



# Environmental Friendly Approach: Atmospheric and High-Pressure Acid Leaching Studies, Low-Grade Laterites Nickel Processing

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## Abstract

This paper examines the performance of Atmospheric Leaching (AL) and High-Pressure Acid Leaching (HPAL) on nickel laterite, classified as limonite. The study, conducted on a laboratory scale, involved temperatures of 35-95°C for AL and 220-250°C for HPAL. Nickel and cobalt contents were found to be 0.7% and 0.04%, respectively. AL achieved an 89% yield of Al with a pH of 0.2 and a 14-hour leaching time, while nickel and iron recoveries reached 92% and 87% after 20 hours, with an acid consumption of 1.2 kg H<sub>2</sub>SO<sub>4</sub> per 100 kg laterite (dry) at pH 0.2. Leaching experiments at 220-250°C for 2 hours showed similar nickel recovery rates, indicating no improvement beyond 240°C. Hematite, a stable compound associated with nickel, hindered its release during HPAL due to its resistance to leaching. Nickel yields remained around 90% in both AL and HPAL tests. Iron behavior differed significantly between the two methods, with HPAL dissolving iron initially but transforming it into hematite in situ, leading to lower net acid consumption compared to AL. The leaching mechanism for iron oxides followed empirical power law kinetics of order 1.5 with activation energies of 36.23 and 25.09 kJ/mol for Ni and Fe, respectively.

## 1. Introduction

Nickel (Ni) is an important metal in modern-day metallurgy due to its unique properties (e.g., corrosion resistance, toughness, strength, and magnetism). Low-grade laterite ore deposits constitute one of the two major sources of Ni and pay metal cobalt (Co), the other being sulphide ores. Globally, the main bulk (~60%) of the world's Ni is extracted from sulphidic ores although they constitute only ~30% of land-based Ni reserves. Ni laterite profile can be divided into four zones (saprolite, transition, limonite, and

ferricrete) with the effect of weathering progressively decreasing from the surface to the bedrock. Depending on the level of maturity of weathering and the climatic zones, the lateritic profile varies from region to region and from zone to zone within the same deposit. A typical mineralogical profile formed as the result of the weathering is shown in Figure 1. The ores may also have high clay and moisture contents and small traces of Cu and Zn, all of which add to their variability and complexity [1-3].

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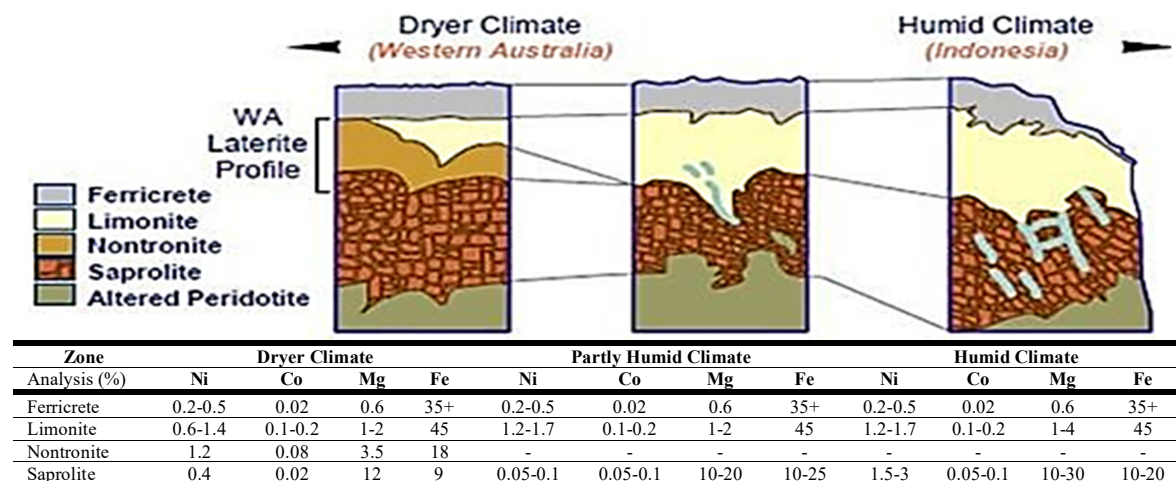


Figure .1. Laterite profiles from different climatic zones reflecting varying degrees of weathering

The Caron process was developed for the processing of limonitic ores or blends comprising limonite and saprolite ores. It is based on the selective reduction of Ni and Co, followed by dissolution in ammonia/ammonium carbonate

solution. A small amount of Fe is reduced forming an alloy with the Ni and Co and the remainder is reduced to magnetite [4, 5]. Roasting takes place at high temperatures (~850 °C) according to the following reaction:



The ore is then quenched in ammonia/ammonium carbonate solution (150-200 °C) which leads to Ni and Co solubilization as ammonia complexes and Fe oxidation and precipitation as Fe (OH). Co and Ni are recovered as Co-Sulphide and Ni-carbonate by steam stripping. Despite its successful application in treating low-grade ores, the Caron process gives low extraction rates (<90% Ni and <80% Co) at very high energy costs due to calcination at high temperatures.

As high-grade ore deposits are being exhausted steadily, there is an increased focus in the minerals industry on the processing of complex, low-grade (<1.5 wt. % cut-off grade) ores [6-8]. Depending on the cut-off grade of Ni and Co in lateritic ores, they may be processed by pyro-metallurgical (>2 wt. %) or hydrometallurgical routes (<2 wt. %) as summarized in [9-11]. Whilst several Ni laterite AL plants are in operation around the world and a plethora of fundamental and applied studies reported on them, there are still major production issues that generally challenge their economic viability [12, 13]. These are relatively longer leach times required (usually 4-10 h), high acid consumptions (>500 kg acid/t of dry ore), and low Ni and Co extraction rates (<80%) [14].

Higher temperatures have been shown to improve Ni and Co extraction kinetics for AL [15-

19]. A study carried on two different Ni laterites, i.e., nontronite and limonite ores, leached for 24 h showed that for a temperature increase from 25 to 95 °C, Ni extraction increased from 55 to 96% and from 30 to 93%, in nontronite and limonite respectively, for 24 h [20]. Studies of saprolitic ore revealed that after 5 min leaching of 10% (v/v) dispersion, at 70 and 95 °C led to a Ni extraction rate of 40 and 84%, respectively [21]. However, other studies have indicated that it may be more economical to leach for a longer time at lower temperatures for some ores rather than applying higher temperatures in shorter times [22].

A review of atmospheric acid leaching by McDonald and Whittington (2008) revealed that the leaching mechanisms and kinetics of such ores may be improved through modification of process variables (e.g., temperature, particle size, salt addition, roasting) [23-24].

HPAL is best suited for the treatment of limonitic ores, although the Al and Mg content should not be too high as they lead to increased acid consumption. It is generally regarded as the main commercial alternative for the Caron Process. During HPAL, the ore is leached in an autoclave with H<sub>2</sub>SO<sub>4</sub> at 240-270 °C to dissolve most of the solid into a solution. The high temperature ensures fast reaction times of 60-90 min and also results in the precipitation of most of the dissolved Fe as

hematite ( $\text{Fe}_2\text{O}_3$ ) or jarosite ( $\text{KFe}_3^{3+}(\text{SO}_4)_2(\text{OH})_6$ ) and Al as alunite ( $\text{KAl}_3(\text{SO}_4)_2(\text{OH})_6$ ). Because of the aggressive leaching conditions, >95% of the Ni and Co and >90% of the Mg and Mn are dissolved into a leach solution. While most of the initially solubilized Fe, Al, Si, and Cr are transferred to the solids, minor amounts still remain in the solution and add to the complexities in the next processing step. The leached slurry is then transferred to flash cooling, neutralization, liquid/solid separation (via Counter Current Decantation (CCD)), solution purification, and Ni and Co recovery [23-26].

