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Extraction of Nickel and Cobalt from Sulawesi Limonite Ore in Nitric Acid Solution at Atmospheric Pressure

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Abstract. Characterization and leaching study of a limonitic laterite ore from Sulawesi has been undertaken in order to find out mineralogical nature and Ni and Co concentration along with dissolution behavior during leaching process in nitric acid. Extraction rates of Ni and Co in different acid concentration and reaction time were also assessed. Mineralogical identification was conducted using optical microscopy and X-ray diffraction (XRD) techniques; whereas Ni and Co content was determined by means of atomic absorption spectrometry (AAS). Results of analyses show that minerals found in lateritic nickel ore studied were dominated by goethite with less quartz, talc, and trace spinel. Goethite was found to be high dissolution rate in nitric acid whereas quartz, talc and spinel were not dissolved during the leaching process. The highest extraction rates of Ni and Co with different acid concentrations at 1 hour leaching time were 13.22% and 24.17% respectively; whereas the maximum Ni and Co extraction rates under 2 molar nitric acid with different leaching times were obtained at 90 minutes with recovery of 24.17% and 56.53% respectively.

1. Introduction

Lateritic nickel ore is formed by chemical weathering of ultramafic rocks situated in the tropical regions. Ore types are classified on the basis of their mineralogy namely: hydrous silicate, clay silicate and oxide ores [1-3]. Global nickel reserves in 2018 reached up to 89 million tonnes, of which about 21 million tonnes or 23.59% originated from nickel laterite deposits in Indonesia [4]. Nickel laterite deposits in Indonesia are distributed in Eastern Sulawesi, Halmahera and West Papua.

At present, nickel laterite ores in Indonesia are processed by using conventional smelting to produce nickel matte and ferro nickel. However, smelting process requires ore with higher grade (>1.5 wt%) of Ni to be economic viable. Therefore, lower grade ores, generally limonite, are not profitable to be processed and they are discarded to the waste dump. Hydrometallurgical route is known to be an alternative option in processing the lower grade Ni-ore so that resources of deposit will be expected to increase. Some advantages associate with using leaching method such as operating at lower temperature and some other valuable metals can be recovered [5]. Some leaching studies on lateritic nickel ores for both saprolite and limonite from Indonesia have been carried out using different leaching agents such as sulfuric acid [6], hydrochloric acid [7], nitric acid [8] and citric acid [9].

The objective of this paper is to find out mineralogical and chemical nature of a limonite ore sample, to analyze dissolution behavior of this Ni-laterite ore and to estimate recovery of Ni and Co during leaching process using nitric acid at atmospheric pressure condition.



2. Sample and Methods

Nickel laterite ore sample used in this study was collected from limonite zone of an abandoned mine in the Latowu village, Batuputih subdistrict of North Kolaka Regency, Southeast Sulawesi Province (Fig.1). The mineralogy of the sample including texture, abundance and types of minerals was initially characterized by means of polarizing microscope (Nikon Eclipse LV100POL) and X-ray diffractometry (XRD; Shimadzu Maxima X-7000); whereas chemical compositions of the sample mainly Ni and Co were analyzed using atomic absorption spectrometry (AAS).

Leaching experiment was performed by using magnetic stirrer (LSH) with the temperature of 85°C, grain size of 100#, liquid to solid ratio of 10:1 and stirring rate 250 rpm. Leaching process was carried out in nitric acid (HNO₃) with different concentrations of 0.5; 1.0; 1.5; 2.0; 2.5; and 3.0 M and leaching time of 30; 60; 90; 120; 150 and 80 minutes. After leaching, pregnant solutions and solid residues were separated by membrane filter with diameter 0.45 micron. Pregnant leach solutions (PLS) were further analyzed by AAS whereas solid residues were characterized by XRD.

