

# Selective precipitation of iron from multi-element PLS produced by atmospheric leaching of Ni-Co bearing laterite

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## ABSTRACT

Laterites are the main resources of oxidized nickel in the world. Nickel and cobalt are embedded in limonite laterites within the goethite structure. Therefore, the removal of iron ions will lead to the simultaneous precipitation of iron, nickel, and cobalt. In our previous study investigating atmospheric leaching of laterite ore using sulfuric acid with the addition of NaCl to the solution, we determined the optimal parameters to minimize the co-dissolution of iron. Based on the determined optimum conditions, a PLS was prepared. In the current study, the effect of pH on iron precipitation from the PLS was investigated using sodium hydroxide as a neutralizing agent. Results indicated that a pH=4 can result in the highest removal of iron from the leaching solution (around 90%) while minimizing the loss of nickel and cobalt. The SEM analysis revealed ferrihydrite as the most important mineral in the final precipitation obtained at pH=4. The results of this study can be used for benchmarking more efficient methods for iron removal from the solution and improving the dissolution kinetics of nickel and cobalt.

**Keywords:** Laterite, Nickel, Cobalt, Leaching, Precipitation

## 1. Introduction

Approximately 72% of the world's nickel resources are lateritic. Currently, more than 60% of the world's nickel is obtained from laterites [1]. Nickel in limonite laterites is rich in ferric iron oxides and also contains small amounts of magnesium and aluminum. As a result, the nickel in these laterites can only be dissolved by acid attack. Nickel extraction in these laterites is dependent on the breakdown of the iron matrix which necessitates a high acid consumption [2, 3]. To reduce capital costs, acid leaching of nickel laterite ores at atmospheric pressure has gained increasing popularity over high-pressure acid leaching [3]. The kinetics of nickel extraction together with the ease of solution processing in further stages are other advantages of acid leaching at atmospheric pressure [2]. However, unlike high-pressure acid leaching during which most of the dissolved iron eventually precipitates as hematite, acid leaching at atmospheric pressure releases iron ions that remain in the solution [1, 2, 4]. The presence of iron in pregnant leach solution (PLS) is a common problem [1, 3] that should be solved before nickel recovery. If the iron is not effectively removed, the production of pure nickel and cobalt in subsequent downstream hydrometallurgical processes would be difficult [3, 4]. In this case, using the solvent extraction method for the treatment of iron from metal solutions with high iron content is less economical [3]. Therefore, iron removal of laterites is usually performed using selective precipitation by changing the pH, although this method can be also costly depending on the iron concentration [1, 3, 4].

The most common leaching agent to leach nickel lateritic ores is sulfuric acid [5]. Roasting pretreatment and addition of chloride salt are some techniques used to reduce acid consumption in atmospheric acid leaching. However, the increased porosity and surface area of the ore

together with improved selectivity of iron can consequently increase the non-selective leaching of nickel [2, 6]. The trivalent iron can be removed by increasing the pH of the leach solution. With an increased pH, iron is precipitated before the nickel and cobalt can be recovered [4]. Albeit, increasing the solution pH and the precipitation temperature is in favor of iron removal, more nickel and cobalt will be transferred to the residue [4, 7]. At higher pHs, sediments form faster and have finer and more stable particles. In addition, the use of higher pHs can increase the capacity while lowering the stabilization costs after precipitation which is beneficial for industrial applications. On the other hand, the advantage of using a low pH is a better solid-liquid separation due to the formation of larger particles. Removal of other metals from the co-precipitation process and/or adsorption depends on the nature of the iron sedimentations that are initially formed. The presence of Fe<sup>2+</sup> in solid particles indicates thermodynamic instability [8]. It can accelerate the breakdown of the Fe<sup>3+</sup> compounds and reduce Fe<sup>3+</sup> ions exposed at the compounds' crystal surface [9]. Before the ferric precipitation begins, the ferrous iron must be oxidized to the ferric iron, usually by bubbling air through the acid solution. Under high pH conditions (pH 8.5–9.5), oxidation and subsequent precipitation of ferrous iron to ferric occurs rapidly [8]. A pH between 2.5 to 3.5 is sufficient for the precipitation of ferric iron, but precipitation of aluminum content in the solution requires a pH between 4 and 5. At a low pH, hydrogen ions compete more effectively for adsorption sites than nickel ions. With an increased pH, the mineral surface obtains a more negative charge that consequently increases the absorption of the nickel. Arai et al. (2008) showed that at pHs below 6, the absorption of nickel into goethite, hematite, and ferrihydrite is less than 0.1 mg/m<sup>2</sup>. Therefore, precipitation with a higher surface area can result in a higher nickel content through adsorption [4]. An increase of the pH to the desired value is dependent on the metal precipitation in the form of hydroxide. Hydroxides of

