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The Analysis of Dissolution Reaction Mechanism of Antigorite, Goethite, and Nickel Oxide from Laterite Ore in Chloric Acid Solution

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Abstract : This work is aimed to reveal the profile of milled laterite ore and the dissolution mechanism of antigorite, goethite, and nickel oxide during leaching of laterite ore in chloric acid. Laterite ore from South East Sulawesi, Indonesia, which is the product of weathering of earth's mantle's upper part, consists mainly of antigorite, goethite, quartz, and nickel oxide. The laterite ore was analyzed by using Scanning Electron Microscope/ Energy Dispersive X-Ray Spectroscopy (SEM/EDS), X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF) to obtain the characteristic of milled ore. The milled laterite ore was leached in hydrochloric acid solution at different temperature and acid concentration. The solid parts of leached samples were analyzed by using XRD and XRF to reveal the phases and the % weight of iron and nickel within the samples, respectively. It is found that in the milled laterite ore, antigorite, goethite, and nickel oxide were not chemically and physically associated. The path of antigorite, goethite, and nickel oxide dissolution reaction with chloric acid is independent of one another. The dissolution reaction of antigorite produced significant solid magnesium hydroxide chloride at 50 ^{o}C as an intermediate product before antigorite was fully being dissolved. Meanwhile, goethite was dissolved significantly at 50 ^{o}C and above, whereas and nickel oxide was dissolved at 30 ^{o}C and above. The dissolution of one-mole antigorite and one-mole goethite consumes nine-mole of hydrochloric acid molecules.

Keywords : laterite, goethite, antigorite, leaching, nickel oxide.

Introduction

Laterite deposit is among the product of weathering of the earth's mantle's upper part.[1] The laterite ore consists of serpentine, goethite, quartz, nickel oxide, and other minor elements.[2-4] The laterite can be processed through the hydrometallurgical route of process. This process requires less energy than the pyrometallurgical process.[5]One of the important stages in the hydrometallurgical process is the leaching stage. The leaching stage

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is the dissolution of laterite ore elements in certain acids. Many published paper related with laterite leaching reports only the dissolution of iron or nickel without analyzing the reaction sequence involving other minerals in laterite.[3,6-8] In this paper, we aim to reveal the reaction sequence during the leaching of laterite ore. The laterite ore was leached in chloric acid solution at various temperatures and concentrations. Scanning Electron Microscope (SEM) was used to reveal elements distribution in the milled ore. The milled and leached ore samples were analyzed by using X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF) to trace the reactions happening during leaching. The result of SEM, XRD and XRF analysis will be the basis of the revealing of the mineral property, reaction sequence, and the effect of temperature and concentration on leaching process.

Experimental

The materials used in the experiment are laterite ore, which was obtained from South East Sulawesi, Indonesia, and chloric acid (HCl p.a., Merck). Laterite ore was dried for 24 hours before milling for 6 hours. The milling equipment used to mill laterite ore is the ordinary tumbling mill. The mill vial was made of stainless steel with an inner diameter of 10 cm and a length of 20 cm. The mill vial was filled with 13 steel balls having an average size of 1.5 cm. The milled powder was screened to give the particle size of 200 mesh (74 microns). The milled powder was analyzed by using Scanning Electron Microscope/Energy Dispersive X-Ray Spectroscopy that (SEM/EDS, Rigaku, Japan) to obtain the mapping data of elements in the milled ore. The milled laterite ore powder was leached in chloric acid solution at a temperature of 30, 50, 70, and 90 ^{o}C, with a chloric acid concentration of 4M. Another experiment was conducted at a chloric concentration of 1.72, 2.4, 4, and 6 M, with a leaching temperature of 90 ^{o}C. After leaching, the leached laterite ore was separated from the solution by using a filtration paper. The leached laterite ore residue is dried for 24 hours before being analyzed by using X-Ray Diffraction (XRD, Shimadzu 7000, Japan) and X-Ray Fluorescence (XRF, Thermo Niton XL3t) to reveal the phases and the % weight of nickel and iron in the leached laterite ore, respectively. The voltage and current of the X-Ray tube of XRD equipment were 40 kV and 30 mA, respectively. The samples were irradiated with CuK-alpha radiation at sampling pitch and scanning speed of 0.02 degrees and 2.4 degrees/minutes, respectively.