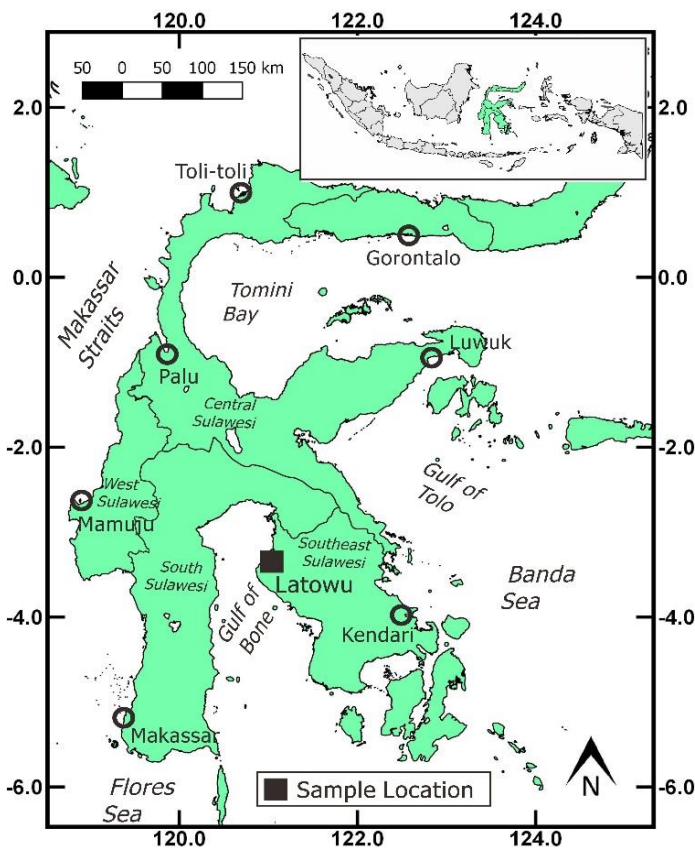


Figure 1 Map showing sample location

hydrothermal or weathering process of mafic minerals mainly pyroxene. Spinel was also identified in sample in trace amount. It is shown by bright in color with unihedral crystal.

Result of chemical analysis of sample by means of AAS shows that sample contains 1.23% Ni. This value indicates low grade so that the ore is not amenable to be processed by means of conventional smelting due to the high cost in operation. Majority of mining companies in Indonesia exploit the Ni laterite ore with minimum of 1.50% Ni. Meanwhile, sample contains about 1.23% Co, indicating relatively high grade. The mean grade of Co in limonite ore is generally about 0.10%.

3. Results and Discussion

3.1 Ore Characterization

Photomicrographs of a limonite sample are depicted in Figure 2, while the XRD pattern is shown in Figure 3. Results of microscopic observation exhibits that sample predominantly contains goethite [α -FeOOH]. The occurrence of goethite was assigned to the irregular and acicular texture. It shows reddish brown and locally yellowish in color. Goethite was also detected with the presence of basal spacing with $d_{hkl} \sim 4.18 \text{ \AA}$; 2.69 \AA and 2.45 \AA .

Other minerals identified in association with goethite were quartz [SiO_2], talc [$\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$] and spinel [$\text{MgAlCrSi}_2\text{O}_4$]. Microscopic appearance of quartz was characterized by darker in color with relatively rounded in texture. The presence of basal reflections with d_{hkl} values of 4.25 \AA ; 3.34 \AA and 1.82 \AA indicate positive identification of quartz. Talc was detected in sample based on XRD data with reflections intensity occurring at d_{hkl} values of 9.40 \AA ; 4.70 \AA and 3.12 \AA . Talc is a phyllosilicate mineral having 2:1 structure. It might be formed by

