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Atmospheric pressure leaching of nickel from a low-grade nickel-bearing ore

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Abstract: This study focused on the extraction of nickel from a low-grade lateritic ore. The characterization of representative samples was done by X-ray diffraction (XRD), X-ray fluorescence (XRF), and microscopic mineralogical studies. Nickel was uniformly distributed in iron (hydr)oxide minerals. The pre-concentration of nickel was attempted by magnetic separation, but was unsuccessful. The effect of the type of lixiviant, acid concentration, S/L ratio, time, and temperature were investigated in the atmospheric leaching process. Based on the experimental data, optimum conditions for the maximum recovery of nickel were determined under the following conditions: 5 M H₂SO₄, 25% (w/v), 90 °C, and two hours. Pre-calcination, for increasing nickel extraction by converting goethite to hematite, was carried out on raw ores at different times (30-180 minutes) and temperatures (180-540 °C). Recovery was found increasing from 69 to 95% under the same conditions. Kinetic studies were conducted by fitting the data with Shrinking Core (SC) models. The study determined, from the estimate of activation energy, that the rate of reaction controlled by chemical reaction.

Keywords: nickel recovery, laterite ore, leaching, calcination, kinetics

1. Introduction

Nickel is an important metal used for a wide variety of applications. The world's various deposits of nickel are in the forms of sulfide and oxide (laterites) ores. Currently, the majority of nickel is obtained by exploiting the sulfide ores (Pawlowska and Sadowski, 2017). However, a continuous depletion of these generally high-grade deposits (sulfide ores) has led to oxides (in form of nickel laterites) become more attractive.

Nickel, obtained from low-grade lateritic deposits, is generally extracted by hydrometallurgical processing, mostly using sulfuric acid as the lixiviant. Other lixiviants have also been examined. For example, the prospects for using hydrochloric acid are reviewed by Harris et al. (2009) and a process based on nitric acid, termed as the DNi Process (McCarthy and Brock, 2015) has been described. Teir et al. (2007) found sulfuric acid to be more efficient than hydrochloric and nitric acid nickel leaching from serpentine ores.

In general, High Pressure Acid Leach (HPAL) is known as the most effective aqueous treatment process for the extraction of valuable metals from laterite ores (Dalvi et al., 2004). But the process requires specialized equipment and high operating temperature (245–270 °C), and pressure (4–5 MPa) (McDonald and Whittington, 2008a). Meanwhile, Atmospheric Leaching (AL) has also been reported for processing various limonitic and saprolitic laterite ores (McDonald and Whittington, 2008a, 2008b). The AL process has low energy consumption and capital costs. However, if sulfuric acid leaching conditions could be optimized, keeping in mind the problems, such as high acid consumption, high dissolved iron content in pregnant leach solution, and high residual acid concentration, the leaching of nickel laterites at atmospheric pressure could compete effectively with HPAL (Buyukakinci and Topkaya, 2009).

Atmospheric leaching of nickel from limonites using sulfuric acid is difficult. Furthermore, high acid consumption is often required to achieve high nickel extraction, which inevitably leads to a high concentration of residual acid in the pregnant solution. This, in turn, creates difficulties in the downstream operations for iron removal (Chang et al., 2016). Further studies were carried out to reduce the residual acid concentration and decrease the iron content by multi-step leaching techniques (Liu et al., 2004; Liu and Krebs, 2006).

Another strategy to reduce the large amount of acid, without jeopardizing nickel recovery, was to pre-treat the lateritic ores. Among the pretreatment methods, the following were explored: reductive roasting (Chang et al., 2008), pre-roasting in the air (Li et al., 2013), sulfation-roasting (Guo et al., 2009), selective reduction - chlorination (Fan et al., 2010), alkali activation pretreatment (Wang et al., 2014; Zhang et al., 2015), calcination (Kim et al., 2010), and para-goethite precipitation (Basturkcü and Acarkan, 2017).

The simplified iron reactions during acid leaching of the pre-reduced limonitic laterite ore can be conceptually presented as below:



Nickel is also released in the solutions during acid leaching of the calcine from reduction roasting and iron dissolution by equation (1). The dissolved ferrous iron is oxidized to ferric iron (Eq. 2), which immediately undergoes hydrolytic reactions (3) and (4) (i.e. the conversion of soluble iron to insoluble FeOOH and Fe₂O₃). The acid consumed by the leaching of FeO in reaction (1) is regenerated by hydrolytic reactions (3) and (4) (Chang et al., 2016).

This study focused on atmospheric pressure acid leaching of a low-grade nickel-bearing ore, obtained from tuff oxides of Sarbisheh, Iran. After chemical and mineralogical analysis, the preliminary leaching of the ore (-0.050 mm) was carried out under various conditions using different types of lixiviant, temperature, time, acid concentration, and solid/ liquid ratio. Particular interest was devoted to the dissolution behavior of pre-calcined ore under optimum conditions. Furthermore, the leaching kinetics of Ni was studied and the corresponding kinetic model was proposed.