Result and Discussion

The chemical composition of laterite that was used in the experiment is shown in Table 1 and the XRD patterns of as recived laterite ore is shown in Figure 1. A significant amount of iron, nickel, magnesium, silicon, and oxygen composes the laterite, as well as other minor elements. XRD pattern shown in Figure 1 reveals that the laterite is composed of Antigorite ($Mg_3Si_2O_5(OH)_4$, ICDD no. 01-089-6205), Goethite (FeOOH, ICDD No. 01-061-0402), and quartz (SiO_2, ICDD no. 03-065-0466).

Table 1.Chemical composition of nickel laterite ore from South East Sulawesi, Indonesia

Element	Fe	Ni	Cr	Mn	Co	Cd	Pd	Zn	K	Balance
% Weight	30.11	3.30	0.80	0.56	0.15	0.11	0.06	0.04	0.17	64.07

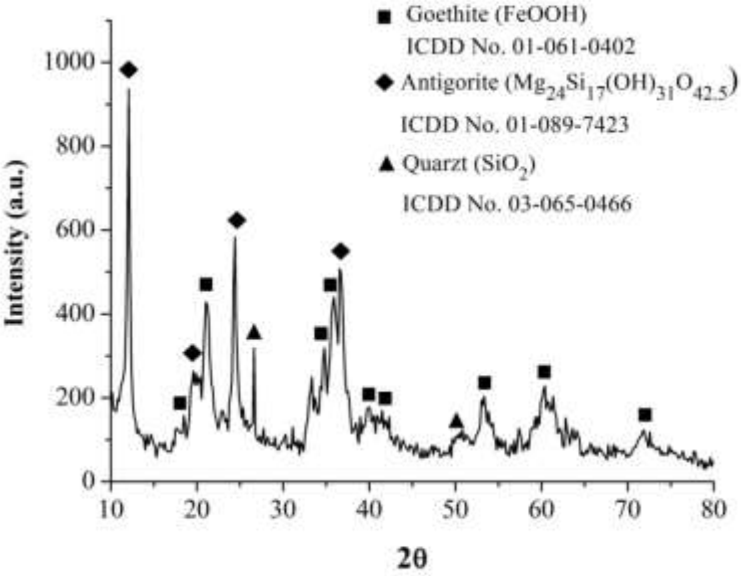

Figure 1. XRD pattern of the as-received laterite ore

Antigorite is one of the minerals in the Serpentine Group that has an alternating wave structure with a superlattice along the a-axis.[9] Goethite is one of the most widespread forms of iron oxides in terrestrial soils, sediments, and ore deposits.[10] The oxygen and hydroxide anions in Goethite are arranged as a hexagonal close-packed structure with Fe3+ cations situated on octahedral interstitial sites.[11] Quartz is an abundant mineral in earth crust; it can be found in igneous, metamorphic, and sedimentary rocks.[12] SiO_2 was formed by primary and secondary magmatic, hydrothermal or sedimentary processes or during diagenesis and metamorphosis.[13] The serpentine group minerals (including antigorite), goethite, and quartz are among the minerals that compose laterite ore from Indonesia.[2-3] Serpentine and quartz are among the minerals that were reported to exist in nickel laterite ore, but different authors report the different forms of iron-bearing minerals, some of which reported that the iron-bearing minerals in laterite are magnetite and hematite, whereas the other reported that goethite is the only form of iron-bearing mineral in laterite.[2-3,14-16] The investigation of our sample, through XRD analysis, has found that iron in laterite is in the form of goethite. Laterite is the product of intense sub-aerial weathering of rock that makes the content of iron and or aluminum higher than the parent rock, whereas silicon content is lower than that of the parent rock.[17] The lateralization divides soil into six horizon layers; one of them is the so-called mottled layer.[18] In the beginning, this layer consists of sodium aluminum silicate group minerals and goethite. The weathering of this layer causes the chemical reaction that converts some part of goethite into hematite, which is indicated by the formation of the mottled spots. The Gibbs free energy formation of hematite is negative and lower than that of goethite, which reveals that the formation of hematite from goethite is thermodynamically feasible.[19] In this particular reaction, heating is needed to accelerate the reaction; i.e. without heating, the reaction rate will be very low.[20] Therefore, the conversion reaction of goethite into hematite in nature is not rapid; it takes years to convert goethite into hematite. These facts lead to the strong assumption that the difference in the result of laterite analysis in the previous researches is possibly caused by the different reaction progress of goethite to hematite conversion in the laterite layer at the particular location where the sample was obtained.

