Recovery of Nickel and Cobalt from a Low Grade Laterite Ore

Batnasan ALTANSUKH1*, Kazutoshi HAGA1 and Atsushi SHIBAYAMA1,2

1Graduate School of Engineering and Resource Science, Akita University, Akita 010-8502, Japan
2Faculty of International Resource Science, Akita University, Akita 010-8502, Japan

Abstract
This study focused on the development of hydrometallurgical process which allows the nickel (Ni) and cobalt (Co) recovery from a low grade laterite ore. The developed process consists of two main stages: leaching and precipitation. In the first stage, an appropriate leach liquor which contains a relatively high nickel and cobalt concentration is prepared by chemical leaching and pressure leaching followed by separation steps, respectively. The effects of sulfuric acid concentration, leaching temperature and time, internal pressure in an autoclave on the efficient leaching of Ni and Co from a laterite ore were presented. In the next stage, the Ni and Co ions present in the liquor are co-precipitated as sulfides by sulfuration with sodium hydrosulfide (pH 3.8).

It was found that talc (Mg₃Si₄O₁₀(OH)₂), goethite (FeOOH) and quartz (SiO₂) are main minerals of the laterite ore which contains 48.12% Fe, 1.8% Al, 0.95% Ni, 0.07% Co, 0.29% Cr and 0.15% Mg. The high efficiency of pressure leaching for Ni and Co from the low grade laterite ore was revealed by comparing the chemical leaching results. The vast majority of Co (>97%) and Ni (55%) from the purified liquor resulting after neutralization with calcium carbonate (CaCO₃) was precipitated as mixed-sulfides by adjusting the NaHS/(Co + Ni) ratio to 3 by the addition of sulfuration agent (NaHS) at the solution pH of 3.8. Based on the experimental results, the efficient recovery of cobalt (Co) and nickel (Ni) from the low grade laterite ore can be achieved using the pressure leaching and neutralization treatment followed by sulfuration.

Key words: laterite ore, nickel, cobalt, pressure leaching, leach liquor, neutralization

1. Introduction

Rare metals such as nickel (Ni) and cobalt (Co) have been widely used in various electronic materials, aerospace, superalloys and automotive batteries, etc, and are an important metals supporting a high-tech industry1-3. About 70% of the Earth’s land-based nickel resource are contained in laterites and the about 30% exist as sulfide ores which account for about 60% of world nickel production compared to about 40% for laterites1,4-7. Laterite ores are mineralogically and chemically complex due to alteration of the host rock, climate, drainage, geomorphological history and composition8,9.

The complexity of the ores led to develop new and more economical processing technology to effectively utilize low grade laterite ores. A variety of methods are used to process laterite ores. These methods can be generally categorized under hydrometallurgical processes and pyrometallurgical processes. The development of hydrometallurgical processes including high pressure acid leaching (HPAL) and atmospheric leaching (AL) are addressed to achieve the optimum resource utilization, replacing the energy intensive pyrometallurgical processes4,10-12. Although HPAL could be give relatively high nickel and cobalt recoveries with negligible iron, aluminium and magnesium concentration due to their hydrolysis and precipitate at high temperature, the expensive autoclaves with high maintenance costs is required11-14. In case of AL process which can be controlled and operate at ambient temperature and at low costs avoiding the need for expensive autoclaves, however the leach liquor resulting from AL contains high concentrations of iron and aluminium15,16. It therefore demands to improve the efficiency of the leaching process for rare metals from low grade laterite ores.
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This study first considered the behavior of nickel and cobalt in selective leaching from a laterite ore in Papua New Guinea by both chemical leaching and pressure leaching. The objective was to compare the results of the two leaching processes. It then focused on the recovery of nickel and cobalt simultaneously as a mixed sulfide form by neutralization and sulfuration. On the other hand, influence of major impurity elements such as iron, aluminium and chromium from the laterite ore on the recovery of Ni and Co was discussed.