2. Material and methods

The nickel-bearing ores used in this study was supplied from low-grade laterite ores at Sarbisheh, Southern Khorasan, Iran. Primary and secondary crushing have done by using laboratory jaw crusher and ball mill, respectively. Representative samples were ground in a disk pulverizer. Mineralogical studies of ore samples were carried out using XRF, XRD, and optical microscopic study of thin and polished sections. The chemical composition of the lateritic nickel ore was analyzed on a ARL PERFORM'X spectrometer in Razi metallurgical research center (RMRC), Tehran. The mineralogical compositions were examined based on BS EN13925-1; 2008 standard method before and after leaching/roasting on a XRD with Cu K α 1 operating at 40 kV, 30 mA and 2 θ = 2-90°.

To prepare the leaching experiments' feed, the particle size of the sample was reduced to 100% finer than 50 μm . Sulfuric acid and hydrochloric acid used in this work were of analytical grade. Based on thermo-gravimetric and differential scanning calorimetry (DSC-TGA) results, a series of calcination experiments was carried out in order to investigate the effect of pretreatment on nickel extraction. Thermal analysis was performed using BAHR STA 503 with heating speed of 5 c/min and a maximum temperature of 750 °C.

Leaching experiments were conducted using sulfuric and hydrochloric acid in a 600 cm³ glass beaker, placed on a hot plate/magnetic stirrer. For each test, dilute acid solution was heated to a desired temperature. A weighed ore sample was placed in the beaker and magnetically stirred at 300 rpm. pH was adjusted by using the Metrohm 827 pH meter (Switzerland). Some samples were drawn at predetermined time intervals and the element concentrations were analyzed by using the AAS 240 Varian Spectrometer. All chemicals were in analytical grade.

3. Results and discussion

3.1 Sample characterization

According to the XRF results (Table 1), the laterite ore was characterized by high iron content ($Fe_{total} = 62.12$ wt.%), low nickel content (0.8 wt.%). The results of the XRD analysis are shown in Fig. 1 and table 2. The results indicated that quartz (SiO_2) and goethite [$FeO(OH)$] and dolomite were the main crystalline phases, whereas no crystalline nickel phase was observed. This suggests that any nickel phase is predominately amorphous or the concentration is below the detection limit, or that the nickel is contained within other minerals. The sample is a typical goethitic-type nickeliferous laterite ore.

Table 1. The chemical composition of laterite ore analyzed by XRF /wt %

Composition	SiO_2	Al_2O_3	MgO	Fe_2O_3	LOI	
Content (%)	24.5	1.12	0.64	62.1	8.1	
Composition	NiO	Cr_2O_3	Co_3O_4	SO_3	CaO	MnO
Content (%)	0.8	0.51	0.1	0.16	1.62	0.49

* LOI is loss on ignition

Microscopic observation showed that the main part of the sample was composed of goethite and hematite (Fig. 2a). Nickel was present in the iron (hydr)oxide minerals (Fig. 2b) in the rock matrix. In Fig. 2c, the ore matrix is covered by hematite minerals. The nickel-containing minerals in rock matrix are illustrated in Fig. 2d. In similar studies by Kim et al. (2010), it was observed that nickel is uniformly distributed and has a very small particle size. The study also found nickel occurring in iron-bearing minerals (Kim et al., 2010). Considering these results, and also preliminary grinding and classification test, vigorous grinding would be required to reduce the size of the samples (down to $\approx 50 \mu m$) in order to liberate goethite particles.

3.2 Pre-concentration by magnetic separation

In the present work, magnetic separation of the low grade ($\sim 1\%$), goethite-rich Ni laterite ores was investigated. Samples of 1 kg mass were ground down to $\sim 50 \mu m$ and two different intensities (0.12 and 0.25T) of dry magnetic separators were used at ambient temperature. Table 3 shows the amount of magnetic material and nickel content in each fraction. However, the Ni distribution did not show any noticeable magnetic upgrade.

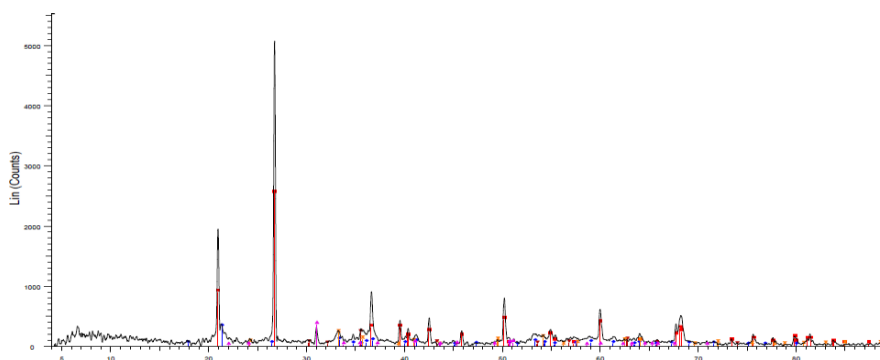


Fig. 1. XRD patterns of the Sarbisheh laterite ore

Table 2. The phases identified by X-ray diffraction

Phase name	Chemical composition
Quartz	SiO_2
Goethite	$FeO(OH)$
Dolomite	$CaMg(CO_3)_2$
Hematite	Fe_2O_3
Magtite	Fe_3O_4

