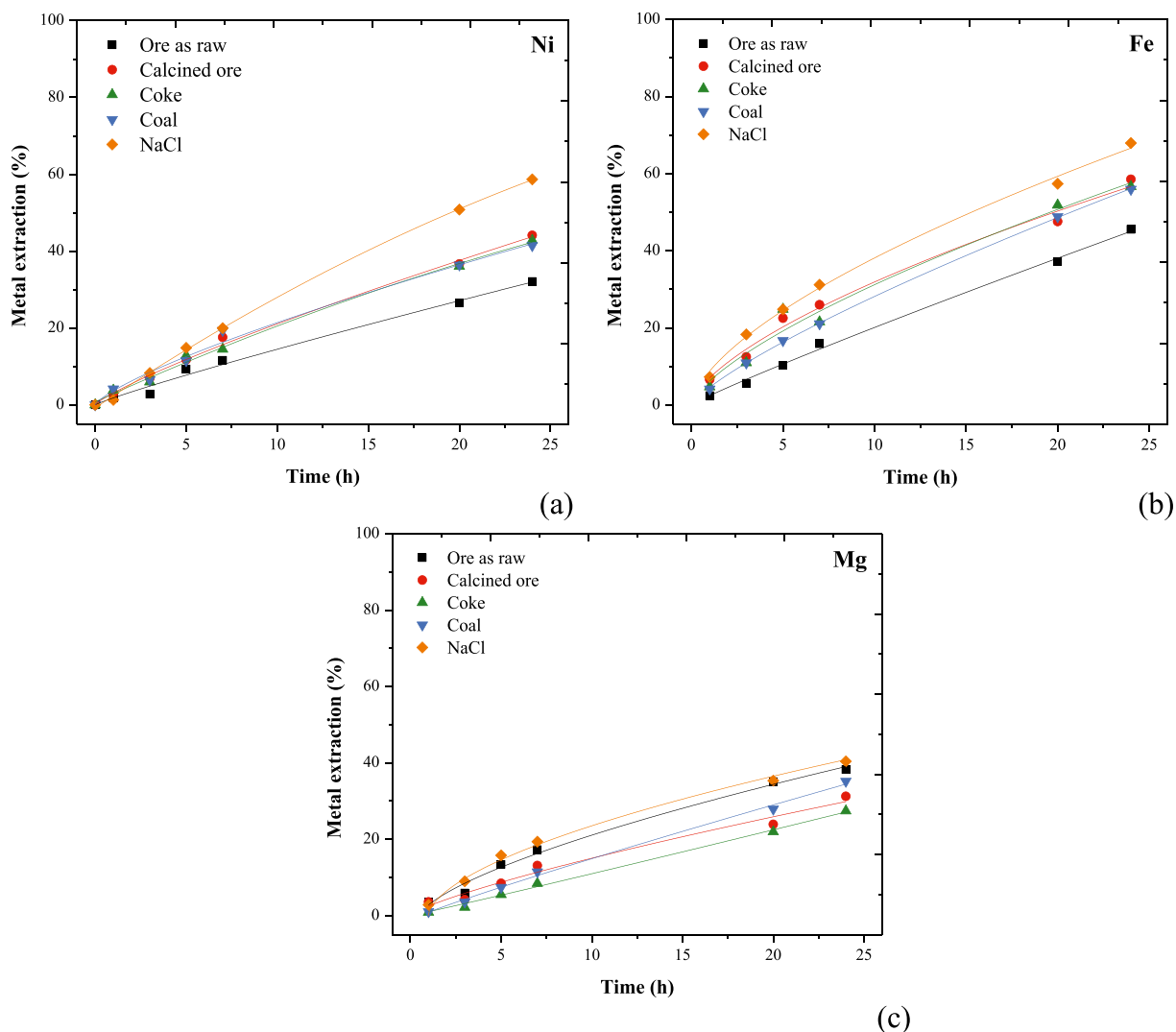


Fig. 5. Ni (a), Fe (b) and Mg (c) leaching for samples treated and not heat treated. Conditions: calcination at 600°C/2 h of raw ore and with coke, coal and NaCl 6% w/w. Leaching: H₂SO₄; 1 M; 500 rpm; 50 g/L; 24 h.