Less problem is faced by increasing the scale of the solution and corrosion during AL compared with the more corrosive HPAL process. AL may be applied to low-grade ores such as limonite and saprolite, however, significant challenges are associated with the leaching mechanisms/kinetics and the subsequent processing of the leach liquor [27-30]. The process is often faced with very high acid consumption rates (>500 kg/t of ore) due to the significant leaching of associated gangue species. The leachate may contain significant concentrations of dissolved species (e.g., Al, Mg, and Fe) which interfere with the extraction of value metals. This requires the concentration of these metal ions to be reduced or using methods that will be more selective to Ni and Co extraction [31].

Several studies on the AL behavior of different Ni laterite ores have shown varying mineralogy and chemistry-dependent behavior [32-33]. A review of atmospheric acid leaching by McDonald and Whittington (2008) revealed that the leaching mechanisms and kinetics of such ores may be improved through modification of process variables (e.g., temperature, particle size, sulphurization, salt addition, roasting). AL studies of serpentinized laterite ore showed that recoveries of up to 74% Ni and 51% Co could be achieved after 2 h at 80 °C [34, 35]. Reports on Ni laterite leaching showed both temperature and time to play a vital role in Ni and Co extraction rates [36, 37]. Increasing leaching time in general leads to an increasing Ni/Co extraction rate but beyond a critical time for a given ore, the rate falls [38, 39]. Higher temperatures have also been shown to dramatically improve the extraction of Ni/Co species at a given reaction time [40, 41]. Due to Ni laterite ores' characteristically variable mineralogy and complexity, ore-body-specific investigation to facilitate their hydrometallurgical process is

commonly required. Several studies have been reported on different strategies to improve the leaching mechanisms and kinetics of lateritic ores [42, 43]. The effect of parameters such as pH, temperature, particle size, and pulp solid loading has been examined. Higher Ni/Co extraction rates have been reported for higher temperatures [44].

This paper described leaching tests that were carried out to confirm that nickel laterite is amenable to nickel leaching. AL and HPAL tests were performed by laterite. This work specifically focused on the process conditions and acid leaching behavior of lateritic minerals. Also, this study investigates the mechanisms and kinetics of isothermal, batch leaching of Ni laterite ore. The effect of time (up to 26 h), and temperature (35 - 98 °C) on the  $\text{H}_2\text{SO}_4$  acid leaching behavior of 37 wt. % solid dispersions at pH0.2 was investigated.

## 2. Materials and Methodology

### 2.1. Pre-Treatment of Ore Sample

#### 2.1.1. Crushing and grinding of samples

Laterite ore sample (80 kg) (Bavanat ore, Fars, Iran) was milled down to a particle size of 1 mm. Afterward, Laterite samples for leaching tests were ground in a ball mill to 80% -56  $\mu\text{m}$  fineness. Grinding time was 20 min and one grinding batch included 3.5 kg laterite, 22 kg balls, and 3.5 kg of water for 50% slurry density.

#### 2.2. Atmospheric leaching tests

Atmospheric leaching tests were carried out in a mechanically agitated 5-liter titanium tank reactor. The test procedures were different in these leaching tests. The first leaching test focused on studying suitable pH for leaching and it was carried out as a descending pH test. The second leaching test was carried out at a constant pH. Concentrated sulfuric acid was used in both leaching tests.

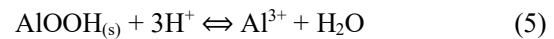
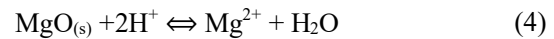
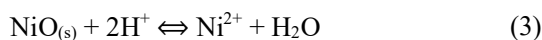
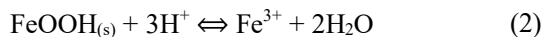
The reactor was equipped with baffles, an A45-type mixer with 4 blades, online pH measurement, temperature measurement and a controller connected to the heating plate and reflux condenser. Acidic feed to the leaching reactor was controlled by pH measurements. Leaching temperatures used were 35-98 °C, the mixing speed was 900 rpm and the pulp density was in the range of 250-370 g/l. Table 1 summarized the main process conditions in the tests. The test work setup could be seen in Figure 2.

**Table 1. Main parameters of atmospheric leaching tests.**

Parameter	Test 1	Tests 2,3,4,5
Temperature, °C	95	35,55,75,98
Leaching time, h	26	20
Laterite mass, g	1000	1110
Initial slurry volume, l	4	3
Initial solid content, g/l	250	370
Mixing speed, rpm	900	900
pH	1.5-0.2	0.2
H <sub>2</sub> SO <sub>4</sub> feed, kg/kg laterite	1.44	1.16

**Figure 2. Atmospheric leaching test setup.**

The main chemical reactions considered to take place in the leaching stage are as follows (Reactions 2-5):



When nickel and magnesium oxides were leached, they stayed in the solution as sulfates. Iron and aluminum dissolved accordingly to ferric sulfate and aluminum sulfate.

### 3. Experimental Study

#### 3.1. Atmospheric Leaching (Test 1)

In the first atmospheric leaching test, the effect of different pH levels on nickel recovery was studied and acid consumption was determined by means of descending pH test. The pH was decreased step-by-step from 1.5 to 0.2 by concentrated sulfuric acid. The target steps were pH 1.5, 1.0, 0.5 (actual pH was 0.25 – 0.34) and 0.2. Leaching time was 6 h for each pH step, except at pH 0.2, where it was 8 h. The test was started by mixing the laterite with water and placing the slurry in the reactor. After heating was started and the leaching temperature was reached, acid pumping was started for pH adjustment. The acidic feed pump was operated through pH measurement and controller. To avoid excess frothing of the slurry, acid feeding was started slowly.

Sampling was conducted at 2, 4, and 6 h for pH 1.5-0.5 steps, and at 2, 4, and 8 h for pH 0.2. Samples were taken as 30 ml slurry samples and filtrated, to obtain solid and liquid samples. Samples were analyzed with ICP-OES. Table 2 summarized the main chemical analyses of the solid assays of atmospheric leaching test 1.