2. Experimental

2.1. Sample

A low grade laterite ore sample from Papua New Guinea was used in this study. Chemical composition and mineralogical constituent of a laterite ore were studied by ICP-OES and XRD techniques. The chemical analysis by ICP-OES of the sample showed that it consists of 48.12% Fe, 1.8% Al, 0.95% Ni, 0.29% Cr, 0.07% Co and 0.15% Mg (Table 1). The mineralogical study by X-Ray diffraction analysis (XRD) confirmed that goethite (FeOOH), quartz (SiO₂) and talc (Mg₃Si₄O₁₀(OH)₂) are main mineral constituents of the laterite ore as shown in Fig. 1. The particle size of the laterite ore ranges between 1.8 and 105 μm with the average particle size (D₅₀) of 24.99 μm (Fig. 2).

2.2. Study of leaching behavior of metals from a laterite ore

The leaching behavior of nickel and cobalt were studied using chemical and autoclave leaching in order to compare the usefulness of the both leaching methods and to develop the recovery process of rare metals (Ni, Co) from low grade laterite ores. The chemicals used in this study (H₂SO₄, CaCO₃, NaHS, Al₂(SO₄)₃, CoSO₄·7H₂O, CrSO₄, FeSO₄·7H₂O and NiSO₄·6H₂O) were of analytical grade.

2.2.1. Chemical leaching at atmospheric pressure

A 10 g laterite ore sample was added into 100 mL of sulfuric acid solution placed in a 200 mL volume of a Teflon beaker and stirred at 500 rpm. The experiments were conducted in different sulfuric acid concentrations (1–4 mol/L), different temperatures (60°C–95°C) and various time intervals (2–4 hours), respectively.

2.2.2. Autoclave leaching at high pressure

The experimental procedure during the pressure leaching are as follows: 1) a 10 g of laterite ore sample was inserted to 100 mL sulfuric acid solution in a Teflon vessel which has a 200 mL capacity and placed it in an autoclave. 2) A pulp density (100 g/L), sulfuric acid concentration (0.5–2 mol/L), agitation speed (750 rpm) and leaching temperature (120–180°C) were maintained respectively. 3) when the temperature stabilized, oxygen gas was supplied into the reactor until the pressure inside of the autoclave reach up to 4 MPa and controlled the leaching time from 0.5 h to 2 h, respectively. The oxygen flow and pressure inside of the autoclave were monitored by pressure gauge on an oxygen cylinder and reactor, respectively. The schematic illustration
of the autoclave used in this study is shown in Fig. 3.

2.3. Recovery of metals from a prepared solution and leach liquor

Optimum conditions for the recovery of Ni and Co from a previously prepared solution contained Ni, Co and Al, Fe, Cr were examined first by neutralization and sulfuration with respect to pH of the solution and weight molar ratios of NaHS/(Co + Ni), respectively. The solution was made using deionized water, chemical grade aluminium sulfate (III), cobalt sulfate (II) heptahydrate, chromium sulfate (II), iron sulfate (II) heptahydrate and nickel sulfate (II) hexahydrate and adjusted its composition to be similar with the leach liquor obtained from pressure leaching under optimum conditions.

The neutralization experiments were performed at fixed volume (50 mL), fixed stirring speed (600 rpm), fixed time (20 min) and fixed temperature (<50°C) while the pH of solutions were adjusted from pH 0.5 to pH 5.0 by neutralizing agent as CaCO₃. The optimum condition was obtained at pH 3.8 for the removal of Al, Fe and Cr from the previously prepared solution.

The precipitation behavior of Ni and Co from the leachate obtained by neutralization of the previously prepared solution was studied in the varying weight molar ratios of NaHS/(Co + Ni) from 1.0 to 3.0 when the fixed experimental conditions were: volume of the solution of 50 mL, pH of the solution of 3.8, stirring time of 20 minutes, stirring speed of 600 rpm, temperature <50°C, respectively. When the weight molar ratio of NaHS/(Co + Ni) was 3, the high recovery rate of metals (Ni and Co) was observed. Therefore, this condition was chosen as an optimum condition for subsequent experiment.