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ferrous ions precipitate at pHs higher than 8.5, whereas trivalent iron hydroxides precipitate at pHs higher than 3.5 [10]. For selective iron precipitation, the hydrolysis of soluble iron can be accelerated by the addition of an alkaline substance, so that the metals precipitate as ferric hydroxide [11].

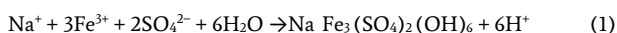
Wang et al. (2011) investigated the factors that influence nickel loss during the removal of iron from acidic leaching solutions containing only nickel and iron at atmospheric pressure. This study showed that, compared to aluminum and chromium, precipitation of ferric iron occurs at a lower pH range. Therefore, higher pH values are often used to achieve maximum removal of aluminum and chromium. The results reported by Agatzini-Leonardou (2009) for recovery of nickel from the leaching solution of nickel-ferrous laterite indicate that increase of the temperature and pH in one-step precipitation is in favor of removing iron, aluminum, and chromium, but more nickel will be lost in the solid fraction [12].

In a later study, Wang et al. (2018) precisely controlled the pH and temperature in a multi-step precipitation process, that enabled the precipitation of iron with minimal nickel loss in the residue [4]. In another study by Nayanthika et al. (2018), the pH was perturbed from 1 to 9 with the aim to remove iron from the leaching solution in the nickel and cobalt dissolution process of laterite. Their results showed that under alkaline conditions (pH $\geq$ 7), very high iron removal efficiencies (80-100%) can be achieved in a short time [13].

The studies by Alkarkhi et al. (2012) and Saka (2014) indicated that temperature has a significant effect on the rate of iron extraction [9, 14]. They found that depending on the temperature, iron in the solution can be precipitated in the form of jarosite, hematite, goethite, or paragoethite. Moreover, high temperatures would be in favor of more stable iron formation [3, 7]. Precipitation of iron in the form of iron hydroxide is also possible, but only a small amount of iron (2 g/L) can be removed from the solution. For precipitation of iron in the form of goethite, the concentration of trivalent iron in the leach solution should be reduced to less than 200 mg/L by adjusting the pH in the range of 2.5-3.0. In this range of pH, precipitation of nickel and cobalt is very low. It is also reported that an autoclave (high temperatures 160-190 °C) is required to precipitate iron in the form of hematite [14].

The ratio of lateritic soil to the extracting agent and time did not play an important role in iron extraction [9]. Saka et al. (2014) showed that the rate of iron removal in the early stages of hydrolysis is higher, although complete hydrolysis of iron takes a long time [14]. In addition, a higher initial Fe/Ni ratio causes more nickel and cobalt loss during the iron removal process and therefore increases the amount of precipitation [2, 3]. The pH value affects the oxidation rate of the ferrous iron, however, pH is also a key factor in nickel loss [2, 14]. With the same initial Fe/Ni ratio, an increase in the pH results in a greater rate of nickel loss [3, 14]. Chang et al. (2010), investigated the iron removal as goethite from the leaching solution of the reduced laterites and found that pHs less than 2.5 can reduce the rate of nickel loss to less than 5% [2].

Hydrolysis of ferric iron produces sulfuric acid and decreases the pH of the solution, which consequently increases the leaching of the nickel [7]. In sulfuric acid solutions, sodium jarosite (NaFe<sub>3</sub>(SO<sub>4</sub>)<sub>2</sub>(OH)<sub>6</sub>) is formed in presence of soluble ferric ions (Fe<sup>3+</sup>) and sodium ions (Na<sup>+</sup>) (eq. 1, [1, 3]):



This reaction produces acid and has a positive effect on the consumption of the process acid [1]. During the precipitation process, a large amount of the produced acid remains intact in the solution. Therefore, a neutralizing agent must be added in order to complete the precipitation reaction [14].

