

SELECTIVE LEACHING OF NICKEL FROM PREREDUCED LIMONITIC LATERITE UNDER MODERATE HPAL CONDITIONS- Part I: Dissolution

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Abstract

High pressure acid leaching (HPAL) is the primary method for extraction of nickel from limonitic laterite. The severe operating conditions, the complexity of facilities, and several technical problems, such as scale formation, have always accompanied the HPAL, however. In this paper, the selective acid leaching of nickel from the pre-reduced limonitic laterite under moderate HPAL conditions was studied. For the calcine with reduction degree of 93.4%, about 88.2% nickel could be selectively extracted at 100°C with an acid/ore ratio of 0.164, leaving less than 5.0g/L iron in the solution. Further increase of leaching temperature, and the ensuing dehydration, transformed goethite in the leaching residue to hematite. When the temperatures were as high as 180°C, the recovery of nickel increased to 95.2%, within one hour, depressing the residual soluble iron to below 1.0g/L ($pO_2 = 1.0$ MPa, acid/ore = 0.287).

Keywords: Nickel; Selective Leaching; Reductive Roasting; Goethite; Hematite

1. Introduction

The first plant for high pressure acid leaching (HPAL) of laterite ores was built in Moa Bay, Cuba [1]. It represented the follow-up to the previously built plant based on Caron process, known by laterite ore pretreatment step by reductive roasting to enable ammoniacal leaching of nickel in the subsequent stage. Since then, it was not until 1990s, and beyond, when the new HPAL plants were built that are currently in operation in Australia, Philippines, New Caledonia, Papua New Guinea and Turkey [2]. All these HPAL plants, regardless of their generational advancement, were very expensive to build, and equally expensive to operate. To minimize the corrosion at the typical high temperature ($T > 250^\circ\text{C}$) and pressure ($p > 4$ MPa) conditions, very expensive construction materials were needed. The operating difficulties and expenses were tied to solving the corrosion issues, safety control, descaling of reactor walls, dealing with the acid neutralization and iron control.

Obviously, the process based on atmospheric pressure leaching (AL) conditions would provide significant advantages over HPAL, and would have been preferred if it were available. The motivation for developing such a process has been in place as can be judged by the very large volume of relevant research

information produced by industry, government research labs, and academia. The main challenges to meet during AL process development have been high acid consumption, selective leaching, iron control and acceptable nickel recovery.

Generally, under AL conditions, nickel is more difficult to leach from limonite than from smectite (clay) and saprolite ores [3]. To achieve high nickel extraction, often high acid consumption is required, as high as one ton of H_2SO_4 per ton of laterite ore, which inevitably leads to high concentration of residual acid in the pregnant solution creating difficulties in the downstream operations for iron removal [4,5].

One of the strategies to reduce the requirement for large amount of acid, without jeopardizing nickel recovery, was to pretreat the lateritic ores in a particular way that would enable a readier nickel leaching compared to the original ore. This would simultaneously work in favor of having an improved iron control. Among the pretreatment methods the following were explored, pre-roasting in the air [6,7,8], sulphation roasting [9], selective reduction-chlorination [10], alkali activation pretreatment [11,12,13], to list a few.

Another approach was to leach lateritic ores with the reductant present in the leaching medium. The expectation was that the reductant used would help with unlocking the nickel hosting minerals in laterite

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ore that would result in improved nickel leaching and selectivity. Among numerous reductants explored, such as $\text{Na}_2\text{S}_2\text{O}_3$, $\text{H}_2\text{C}_2\text{O}_4$, SO_2 , Fe^0 and Fe^{2+} [14,15, 16,17,18,19,20] only the use of metallic iron [18] produced somewhat satisfactory results.

As the lixiviant, sulfuric acid has been used the most. Other lixiviants were explored, as well. For example, the prospects for using hydrochloric acid are reviewed by Harris et al. [21]. Most recently, a process based on nitric acid, termed as DNi Process [22] is being evaluated toward its potential for process development for treatment of laterite ores under atmospheric pressure leaching conditions.

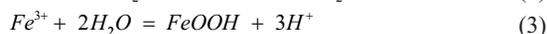
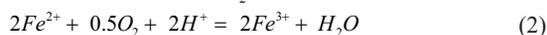
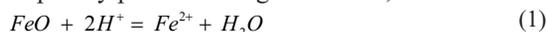
Finally, considering vast quantities of low grade limonitic laterite ores, it should be pointed that the most ideal process for recovery of nickel would have been heap leaching with sulfuric acid, the main advantage being minimal capital and operating expenditures. However, low recoveries, lengthy leaching times, high acid consumption, nonselective leaching, decommissioning challenges, and environmental problems are the most serious hurdles toward its commercialization [23].