**Table 2. Solid assays of atmospheric leaching test 1**

Time (h)	pH act	Acid/Lat. (kg/kg)	Mg (%)	Al (%)	Ca (%)	Cr (%)	Mn (%)	Fe (%)	Co (%)	Ni (%)	Cu (%)	Zn (%)	S (%)	SiO <sub>2</sub> (%)
0			3.74	1.44	5.59	2.17	0.19	23.6	.036	0.70	0.012	0.024	<0.05	27.4
2	1.50	0.43	1.24	1.71	4.49	2.24	0.09	24.6	0.030	0.61	0.01	0.030	3.5	29.5
4	1.45	0.45	1.18	1.63	4.90	2.10	0.07	24.0	0.030	0.55	0.01	0.030	3.6	29.4
6	1.49	0.46	1.18	1.64	4.98	2.17	0.07	23.8	0.030	0.52	0.01	0.030	3.6	29.2
10	1.00	0.74	1.28	1.72	4.76	2.83	0.06	21.0	0.020	0.39	0.01	0.030	3.7	34.0
12	1.01	0.79	1.22	1.69	4.92	2.58	0.06	20.0	0.020	0.35	0.01	0.030	3.9	36.4
14	1.12	0.82	1.35	1.71	5.55	3.06	0.06	18.2	0.010	0.30	<0.01	0.030	4.3	38.0
16	0.25	1.25	1.32	1.45	6.96	2.60	0.04	10.7	0.010	0.18	<0.01	0.050	5.37	35.1
18	0.34	1.25	1.48	1.58	7.48	3.13	0.03	7.6	0.010	0.14	<0.01	0.050	5.95	40.1
20	0.31	1.25	1.43	1.46	7.47	2.90	0.02	5.7	0.010	0.11	<0.01	0.050	6.32	45.6
22	0.16	1.44	1.47	1.57	7.56	2.60	0.03	3.9	0.010	0.08	<0.01	0.050	6.16	46.1
24	0.22	1.44	1.41	1.49	7.86	3.20	0.02	3.3	0.010	0.07	<0.01	0.050	6.37	39.5
28	0.22	1.44	1.32	1.34	7.29	3.00	0.02	2.6	0.010	0.06	<0.01	0.050	6.26	47.1
Leach residue	-	-	1.27	1.41	9.09	4.40	0.02	2.9	0.010	0.05	<0.01	0.040	7.6	48.8

Nickel concentrations in solids in regards to pH versus leaching time were shown in Figure3. Metal yields during the leaching at different pHs and acid dosages were illustrated in Figure4. The metal

yields were calculated against the SiO<sub>2</sub> content of the laterite. SiO<sub>2</sub> did not leach from the laterite and could therefore be considered as an inert substance.

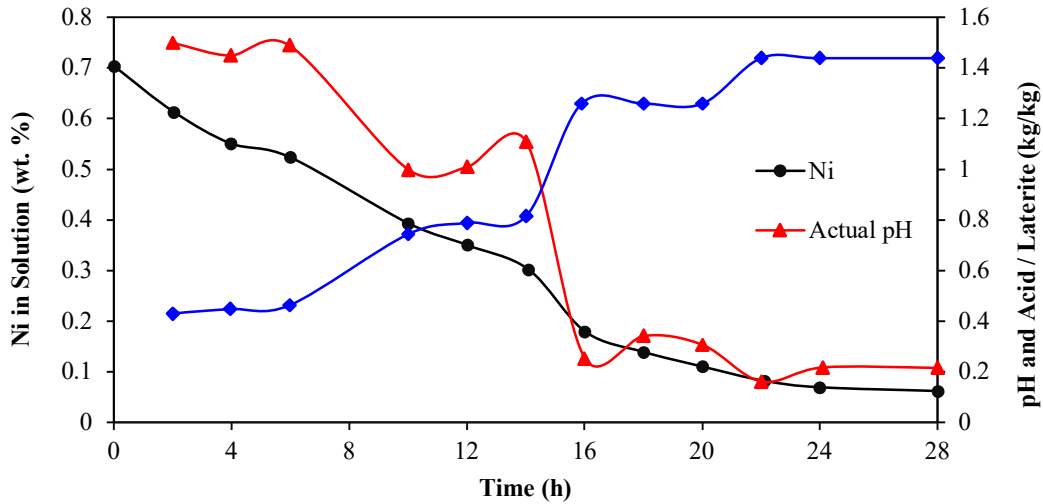


Figure 3. Nickel concentrations in solids, solution pH and acid dosage during the leaching.

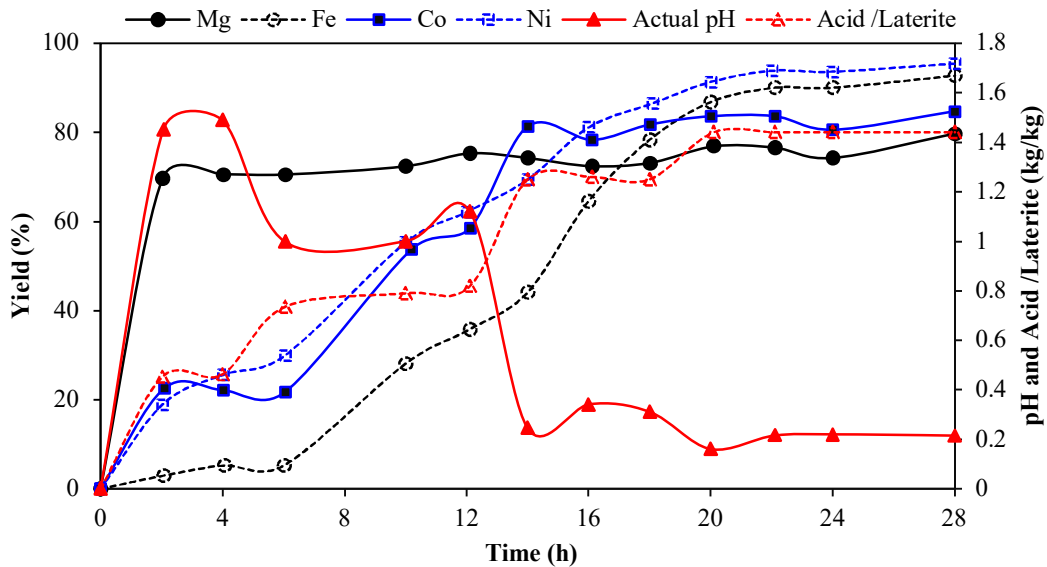


Figure 4. Metal yields (solid based) at different pHs and acid dosages during the leaching.

Nickel did not dissolve significantly at pH 1.5, at pH 1 nickel dissolution remained still incomplete and rather slow. It could be observed that nickel was leached efficiently when the pH was approximately 0.2. The nickel yield at the end of the test was 95%. The iron yield was also high, it was 94 % at pH 0.2. The leaching yield for cobalt was 84%. Magnesium was soluble already at pH 1.5. Sulfuric acid consumption at the end of the test was 1.44 kg acid/1 kg laterite.

### 3.2. Atmospheric Leaching (Tests 2, 3, 4, 5)

The second atmospheric leaching test was carried out at a constant pH of 0.2 to study leaching kinetics. The pH of the system was selected based on test 1 to ensure leaching efficiency for the nickel. The test was started by pulping the laterite to water and placing the slurry in the reactor. Heating was commenced and after the leaching temperature was reached, acid pumping was

started to reach the target pH of 0.2. The acid feed pump was operated through pH measurement and controller. To avoid excess frothing of the slurry, acid feeding was started slowly. Sampling was conducted at 2 h, 6 h, 10 h, 14 h, and 20 h. Samples were taken as 30 ml slurry samples and filtrated, to

obtain solid and liquid samples. Solution samples were analyzed with ICP-OES and solid samples were analyzed with ICP-OES after total dissolution or fusion melt. Metal concentrations in the solution during the leaching were illustrated in Figure 5.