Aliprandini et al. (2016) studied the selective chemical precipitation of different metals, using NaOH to assess the impact of pH. Precipitation experiments were performed at 25 °C for 5 minutes on two different solutions of leached ore with sulfuric acid. The results showed that the solution prepared using Fe<sup>2+</sup>, nickel, cobalt, and iron metals were observed in the solution phase even at pHs greater than 4. For the

solution prepared using Fe<sup>3+</sup>, cobalt and iron began to precipitate at pH=2.5 which could consequently increase the precipitation rate with increasing the pH. For this solution, nickel ions were precipitated at pH=5.5 [5]. In a study by Miettinen et al. (2019), iron precipitation as jarosite was performed for limonite laterites, using nickel-containing silicate laterites for neutralization. The acid produced from the precipitation reaction of jarosite dissolves nickel and other metals (such as magnesium) from silicate laterites. Acid consumption in this method was 0.4 kg per 1 kg of laterite compared to 0.6-0.8 kg required for the direct leaching method. Iron dissolution in this method was only 1.5-3% whereas for the two laterite samples in the direct leaching method it was 15-30% [1].

In addition to ferric iron, Cr (III) and Al are among the most problematic impurities generated from nickel laterite processing due to their interference with downstream Ni/Co recovery and purification process [15]. The co-precipitation of Ni and Co with trivalent metal cations, and adsorption of Ni and Co by the hydrous oxides of Fe (III), Cr (III), and Al, are possible mechanisms for metal loss during partial neutralization.

Our literature review indicated that to date, there is no study to assess the removal of iron from iron-rich laterites. Previous studies showed that a higher initial Fe/Ni ratio in laterites causes more nickel and cobalt loss during iron removal, and therefore precipitation amount increases. This is important, especially for iron-rich laterites, wherein one hand, increasing the precipitation rate will lead to the removal of more iron from the leaching solution, and on the other hand, more nickel and cobalt will be transferred to the residue.

In this study, the effect of pH on iron precipitation from iron-rich laterite samples leached by sulfuric acid was investigated, using sodium hydroxide as a neutralizing agent. The research mainly aimed to study the iron removal from the leaching solution of iron-rich laterites. Moreover, we aimed to minimize the loss of nickel, cobalt, and iron precipitation in the residue and investigate the impact of Cr (III) and Al impurities on Ni and Co loss.

## 2. Materials and methods

### 2.1. Sample and characterization

The laterite sample used in this study was provided from east Sarbisheh located in South Khorasan Province in Iran with an approved reserve of 3,700,000 tons. The average grades of nickel, cobalt and iron in the sample were 1.74%, 0.14%, and 40.83%, respectively. It was rich in nickel, cobalt, and high iron content. Elemental analysis of the laterite sample after calcination in the furnace at 500 °C for 2 hours (performed to achieve higher nickel and cobalt recoveries) showed that the average grade of nickel, cobalt and iron in this sample were 2.3%, 0.17%, and 42.66% respectively. Calcination converts goethite (FeOOH) to hematite (Fe<sub>2</sub>O<sub>3</sub>) and produces pores and cracks that help nickel to be easily dissolved by the leaching agent [16]. Therefore, we used the calcined sample as the feed sample for all the leaching tests as well as for iron precipitation experiments. Results of the particle size analysis (wet method) using a Particle Size Analyzer (Micro-Tec Plus) showed that the laterite particles used in this study were fine-grained and smaller than 38 microns. The d<sub>25</sub>, d<sub>50</sub>, and d<sub>80</sub> were 5.6, 15.1, and 30.1 μm before calcination, and 2.5, 8.6, and 25.2 μm after calcination. XRD analysis (MPD 3000) showed that hematite, calcite, quartz, and goethite were the most important crystalline phases in the studied sample. Results of the XRF analysis (MAGIX-PRO) showed 61.4% of Fe<sub>2</sub>O<sub>3</sub> in the studied laterite sample, indicating the high amount of iron in the sample. Moreover, we found 3% NiO and 0.2% Co<sub>3</sub>O<sub>4</sub> together with 9.2% SiO<sub>2</sub> and 5% Al<sub>2</sub>O<sub>3</sub> in the chemical composition of the sample (Table 1).