Despite numerous explored strategies toward implementation of atmospheric pressure leaching of nickel laterites none of the methods that have been studied thus far has been commercialized for the treating of low grade limonitic laterites.

In this work, our strategy was to return to the proven HPAL process but seek the methods that could lead to its use under less severe temperature and pressure conditions. The judging criteria would be uncompromised nickel recovery, improved nickel leaching selectivity, and improved overall iron control. The approach that was selected was to use the reduction roasting step as in the Caron process, but with the difference that the produced calcine would serve as the feed in HPAL. The expectation was that reduction roasting would make nickel more amenable to leaching, thus placing more moderate requirements toward temperature and pressure in HPAL.

As nickel in limonitic laterites is mainly associated with iron oxides, such as goethite - FeOOH , the task of reductive roasting is to convert the host mineral to a lower more active form of oxide, wustite- FeO , that would be easier to dissolve than the raw ore in the traditional HPAL. This strategy is based on the fundamental fact that with respect to laterites nickel dissolution is directly correlated with iron dissolution.

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



During acid leaching of the calcine from reduction roasting, along with iron dissolution by equation (1), nickel is also released in the solutions. Simultaneously, the produced ferrous iron is oxidized to ferric state, reaction (2), which immediately undergoes hydrolytic reactions (3) and (4), i.e. the conversion of soluble iron to insoluble FeOOH and Fe_2O_3 . The net effect is selective leaching of nickel against iron, just like in the traditional HPAL, but this time under much milder temperature and pressure conditions. The acid consumed by leaching of FeO in reaction (1), is regenerated by hydrolytic reactions (3) and (4).

2. Experimental

2.1 Materials

A limonitic laterite ore (P80=74 μm) from Philippines, used in this study, had the following composition: 48.82 %Fe, 1.0 %Ni, 0.137 %Co, 0.485 %Mg, 0.592 %Ca, 3.46 %Al, 1.09 %Mn, 2.47% SiO_2 , 0.173 %S. The ore pelletized to about 10 mm diameter was subsequently subjected to reduction in a furnace with alumina tube under CO/CO_2 atmosphere. Calcination in the temperature range of 800-1000 $^\circ\text{C}$ produced calcine with the different reduction degree. The XRD patterns of the raw limonite ore and the reduced calcine with a reduction degree of 93.4% are shown in Fig. 1. The main mineral in the raw ore is goethite, with some other non-identified minor phases. The reduced calcine consists of wustite, minor magnetite, and Fe-Ni alloy.

The reduction degree of the calcine was defined as the mass ratio of the analyzed low valance iron to the total iron in the calcine, $m_{\text{Fe(II)+Fe(0)}/m_{\text{total Fe}} \times 100\%$. After reductive roasting, the samples of the reduced

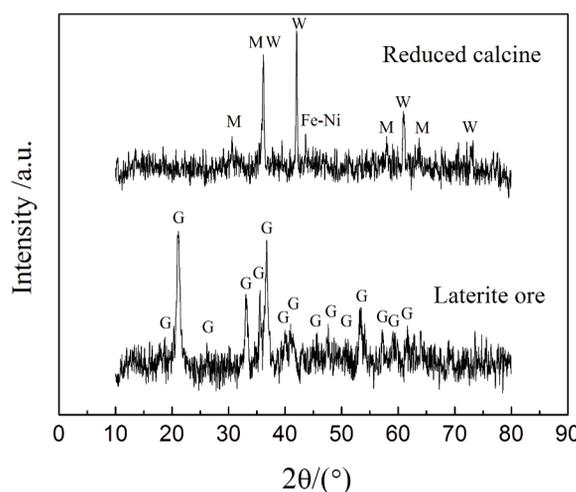


Figure 1. XRD patterns of raw laterite ore and the reduced calcine with the reduction degree of 93.4% (G: goethite, W: wustite, M: magnetite, Fe-Ni: iron-nickel alloy)



calcines were firstly dissolved with 6M HCl and then analyzed by titration with $K_2Cr_2O_7$ to determine the concentrations of Fe^{2+} and total iron.

In most of the leaching tests, the calcine with a reduction degree of 93.4% was used. Two other calcines with the reduction degree of 80.2% and 73.1% were also used to examine the importance of the extent of reduction. The purity of oxygen gas was 99.6%. All other reagents were of analytical purity.

2.2 Acid Leaching of Calcine

The atmospheric leaching was conducted in a two-liter seven-necks flask heated by electric heating plate. The stirrer shaft and central neck of the flask were assembled with a mercury seal to minimize the evaporation of water. Other necks of the flask were sealed by plastic plugs to hold the thermal probe, sampling tube and gas inlet/outlet. A water-cooled condenser prevented water loss by evaporation. For the batch oxygen pressure leaching test, autoclave equipped with one liter zirconium made container was used.