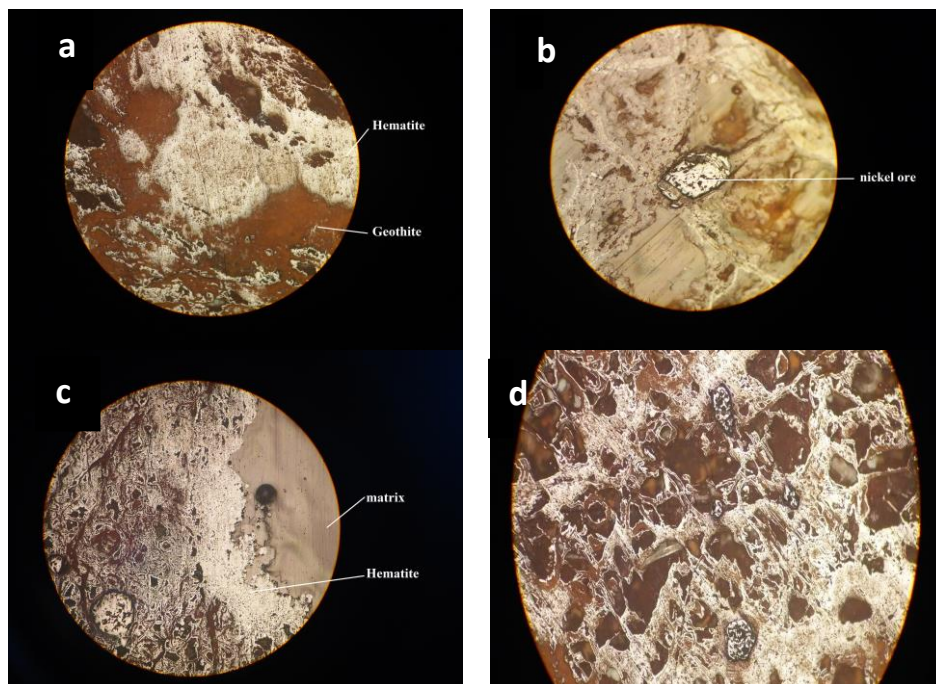


Fig. 2. Polished sections of lateritic ore; a) main phase of sample, b) nickel in the rock matrix, c) association of the phases, d) nickel-containing minerals in rock matrix

Table 3. The effect of magnetic separation on Ni concentration

Fraction	Weight percentage (%)	Ni content (%)
Feed	100	1.31
Concentrate at 0.12 T	1.42	0.594
Concentrate at 0.25 T	2.81	0.703
Tailing	94.7	1.323

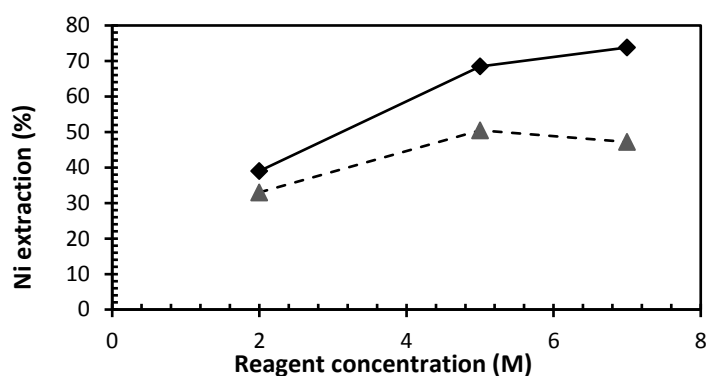


Fig. 3. The effect of the lixiviant type on nickel dissolution: ◆ sulfuric acid, ▲ hydrochloric acid. Operation conditions: 90 °C, 2 h, S/L= 0.25 g/cm³, 300 rpm

3.3 Ni extraction by leaching

3.3.1 Effect of the type of acid

A series of leaching experiments was conducted with different concentrations of sulfuric and hydrochloric acid as lixiviant. Conditions, such as temperature (90 °C), time (two hours), stirring speed (300 rpm), and S/L ratio (0.25 g/cm³), were kept constant while conducting the experiments. The best conditions for the maximum recovery were obtained by using H₂SO₄ (see Fig. 3). Therefore, sulfuric acid solution was used for nickel extraction in a comprehensive study.

3.3.2 Effect of acid concentration

The effect of H_2SO_4 concentration on Ni recovery was studied at 90°C over two hours. The S/L ratio was changed from 0.1 to 0.4 g/cm^3 . Generally, an increase in acid addition for atmospheric leaching improves the metal extraction from laterite ores (Buyukakinci and Topkaya, 2009) and causes more nickel dissolution (Fig. 4). Therefore, with an increasing H_2SO_4 concentration from 2 to 5 M, the extraction of Ni was increased from 15 to 68%. This effect is also observed at higher S/L ratios, which will be discussed in more detail in the next section.

