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The effect of calcination as pre treatment to enhance the nickel extraction from low-grade laterites

A. Garces-Granda[a,*], G.T. Lapidus[b], O.J. Restrepo-Baena[a]

[a] Universidad Nacional de Colombia, Colombia
[b] Universidad Autónoma Metropolitana, Mexico

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ABSTRACT

The effect of a thermal pre treatment on the leaching behavior of a low-grade nickeliferous laterite ore, obtained from the north of Colombia, was investigated. The lateritic ore was first calcined at different temperatures, and then leached for 48 h in 3.2 M hydrochloric acid. An increase in nickel extraction from 15.8% to 57.1%, 98.0%, 91.9% and 26.1% was achieved in the leach, when the ore was previously calcined at 250 °C, 430 °C, 550 °C and 800 °C for 2 h, respectively. The increased nickel extraction from the pre-treated ore was believed to be partly due to increased surface area created through water removal from the goethite structure during the dehydroxylation process. Nickel showed congruent dissolution with Fe, indicating that Ni was uniformly incorporated into the iron oxides structure. Consequently, the use of calcination as a pre treatment can enhance the nickel extraction and significantly decrease the time required for the atmospheric acid leaching step.

1. Introduction

Nickel naturally occurs in sulfides or laterites. According to the U.S. Geological Survey, (2017), about 60% of the world nickel resources are laterites, while the remaining 40% are sulfides. By 2008, total Ni production from laterite ores had risen to 46% of global supply and it exceeded 50% in 2010 (Butt and Cluzel, 2013). Laterites are the result of soil degradation generated by climatic changes, water filtration and other factors (Wilman et al., 2009), increasing the nickel content by increasing the depth of the deposit (Girgin et al., 2011). These can be classified into limonites or saprolites, depending on the iron and magnesium content (Dalvi et al., 2004; Oxley et al., 2016; Thubakgale et al., 2013).

Limonites refer to laterites with high iron contents (at least 40% by weight) and low magnesium contents (0.5–5%). In these, divalent iron has been oxidized and precipitated as microcrystalline ferric (oxy)hydroxides (predominantly goethite [α-FeOOH] with lesser amounts of hematite [α-Fe_2O_3]); together they comprise the dominant Ni host mineralogy, showing contents of up to 1.5% (Apostolidis, 1974; Landers et al., 2009).

Different publications (Landers and Gilkes, 2007; Landers et al., 2009; Li et al., 2013; Li et al., 2009; O'Connor et al., 2006) have described a significant enhancement of nickel extraction by atmospheric leaching, after simply heating nickel laterite at different temperatures, as a means of converting the hydrated iron oxides to hematite, thus avoiding the requirement for expensive autoclave technology.

Due to the removal of free and combined moisture and to the partial collapse of the phase structure, a calcination step could alter the mineralogical composition of the ore and increase the surface and porosity of the raw ore, thus making it more amenable to leaching (Landers and Gilkes, 2007; Li et al., 2009; Olanipekun, 2000). Landers and Gilkes (2007) also postulated that during progressive dehydroxylation of goethite some of the associated metals may not be compatible with the various hematite-like structures and may be ejected from the lattice into the abundant voids or onto the crystal surface. In these accessible locations, they will be much more easily dissolved by acid solutions.

In this study, a calcination step at different temperatures has been employed to evaluate its benefit on the leachability of nickel from a low-grade Colombian laterite. This work describes the effects of prior heating of the limonitic ore on the mineral structure and the dissolution rate of Ni in 3.2 M HCl solution.

2. Experimental

2.1. Materials

A low-grade nickel laterite ore from the north of Colombia was used for this study. Chemical and mineralogical compositions of laterite were analyzed by atomic absorption (AA), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The chemical analysis result of

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* Corresponding author.
E-mail address: adpgarces@gmail.com (A. Garces-Granda).

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Table 1
Chemical analysis results by AA.

%Ni	%Fe	%MgO	%SiO_2	%Al_2O_3	%Co	SiO_2/MgO
1.15	41.1	1.51	14.29	6.9	0.122	9.45

the sample is presented in Table 1, where it can be noted that the nickel laterite contains 1.15% Ni, 0.122% Co and 41.1% Fe as the major elements of interest. The large SiO_2/MgO ratio and high iron content indicate that the ore consists mainly of limonite-type laterites.

The mineral sample contains iron in the form of goethite (FeO(OH)), wustite (FeO) and chromite ($FeCr_2O_4$), magnesium silicates in form of nepouite $(Ni, Mg)_3Si_2O_5(OH)_4$ and silica contents as quartz and cristobalite, according to the XRD analysis. However, it was possible to determine that nickel is associated, in most cases, with iron oxides and there is no evidence of the existence of a single nickel phase.

2.2. Methods

2.2.1. Calcination

For the thermal pre treatment, approximately 50 g of particles, passing 37 µm, was placed on a ceramic plate. The plate was placed in a furnace, pre-heated to the selected temperature (250 °C, 430 °C, 550 °C and 800 °C), for 2 h. The furnace was opened at specified times to perform mixing of the charge. After roasting, the samples were removed from the furnace and allowed to cool to room temperature.