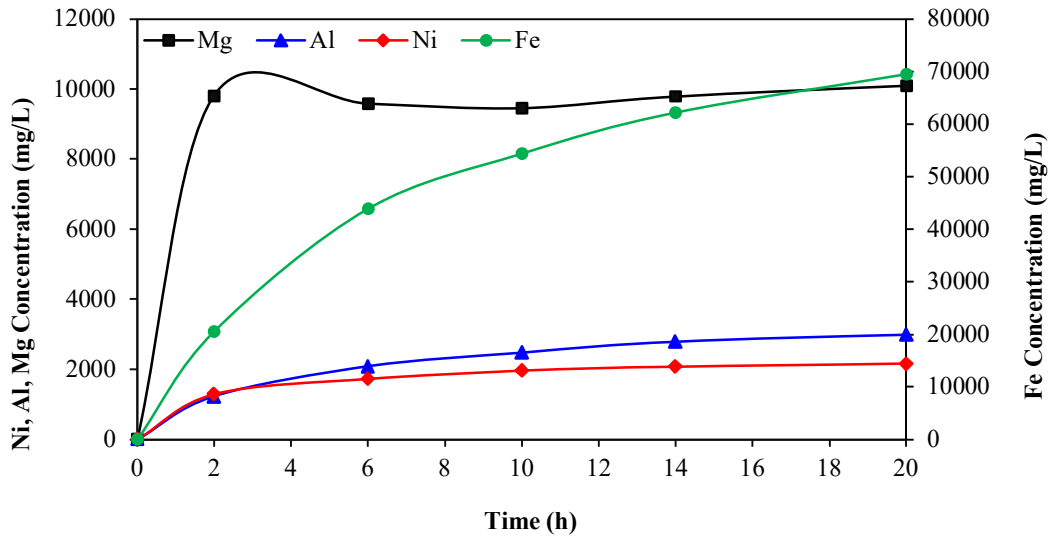


Figure 5. Metal concentrations during the leaching, pH of the system was 0.2

Magnesium leached rapidly and it reached maximum concentration already at the beginning of the test. Magnesium concentration of the solution was approximately 10 g/l. Iron

concentration was very high at the end of the test, it was 70 g/l. Nickel concentration reached a concentration of 2 g/l. Metal contents of the solids during the leaching were presented in Figure 6.

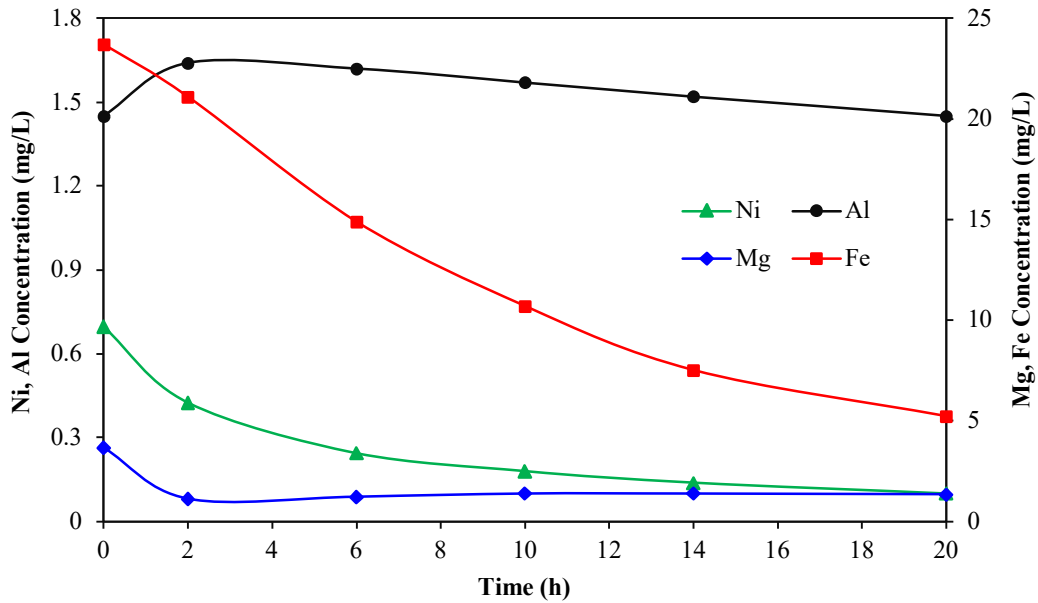


Figure 6. Metal concentration of solids during the leaching, pH of the system was 0.2.

Nickel content in the solids decreased during the test from 0.7% to 0.10% by weight. Fe concentration changed from 23.6% to 5.2% by weight. Solid-based yields for Ni, Al, Fe, Mg, and Co were presented in Figure 7. The metal yields

were solid-based yields and they were calculated against the SiO<sub>2</sub> content of the laterite. SiO<sub>2</sub> did not leach from the laterite and could therefore be considered as an inert substance.

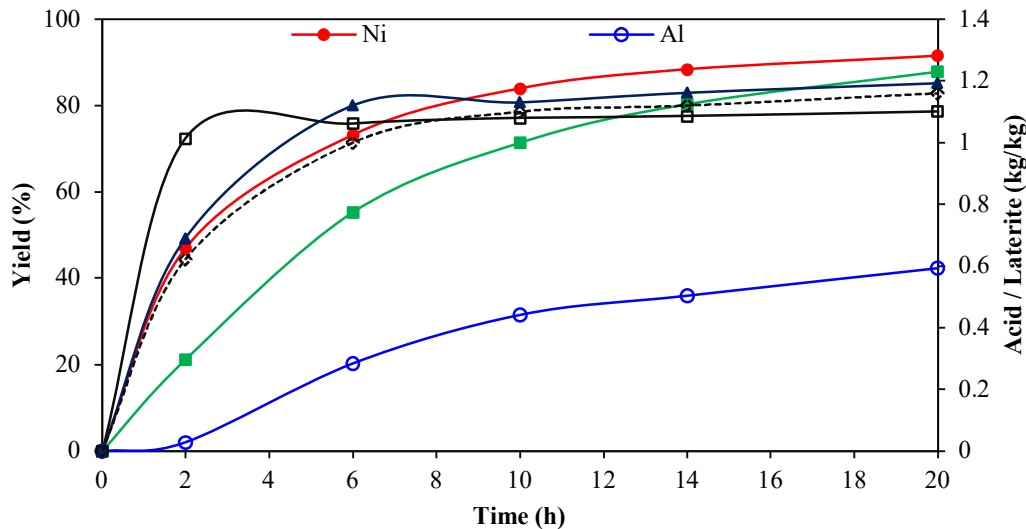


Figure 7. Solid based yields and acid consumption in the atmospheric leaching test, pH of the system was 0.2.

Magnesium leaching was rapid as magnesium leached within 2 h. Iron and nickel leaching was slower. Iron required approximately 20 h leaching time for 87% yield. A leaching time of 14 h was needed for nickel to achieve 89% leaching yield. After 20 h leaching time, nickel yield reached 92% yield. Aluminum was not leached effectively resulting in a leaching yield of 42%. The leaching yield for cobalt was 86%. The acid consumption at the end of the test was 1.16 kg acid/1 kg laterite.

The leaching kinetics of the major elements (Ni, Fe) from the Fe-oxide minerals were analyzed using the shrinking core (SC) and shrinking particle (SP) models.