**Table 1.** Chemical composition of iron-rich laterite ore obtained from XRF (weight percentage).

Composition	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	MnO	Cr <sub>2</sub> O <sub>3</sub>	NiO	Co <sub>3</sub> O <sub>4</sub>	L.O.I.
(%)	9.2	5.0	61.4	4.0	0.4	0.4	0.7	0.9	3.0	0.2	14.6

## 2.2. Experimental method for iron precipitation

Leaching experiments with sulfuric acid were performed in a one-liter glass reactor at atmospheric pressure. Schematic layout and pictures of the reactor used for leaching experiments are presented in previous articles [6, 17].

Our previous investigations revealed that the optimal leaching conditions with sulfuric acid are as follows: S/L=0.1, stirring speed=370 rpm, temperature=90 °C, sulfuric acid concentration=5 M, leaching time=2 h and NaCl content=25% of solid weight [6, 17]. Precipitation experiments were performed using the solution obtained from the filtration of leaching pulp under optimal conditions. Thus, the PLS used for precipitation experiments were the product of the agitation leaching process of the laterite sample using sulfuric acid under optimal conditions, at atmospheric pressure. The main elements of the obtained PLS were analyzed and are listed in Table 2. With the initial Fe/Ni ratio of 5.7, the PLS included 1.89 g/L Ni, 0.13 g/L Co, and 10.78 g/L Fe. The pH of the filtrated solution, without pH adjustment, before starting precipitation experiments for all samples in Table 4 was 0.4.

pH in precipitation experiments was adjusted by adding sodium hydroxide (0.1 M NaOH), which neutralizes the acid released from iron hydrolysis. The effect of pH on iron precipitation rate was investigated at pH=2, 3, 4, 5, and 6. While adding the precipitate agent, the chemical reaction between the acidic environment and the alkaline additive generates heat [3], and therefore, in our experiments, the solution was stirred for 5 minutes using a magnetic stirrer to maintain the constant temperature in the total solution.

After iron precipitation, the samples were filtered using No.42 Whatman filter papers, and the obtained liquid was analyzed for nickel, cobalt, and iron. All experiments were repeated twice and the results were averaged. The average error of the atomic absorption spectroscopy (AAS) instrument was 0.5% and the experimental error to evaluate Ni, Co, and Fe concentration was 3%.

**Table 2.** Elemental analysis of the main elements in the obtained PLS (pH 0.4).

Concentration (g/L)							
Co	Ni	Fe	Al	Cr	Mg	Na	Mn
0.13	1.89	10.78	2.71	1.9	1.42	19.64	0.64

## 3. Results and Discussion

### 3.1. Leaching experiments

Previous investigations on the studied laterite sample showed that with increasing temperature and the NaCl amount, the nickel and cobalt recovery rates generally increase. On the other hand, an increase in the NaCl amount significantly decreases the iron dissolution in the solution. Therefore, the addition of NaCl has great importance in the removal of iron from the leaching solution of iron-rich laterites. The optimum amount for NaCl additive was 25% (weight ratio) of the laterite solid used in the experiment [17]. Table 3 compares the nickel and cobalt recovery rates and the iron dissolution rate with and without the NaCl additive. We found that the nickel and cobalt recovery increased after adding NaCl. However, iron dissolution using sulfuric acid reduced from 96.1% to 32.9% when adding 25% (weight ratio) NaCl.

**Table 3.** Comparison of nickel and cobalt recovery rates and iron dissolution rate with and without adding NaCl (sulfuric acid concentration= 5 M, S/L=0.1, stirring speed= 370m rpm, leaching time= 2 h, temperature= 90 °C).