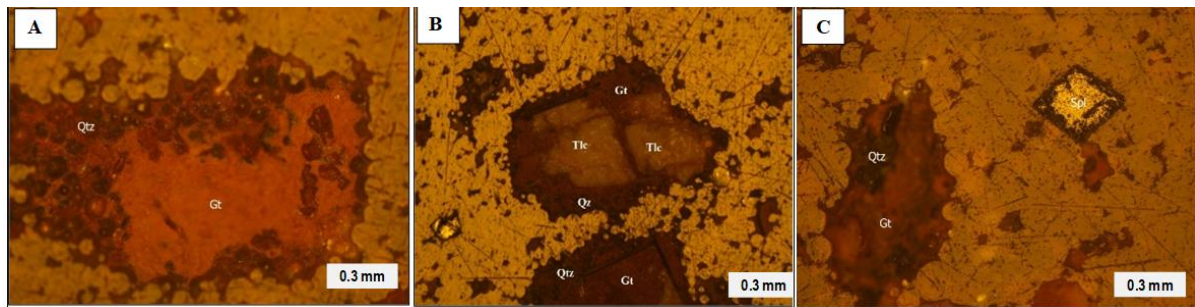


Figure 2. Photomicrographs of limonite ore sample showing the irregular texture of goethite (Gt) in association with quartz (Qtz), (A). Talc also present with goethite replacing the rim (B). Euhedral spinel, possibly chromite still preserves the crystal form, however the color starts to change on the margin of spinel crystal into goethite (C)

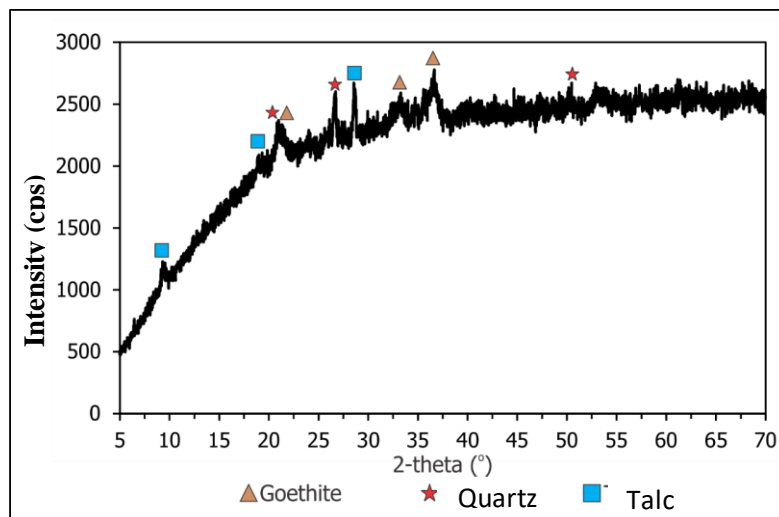


Figure 3. X-ray powder diffraction pattern of limonite ore sample used in this study

3.2 Dissolution Behavior of Limonite Ore

The XRD patterns of some solid residues after leaching of limonite ore in nitric acid solution are provided in Figure 4. It was shown that reflection intensity with $d_{hkl} \sim 4.16 \text{ \AA}$ decreases after leaching process. It indicated that goethite as Ni and Co bearing mineral in limonite ore partially dissolves during reaction with nitric acid which is called incongruent dissolution. In contrast, intensity of peaks with $d_{hkl} \sim 9.30 \text{ \AA}$ and $\sim 3.12 \text{ \AA}$ strongly increases as compared with the peaks at raw ore sample. This indicates that talc did not dissolve. Similarly, quartz with basal spacing occurs at $d_{hkl} 3.34 \text{ \AA}$ exhibits the increasing intensity in solid residue.

Dissolution rate of minerals in acid solution is affected by some factors such as crystallinity, impurities, grain size and porosity [10]. Goethite is characterized by poorly crystalline minerals as the result of chemical weathering of ultramafic rocks. Goethite structure is formed by hexagonal closed-packing arrangement of O^{2-} and OH^- anions, stacked with Fe^{3+} occupying half of octahedral site [11]. Isomorphous substitution of divalent cations (Ni, Co and Zn) in goethite is not only affecting its physical properties such as color, unit cell dimension and magnetic susceptibility but also they can modify the solubility and dissolution kinetics. Crystal shape, crystal size, and crystallinity may also influence the dissolution rate of goethite.