After that the optimum conditions obtained from the neutralization and sulfuration experiments were used for the recovery of the Al, Fe, Cr and Ni, Co from the leach liquor resulting after pressure leaching under optimum condition (Section 2.2.2).

2.4. Characterization and chemical analysis

The chemical composition of all solutions prepared was determined by an inductively coupled plasma optical emission spectrometer (ICP-OES, SPS 5510 III Nanotechnology Inc). The qualitative analysis of the sample and all solid residues were carried out on a X-ray diffractometer (XRD, RINT-2200V, Rigaku) with a Cu Ka irradiation source (λ = 1.5405 Å) at 40 kV and 50 mA at a continuous scan mode between 10° and 80° 2θ. The particle size distribution of the sample was measured by micro-track particle size measurement equipment (MT3000 II, Nikkiso Instrumentation Co., Ltd). The pH of the sample solutions was monitored in every experiment using a laboratory pH meter.

3. Results and discussion

3.1. Chemical leaching at atmospheric pressure

3.1.1. Chemical leaching—effect of temperature

The effect of temperature on leaching behavior of metals from a sample of the laterite ore was investigated at the initial concentration of sulfuric acid of 2 mol/L, pulp density of 100 g/L, stirring speed of 500 rpm and leaching time of 2 hours with various temperature ranging from 60°C to 95°C. As shown in Fig. 4, the experimental results
showed that leaching rates of the metals were increased with increasing the temperature. The leaching rate of iron (Fe) increased drastically with increasing the temperature from 60°C to 95°C. The vast majority of the iron (>99% Fe) and aluminium (80% Al) from the sample was dissolved in 2 mol/L H₂SO₄ at above 80°C, whereas the highest leaching rate of nickel (90% Ni) and cobalt (40% Co) as target metals was observed at 90°C and 95°C, respectively.

3.1.2. Chemical leaching—effect of sulfuric acid concentration

Effect of sulfuric acid concentration on the leaching rate of rare metals (Ni and Co) was also examined at a fixed pulp density of 100 g/L, fixed leaching temperature of 80°C, fixed leaching time of 2 h, fixed stirring speed of 500 rpm, and varying the concentration of sulfuric acid from 1 mol/L to 4 mol/L. As it can be seen in Fig. 5, an increase in the sulfuric acid concentration seems to affect much leaching rate of metals. At the lowest H₂SO₄ concentration (2 mol/L) the leaching rate of iron (Fe) was reached nearly 97% that was kept up to 4 mol/L H₂SO₄ concentration. The leaching rates of the other metals appeared to be rapid while the H₂SO₄ concentration increased until 4 mol/L, however leaching rates of the metals were less than 85%. The results presented in Fig. 5 showed that in 1 mol/L H₂SO₄, Ni and Co dissolution reached 38% and 15% and increased to 75%, 35% and 85%, 72% in 2 mol/L and 4 mol/L H₂SO₄ respectively.

On the basis of results presented in this section, it revealed that although the highest dissolution of Ni and Co was observed by chemical leaching under the optimum condition, which is an inefficient approach for dissolution of the Ni and Co from low grade laterite ores because of no selective leaching of the rare metals (Ni, Co). It is obvious that H₂SO₄ is a highly corrosive strong mineral acid especially a high concentration of H₂SO₄ can cause very serious damage upon contact it. In this study we introduced a fast and efficient method with relatively high Ni and Co dissolution, on the other hand, lower solubility of impurities (Fe, Al and Cr) from the laterite ore.

3.2. Pressure leaching using an autoclave

In this section the optimum conditions which are reaction temperature, H₂SO₄ concentration, pressure inside of the autoclave and leaching time were examined for leaching of Ni and Co from a laterite ore sample using an autoclave.