The leaching of laterite in the chloric acid solution dissolved some elements in laterite as shown in Figure 2 and Figure 3. These figures also show that the leaching behavior of laterite in the chloric acid solution was affected by the leaching temperature and concentration of acid. The XRD peaks intensity corresponding to antigorite and goethite decreased after the laterite was being leached at 30 °C. It indicates that at 30 °C some parts of antigorite and goethite had dissolved. The new peaks with significant intensity corresponding to $Mg_3Cl_2(OH)_4.2H_2O$ (ICDD no. 00-012-0133) shown up. It is the product of a reaction involving antigorite, which can be written as follows.

$2Mg_3Si_2O_5(OH)_{4(s)}+4HCl_{(aq)} \rightarrow 2Mg_3Cl_2(OH)_4.2H_2O_{(s)} +4SiO_{2(s)}+ 5H_2O$ (1)

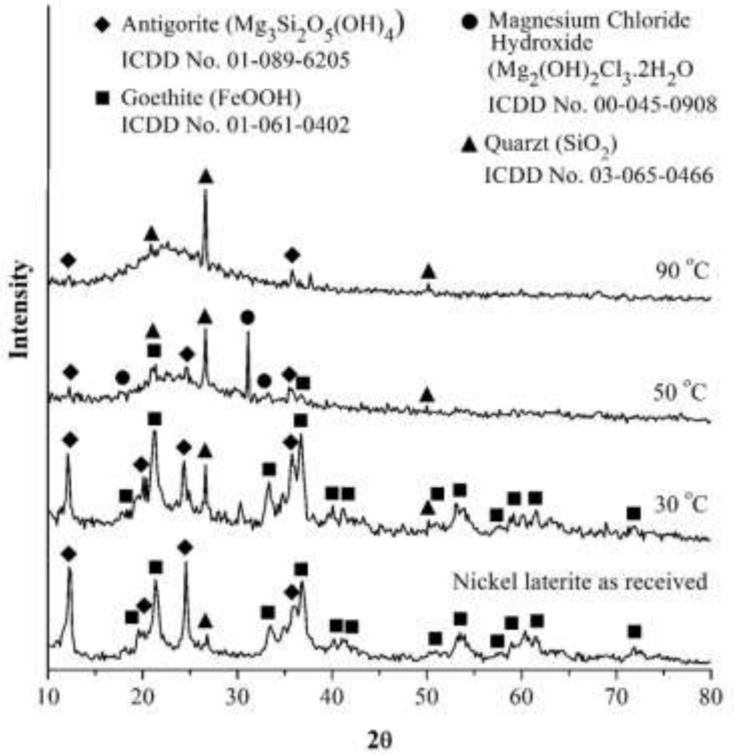

Figure 2. XRD patterns of laterite ore and those that were leached at 30, 50, and 90 °C (HCl Concentration = 4M, leaching time = 4 hrs)