In an experimental procedure, the specific mass of calcine slurred with 600 ml water, according to the desired liquid/solid (L/S) ratio, was heated preset to a fixed temperature. Then the calculated amount of concentrated H_2SO_4 was added according to specific acid/ore (A/O) ratio.

The oxygen cylinder was used to supply oxygen. The oxygen was introduced after autoclave reached the preset temperature. The pressure of oxygen was controlled by a pressure reducing valve. The pressure difference on the gauge represents oxygen overpressure, which was kept constant during the pressure leaching experiments.

2.3 Analytical methods

The solution samples, obtained by filtering of withdrawn slurry samples at particular time intervals, were used to analyze dissolved metals. The residues were also analyzed to provide the complete information on the mass balance, particularly with respect to impurities such as Ca, Mg, and Al. The analysis was performed by using ICP-AES. The concentrations of ferrous ion and total iron were determined by redox titration method as mentioned previously. The residual concentrations of sulfuric acid in the leaching solutions were determined by titration with NaOH solution using methyl orange as indicator and using sodium salt of cyclohexane -1,2-diaminetetraacetic acid (CDTA) to mask the ferric ion. For the solution samples with relatively low acid concentration, a Sartorius PB-10 type digital pH meter equipped with automate temperature compensation probe, was used to measure the pH at

room temperature. The phase composition was identified by XRD and the morphology of the leaching residue was characterized by SEM. Sulfur content in the leaching residue was determined by a C-S analyzer.

3 Results and discussion

3.1 Leaching under Atmospheric Pressure of Oxygen

Leaching of the calcine with reduction degree of 93.4 % under atmospheric pressure was conducted at 95°C, A/O 0.46, L/S 20:1, O_2 flow $1.5L \cdot min^{-1}$. Fig. 2(a) shows the concentration curves of iron and nickel vs. time. The pH vs. time is shown in Fig. 2(b). Twenty minutes after the addition of sulfuric acid, the concentration of iron rapidly reached about 13g/L, which equals to 33.6 % extraction of the iron in the calcine. This means that the reduction made calcine responsive to leaching with sulfuric acid. Iron dissolved nearly all as ferrous iron. The extraction of nickel at the first twenty minutes was only 16.3 %, which is low compared with the extraction percentage of iron. Considering high pH during this time, part of the leached nickel might have re-precipitated, or adsorbed onto the residue, as indicated in another study [24].

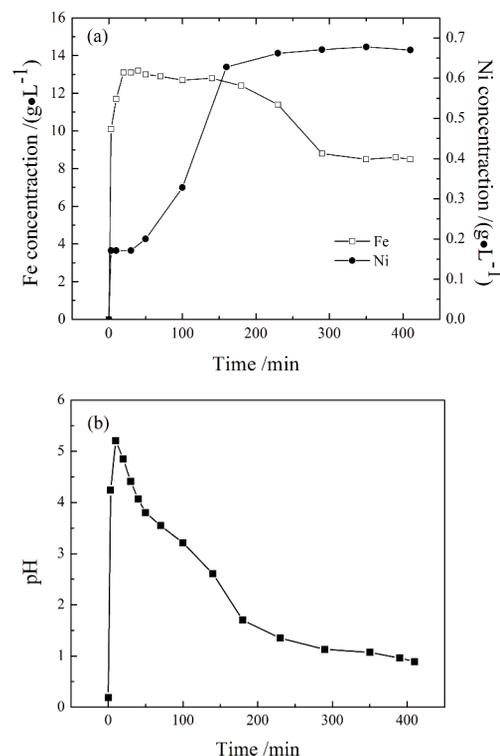


Figure 2. (a) Leaching of reduced calcine at 95°C under atmospheric pressure oxygen; (b) pH variation with time. (Conditions: calcine with reduction degree of 93.4%, A/O 0.46, L/S 20:1, O_2 flow $1.5L \cdot min^{-1}$)



Upon initial increase, subsequently, the iron concentration was slowly decreasing mainly due to the oxidation of ferrous to ferric iron and the hydrolysis of ferric iron. The hydrolysis of ferric iron reduces the pH of solution enabling further leaching of nickel from the calcine. Due to the slow oxidation of ferrous to ferric iron, the rate of additional nickel dissolution is also slow. After six hours, the solution stabilized to about pH=1, effectively stopping further oxidation/hydrolysis. The final leach solution contained 0.67 g/L Ni, corresponding to 63.8 % Ni extraction. The solution still contained about 8.5 g/L of iron. The low rate of leaching and the relatively low extraction rate of Ni made the atmospheric leaching less attractive.