3.2.1. Pressure leaching—effect of temperature

The effect of temperature on the leaching rate of metals (Ni, Co, Al, Fe and Cr) from a laterite ore was investigated within the temperature range from 120°C and 180°C under fixed pulp density of 100 g/L, fixed sulfuric acid concentration of 2 mol/L and fixed initial oxygen pressure of 2 MPa as well as fixed leaching time of 1 h at stirring speed of 750 rpm. Concentrations of the metals were analysed in the samples taken during the leaching and whose results are shown in Fig. 6. It can be seen from Fig. 6 that the leaching rates of Ni, Co and Al increase slightly with increasing temperature from 120°C to 180°C, respectively. At elevated temperature, the leaching rate of Fe decreases from 88% to 60%, whereas Cr dissolution increases from 65% to 75%. The results suggested that the leaching temperature did not seem
to affect much the leaching rate of the rare metals (Ni, Co) however it led to select of 180°C for the next experiment as an optimum condition because of lower dissolution of impurities especially Fe from the ore.

Under the condition, iron control become possible as a result of precipitation of iron hydroxide sulfate (Fe(OH)SO₄) that confirmed by XRD analysis (Fig. 7).

**3.2.2. Pressure leaching—Effect of H₂SO₄ concentration**

The effect of sulphuric acid concentration on the dissolution of rare metals (Ni, Co) and accompanying metals (Al, Fe, Cr) was investigated in the range of 0.5 mol/L to 2 mol/L at the fixed temperature of 180°C, pulp density of 100 g/L and oxygen partial pressure of 2 MPa, leaching time of 1 h, stirring speed of 750 rpm. The observed effect of the sulfuric acid concentration on the leaching behavior of the metals from the laterite ore is shown in Fig. 8. To elevate the dissolution of the metals from the laterite ore, high concentration of the metals was observed at 2 mol/L H₂SO₄ condition, whereas concentrations of the metals especially Al, Cr and Fe as impurities were reduced drastically with decreasing the H₂SO₄ concentration to 0.5 M. Although the concentration of cobalt (Co) was almost constant at the different sulfuric acid concentrations. It was observed that sulfuric acid concentration was found to have a noticeable effect on the leaching of the metals except than Co from the laterite ore. Therefore the results suggested to selection of 0.5 mol/L concentration of sulfuric acid for the subsequent experiment. XRD measurement results of the residues are also shown in Fig. 9, which also verified that the formation of ferric hydroxide sulfate (Fe(OH)SO₄) takes place at higher H₂SO₄ concentration (2 mol/L), whereas ferric oxide (Fe₂O₃) occurs at lower H₂SO₄ concentration (0.5 mol/L) during the leaching under elevated temperature (180°C).

**3.2.3. Pressure leaching—Effect of process (internal) pressure**

The effect of process pressure inside of the autoclave on the metals dissolution was also examined in the range from 2 MPa to 4 MPa by using O₂ gas supply, while the pulp density (100 g/L), sulfuric acid concentration (0.5 mol/L), leaching temperature (180°C), leaching time (1 h) were
fixed constant. The results obtained are shown in Fig. 10. On the basis of results presented in Fig. 10, it can be observed that the leaching rates of Co, Al and Cr were almost constant with the change in pressure. In the condition, changes in pressure have little effect on the dissolution of nickel (Ni) and iron (Fe) from the sample. The highest leaching rate for the target metals (Ni, Co) was selected at the pressure of 2 MPa, where Co and Ni were solubilized nearly 98% and 60%, respectively.

### 3.2.4. Pressure leaching—Effect of reaction time

The experiment were conducted in the various time ranging from 0.5 h to 2 h while fixed internal pressure in the autoclave of 2 MPa, temperature of 180°C, sulfuric acid concentration of 0.5 mol/L, pulp concentration of 100 g/L and agitation speed of 750 rpm. Fig. 11 shows the effect of leaching time on the dissolution of the metals from the sample. The leaching rates of Al, Cr and Ni especially Ni from a laterite ore increase with leaching time up to 1 h but they decrease with increasing the time further to 2 h, respectively. On the other hand the leaching rate of iron decreases with increasing the time from 0.5 h to 2 h as the result of precipitation of iron oxide (Fe₂O₃) or goethite (FeOOH) and it was confirmed by XRD measurement. The results presented in Fig. 11 led to select of 1 h as a best condition for leaching of Ni and Co by pressure leaching.