This study investigates the mechanisms and kinetics of isothermal, batch leaching of Ni laterite ore. The effect of time (up to 26 h), and temperature (35 - 98 °C) on the H<sub>2</sub>SO<sub>4</sub> acid leaching behavior of 37 wt. % solid dispersions at pH 0.2 was investigated.

The iron oxides' leaching mechanism, on the other hand, was chemical and diffusion-reaction controlled, following an empirical power law kinetics of order 1.5 with E<sub>a</sub> of 36.23 and 25.09 kJ/mol, respectively, for Ni and Fe.

### 3.3. High-pressure acid leaching tests (HPAL)

The leaching performance of laterite was also evaluated in two High-Pressure Acid Leaching (HPAL) tests. The main parameters of HPAL tests were presented in Table 3. HPAL tests were carried out at 220 °C and 250 °C temperatures in a 3.8 L titanium autoclave. The autoclave was equipped with an agitator, temperature measurement and control, heating system, cooling coils, and pressure sensor. The autoclave system was connected to the data collection system to log operating parameters such as temperature and pressure. The mixer type used in the first autoclave test was the conventional titanium A45-type lower- and upper impeller with 4 blades. In the second autoclave test, an upper impeller was likewise A45-type, while a lower impeller a proprietary titanium OKTOP mixer of GLS-type was used.

Table 3. Main parameters of HPAL leaching tests.

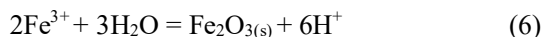
Parameter	Tests
Temperature, °C	220,230,240,250
Leaching time, h	2
Slurry volume, l	2.9
Laterite mass, g	1120
Solid content, g/l	386
H <sub>2</sub> SO <sub>4</sub> feed, kg/kg laterite	0.58



Figure 8. HPAL tests setup.

The slurry was charged into autoclave reactor and heating towards test temperature was started. After the test temperature was achieved, it was set as the start of the test. The duration of the pressure leaching test was 2 h, and sampling was carried out at time points 0 h, 0.5 h, 1 h, and 2 h. In test 1, the first slurry sample was taken at a temperature of 117 °C. In test 2, the first slurry sample was withdrawn at 0 h when 250 °C was reached. Samples were taken as 30-50 ml slurry samples to obtain solid and liquid samples after filtration. Redox and pH of each sample were measured. Both solution and solid samples were analyzed with ICP-OES for Ni, Cu, Co, Zn, Mn, Fe, Cr, Al, Ca, Mg, Si, and S. Final leach residue was also analyzed with XRD.

The main chemical reactions taking place in the leaching were Equations 1-4. In addition, in HPAL conditions ferric iron precipitated to hematite and simultaneously released acid according to Reaction 6:



### 3.4. Atmospheric leaching kinetic

The following equations (1-3) are used to describe the kinetics of atmospheric leaching with organic and mineral acids by shrinking core models.

$$X=kt \quad (1)$$

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

$$1-(1-0.45x)^{1/3}=kt \quad (3)$$

In the above formulas,  $t$  is the reaction time (minutes or hours), and  $k$  is the apparent rate constant.

Shrinking core models are used to calculate the kinetics of atmospheric leaching with organic and mineral acids.

### 3.5. Kinetic investigation with hydrochloric acid

The shrinking core model was used to calculate the kinetics of the atmospheric leaching process with hydrochloric acid. The nickel leaching process is controlled by product release and chemical reactions. On the other hand, iron extraction is influenced by the control step of the chemical reaction. Overall, the chemical reaction mechanism is the main controlling step in the extraction of both elements.

### 3.6. X-ray analyses (XRD)

X-ray analyses (XRD) were used to recognize the type of metal minerals present in the sample (Figure 9).



# XRD Phase Analysis

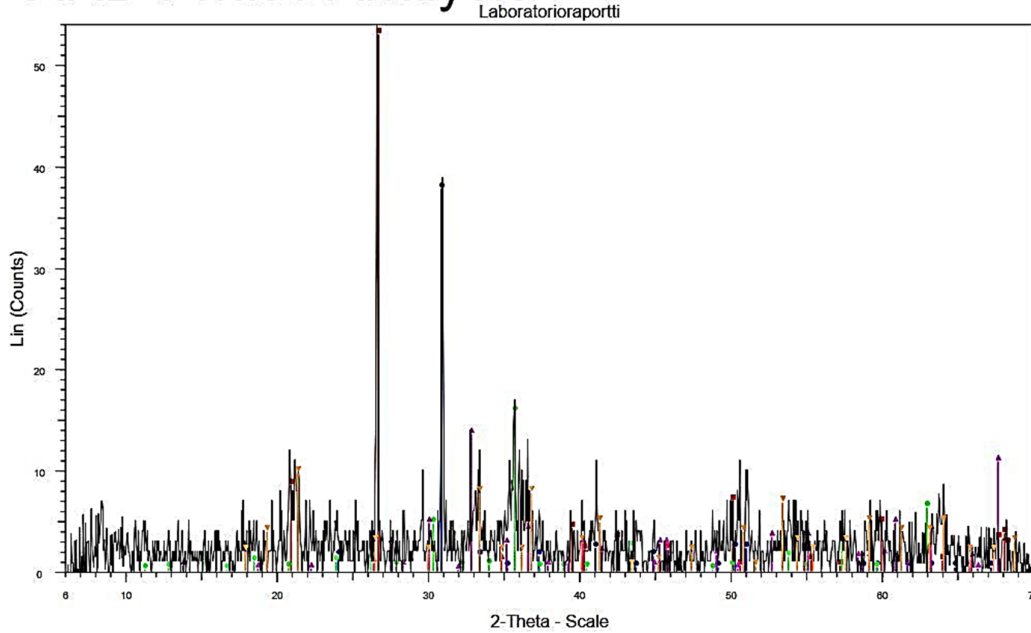


Figure 9. XRD analyses graph.

### 3.7. Results and Discussion

Metal concentrations in solution and solids were presented for the HPAL test at 240 °C in Table 4 and at 250 °C in Table 5.

Nickel was efficiently leached from the laterite. Its concentration in the solid at the end of the tests was only 0.08% by weight. Magnesium and aluminum were also soluble in HPAL conditions. Iron content in the solids remained high during the

leaching. This was expected because iron went through consecutive leaching and precipitation reactions during the process. Iron first was dissolved but then also re-precipitated as hematite in HPAL conditions. A comparison of the yields for the selected metals was shown in Figure 10. The metal yields were solid-based yields and they were calculated against the SiO<sub>2</sub> content of the laterite. SiO<sub>2</sub> did not leach from the laterite and could therefore be considered an inert substance.

Table 4. Metal concentrations in solution and in solid in HPAL test at 240 °C.

T (h)	T (°C)	Ni (mg/g)	Ni (w%)	Fe (mg/g)	Fe (w%)	Al (mg/g)	Al (w%)	Co (mg/g)	Co (w%)	Mg (mg/g)	Mg (w%)
Feed	20	0	0.70	0	23.6	0	1.44	0	0.036	0	3.74
Heating	117	887	0.54	9240	23.5	939	1.59	47	0.030	12600	1.12
0.5	240	2520	0.10	6320	26.8	3810	0.84	113	0.010	13600	0.84
1	240	2600	0.09	5510	26.5	3970	0.90	117	0.006	13900	0.84
2	240	3120	0.08	6310	26.6	4740	0.87	134	0.005	16500	0.43

Table 5. Metal concentrations in solution and in solid in HPAL test at 250 °C.