3.2 Transformation of Iron to Goethite at High Oxygen Pressure

The slow oxidization kinetics of ferrous iron in the acidic solutions is a well-known issue in hydrometallurgy. One of the methods to increase the rate of ferrous iron oxidation is by increasing the partial pressure of oxygen. The results from leaching of calcine with a reduction degree of 93.4 % under 1.0 MPa O₂ are shown in Fig. 3, where the extraction of Ni and concentrations of free acid and iron in solution are given vs. time. The concentration of iron reached 26.5 g/L only five minutes after the addition of sulfuric acid. Nearly all of iron was present in ferrous form. Under the effect of high pressure of oxygen, the ferrous iron was rapidly oxidized, followed by hydrolysis of ferric iron and the generation of sulfuric acid. Under these conditions, the solid hydrolysis product is goethite, as verified by the XRD analysis. The generated acid then leached more Ni from the calcine. At sixty minutes, 84.6% of Ni was extracted, with the total iron and sulfuric acid being 3.78 g/L and 5.54 g/L, respectively. Further increase of time had no marked effect on leaching properties.

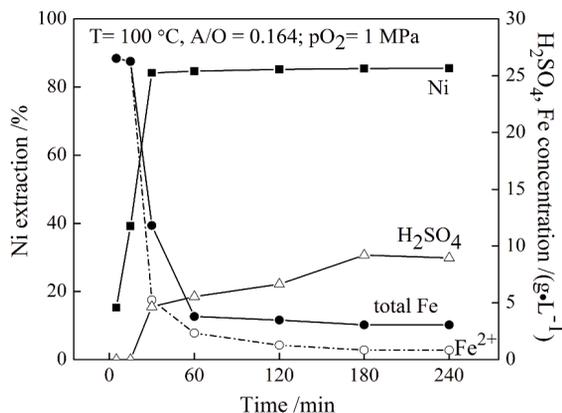


Figure 3. Extraction of Ni and concentrations of free acid and Fe in solution during leaching of calcine with reduction degree of 93.4% and L/S of 4:1

The effect of A/O ratio on the leaching results of calcine with the reduction degree of 93.4 % under 1.0 MPa O₂ at 100°C, L/S 10:1 for one hour is presented in Fig. 4. The increase of A/O ratio from 0.123 to 0.164 enhanced the extraction of Ni from 76.2% to 88.2%. In the same time, the total iron and sulfuric acid were 2.3 g/L and 2.2 g/L, respectively. Further increase of A/O ratio had little effect on extraction of Ni, except for raising of the residual concentrations of iron and sulfuric acid.

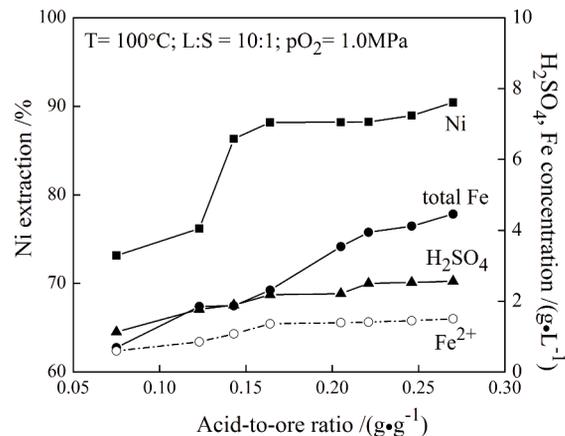


Figure 4. Effect of acid/ore ratio on extraction of Ni and concentrations of free acid and Fe in solution for a 60 min leach of calcine with a reduction degree of 93.4%

The extraction of impurities as a function of the A/O ratio is given in Fig. 5. Calcium and magnesium were more readily leached out than aluminum. Over 90% of Ca and Mg were extracted at A/O ratio of 0.205, while the extraction of Al did not exceed 31.7% even at A/O ratio of 0.270. The low extraction rate of aluminum may result from the refractory nature of aluminum-containing minerals after pretreatment by roasting.

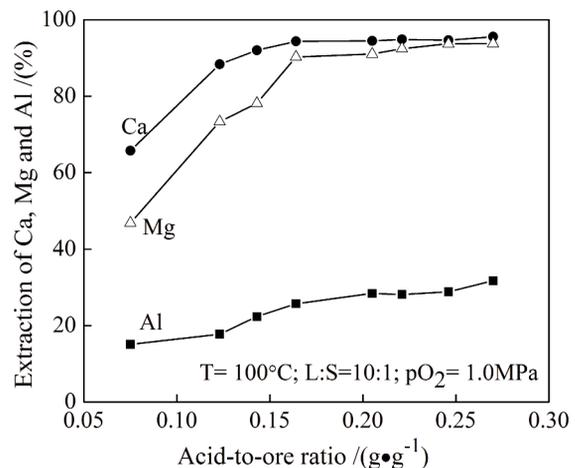


Figure 5. Effect of acid/ore ratio on extraction of Ca, Mg and Al for a 60 min leach of calcine with the reduction degree of 93.45%