3.3.3 Effect of solid/liquid ratio

The effect of solid loading upon Ni extraction is visible in Fig. 5. As the S/L ratio increased, the extraction was raised to a maximum level. In this study, Ni extraction was increased for different H_2SO_4 concentrations with S/L ratio from 0.1 to 0.25 g/cm^3 .

At a higher S/L ratio, stronger acid ($\geq 5\text{ M}$) was required to improve the extraction of valuable metals, whereas, at a low acid concentration (2 M), it was not possible to dissolve nickel from laterite ore, with the extraction reducing sharply at higher S/L ratio (0.4 g/cm^3). The mixture became thick and pulpy at high acid concentration and S/L ratio (i.e. 7 M and 0.4 g/cm^3), making it impossible to agitate. Hence, $\text{S/L} = 0.25\text{ g/cm}^3$ (25% w/v pulp density) was selected for the rest of the experiments.

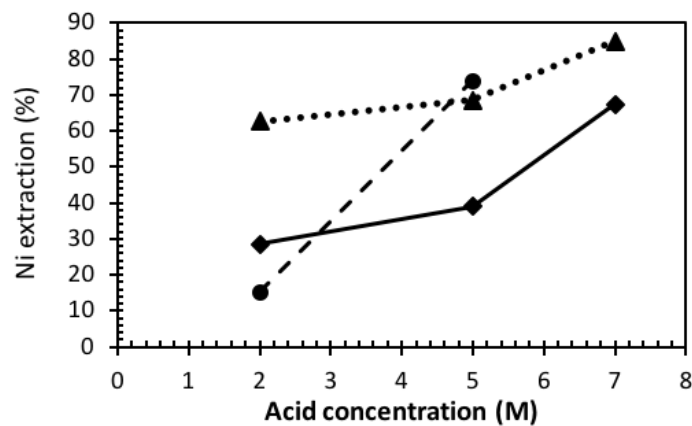


Fig. 4. The effect of acid concentration on Ni extraction for various S/L ratios: ◆ 0.1, ▲ 0.25, ● 0.4 g/cm^3 . Operation conditions: 90°C , 120 min, 0.1-0.4 g/cm^3 , 300 rpm

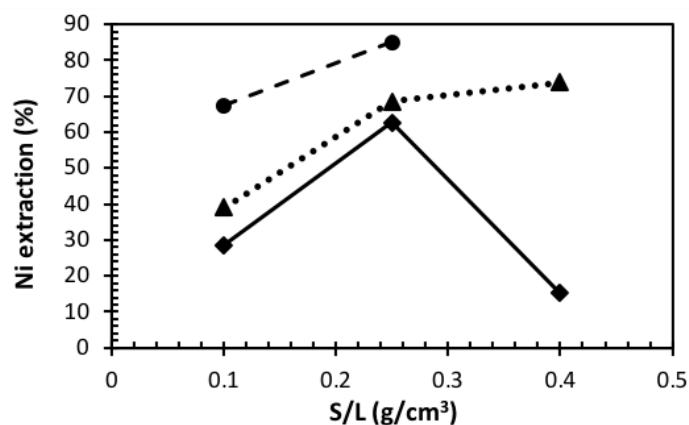


Fig 5. The effect of S/L ratio on Ni extraction. Operation conditions: 90°C , 120 min, ◆ 2, ▲ 5, ● 7 M H_2SO_4 , 300 rpm

3.3.4 Effect of temperature

The effect of temperature on nickel dissolution from the laterite ore was investigated over a temperature range of $45\text{--}60^\circ\text{C}$ at acid concentration of 5 M, stirring speed of 300 rpm, and L/S equal to $4\text{ cm}^3/\text{g}$. Higher leaching temperature generally leads to reduced batch time and increased extractions

(MacCarthy et al., 2014, 2016). However, for some systems, it may be more economical to leach for a longer time at lower temperatures rather than at the higher ones. Therefore, samples were taken at different intervals to study the time and temperature effects simultaneously.

Temperature has a significant effect on the rate of Ni dissolution (Fig. 6). Weak dissolution of nickel at a low temperature (45-60 °C) was observed. The increasing reaction time had negligible influence on the leaching efficiency. However, Ni extraction sharply increased at 60-90 °C. It was noticeable that an increase in time improved the nickel extraction at ambient temperature. In fact, at a temperature of 40 °C, 43% of nickel was extracted after three hours, whereas 54% of nickel was recovered at a temperature of 90 °C after 30 minutes.