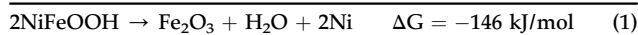
a smaller particle size. The mineral granulometry was set at 75 μm for the analysis of the other variables of the study. The difference between the sizes of 63 and 75 μm is insignificant (which requires less mineral preparation if it is used in the future on an industrial scale).

3.3. Effect of mineral calcination on nickel dissolution

The acid leaching test results showed that calcination at 600°C produced positive impacts on the Ni and Fe recovery (Fig. 5). The thermal treatment could generate an increase in the mineral porosity, which would increase the specific surface area and consequently its reactivity. The H₂SO₄ attack could be possible along the areas of weakness (pores,

cracks or dislocations) which resulting from the mineral matrix modification of the laterite with calcination.

The increase in nickel extraction may be due to the fact that part of the metal occluded in the goethite passes towards the surface of the iron oxides once the thermal conversion of the hydroxide occurs during heating (above 250°C, Fig. 1).



This situation explains the similarity in the leaching speed of both elements, which can be seen in Fig. 5 and which is independent from that of magnesium.

According to what was reported by Li et al. [21], in some laterite samples, nickel can be uniformly

distributed in the oxide crystals iron, as occurs with this mineral. However, part of the liberated nickel may be incorporated into the iron oxide structure, which inhibits a total extraction of the metal. Added to this is some amount of nickel that may be contained in the silicate structure.

The addition of agents such as coke, coal, and NaCl in the calcinations was also analyzed. The resulting metals are more susceptible to leaching in the media of the chlorinating calcination. Unlike the extraction increases of 26 and 22% for nickel and iron respectively in chlorinated media (relative to raw ore), coke and coal calcinations do not improve metal solubilization. Its effect on calcination is almost nil since the extractions are similar to those obtained by calcination without additives. It may be that after the ore was pretreated, remnants of both coal and coke may have remained in the calcined ore samples, which generated chemical interferences in the leaching. Carbonaceous materials tend to reabsorb metals into the pulp after dissolving them in solution. This phenomenon is common in cyanide gold leaching [22] and is known as “preg-robbing”.

The micrographs in Fig. 6 show the presence of darker-colored particles (Fig. 6A) after leaching. These particles have an amorphous structure with a macro-porous surface (Fig. 6B, C) and according to the chemical analysis of particles by EDS (Fig. 6D) it was found that the particles have a carbon content of 92 wt%. All of the above indicates the presence of carbonaceous material remaining from the calcination, which represents the sequestration of nickel ions during leaching.

This situation could have inhibited the oxidation and reduction of the mineral during the thermal treatments with coke and coal, respectively. To avoid resorption action chlorine or kerosene could be used to deactivate carbonaceous materials under strongly oxidizing conditions or use pressure oxidation [23], however, this is outside the scope of this study.

Figure 5 also shows that a small percentage of nickel is present in a simple and easily soluble form, capable of being dissolved without additional agents or pretreatments. The extraction curves show that the leaching kinetics of the laterite is slow, therefore a greater metallic extraction would require