	Ni Recovery (%)	Co Recovery (%)	Fe Dissolution (%)
adding 25% (weight ratio) NaCl	99.6	97.5	32.9
without NaCl	95.3	85.7	96.1

### 3.2. Precipitation experiments

Table 4 presents the initial and final chemical compositions of the leach solution for the precipitation experiments. As can be seen, starting from the initial pH of 2 and before reaching the optimum initial pH for

iron precipitation (pH=4), the iron concentration in the leach solution decreased whereas the nickel and cobalt concentrations increased. This can be explained by the fact that at the optimum initial pH, a large amount of iron will be removed from the solution and will be precipitated in the residue while less nickel and cobalt will be transferred to the residue together with the iron. With a pH lower than the optimum initial pH, better solid-liquid separation occurs due to the formation of larger particles [8] that consequently increase the concentrations of nickel and cobalt. Compared with pH=4, at pH=5 or 6, sediments form faster [8] and therefore surface adsorption of nickel and cobalt increases into the residue. The optimum initial pH for iron precipitation determined from the results of the five experiments (Table 4) was pH=4. When the initial pH of the solution was adjusted to 5 and 6 for precipitation experiments, due to the fineness of the sample and the mix of leaching solution and sodium hydroxide, the resultant pulp was difficult to filter. At pHs  $\geq 5$ , filtration by a vacuum filter using No.42 Whatman filter paper required a long time. On the other hand, as mentioned in section 2.2, NaCl was used as an additive to prepare the PLS solution for precipitation experiments. It is known that the chlorine ion in NaCl can activate the dissolution sites. A surface complex formed by chlorine and iron ions increases the pH from 2 to 4 and causes the partial release of nickel and cobalt bounded to the iron into the solution (reactions (2) and (3)). As a result, the concentration of nickel and cobalt in the solution increases, and better precipitation of iron in the residue can be achieved [18]. At pH=5 or 6, by passing the zero-charge-point of iron-bearing minerals, the complexation of chlorine ions with iron stops which consequently reduces the concentrations of nickel and cobalt in the solution [19].



**Table 4.** Final concentrations of Co, Ni, and Fe in the PLS (Precipitation time= 60 min).

Sediment color	Final concentration (g/L)			Initial pH of the solution with pH adjustment
	Co	Ni	Fe	
light brown	0.14	2.0	0.05	2
Dark brown	0.14	2.1	0.03	3
Dark brown	0.15	2.3	0.01	4
hepatic	0.13	1.9	0.07	5
hepatic	0.12	1.7	0.07	6

For all the experiments, the iron content in the final solution was less than 0.1 g/L (Table 4), which confirms the completion of the iron removal. The sediment color also changed at different initial pHs, so that the formed sediment becomes darker with increasing pH. from pH=2 to 4, due to the increase in iron precipitation, the residue color changes from light brown to dark brown. At initial pH=5 or 6, the residue color changes to hepatic because the aluminum in the solution also precipitates [4].

The rate of Ni and Co loss in the residue at initial pH=4 for the PLS is presented in Table 5. Interestingly, an inappreciable amount of Ni and Co precipitated at the optimal pH (Table 5).

**Table 5.** Loss of Ni and Co (pH=4).

% Fe precipitation	Ni, mg/g	Co, mg/g	Ni loss, %	Co loss, %
90.72	0.327	0.176	0.86	2.43

Figure 1 depicts the influence of pH on iron, nickel, and cobalt precipitation. As can be seen, at pH=4, more than 90% of the iron can be removed from the solution following a one-step precipitation process. The pH value had a considerable impact on the loss of nickel into the residue. The loss of nickel and cobalt increased as the pH increased. At pH=4, while the highest rate of iron precipitation occurred, the loss of nickel and cobalt into the residue was also higher compared to the other pHs. The loss of nickel and cobalt to the residue can be postulated by simultaneous precipitation of these metals together with iron. For pH values higher than 4, the precipitation process took longer due to the



formation of a gel-like structure, and the precipitation rate of iron, nickel, and cobalt decreased when compared to pH=4. For pHs between 2 and 4, the nickel and cobalt concentrations have increased after the iron precipitation (Table 4), however, in the iron-rich limonite laterites, trapping of nickel and cobalt in iron compounds resulted in an insignificant fraction of Ni and Co that co-precipitated with iron into the residue.