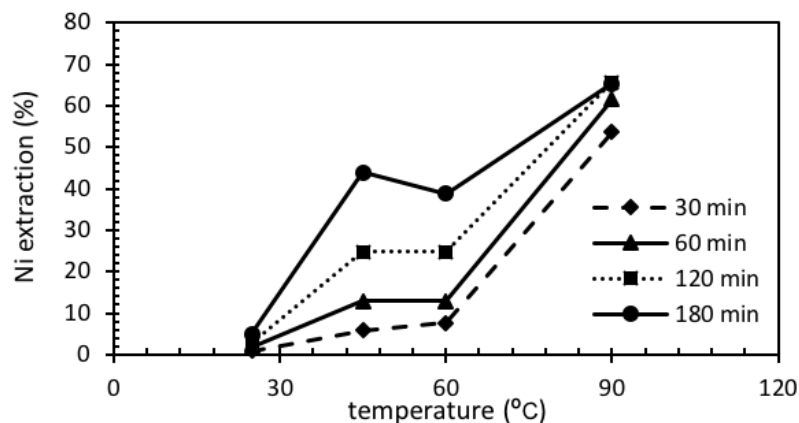


Fig. 6. The effect of temperature on Ni extraction. Operation conditions: 5 M H₂SO₄, 0.25 g/cm³, 300 rpm

3.3.5. Effect of contact time

In order to investigate the effect of contact time, some experiments were performed with 5 M H₂SO₄ solution at 90 °C and L/S ratio of 4 cm³/g. Samples of 5 cm³ were taken at regular intervals during the leaching. The results of nickel extraction vs. time are shown in Fig. 7. At the beginning of the leaching, the extraction rate was fast. During the first 30 minutes, Ni extraction was 53.7% and it reached 61.5% after 60 minutes. However, negligible further dissolution was found after 120 minutes.

It can be concluded that the extraction of nickel from the laterite ore reached 70% at optimum conditions. Therefore, some pre-treatment steps should be undertaken if the leaching of Ni from these resources is to be improved.

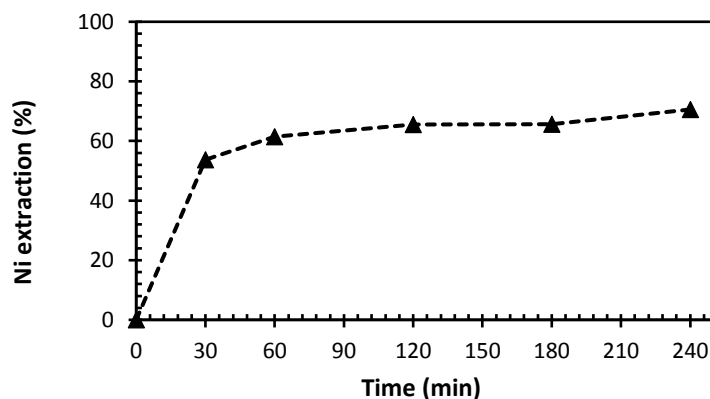


Fig. 7. The effect of the contact time on Ni extraction; operation conditions: 5 M H₂SO₄, 90 °C, 0.25 g/cm³, 300 rpm

3.3.6 Effect of pre-calcination

The effect of pre-calcination on nickel extraction was studied in the present work. When the laterite sample was heated, the dominant mineral phase goethite converts to hematite via the following reaction (Teir et al., 2007):



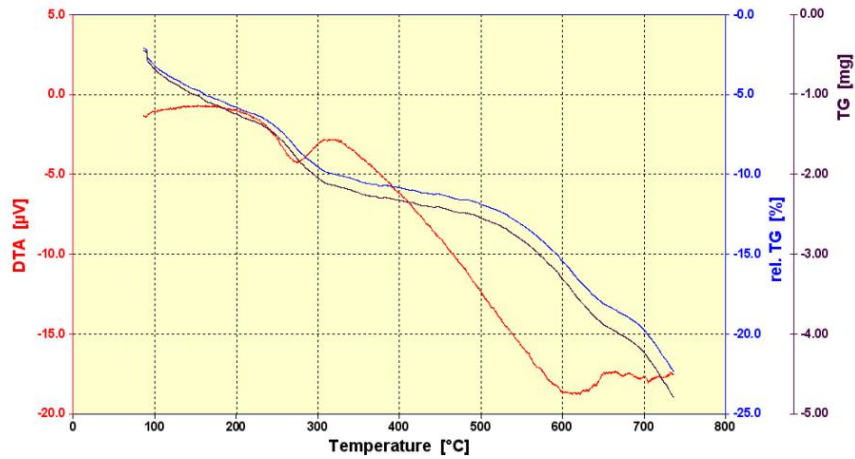


Fig. 8. TG/DTA curve for Sarbisheh nickel laterite ore

Table 4. The effect of calcination temperature (2 h) on Ni extraction (90 °C, 2 h, 0.25 g/cm³ S/L, 5 M H₂SO₄, 300 rpm)