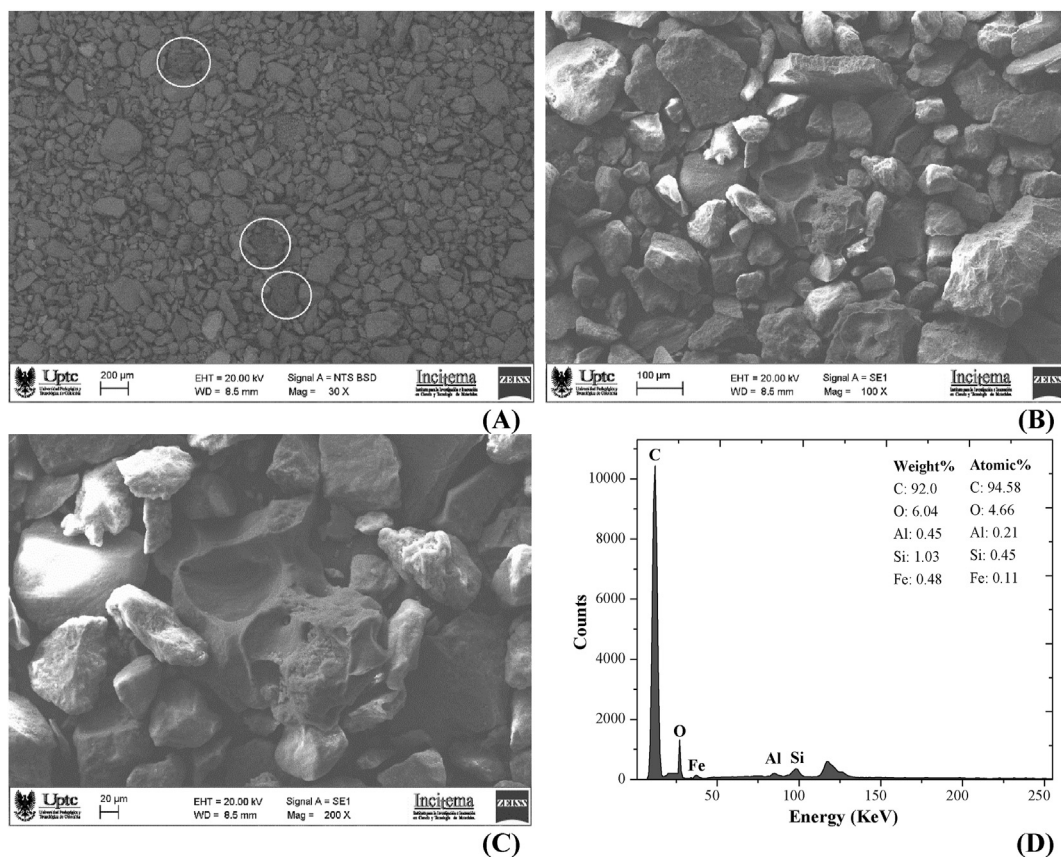


Fig. 6. SEM micrographs (A–C) of leached laterite with presence of carbonaceous material and EDS spectrum of reducing agent particle (D).

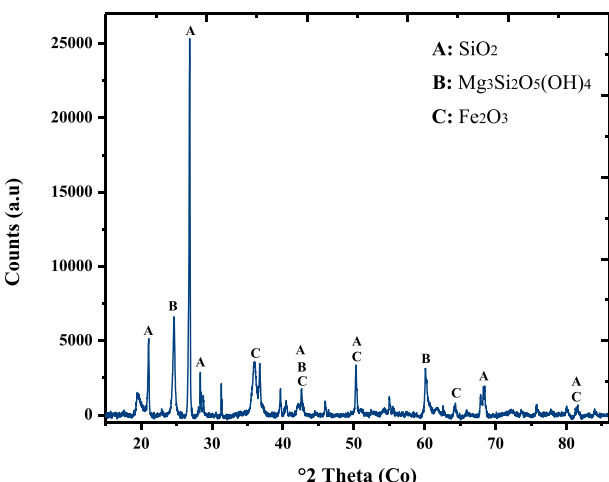


Fig. 7. XRD pattern of the calcined and leached laterite sample.

increasing the working time and optimizing the conditions and activations of the mineral and the leaching.

Regarding magnesium, heat treatments did not affect its leaching characteristics. Figure 7 shows no significant change in magnesium silicate. As indicated above, the element is encapsulated within the silicate framework (Fig. 3), which normally has low solubility. Calcinations at 600°C are not sufficient for the decomposition of the mineralogical specie.

3.4. Effect of the NaCl:laterite ratio on calcination

The influence of the NaCl: mineral ratio on the leaching of Ni, Fe, and Mg is investigated as shown in Fig. 8.