3.3 Transformation of Iron to Hematite at High Oxygen Pressure

3.3.1 Critical temperature of goethite dehydration to hematite

Iron in solution can be precipitated either as goethite or as hematite, mainly depending on the precipitating temperature. The high temperature favors the formation of hematite as the most stable form of iron. To examine the dehydration temperature of goethite to hematite, leaching tests at different temperatures were conducted using the calcine with the reduction degree of 93.4 % under 1.0 MPa oxygen for 60 min with A/O of 0.164, L/S of 10. The XRD patterns of the leaching residues formed at different temperature are given in Fig. 6, which indicate that the leaching residue produced at 110°C is mainly goethite with minor unleached magnetite. Increase of temperature to 120°C transforms almost all of goethite into hematite.

The SEM morphology images of the leaching residues formed at different temperature are assembled in Fig. 7. The iron precipitation formed at

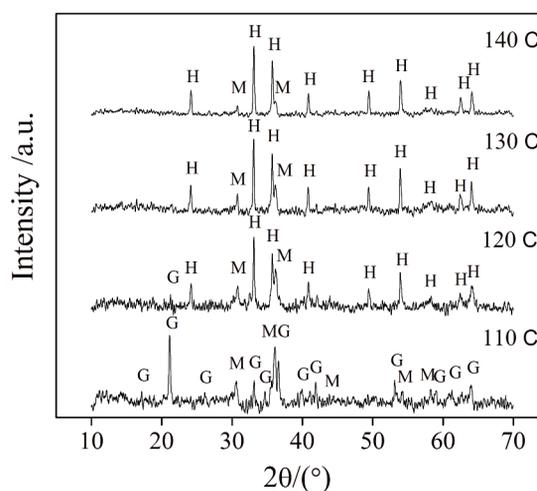


Figure 6. XRD patterns of leaching residues formed at different temperatures after leaching of calcine with a reduction degree of 93.4% for 60 minutes at $pO_2 = 1.0\text{MPa}$, $A/O = 0.164$ and $L/S = 10$. (G: goethite, M: magnetite, H: hematite)