Since the studied sample was of the iron-rich laterite type, increasing the amount of iron precipitation in the residue was the most important goal of this study. For pH=4, a comparison of the Ni and Co content in the residue after precipitation with those measured in the solution before precipitation indicates a small loss of nickel and cobalt together with iron precipitation (21.8% and 17.3%, respectively) (Figure 1). Therefore, according to the results of the five precipitation experiments performed to obtain the maximum iron precipitation (presented in Table 4 and Figure 1), pH=4 is the most suitable initial pH.

At pH=6, 38.6% of iron was precipitated while 10% nickel and 7.7% cobalt were lost (Figure 1). However, at pHs higher than 6, the residual sediment on the filter paper was jelly. Even after an hour, only a small portion of the liquid had passed through the filter, and the residue on the filter paper became crystalline, rigid, and firm.

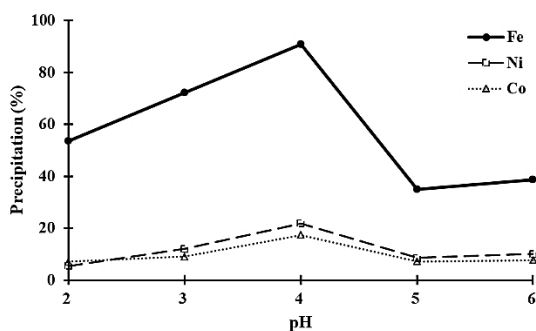


Figure 1. The effect of pH on precipitation of iron, nickel, and cobalt.

As the pH increases from 2 to 4, the absorption of nickel and cobalt into the solution increase [4]. For pH=5 or higher, due to the reduced precipitation of iron, nickel, and cobalt precipitation with iron also decreases. As mentioned before, filtration at pHs > 6 took a long time.

Chemical analysis of the residue obtained from the iron precipitation experiment at initial pH=4 showed a low nickel and cobalt content (Table 6). In addition, we could not find large amounts of Mg and Al in the residue. In contrast, there was a high amount of sodium (17.28%) due to the use of NaOH additive.

Table 6. Chemical analysis of the residue obtained from iron precipitation experiment at pH=4.

Fe (%)	Ni (%)	Co (%)	Mg (%)	Na (%)	Al (%)	*LOI (%)
8.90	0.015	< 0.001	0.034	17.28	0.862	9.66

\*Loss of ignition at 1000 °C

In order to explain the precipitation mechanism, SEM (Figure 2) and XRD (Figure 3) analyses were performed on the residue obtained from the iron precipitation experiment at optimum initial pH (pH=4). According to XRD analysis, at pH=4, the main mineral in the final precipitation was ferrihydrite (approximately 70%) which is an amorphous mineral. Ferrihydrite is hydrated ferric iron and can be formed by rapid hydrolysis of  $Fe^{3+}$  solution [3]. The light grains (white grains) in Figure 2 are iron-bearing minerals, which were more than other grains in the residue. These are fine-grain, amorphous, and mainly composed of ferrihydrite.

According to the Pourbaix diagram, the  $Fe^{3+}$  stability zone decreases with increasing temperature. Therefore, even if the acidity is high, iron ions such as  $Fe^{3+}$  will still precipitate if the temperature increases [14]. Dyer et al. (2012) found that lower pH leads to faster hydrolysis, which produces finer particles that appear to be more amorphous. The formation of more amorphous sediments leads to the loss of less nickel

and cobalt [3]. Figure 2 also confirms the amorphous and fine-grained nature of ferrihydrite.

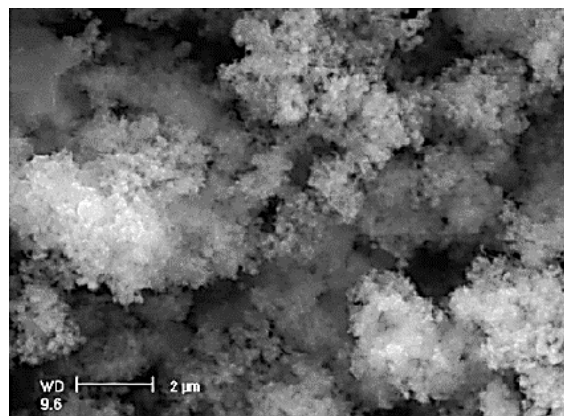


Figure 2. SEM image of the residue obtained from the precipitation experiment at pH=4.