As leaching temperature increased up to 50 °C, the intensity of peaks corresponding to antigorite decreased down to a very low level. At the same time, the peaks corresponding to $Mg_3Cl_2(OH)_4.2H_2O$ disappeared, and the new peaks corresponding to $Mg_2(OH)_3Cl_3.2H_2O$ (ICDD no. 00-045-0908) appeared. The decreasing of peaks corresponding to antigorite indicates that reaction (1) took place rapidly. The disappearance of peaks corresponding to $Mg_3Cl_2(OH)_4.2H_2O$ and the appearance of new peaks corresponding to $Mg_2(OH)_3Cl_3.2H_2O$ indicates that $Mg_3Cl_2(OH)_4.2H_2O$ reacted to become $Mg_2(OH)_3Cl_3.2H_2O$, in which the reaction can be written as follows.

$4Mg_3Cl_2(OH)_4.2H_2O_{(s)} + 10HCl_{(aq)} + 3O_2 \rightarrow 6Mg_2(OH)_3Cl_3.2H_2O_{(s)}$ (2)

The increasing of leaching temperature up to 90 °C leads to the dissolution of the entire phases, leaving quartz as the main phase in the XRD patterns of the leached laterite ore. The dissolution reaction that follows reaction (2) can be written as follows.

$6Mg_2(OH)_3Cl_3.2H_2O_{(s)}) + 6HCl \rightarrow 12MgCl_2 + 24H_2O + 3O_2$ (3)

Reaction (1), (2), and (3) can be integrated to obtain the single reaction that represents the all initial reactants and all final reaction products. Reaction (1), (2), and (3) can be integrated to produce the following reaction.

$Mg_3Si_2O_5(OH)_{4(s)} + 6HCl_{(aq)} + H_2O \rightarrow 3MgCl_{2(aq)} + 6H_2O + 2SiO_{2(s)}$ (4)

The solid product of reaction (4) is SiO_2. The intensity of peaks corresponding to SiO_2 consistently increases as leaching temperature increases from 30 to 90 °C. The XRD database confirms that the crystal structure of this SiO_2 is quartz, but on the other hand, quartz is also the mineral that was already existed in the as-received laterite ore. There are no reports about the formation of quartz at low temperature; and quartz can only be formed at 970-1100 oC with amorphous SiO_2 as starting material.[21] Besides, other researchers have reported that only amorphous SiO_2 was formed through decomposition and precipitation reaction involving the compounds that contain Si-O network in aqueous solution at low temperature.[22-27] The appearance of a broadening peak in the XRD patterns of leached samples indicates the formation of an amorphous phase, which can be attributed to amorphous SiO_2. This amorphous SiO_2 is an actual product of reaction (1). This makes the crystalline quartz SiO_2 shown in Figure 1 is not the product reaction of reaction (1). The crystalline quartz SiO_2 is the mineral that has already existed in the as-received laterite ore. Therefore, the increasing of the intensity of peaks corresponding to crystalline quartz SiO_2 is merely the result of the decrease and disappearance of the intensity of peaks corresponding to other phases.

FeOOH is also a mineral that conducted a reaction during the leaching process of laterite ore. The leaching of laterite at 30 ^oC increased the intensity of peaks corresponding to FeOOH, but since FeOOH is not the product of any reaction, the increase of the intensity of peaks corresponding to FeOOH is not the indication of the increasing of FeOOH, The increasing of FeOOH is merely caused by the decreasing of peaks corresponding to $Mg_3Si_2O_5(OH)_4$. The decomposition reaction of FeOOH took place rapidly at temperature 50 ^oC, which is indicated by a significant decrease of the intensity of the peak corresponding to FeOOH. Since the intensity of peaks corresponding to ferrous or ferric phase does not appear in the XRD pattern of the sample being leached at 50 °C, the product must be a water-soluble ferrous phase, such as $FeCl_3$. The reaction involving FeOOH can be written as follows.

$$FeOOH_{(s)} + 3HCl_{(aq)} \rightarrow FeCl_{3(aq)} + 2H_2O \qquad (5)$$

As temperature increases up to 90 ^oC, reaction (5) took place more rapidly, resulting in the complete dissolution of iron in FeOOH into the chloric acid solution.