T (h)	T (°C)	Ni (mg/g)	Ni (w%)	Fe (mg/g)	Fe (w%)	Al (mg/g)	Al (w%)	Co (mg/g)	Co (w%)	Mg (mg/g)	Mg (w%)
Feed	20	0	0.70	0	23.6	0	1.44	0	0.036	0	3.74
0	250	2140	0.12	4700	26.5	2820	1.20	111	0.010	11200	0.79
0.5	250	2430	0.09	3740	27.3	3190	1.15	118	<0.01	12900	0.53
1	250	2130	0.09	3220	26.2	2860	1.02	99	<0.01	11100	0.48
2	250	2060	0.08	2950	26.9	2920	1.09	92	<0.01	11000	0.45

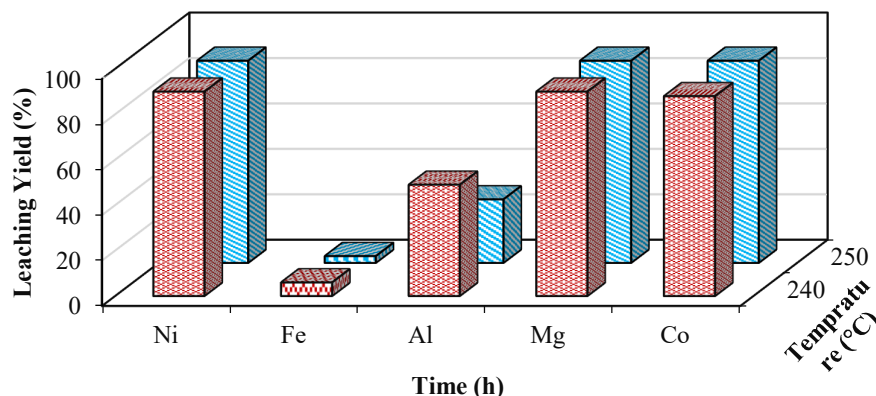


Figure 10. Leaching yield comparison of HPAL tests. Yields are calculated based on solid analysis except for iron which was calculated based on the solution analysis (Co yield at 250 °C was higher than 89%).

### 3.8. Mineralogy of Leach Residues

In order to identify the main phases of the leach residue and distribution of un-dissolved nickel, leach residue samples from atmospheric leaching and HPAL tests were examined using SEM analyses.

### 3.9. Atmospheric Leaching Test Residue

The main mineral phases found within the atmospheric leaching test residue were quartz and chromite. Goethite and hematite were commonly seen but in much less quantities than quartz and chromite. Goethite was found somewhat more readily than hematite. Other minerals, such as Fayalite and anhydrite were observable in trace

amounts. In this sample, nickel was found to remain mainly within the partially leached goethite and hematite (Figure 11). Nickel was found within hematite and goethite in concentrations ranging from 0.4 to 1.7 wt. % Ni and an average composition of 0.95 wt. % Ni. Grain sizes of hematite and goethite found range from approximately 10  $\mu\text{m}$  in width to over 200 $\mu\text{m}$  in width. The smaller grain sizes showed textures of partial leaching whereas the largest grain sizes showed very little leaching textures. Fayalite was only rarely seen as an accessory mineral locked within quartz and was also found to contain some nickel (0.83 wt. % avg.). No other minerals were found to contain any amounts of Ni.

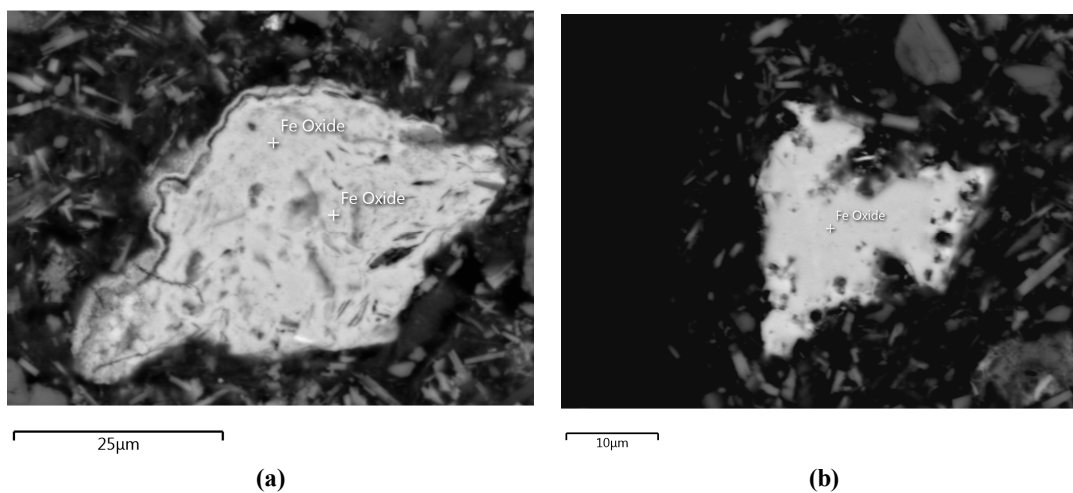


Figure 11. a). Partially leached grain of goethite. b) Partially leached grain of hematite.

Chemical composition of leach residues from atmospheric leaching were presented in Table 6.

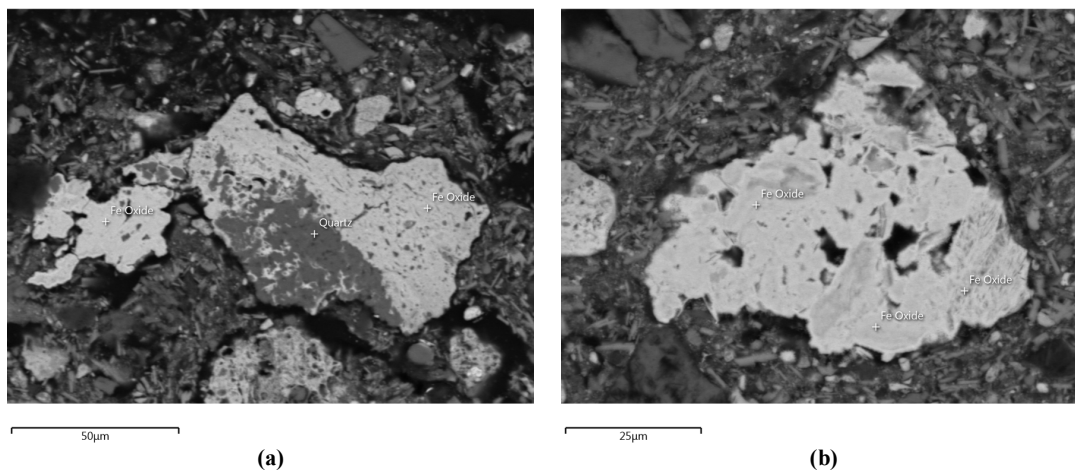
**Table 6. Chemical composition of leach residues from atmospheric leaching tests.**

Test	Mg	Al	Ca	Cr	Mn	Fe	Co	Ni	Cu	Zn	S	SiO <sub>2</sub>
	%	%	%	%	%	%	%	%	%	%	%	%
Test 1	1.27	1.41	9.09	4.4	0.02	2.9	0.01	0.05	<0.01	0.04	7.6	48.8
Test 2	1.39	1.48	8.85	3.7	0.028	5.4	0.01	0.10	0.004	0.048	7.3	33.9

**3.10. HPAL Test Residue**

In contrast to the atmospheric leaching sample, the HPAL sample contained hematite that was easily observable and no observable amounts of goethite. The amount of nickel-bearing hematite appearing within the HPAL sample was more than in the atmospheric leaching test sample overall. The average amount of Ni contained within the analyzed hematite grains was 0.76 wt. %. Hematite grains contained a range of 0.3 to 1.2 wt. % Ni.