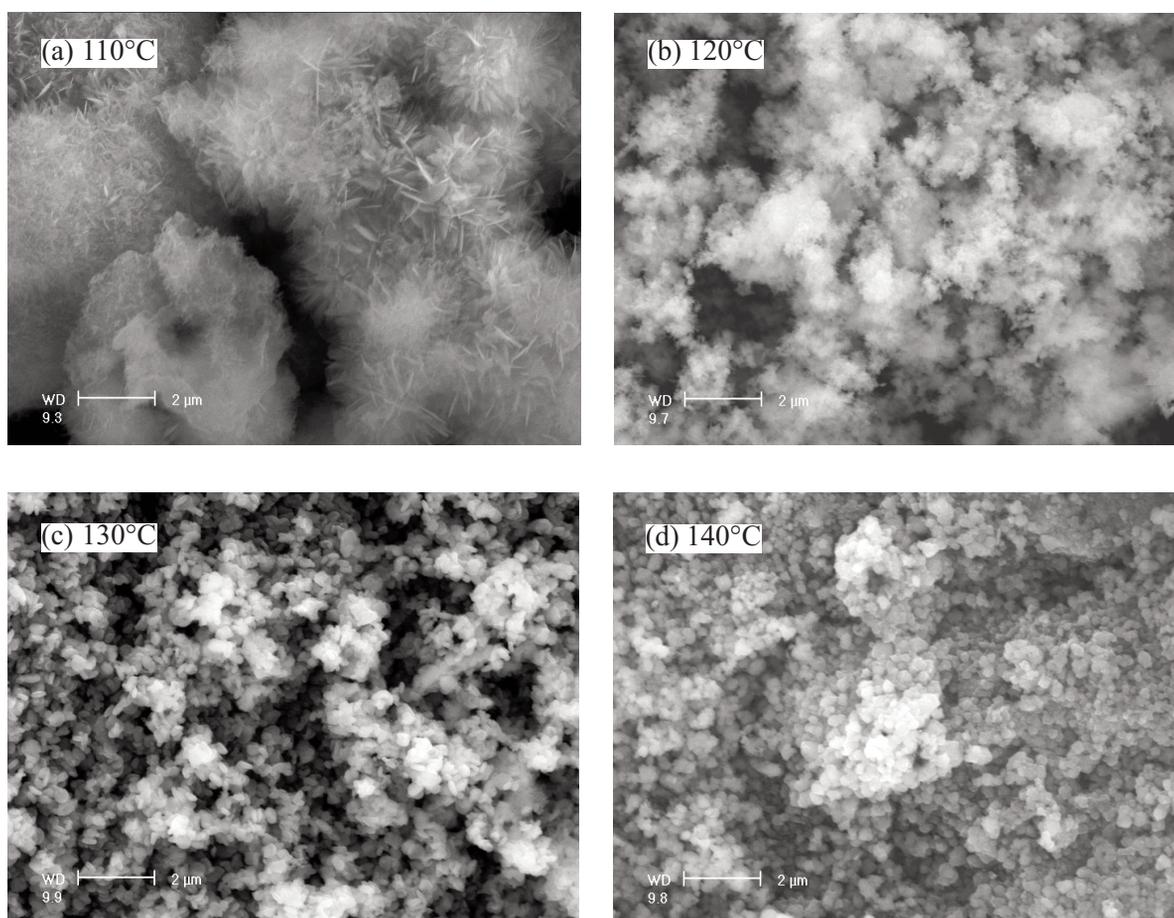


Figure 7. SEM morphology images of leaching residues formed at specific temperature after leaching for 60 minutes under $pO_2 = 1.0\text{MPa}$, $A/O = 0.164$ and $L/S = 10$

110 °C presented as the needle-like crystals as seen in Fig. 7(a) is the morphology typical characteristic to goethite. At 120 °C, a transition of needle-like to the spherical shape could be found, Fig. 7(b). Further increase of temperature to 130°C and 140°C caused the completion to spherical hematite precipitation, as seen in the respective Fig.7(c) and Fig. 7(d).

The results in Fig. 6 and Fig. 7 are a little different from the early equilibrium investigation of $\text{Fe}_2\text{O}_3\text{-SO}_3\text{-H}_2\text{O}$ system by Posnjak and Merwin [25], who in very dilute solutions found turgite ($\text{Fe}_2\text{O}_3 \cdot n\text{H}_2\text{O}$) as the phase being formed between 130°C and 150°C. Our assumption is that the dependence of hematite formation on temperature is influenced by the availability of hematite nucleation centers in iron-containing solutions. This is based on the research of Riveros and Dutrizac [26], who found that the minimum temperature to precipitate hematite from ferric chloride solutions in the presence of hematite seed is about 125°C. Their further research [27] also showed that hematite can be formed from ferric chloride media in preference to ferric oxyhydroxides at temperatures as low as 60°C if the precipitation conditions are controlled by seeding. Another example came from Lu and Dreisinger [28], who demonstrated that during the pressure oxidation of ferrous iron at 155°C, or higher, hematite was the only precipitate. In our study, the precipitation of hematite from the pressure oxidation of ferrous iron at temperature as low as 120°C maybe benefited by the calcine acting as the heterogeneous nucleation centers.

In addition to the increased iron oxides formation kinetics, the temperature increase also widens the predominance stability regions for oxides in question, which can be readily seen on the Eh-pH diagrams (not presented here) constructed under the relevant leaching conditions. The oxides predominance region widening occurs on expense of nickel stability region. For example, at room temperature, nickel can be soluble up to pH 6, while at 150°C nickel is solubilized only below about pH 2.1.

3.3.2 Effects of calcine reduction degree and acid/ore ratio

Three kinds of calcine with the reduction degree of 73.1 %, 80.2 %, and 93.4 %, respectively, were used to test the leaching as a function of acid/ore ratio at 180 °C. The leaching results are given in Fig. 8. For each calcine, the extraction rate of nickel and the residual concentration of iron increased with the increase of A/O ratio. Also, for a given A/O ratio, the calcine with higher reduction degree responded with higher nickel extraction rate. Thus, for the calcine with reduction degree of 93.4 %, more than 95 % of nickel can be extracted by increasing the A/O ratio to

0.287. In the meantime, the residual ferrous and total iron concentrations were relatively low, 0.42g/L and 0.31 g/L, respectively.

The leaching residues of Fig. 8(c) were analyzed for the content of aluminum and sulfur, and the results expressed as the function of A/O ratio are given in Fig. 9.