Figure 3 shows the XRD patterns of laterite ore leached at 90 oC in various concentrations of hydrochloric acid. The figure shows that the dissolution of $Mg_3Si_2O_5(OH)_4$ and FeOOH become rapid as hydrochloric acid increases. At a low concentration of hydrochloric acid (1.72 M HCl), a significant amount of $Mg_3Si_2O_5(OH)_4$ and a large amount of FeOOH seem to have remained uncreated. The increasing of chloric acid concentration up to 2.4 M and 4 M decrease the intensity of peaks corresponding to $Mg_3Si_2O_5(OH)_4$ and FeOOH rapidly, and at chloric acid concentration 6 M those peaks have disappeared, leaving only crystalline SiO_2 (quartz) and amorphous SiO2.

The summation of reaction (4), and reaction (5) is presented as equation (6). The reaction shows that it takes nine moles of hydrochloric acid to dissolve each one mol of $Mg_3Si_2O_5(OH)_4$ and FeOOH.

$$FeOOH_{(s)}+Mg_3Si_2O_5(OH)_{4(s)}+9HCl_{(aq)} \rightarrow FeCl_{3(aq)}+3MgCl_{2(aq)}+2SiO_{2(s)}+7H_2O \qquad (6)$$

A large amount of hydrochloric acid is needed to leach laterite ore. The continuity of the process depends on the availability of hydrochloric acid during the leaching process. At low chloric acid concentration (1.72 M), it seems that the availability of chloric acid is not enough to dissolve $Mg_3Si_2O_5(OH)_4$ and FeOOH entirely. The lack of chloric acid also makes $Mg_2(OH)_2Cl_3.2H_2O$ could not be dissolved. $Mg_2(OH)_2Cl_3.2H_2O$ was supposed to dissolve in chloric acid at 90 ^oC. The unreacted $Mg_2(OH)_2Cl_3.2H_2O$ indicates that chloric acid was not available to dissolve $Mg_2(OH)_2Cl_3.2H_2O$. The increase of chloric acid concentration makes the chloric acid available to complete the leaching reactions. At chloric acid concentrations 2.4 and 4 M, $Mg_3Si_2O_5(OH)_4$ decrease rapidly down to the lowest level, and at chloric acid concentration 6 M, $Mg_3Si_2O_5(OH)_4$ dissolved completely. Meanwhile, the amount of FeOOH also decreased rapidly down to the lowest level and dissolved completely at chloric acid concentrations 2.4 and 4 M, respectively.

The reaction path of each phase in the laterite ore leaching can also be traced by observing iron and nickel dissolution from the laterite ore. Figure 4 shows the % weight of iron and nickel in the laterite ore after being leached at temperatures 30 - 90 ^oC. Iron was slightly dissolved at 30 ^oC but it was rapidly dissolved as temperature increase up to 50 and 90 ^oC. Based on the Eh-pH diagram of iron and nickel, the area of nickel ion is larger than that of iron.[28]This indicates that nickel is more soluble than iron. Figure 4 confirms that nickel is more soluble than iron. The iron dissolution pattern shown in Figure 4 is agreed with the intensity change of peaks corresponding to FeOOH (shown in Figure 2), i.e. the change in the XRD intensity of peaks corresponding to FeOOH in Figure 2 is agree with the change iron content in Figure 4.