Temperature (°C)	Nickel extraction (%)
0	69
180	72
280	85
540	95

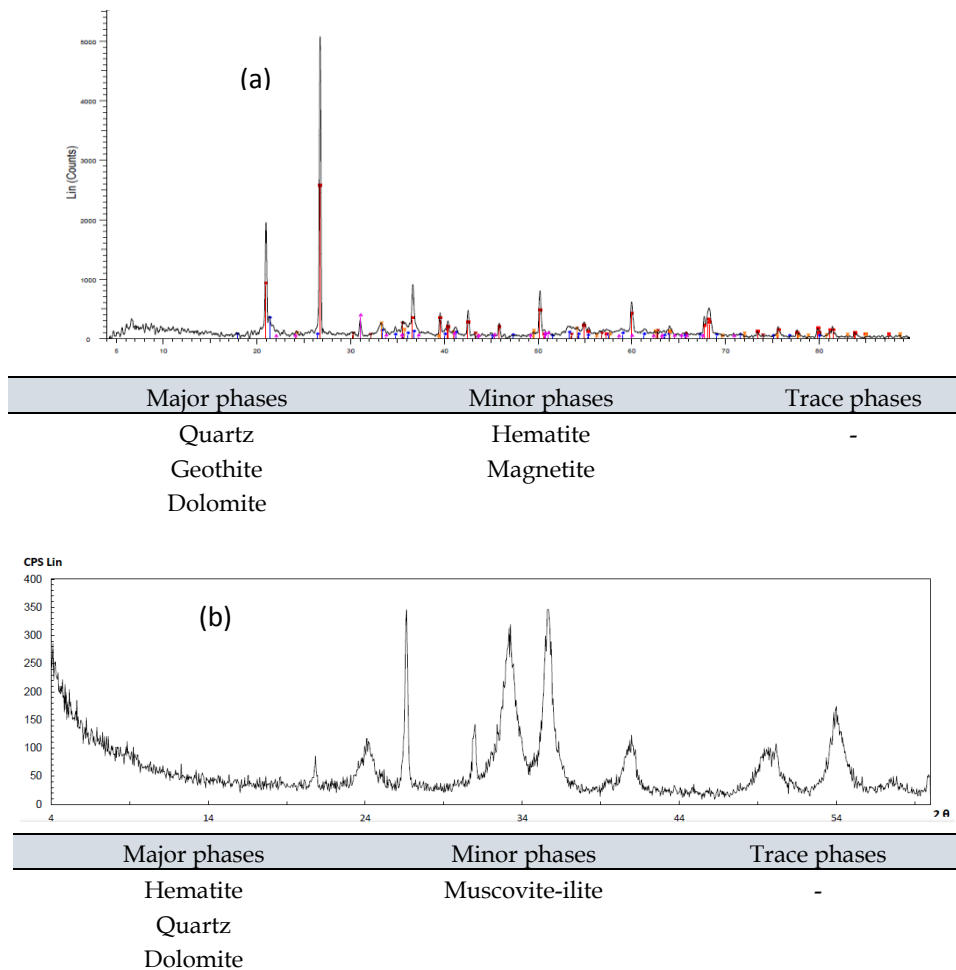


Fig. 9. XRD analysis of a sample (a) before and (b) after calcination at 540 °C for 120 min

Thermogravimetric Analysis (TGA) and differential thermal analysis (DTA) were used to examine the thermal behaviour of the laterite. DTA-TG curves (Fig. 8) showed two main endothermic peaks at 280 and 610 °C corresponding to the structural changes. The first main peak at 280 °C corresponded to the dehydroxylation of goethite to hematite. The temperature of dehydration of pure goethite varies between 274 and 305°C, depending upon the particle size, crystallinity (MacCarthy et al., 2014, 2016), type and extent of metal substitution into the goethite structure. The endothermic peak at about 610 °C corresponded to the removal of the hydroxyl group or the partial decomposition of silicates. In other ores, this temperature varied from 500 to 800 °C (MacCarthy et al., 2014, 2016).

Based upon the TG and DTA curves, the calcinating was conducted at 180, 280, and 540 °C for different time spans. After calcination, the samples were dissolved at optimum conditions (5 M H₂SO₄, 90°C, 2 h, S/L 0.25 g/cm³) and analyzed by AAS in order to determine the nickel concentration. The results are presented in Table 3. As shown, pre-calcination of the ore, followed by the leaching, resulted in higher nickel recovery in comparison to direct leaching of raw limonite ore. Also, nickel extraction during leaching using optimum conditions significantly improved with an increase in calcination temperature. Here, the nickel extraction increased to 95.3% for calcination temperature of 540°C (Table 4).

As goethite was not observed in XRD analyses of calcined ore at 540°C (Fig. 9), this confirms that the host mineral (goethite-FeOOH) converts to Fe₂O₃, which was observed, and which was easier to dissolve. This strategy was based on the fundamental fact that nickel dissolution is directly correlated with iron dissolution. During the leaching process, Iron, nickel and cobalt have been analyzed at the H₂SO₄ concentrations of 5 M. As seen in the Fig. 10, by increasing the acid concentration, the recovery of iron and nickel rises up. Therefore, this Fig. confirmed that nickel presents in the iron hydr(oxide) minerals.

The effect of pre-calcination time was also investigated. Table 5 shows the nickel recoveries for ore calcined at 280 and 540°C. The highest dissolution of the nickel is achieved for pre-calcined ores was after sufficient duration (120 min) and high temperature (540°C).