As a result of experiments explained in section 3.2, optimum conditions were found for dissolution of the Ni and Co from a low grade laterite ore and were summarized in Table 2. It was revealed that the vast majority of cobalt (>99% Co) and more than 60% nickel (Ni) dissolved from the laterite ore under the optimum conditions in which suppressed of the leaching rate of iron, chromium and aluminum up to 10%, 30% and 70% respectively. Therefore pressure leaching can be considered as an efficient method for the recovery of Ni and Co selectively from low-grade laterite ores.

### 3.3. Recovery of Ni and Co from leachate by neutralization and sulfuration

A process for the recovery of rare metals (Co, Ni) from the leachate by neutralization followed by sulfuration and behavior of the impurities in the process were discussed in this section.

#### 3.3.1. Recovery of Ni and Co from a prepared solution

Optimum conditions for the recovery of rare

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**Table 2** Optimum condition for dissolution of rare metals from laterite ore by pressure leaching

| Parameters                  | Condition | Metals concentration, mg/L |
|-----------------------------|-----------|-----------------------------|
| Concentration of sulfuric acid, mol/L | 0.5       | Al  70 | Co  100 | Cr  30 | Fe  10 | Ni  60 |
| Leaching time, h            | 1         |                |      |      |      |
| Leaching temperature, °C    | 180       |                |      |      |      |
| Oxygen pressure, MPa        | 2         |                |      |      |      |

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metals (Ni, Co) by neutralization and sulfuration were first studied using the prepared solution, which consists of deionized water containing some elements such as Co, Ni, Al, Cr and Fe as mentioned in Section 2.3. The concentrations of metals in the solution are 102 mg/L Co, 569 mg/L Ni, 1385 mg/L Al, 55 mg/L Cr and 4859 mg/L Fe, which are approximately similar with leach liquor obtained by pressure leaching (Section 2.2.2).

The neutralization experiment was conducted to remove of Al, Fe and Cr as impurities from the prepared solution using the stated condition in Section 2.3. The results obtained are shown in Fig. 12, which shows that Al, Fe and Cr were removed completely as a mixed-precipitate from the solution in the range of pH between pH 3.5 and pH 3.8, although about 20%–40% of Co and Ni were also precipitated together with Al, Fe and Cr by neutralization. The results obtained led to selection of pH 3.5–3.8 as an appropriate pH for removal of Al, Fe and Cr from the solution forming the precipitate as the result of adhesion of CaCO₃ with hydrous Fe, Al and Cr compounds. Leachate after neutralization of Al, Fe and Cr was used in the sulfuration study for the recovery of Ni and Co from it using NaHS as a sulfuration agent.

The recovery of Ni and Co from the leachate was discussed depending on the varying weight molar ratios of NaHS/(Co + Ni) from 1.0 to 3.0 under the optimum condition. It can be seen in Fig. 13 that the Ni and Co recovery increased with increasing the weight molar ratios of NaHS/(Co + Ni). The result showed that weight molar ratio of NaHS/(Co + Ni) was found to have an effect on the recovery of metals from the leachate. Rare metals (Ni, Co) and impurity metals (Al, Fe) from the leachate solution can be recovered nearly 100%, whereas no Cr recovery was detected while the NaHS/(Co + Ni) ratio was 3. The results obtained led to selection of NaHS/(Co + Ni) weight molar ratio to 3 as optimum condition for the subsequent experiments.