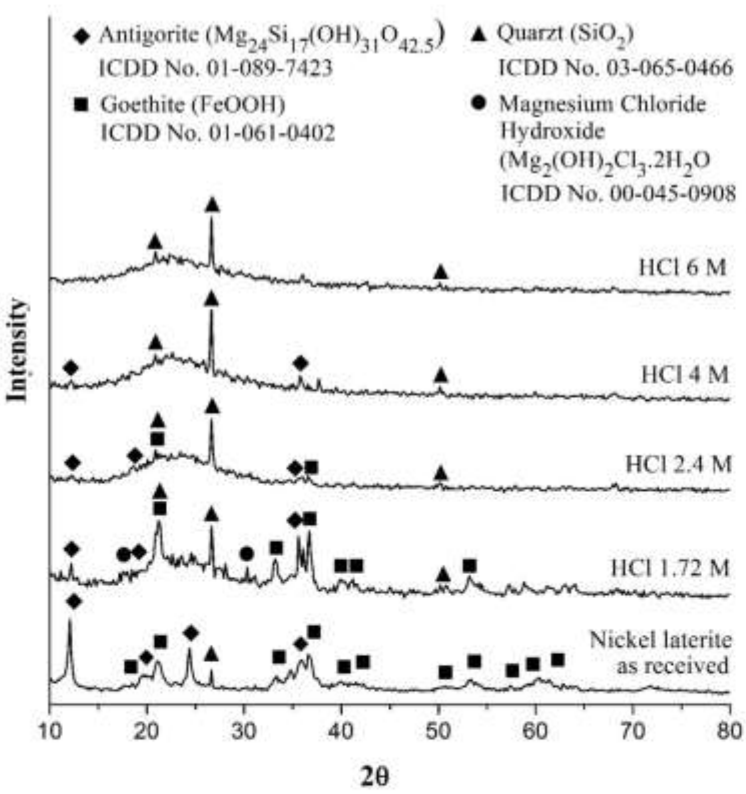

Figure 3: XRD patterns of laterite ore and those that were leached in HCl at concentration of 1.72, 2.4, 4, and 6 M (Leaching temperature = 90 °C, leaching time = 4 hrs)

The content of nickel in laterite ore is very little, therefore the peaks of XRD intensity corresponding to nickel-bearing minerals cannot be found in the XRD patterns of laterite ore. Although the nickel reaction mechanism during leaching cannot be traced through XRD analysis to the leached laterite ore, it can be traced through the analysis of the nickel element in the leached ore. Our previous research work dealing with the mapping analysis of elements in laterite has reported that nickel scattered in laterite ore as nickel oxide (NiO).[5] Nickel did not chemically associate with both antigorite and goethite, but the nickel might associate physically with goethite.[5] The comminution process of laterite ore through milling is believed to have a role in releasing nickel oxide that was physically attached to goethite As shown in Figure 4, the dissolution of nickel at 30 °C was more rapid than that of iron. It indicates that in the milled laterite ore, nickel oxide was not chemically associated with goethite, and nickel scattered independently in laterite ore as nickel oxide. Thus, reaction (4), reaction (5), and the dissolution reaction of nickel oxide took place independently with different reaction speeds.

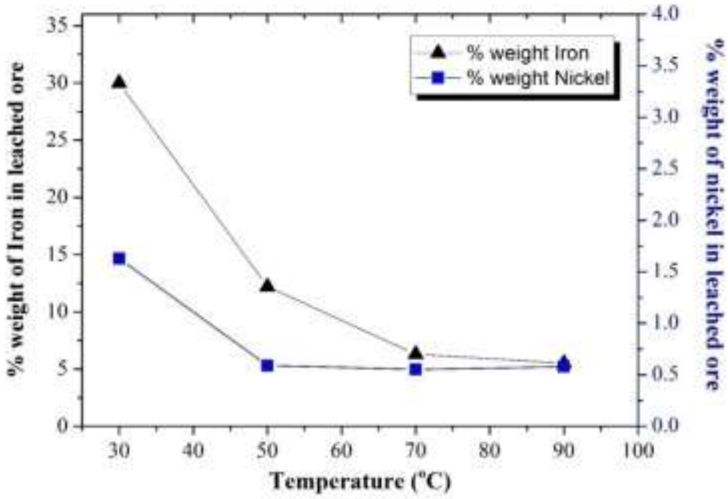

Figure 4: Weight percentage of iron and nickel in the solution as a result of laterite ore leaching at different temperatures

Figure5 shows the % weight of iron and nickel in the laterite ore after being leached at 90 °C by using different concentrations of hydrochloric acid. At hydrochloric acid concentration 1.72 M, iron dissolved slightly, whereas nickel dissolution has already reached its maximum value.