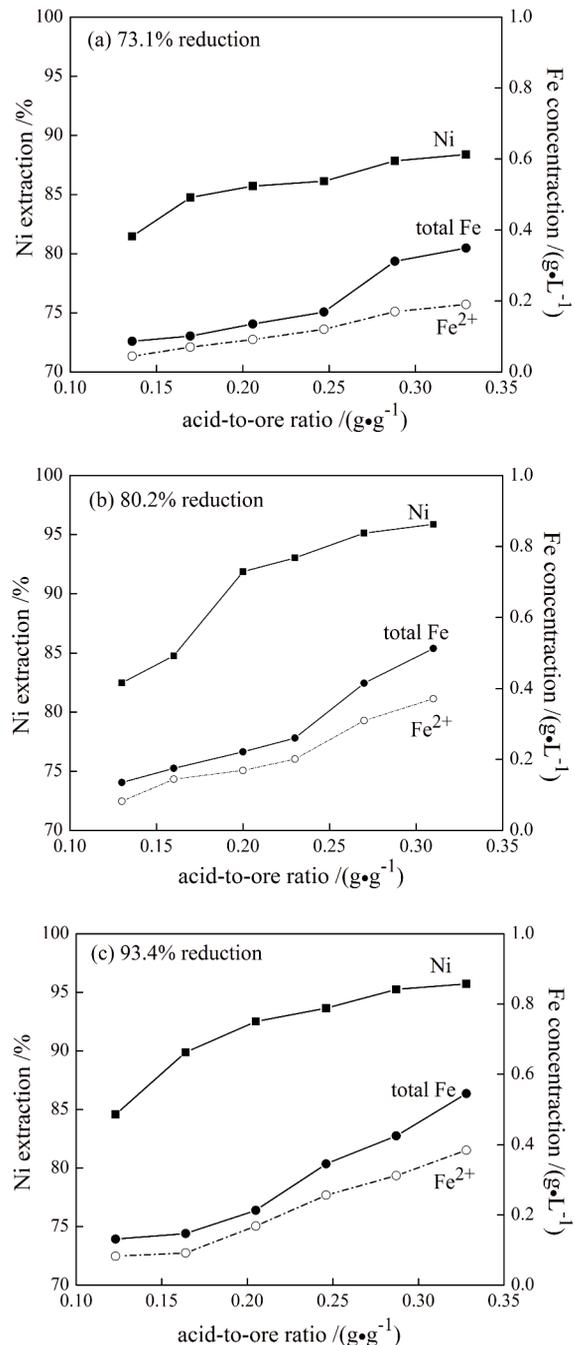


Figure 8. Effect of A/O on leaching of calcines with three different reduction degrees. Leaching conditions: Duration= 60min, $p\text{O}_2=1.0\text{MPa}$, $T= 180^\circ\text{C}$, $L/S= 10$



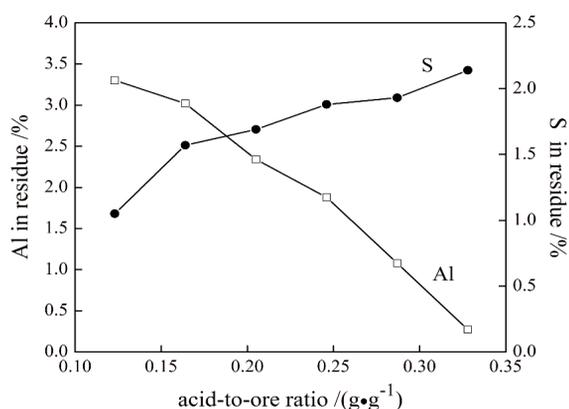
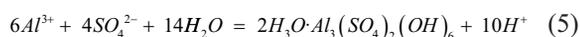


Figure 9. Effect of A/O ratio on the content of Al and S in residues corresponding to the leaching conditions given in Fig. 8c

The sulfur content in the residue increased with the A/O ratio, while the aluminum content changed in a reverse direction. From the opposing change of directions of aluminum and sulfur content in the residue, an important conclusion can be made that aluminum in the residue did not exist in the alunite form. If the aluminum in the calcine dissolved and then re-precipitated as alunite [29], as shown in Eq. 4,



the ratio of S% to Al% in the residue would have been 0.79. Also, the concentration change trend for both, S% and Al%, would have been the same. Clearly, per Fig. 9, neither of these was the case. The possible explanation for the increase of sulfur content in the residue with the increase of A/O ratio is that some sulfate was trapped within the hematite crystal in the fast precipitation process. Or, more likely, some basic sulfate iron precipitated when the sulfate concentration in the solution was high.

By further comparing the behavior of aluminum in Fig. 6 and Fig. 9, it can be inferred that the leaching temperature also had a remarkable influence on the leaching behavior of aluminum. This would be the consequence of conversion of AlOOH like mineral to the more refractory Al_2O_3 during the reductive roasting. The calcined aluminum oxide, being highly acid resistant, can be dissolved only under more concentrated acid conditions at high temperature, such as 180°C in Fig. 9, where the aluminum concentration decrease in the leach residue with A/O increase is clearly observed.

The XRD patterns of two typical residues produced during leaching at 180°C with A/O of 0.246 and 0.328 are shown in Fig. 10. No phase of the alunite related minerals could be found. The formation of alunite would have been of great concern during the operation of HPAL for leaching of nickel from reduced laterite calcines because it would have resulted in an

unavoidable scaling of interior autoclave walls.