Some Fayalite was also found within the HPAL sample, but none was found to contain any Ni. Grain sizes of hematite were found to be similar to the grains found within the atmospheric leaching test. Similarly, the smallest grain sizes show more signs of partial leaching than those of the larger grain sizes (Figure 12). Quartz was found to be abundant, appearing a majority of the time as 20 μm to 50 μm liberated grains. Quartz often appeared locked into hematite or as small inclusions within.



**Figure 12. a) Large particle consisting of quartz and hematite. The grain to the left was partially-leached hematite. b) Partially leached grain of hematite.**

Chemical compositions of leach residues were presented in Table 7.

**Table 7. Chemical composition of leach residues from HPAL leaching tests.**

Test	Mg	Al	Ca	Cr	Mn	Fe	Co	Ni	Cu	Zn	S	SiO <sub>2</sub>
	%	%	%	%	%	%	%	%	%	%	%	%
Test at 240 °C	0.68	0.97	6.12	0.75	0.05	23.8	0.010	0.11	<0.01	0.030	5.91	24.2
Test at 250 °C	0.35	0.88	6.31	1.44	0.04	24.5	0.004	0.07	<0.005	0.019	5.88	29.9

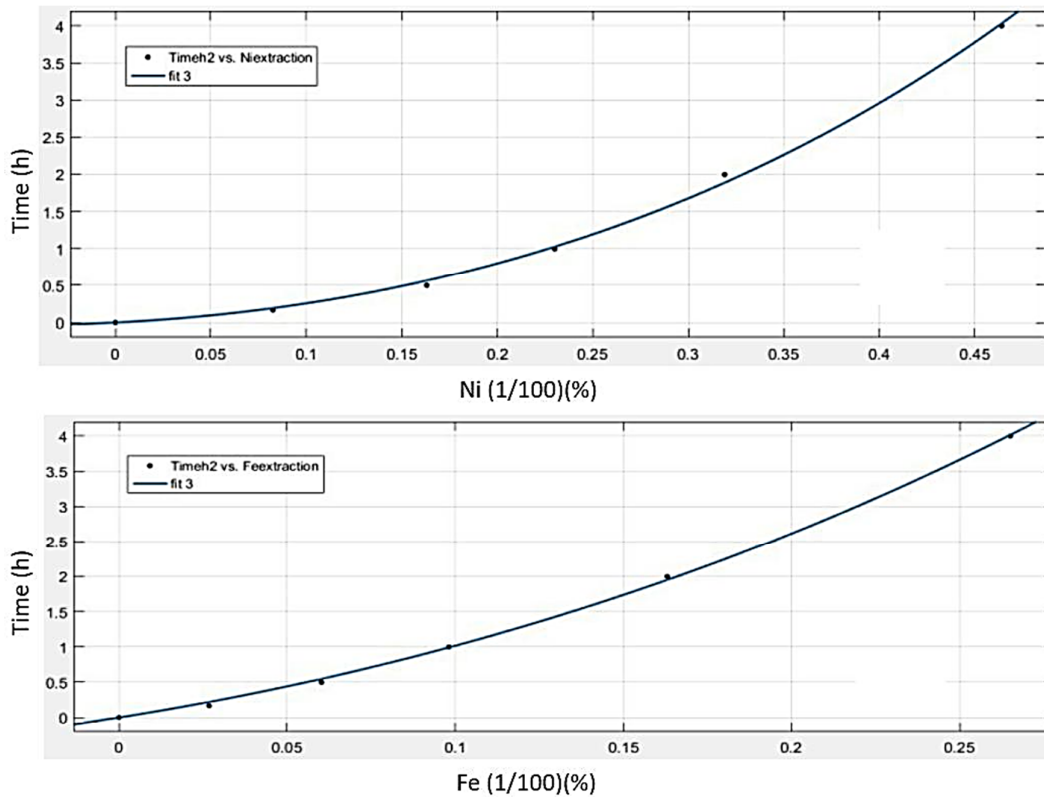
**3.11. Kinetic atmospheric leaching investigation with hydrochloric acid**

The data obtained from fitting the laboratory results of iron and nickel extraction by hydrochloric acid are shown in the table below.

In the Figure 13, the obtained data by the equation at 95°C temperature for two elements Fe and nickel is presented.

**Table 8. The data obtained from the results of iron and nickel extraction by hydrochloric acid**

Element	Temperature(C)	$\tau_F$ (h)	$\tau_P$ (h)	$\tau_R$ (h)	R <sup>2</sup>
Nickel	95	0	20.4692	149.7555	0.9997
	80	0	19.4675	155.7235	0.9993
	70	0	3.2759	266.1177	0.9995
	60	0	0.0053	292.5230	0.9997
Fe	95	0	0	313.4664	0.9996
	80	0	0	348.5264	0.9991
	70	0	0	655.0948	0.9985
	60	0	0	759.9194	0.9983



**Figure13. fitting of laboratory data for two elements nickel (a) and Fe (b), at a temperature of 95 °C.**

**3.12. Kinetic atmospheric leaching investigation with sulphuric acid**

The kinetic atmospheric leaching investigation with sulphuric acid results illustrated in Table 9.

In the figure below the obtained data by the equation at 95°C temperature for two elements Fe and nickel is presented.

**Table 9. The data obtained from the results of iron and nickel extraction by hydrochloric acid.**

Element	Temperature(C)	$\tau_F$ (h)	$\tau_P$ (h)	$\tau_R$ (h)	R <sup>2</sup>
Nickel	95	0	138.4310	27.9156	0.9790
	75	0	151.4103	27.5679	0.9953
	55	0	171.7892	30.9006	0.9895
	35	0	184.4375	32.7550	0.9997
Fe	95	0	126.4220	40.9860	0.9596
	75	0	132.7146	41.6791	0.9791
	55	0	135.4285	43.8305	0.9885
	35	0	138.1160	46.2598	0.9683