Thermogravimetric and differential thermal analysis were conducted on the original sample in air using a PIRYS Perkin Elmer equipment. The temperature was scanned between 30 and 800 °C with a heating ramp of 10 °C/min. In addition a Brunauer-Emmett-Teller (BET) analysis was made in order to determine the specific surface area for all samples calcined and uncalcined.

2.2.2. Leaching

For leaching tests, 1 g of calcined or uncalcined sample was transferred into a leaching reactor containing 100 mL of 3.2 M HCl at room temperature (25 °C) and atmospheric pressure (Patm = 79 kPa). Magnetic stirring was sustained at a constant rate at 300 rpm for a period of 48 h in all experiments. The reaction temperature was maintained constant during the dissolution process by placing the reaction vessel in a water bath. Successive 1 mL aliquots of the suspension were withdrawn at set times (1, 3, 5, 7, 9, 12, 24 and 48 h) using a 3 mL plastic syringe, and filtered through a 0.20 µm Millipore filter. Samples of the leached solution were analyzed for Ni and Fe by atomic absorption spectrometry (Varian, SpectrAA 220 fs).

3. Results and discussions

3.1. Mineralogical analysis

From the results of thermogravimetry (TG) and differential thermogravimetry (DTA) analysis shown in Fig. 1, only one intense peak was observed at approximately 230 °C, which is the result of a change in structure, and corresponds to a weight loss of ~5%.

The weight-loss measurements from TGA over the temperature range of 110 to 400 °C were approximately 12% and mostly represented the loss of structural water (i.e. bound OH^-) from goethite. On further heating (400 to 800 °C), an additional weight loss of 2.3% associated with the complete dehydroxylation of goethite was observed. This additional weight loss may be due to the loss of excess structural OH^- associated with minor impurities such as magnesium silicates.

In order to better understand the results of the TG/DTA, an XRD analysis was also carried out. The aim was to observe the structure evolution of the ore as a function of the calcination temperature. The effect of calcination on mineral transformation of laterite ores at different temperatures is shown in Fig. 2.

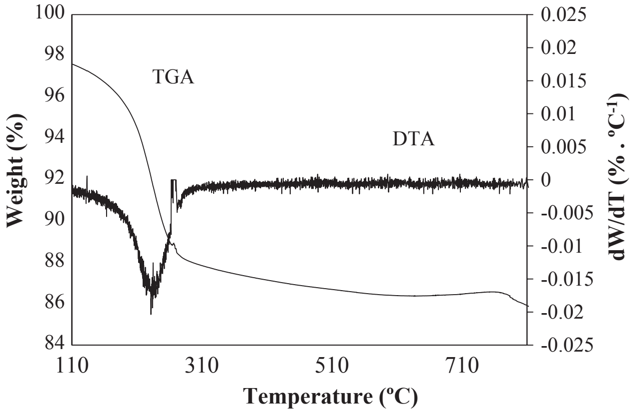

Fig. 1. Thermogram for the raw ore.

X-ray diffraction indicates that, on heating at 250 °C for 2 h, goethite started to alter to hematite. This is consistent with thermal analysis results (Fig. 1), where the dehydroxylation of goethite is described by a single endotherm at ~230 °C, indicating a single reaction.

Fig. 2 shows that the transformation of goethite into hematite occurs with increasing calcination temperature, the peaks of goethite totally disappear when the sample is calcined at 430 °C. According to (Kim et al., 2010), this transformation is almost completed at 800 °C.

3.2. Effect of calcination temperature on nickel dissolution

The effect of thermal pre treatment on nickel and iron extraction by hydrochloric acid is shown in Fig. 3.

When the sample was previously calcined at 250 °C, 430 °C, 550 °C or 800 °C for 2 h, nickel extraction was enhanced significantly from the untreated sample at 15.8% to 57.1%, 98.0%, 91.9% and 26.1%, respectively after 48 h of atmospheric leaching. This increase can be attributed to the increase of the surface area, reflecting the development of micropores in the particles. A greater porosity and the change in the reactivity of the mineral, due to high density of structural defects in the newly transformed and poorly ordered hematite, make the resulting solid more amenable to leaching, even from the initial moments (Landers and Gilkes, 2007; Li et al., 2009; Olanipekun, 2000).

To verify this assumption, calcined and uncalcined samples of the mineral were subjected to a Brunauer-Emmett-Teller (BET) analysis (Table 2). The specific surface area practically doubled when the sample was calcined at 430 °C for 2 h, increasing from 84 m^2/g to 143 m^2/g, but when the sample was calcined at 800 °C, the specific surface area decreased to 77 m^2/g.

Comparing the leaching results of uncalcined and calcined ore at different temperatures, calcinations at temperatures up to 430 °C appears to provide the optimum nickel extraction; on the other hand, higher temperatures are detrimental to iron, as well as nickel extraction. On heating to 800 °C, hydrohematite transforms to well ordered (stoichiometric) hematite [Fe_2O_3] characterised by the complete removal of OH^- of the oxide phase (Landers et al., 2009), decreasing the surface area. According to Li et al. (2009), the leaching rate can be related to the specific surface area.

As may be observed in Fig. 3, the nickel extraction from the ore calcined at 430 °C is the highest, probably because calcination opens the main gangue mineral structure and goethite, allowing a rapid interaction between the leachant and the nickel species during leaching. For this reason, further experiments employed this calcination temperature.

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