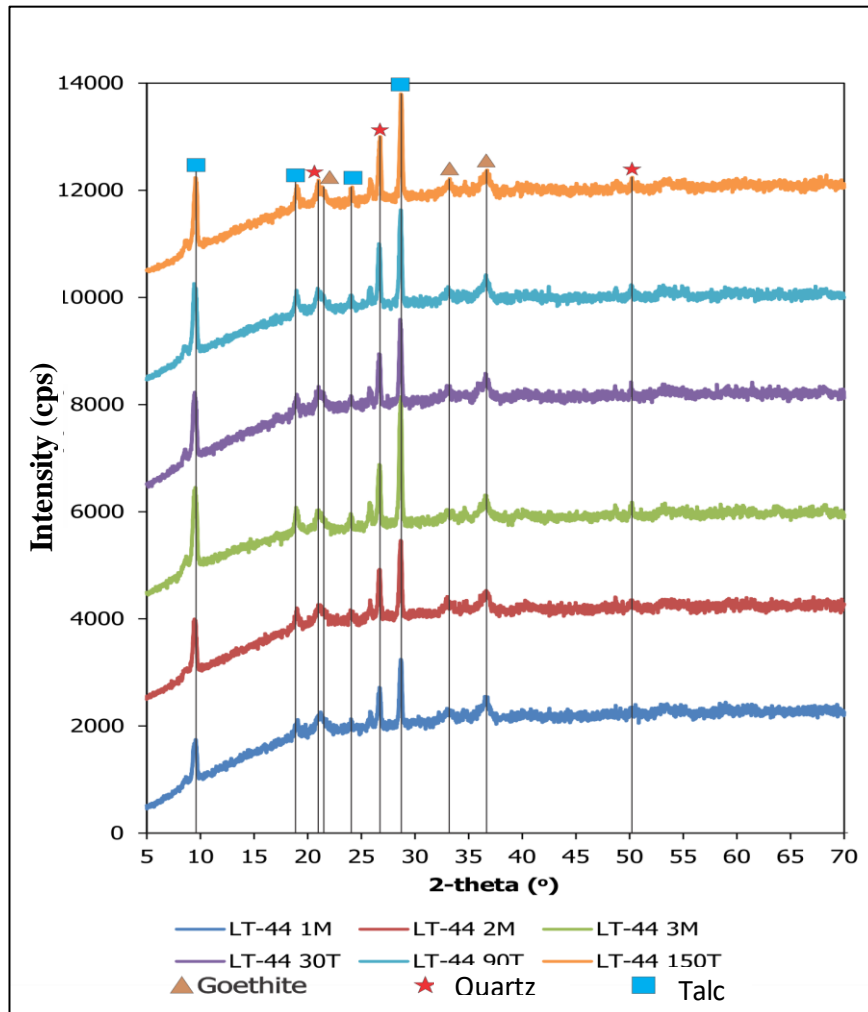


Figure 4. X-ray powder patterns of solid residues after leaching at different concentrations and reaction times

It is suggested that Ni and Co is generally present in goethite in three modes: (i) associated with poorly crystalline or amorphous goethite, (ii) adsorbed onto goethite surface, and (iii) substituted for Fe in goethite structure. Nickel and cobalt atom that located in the single coordination hydroxyl groups would be easier to liberate by acid than in double or triple coordination hydroxyl groups [12].

In the case of talc, quartz and spinel, these minerals do not not expected to be the host of Ni and Co. Hence, they are not necessary to dissolve during leaching process.