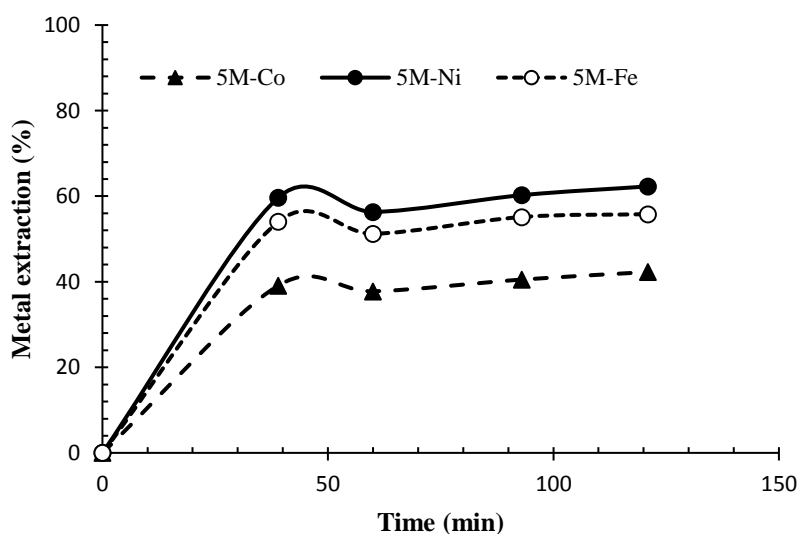


Fig. 10. The extraction of nickel, cobalt and iron by sulfuric acid concentration of 5 M

Table 5. The effect of calcination time on Ni extraction; operation conditions: 90 °C, 2 h, 0.25 g/cm³ S/L, 5 M H₂SO₄, 300 rpm

Time (min)	Nickel extraction (%)	
	(280 °C)	(540 °C)
30	68.4	N
60	74.2	73.6
120	85.5	95.3

3.3.7 Kinetics of nickel dissolution by sulfuric acid

To determine the controlling mechanism for the acid leaching of the laterite ore, an attempt was made to fit the experimental data obtained from leaching to the Shrinking Core (SC) models (Eqs. (6) - (8)). This model assumes that the solid particle retains its bulk size while its unreacted core shrinks progressively with reaction time, leading to the formation of a peripheral porous layer (MacCarthy et al., 2014). The overall acid leaching reaction involves the following: proton diffusion through the liquid film and the reacted porous layer, chemical reaction at the surface of the unreacted core, and volume diffusion of reaction products through the porous layer and into the liquid film back into the bulk solution. The leaching rate is controlled by the slowest of these processes (MacCarthy et al., 2016; Wanta et al., 2016). Eqs. (6) to (8) summarize the shrinking core models.

$$x = kt \quad (6)$$

$$1-3(1-x)^{2/3}+2(1-x) = kt \quad (7)$$

$$1-(1-x)^{1/3} = kt \quad (8)$$

where x is the fraction of the reacted nickel, k (min^{-1}) is reaction rate constant, and t (min) is the leaching time. Eq. (6) assumes the rate controlling steps are bulk diffusion while Eq. (7) assumes it to be the diffusion of leaching reagent through the product layer on the surface of a mineral particle. Eq. (9) assumes that the leaching rate controlling step is the chemical reaction occurring on the surface of a mineral particle.

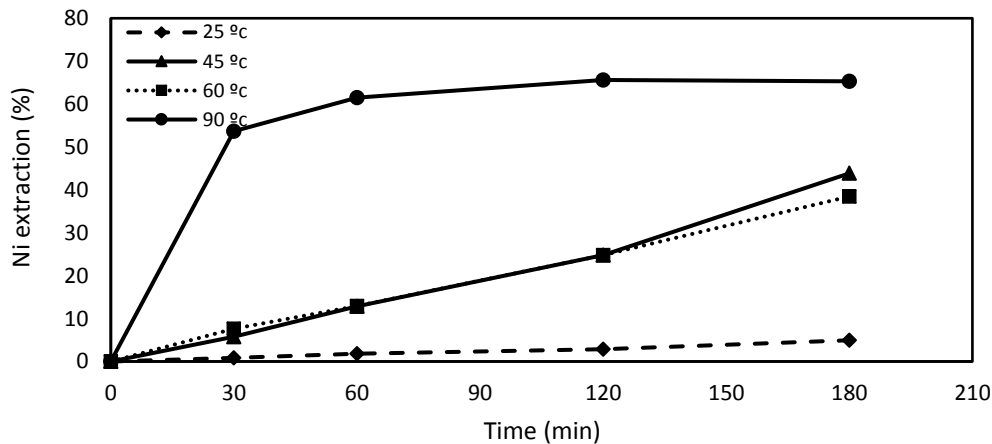


Fig. 11. The effect of temperature on Ni extraction at different time intervals.
Operation conditions: 0.25 g/cm³ S/L, 5 M H₂SO₄, 300 rpm