3.3.3 Effect of leaching temperature and oxygen pressure

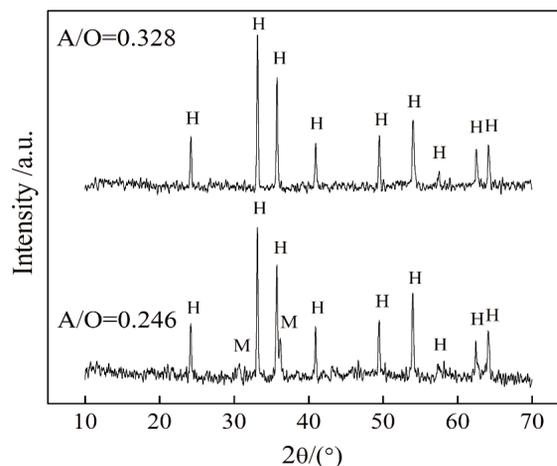


Figure 10. XRD patterns of residues produced by leaching of calcine with the reduction degree of 93.4%. Conditions: $pO_2=1.0\text{ MPa}$, $T=180^\circ\text{C}$, time= 60 min. (H: hematite, M: magnetite)

Fig. 11 and Fig. 12 illustrate the effect of leaching temperature and oxygen pressure on the recovery of nickel, and the final concentration of iron in solution. The effect of these two parameters was examined on calcine with the reduction degree of 93.45% by leaching for 60 min with A/O= 0.205 and L/S= 10. Temperature had a more pronounced effect than pressure on the increase of nickel recovery and the decrease of iron in leach solution.

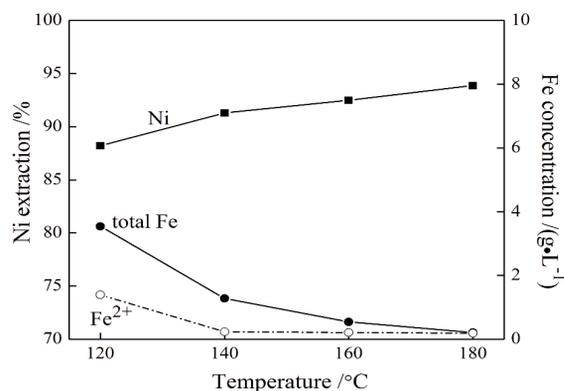


Figure 11. Effect of temperature on extraction of nickel and residual iron in the leach solution. Calcine with the reduction degree of 93.45% was leached for 60 minutes at $pO_2= 1.0\text{ MPa}$, A/O= 0.205, and L/S= 10

5. Conclusions

This work investigated the benefits of using reduced limonitic laterites as the feed to high pressure acid leaching (HPAL). The key parameters examined



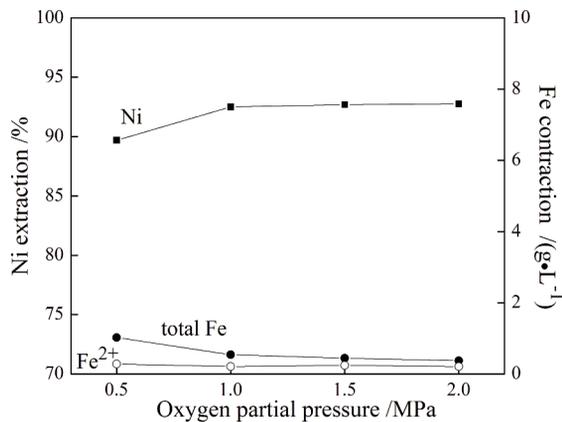


Figure 12. Effect of pressure on extraction of nickel and residual iron in the leach solution. Calcine with the reduction degree of 93.45% was leached for 60 minutes at $T=160^{\circ}\text{C}$, $A/O=0.205$, and $L/S=10$

were the degree of laterite ore reduction, the effect of temperature, oxygen pressure, and acid to ore ratio. It was found that as high as 95.2% Ni can be leached when the ore was reduced 93.4%, $A/O = 0.287$, 1.0MPa oxygen and the temperature was “only” 180°C . Iron precipitation as hematite reduced the residual iron concentration to less than 1.0 g/L. At even lower temperature, $T=100^{\circ}\text{C}$, about 88.2% of nickel could be extracted within one hour of leaching under 1.0 MPa of oxygen pressure and an acid to ore ratio of only 0.164. Under these conditions, iron precipitated as goethite, leaving somewhat higher concentration of iron in solution, less than 5 g/L.

Impurities such as calcium and magnesium nearly completely dissolved into the solution. On the other hand, solubility of aluminum was sensitive to acid/ore ratio and the leaching temperature. Aluminum in the leaching residue existed in other than the alunite form.

Our research indicates that high temperature and pressure requirements in HPAL can be significantly decreased if laterites are reduction pretreated prior to leaching, without having a negative impact on the overall nickel recovery, still maintaining a net acid consumption comparable to that of traditional HPAL. In addition, the resulting pregnant leach solutions with acceptably low iron concentration make the downstream treatment feasible.

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