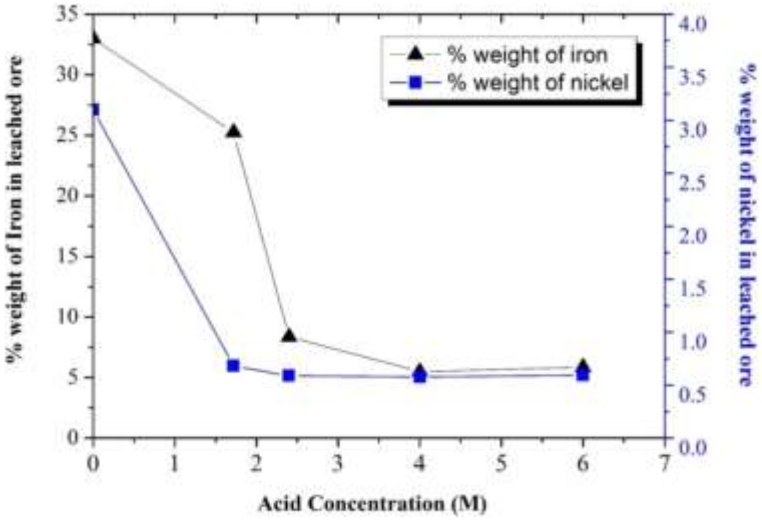

Figure 5. Weight percentage of iron and nickel leached in different concentration of hydrochloric acid

Iron dissolution increases significantly as hydrochloric acid concentration was increased up to 2.4 M. The maximum dissolution of iron was reached at hydrochloric acid concentration 4 M and beyond. Since XRD patterns in Figure 2 and Figure 3 indicate that antigorite seems to be more reactive than goethite, the reaction of antigorite and hydrochloric acid (reaction (4)) takes place before that of goethite (reaction (5)). Reaction (4) states that it takes six moles of hydrochloric acid to dissolve one mol of antigorite. At low hydrochloric acid concentration (1.72 M), hydrochloric acid was not enough to conduct all reactions. The speed of reaction (4) (the dissolution of antigorite) is higher than that of reaction (5) (the dissolution of goethite). Therefore, hydrochloric acid was mainly consumed to dissolve antigorite. The dissolution of goethite could be conducted rapidly when the acid was still available after that antigorite was being leached.

Conclusion

The laterite ore from South East Sulawesi Indonesia mainly consists of serpentine (one of them is antigorite), goethite, quartz, and nickel oxide. Depending on the atmospheric condition, goethite can be transformed to hematite in some particular location. Antigorite, goethite, and nickel oxide might be associated physically, but they are not chemically associated. The milling of laterite ore releases the physical association among these minerals. During the leaching process, antigorite, goethite, and nickel oxide dissolve with different reaction speeds in chloric acid. Magnesium hydroxide chloride is the solid intermediate product of antigorite reaction with chloric acid before the full dissolution of antigorite. The significant goethite dissolution takes place at a temperature of 50 ^oC and above, whereas the significant dissolution of nickel oxide takes place at a temperature of 30 ^oC and above. The dissolution of laterite ore needs a large amount of hydrochloric acid; it takes nine moles of hydrochloric acid to dissolve one mole of antigorite and one-mole goethite. The lack of hydrochloric acid in the leaching of laterite ore at a temperature of 90 ^oC and above is the only reason for the low dissolution of antigorite and goethite in laterite ore.

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