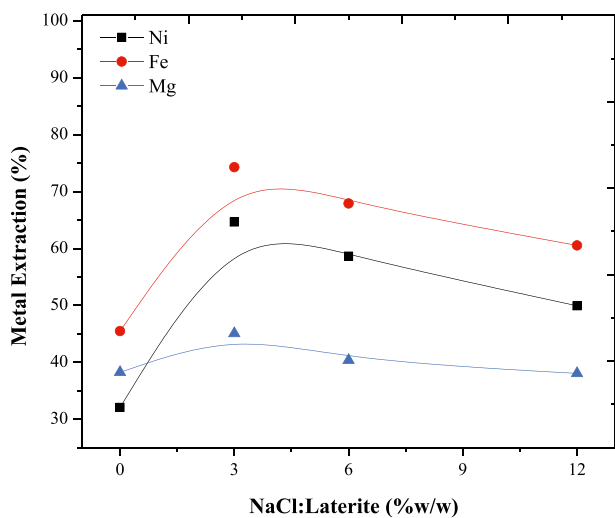


Fig. 8. Effect of the NaCl-laterite ratio in calcination, on the Ni, Fe and Mg leaching.

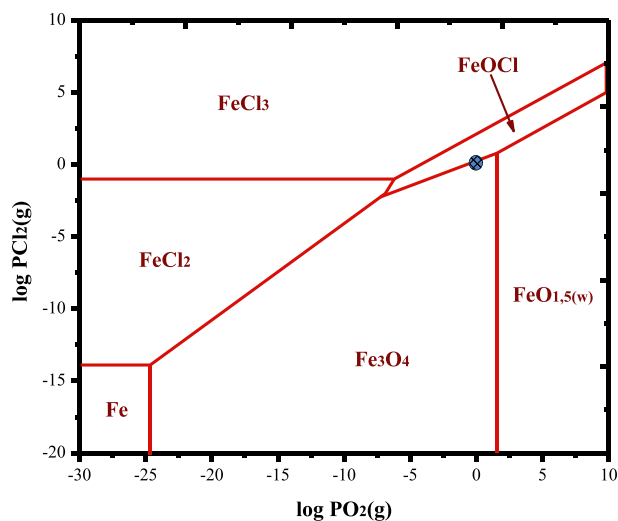


Fig. 9. Phase stability diagram for Fe. Conditions: Pressure of O₂ = 0.21 atm and Cl₂ = 0.79 atm; Temperature: 600°C. Designed with HSC 5.1 software and adapted by authors.

The metallic leaching rates are inversely proportional to the increase in the %w/w used. The addition of the chlorinating agent in the lowest proportion (3%) does have a significant effect with respect to the leaching of the calcined mineral without the addition of NaCl.

This influence can be attributed to the stability of the phase formed during calcination. The mineral matrix undergoes morphological changes during heat treatment, generating imperfections in the iron oxide structures. In these zones, Cl⁻ ions can easily be included in the structure giving way to the formation of FeOCl (Fig. 9).

This compound is much more flexible than that iron oxides due to its layered structure and the weak interaction between them [24]; this allows the release of iron and nickel ions. But, a large presence of these Cl⁻ ions leads to the stabilization of the FeOCl structure, increasing the probability that nickel is included again in the iron oxide during recrystallization. This same situation would occur if the heat treatment time were increased [16,25].

4. Conclusions

The thermal treatments could have modified the morphology of the mineral, increasing its reaction surface area. For this reason, a rapid interaction between H₂SO₄ and nickel species occurred, increasing their dissolution.

It was evidenced that of the three additives added to the calcinations, NaCl is the one that allows increasing metal extractions. The instability of the mineral matrix during treatment leads to the FeOCl

formation which allows the release of iron and nickel ions.

Regarding coal and coke, possible traces of them in the calcined ore would have generated an effect similar to preg-robbing; chemical interferences in the leaching generated by the carbonaceous materials would have conditioned the increase in the extraction of nickel.

Calcination of low-grade laterite in a chloride media is a promising strategy in nickel extraction terms and low environmental impact (moderate operating conditions).

Ethical statement

The authors state that the research was conducted according to ethical standards.

Funding body

This research received no external funding.

Conflict of Interest

The authors declare no conflict of interest.

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