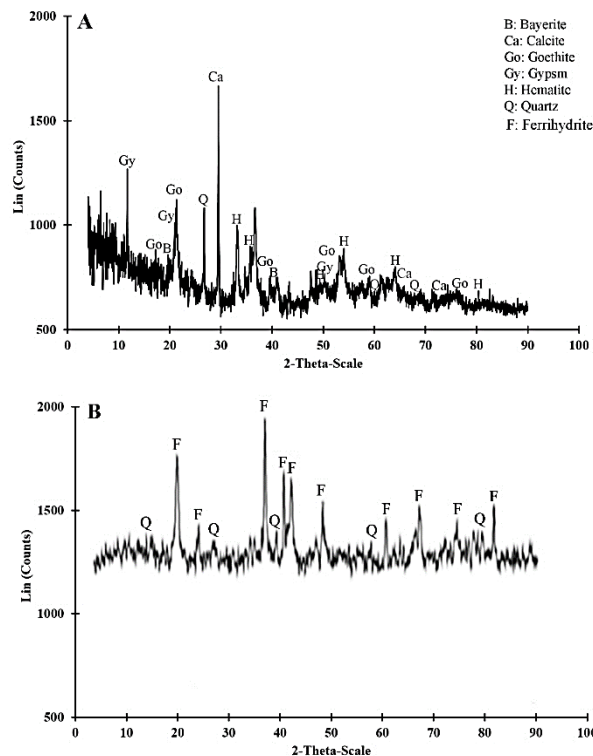


Figure 3. XRD analysis of the A: feed sample and B: sediment formed at pH=4.

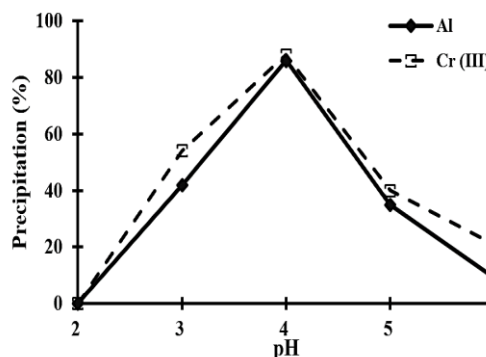


Figure 4. The effect of pH on precipitation of Al and Cr (III) impurities.

The precipitation percentage of Al and Cr (III) impurities obtained at different initial pHs of the PLS is shown in Figure 4. Al precipitation started at  $\text{pH} > 3$ . At  $\text{pH} = 4$ , more than 90% of the iron, 86% of Al, and 88% of Cr can be removed from the solution during the precipitation process. The Al and Cr (III) precipitation patterns are similar to the Fe (III) precipitation as presented in Figure 1.

$\text{Al}^{3+}$  is a harder base than  $\text{Fe}^{3+}$  [20]. The  $\text{Ni}^{2+}$  ion, being a harder cation than  $\text{Co}^{2+}$  would have a higher affinity for hard bases. Therefore,  $\text{Ni}^{2+}$  would be expected to associate with Al oxides and  $\text{Co}^{2+}$  with Fe (III) oxides according to HDAB theory [20]. As a result, to increase the precipitation rate of iron and dissolution rate of cobalt, further research is needed to investigate a trend of cobalt changes during precipitation experiments of iron at each pH throughout the precipitation duration.

#### 4. Conclusion

In this study, the effect of pH on iron precipitation from an iron-rich laterite sample leached by sulfuric acid was investigated. The results showed that  $\text{pH} = 4$  provides the optimal situation that results in the highest removal of iron from the leaching solution, while keeping the simultaneous precipitation of nickel and cobalt in a small range. With the initial  $\text{pH} = 4$ , more than 90% of iron was removed from the solution, and the most important mineral in the final precipitation was ferrihydrite. We also found that the pH value affects the particle size distribution of the final solid residue, the overall kinetics of the process, and the stability of the formed solid residue.

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