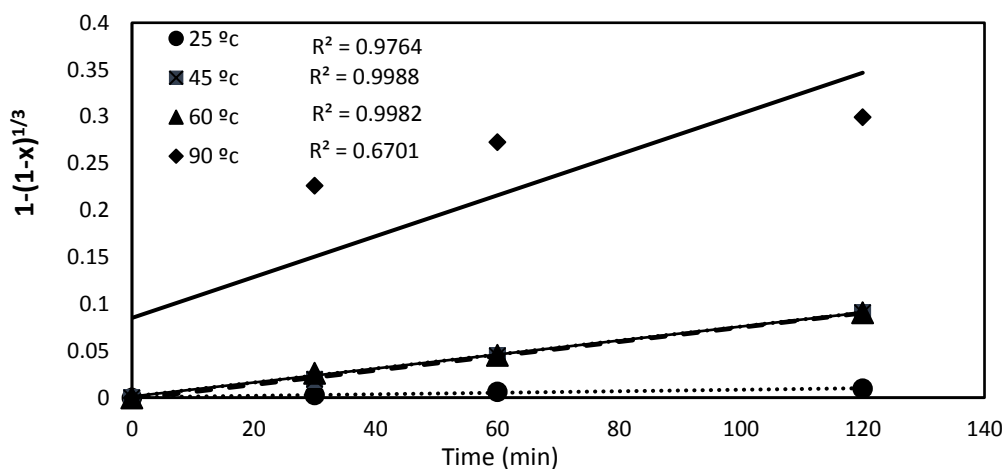


Fig. 12. Chemical control model for kinetics of leaching of nickel at different temperatures

Fig. 11 shows nickel recovery at different temperatures. The data suggest that rising temperature from 60 to 95 °C dramatically increased the extraction of Ni from 12 to 61% after one hour of leaching.

The experimental data obtained from leaching (Fig. 11) were fitted to Eqs. (6) - (8). It was found that the kinetic data showed a poor fit to both the models. However, the correlation coefficients for chemical control model were higher than those of diffusion control models (Fig. 12). According to Fig. 12, during the whole reaction time, as expected, the extraction rate increased evenly with an increase in temperature. This indicates that the rate of reaction was controlled by chemical reaction on the surface of the ore particles.

Based on the rate constants obtained, the activation energy (E_a) for the reaction can be determined by using the Arrhenius law (Eq. (9)):

$$K = K_0 e^{-E_a/RT} \quad (9)$$

where E_a (kJ/mol) is the activation energy, R (8.314 kJ/mol) is the ideal gas constant, k_0 (min^{-1}) is the pre-exponential factor, and T (K) is absolute temperature. For volume diffusion controlled processes, the E_a is generally low (<20 kJ/mol) while for chemical reaction controlled processes it is high (>40 kJ/mol) (Habashi, 1969). Fig. 13 shows the plot of $\ln K$ versus $1000/T$. The plot has an acceptable correlation coefficient (R^2) of 0.9556 and the activation energy of 46.9 kJ/mol was indicative of a temperature-dependent reaction and chemical reaction-controlled leaching behavior.

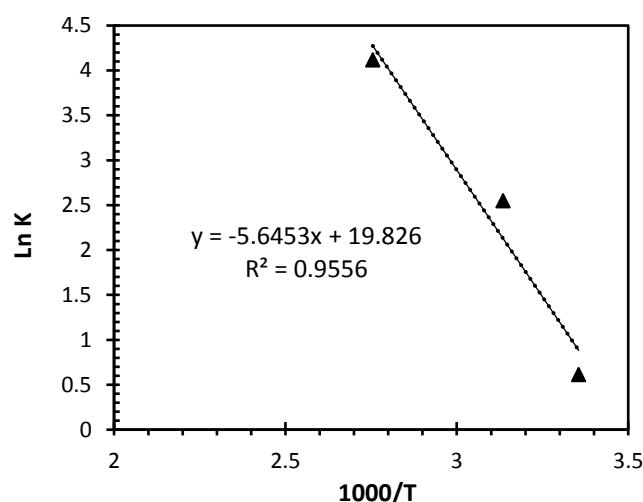


Fig. 13. Plots of $\ln(k)$ vs $1000/T$ for leaching of nickel from laterite ore

4. Conclusions

This study investigated the recovery of nickel from a low-grade laterite ore, and it was found that:

- Goethite and hematite are dominant minerals of the sample and nickel is distributed throughout the ore.
- Pre-concentration of laterite ore by magnetic separation was conducted, but no upgrading of nickel was obtained.
- When lateritic ore is heated, the dominant mineral phase, goethite, converts to hematite, which is easier to dissolve. Therefore, pre-calcination is an effective step to increase nickel extraction.
- The TG/DTA analysis of the laterite sample shows two main endothermic peaks. The dehydroxylation of goethite to hematite takes place at 280 °C and XRD analysis before and after calcination confirmed this transformation.
- Nickel extraction using sulfuric acid reach higher values in comparison to hydrochloric acid leaching. The effect of experimental parameters on Ni recovery was studied and optimum conditions were determined as 0.25 g/cm³ S/L, 5 M H₂SO₄, 90 °C, and two hours.
- The highest dissolution of the nickel from limonite ores was achieved by pre-calcination of ore at an adequate duration (120 min) and a high temperature (540 °C).
- Experimental data were fitted to the shrinking core (SC) models. The results indicated the rate of reaction is controlled by chemical reaction at the surface of the ore particles, consistent with the activation energy which was determined to be 46.9 kJ/mol.

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