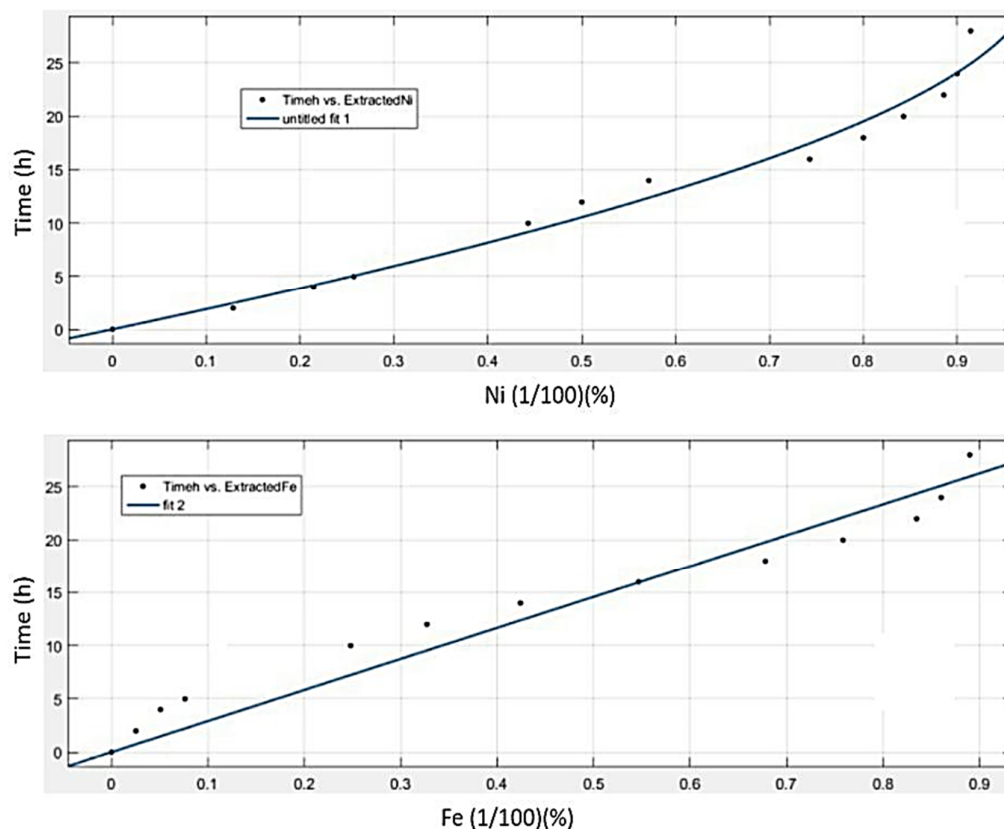


Figure 14. Data graph for two elements nickel (a) and Fe (b), at a temperature of 95 °C.

3.13. X-Ray analyses (XRD)

The results of XRD analysis illustrate that the sample contains 41% quartz, 29.1% dolomite,

12.1% maghemite, 10.3% hematite, and 7.5% gothite (according to Table 10).

Table 10. Data obtained from XRD analyses.

Composition	chemical formula	percentage of minerals
quartz	SiO <sub>2</sub>	%41
dolomite	Ca (Mg,Fe)(CO <sub>3</sub> )	%29.1
maghemite	Fe <sub>2</sub> O <sub>3</sub>	%12.1
hematite	Fe <sub>2</sub> O <sub>3</sub>	%10.3
gothite	Fe <sub>2</sub> O <sub>3</sub> : H <sub>2</sub> O	%7.5

4. Conclusions

The nickel content in the laterite sample was 0.7 wt. %, in the leaching tests, it was observed that nickel laterite was amenable to leaching by means of atmospheric and pressure leaching. A high nickel yield (92%) was obtained in atmospheric leaching with sulfuric acid consumption of 1.16 kg acid/1 kg laterite. After the atmospheric leaching, the Fe concentration in the solution was very high at 70 g/l. Precipitation of iron in the following process steps would require a large amount of neutralizing and precipitation chemicals. This was a common drawback of the atmospheric leaching

of nickel laterites. In HPAL acid leaching, temperature increase from 220 °C to 250 °C did not improve the nickel leaching yield which was approximately 90% at both temperatures (based on solid analysis). According to mineralogical analyses, part of the nickel existed in the hematite lattice. Hematite was a stable mineral in HPAL conditions; therefore, nickel might not be leached totally from hematite minerals. Iron concentration in the solution after HPAL was drastically lower than after atmospheric leaching being 3-6 g/l. Iron leaching yield in HPAL was much lower than in atmospheric leaching because iron behavior in HPAL was very different than in atmospheric

leaching. In HPAL conditions iron first dissolved but transformed then to hematite releasing acid simultaneously. Therefore, net acid consumption in HPAL was clearly lower than in atmospheric leaching and acid economics was therefore more beneficial. Acid dosages were 0.57-0.58 kg acid/1 kg laterite. There was still potential to decrease the acid dosage for this material. In these preliminary tests, the acid dosage was not optimized. Even though the leach recoveries were rather similar in the atmospheric leaching and HPAL options, it is recommended to continue the next steps with the HPAL option. The facts against atmospheric leaching options were:

- High acid consumption
- Long leaching time
- High iron concentration in the solution after leaching, which led to high neutralization/precipitation chemical consumption
- The iron precipitation would be large at the plant scale requiring lots of equipment and resulting in high capital cost
- Some nickel might be lost when it could co-precipitate with iron

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## روش دوستدار محیط زیست: مطالعات اسیدشویی تحت فشار اتمسفر و بالا، فرآوری نیکل لاتریتی کم عیار

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## چکیده:

این مقاله به بررسی عملکرد لیچینگ در فشار اتمسفری (AL) و لیچینگ اسیدی با فشار بالا (HPAL) نیکل لاتریتی که به عنوان لیمونیتی طبقه‌بندی شده، می‌پردازد. این مطالعه در مقیاس آزمایشگاهی انجام شد، شامل تغییرات دمای ۳۵-۹۵ درجه سانتی‌گراد برای AL و ۲۲۰-۲۵۰ درجه سانتی‌گراد برای HPAL است. میزان عیار نیکل و کبالت به ترتیب ۰/۷ و ۰/۰۴ درصد است. روش AL به بازده ۸۹ درصدی Al با pH 0.2 و زمان شستشوی ۱۴ ساعت دست یافت، در حالی که بازیافت نیکل و آهن پس از ۲۰ ساعت با مصرف اسید ۱.۲ کیلوگرم H<sub>2</sub>SO<sub>4</sub> در هر ۱۰۰ کیلوگرم لاتریت (خشک) به ۹۲ درصد و ۸۷ درصد رسید. در pH 0.2 آزمایش‌های لیچینگ در دمای ۲۲۰-۲۵۰ درجه سانتی‌گراد به مدت ۲ ساعت، نرخ بازیابی نیکل مشابهی را نشان داد، که نشان‌دهنده عدم بهبود بیش از ۲۴۰ درجه سانتی‌گراد است. هماتیت، یک ترکیب پایدار مرتبط با نیکل، به دلیل مقاومت در برابر لیچینگ، مانع از انحلال آن در طول HPAL شد است. بازیابی نیکل در هر دو آزمایش AL و HPAL حدود ۹۰٪ به دست آمد. رفتار آهن به طور قابل توجهی بین دو روش متفاوت بود، HPAL در ابتدا آهن را حل می‌کند اما آن را در محل به هماتیت تبدیل می‌کند که منجر به مصرف اسید کمتر در مقایسه با AL می‌شود. مکانیسم شستشو برای اکسیدهای آهن از سینتیک تجربی قانون توان از مرتبه ۱.۵ با انرژی فعال سازی ۳۶.۲۳ و ۲۵.۰۹ کیلوژول بر مول نیکل و آهن پیروی می‌کند.

**کلمات کلیدی:** لاتریت ها، نیکل، شستشوی جوی، HPAL، روش دوستدار محیط زیست.