3.3.2. Recovery of Ni and Co from the leach liquor after autoclave leaching

The recovery of rare metals (Co, Ni) from the leach liquor, which contains 89 mg/L Co, 550 mg/L Ni, 1206 mg/L Al, 108 mg/L Cr and 5689 mg/L Fe by using optimized neutralization and sulfuration processes is discussed as below. The liquor obtained from autoclave leaching (Section 3.2) under optimum conditions was used in this study. The neutralization experiments were conducted using the predetermined condition as explained in Section 2.3 and 3.3.1 respectively. The pH of the leach liquor was adjusted by the addition of calcium carbonate for removal of impurities (Al, Fe and Cr) from the liquor under optimum conditions. Fig. 14 showed that the most majority of Al, Fe and Cr as impurities were removed by adjusting the pH of the leach liquor to 3.8 by the addition of a neutralization agent (CaCO₃) as the result of adhesion of CaCO₃ with hydrous Fe, Al and Cr compounds. Although small amount Co (2%) was removed from the liquor under the condition. On the contrary, a leachate obtained by filtration contains about 98% Co and 60% Ni, which is used in the sulfuration
experiment.

The sulfuration experiments were carried out using the predetermined condition as explained in Section 2.3 and Section 3.3.1. The weight molar ratio of NaHS/(Co + Ni) was adjusted to 3 by the addition of NaHS under the fixed conditions for the recovery of Ni and Co from the leachate. It can be seen in Fig. 15 that Ni and Co into the leachate after neutralization were recovered nearly 100% by sulfuration treatment. The solid and leachate were separated by filtration after the experiment and the results obtained by ICP-OES are summarized in Table 3. It was observed that Ni (885 mg/L) and Co (130 mg/L) are main compositions of the precipitation which also contains small amount Al (5 mg/L) and Fe (134 mg/L) as impurities. The results presented in Table 3 revealed that sulfuration is not suitable method for selective recovery of pure precipitation of Ni and Co from the leachate resulting after neutralization. The following flowchart was proposed for the recovery of Ni and Co from laterite ores as shown in Fig. 16. It can be seen that the vast majority of the cobalt (>98%) and Ni (>55%) from low grade laterite ores can be recovered by combined pressure leaching and neutralization followed by sulfuration treatments.

More work will be carried out to improve the dissolution of Ni from the laterite ore and selective recovery of Ni and Co from pregnant solutions by solvent extraction, ion-exchange resin and hydrogen reduction or precipitation.

4. Conclusions

This study focused on the development of hydrometallurgical process which allows to recover Ni and Co from a low grade laterite ore by combined leaching and precipitation methods. Experimental results revealed that chemical leaching is an inefficient method due to its lower selectivity for dissolution of nickel (Ni) and cobalt (Co) from the laterite ore. However the vast majority of copper (>99% Co) and more than 60% of nickel (Ni) could be dissolved from a laterite ore by pressure leaching under the optimum conditions in which suppressed the concentration of iron, chromium and aluminium into the leach liquor up to 10%, 30% and 70% respectively. By neutralization, the impurities of Fe, Cr and Al were removed almost completely by adjusting the pH of the leachate to 3.8 with the addition of a neutralizing agent (CaCO₃). As a result, the Ni and Co concentration

| Sample                      | Metal concentrations, mg/L |
|-----------------------------|-----------------------------|
|                            | Al  | Co  | Cr  | Fe  | Ni  |
| Leach liquor after HPAL     | 1206|  89 | 108 | 5689| 550 |
| Leachate after neutralizing |  29 |  80 |  85 |  322|  623|
| Leachate after sulfuration  |   3 |   0 |   0 |   0 |   1 |
| Precipitation obtained      |   5 | 130 |   0 | 134 | 885 |
in the purified leachate (liquor) was 60% and 98% while the concentration of Fe and Al was lower than 0.2% and 1%, respectively. The vast majority of Co (>98%) and Ni (55%) from the purified liquor was precipitated as a mixed-sulfide form by adjusting the NaHS/(Co + Ni) ratio to 3 by the addition of sulfuration agent (NaHS) at the solution pH of 3.8 under the optimum conditions. It is proposed that the efficient recovery of Ni and Co from low grade laterite ores can be achieved using the pressure leaching (autoclave leaching) and neutralization followed by sulfuration treatment.

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