3.3 Extraction of Ni and Co from Limonite Ore

The influence of nitric acid concentration on extraction rate of Ni and Co was investigated at temperature 80°C and leaching time of 1 hours. It is shown that with an increase of HNO₃ concentration, followed by the increase of extraction rate of Ni and Co (Figure 5). However, leaching rate of Co was more twice than Ni. Cobalt likely present in association with manganese wad or asbolane. During leaching process, Co and Mn were released from host minerals. Eventually, Co extraction from laterite ore is easier than Ni due to the high concentration of Fe and relatively easy dissolution of manganese in acid [13].

The effect of leaching time on the extraction rate of Ni and Co was also investigated. As shown in Figure 6, it was indicated that the highest extraction rate for both Ni and Co took place at 90 minutes reaction in 2 M nitric acid. Maximum 24.17% of Ni extraction rate were reached at these conditions. This value is far lower than extraction rate of Co which attain up to 56.53%. However, the increase of

leaching time was not accompanied by the increase in extraction rate, even it shows strongly decrease in leaching rate either Ni or Co at 120 minute reaction (Figure 6). Extraction rates of Ni and Co reveal slowly depletion after 2 hours reaction.

Results of leaching experiment exhibit that extraction rates of Ni and Co were still low. This might be due to incomplete dissolution of goethite as Ni and Co carrier. It is therefore suggested that dissolved Ni and Co in pregnant leach solution (PLS) most likely derived from adsorbed metals on goethite surface. Another possibility of low extraction rates of Ni and Co in this experiment is that formation of fine grained silica during laterization process or formation of amorphous silica during leaching process might readsorb Ni and Co from PLS. Cobalt extraction was much better than nickel recovery in this experiment.

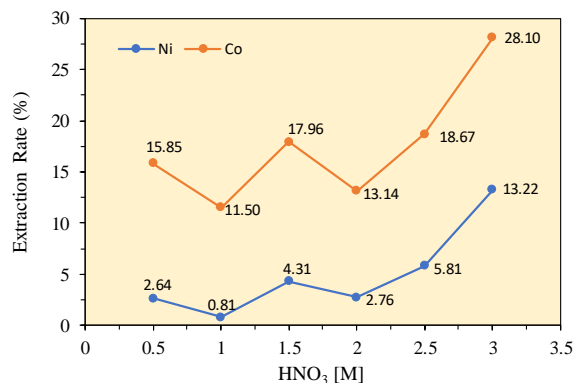


Figure 5. Graph displaying the influence of nitric acid concentration on the extraction rates of Ni and Co from limonite ore

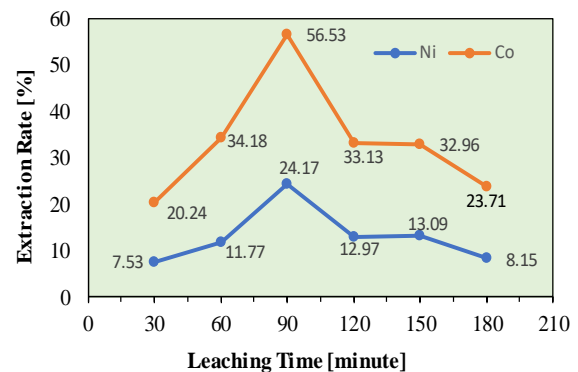


Figure 6. Graph showing the effect of leaching time on the extraction rates of Ni and Co

4. Conclusions

Based on analysis and discussion in previous section, some conclusions can be drawn as the following:

1. Studied limonite ore contains mainly goethite [α -FeOOH] with subordinate quartz, talc and trace spinel.
2. Goethite as Ni and Co bearing phase shows partial dissolution in nitric acid leading to release less nickel and cobalt from goethite crystal, in contrast, quartz, talc and spinel did not indicate dissolution.
3. The highest extraction rate of Ni was 24.17 % more than twice lower than Co extraction of 56.53% which were achieved with using 2 M HNO₃ at 90 minutes reaction. However, Co recovery was much higher than Ni.

Acknowledgement

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