Leaching Kinetics and Mechanism of Laterite with NH$_4$Cl-HCl Solution

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Received: 10 July 2020; Accepted: 21 August 2020; Published: 26 August 2020

Abstract: Following the growing demand for Ni and Co and the dwindling supplies of sulfide nickel ore, attention has turned toward the more efficient exploitation and utilization of laterite ore. Using ammonium chloride acid solution to leach is an effective method. Our research concerned investigations on the leaching mechanism and leaching kinetics of laterite. XRD was used to demonstrate the leaching mechanism through analysis of the pattern of the leaching residue and raw ore, showing that acid concentration affects the leaching process more significantly than other factors, and that valuable metals are mainly released from goethite and serpentine. The leaching order of these materials are as follows: Goethite > serpentine > magnetite and hematite. The leaching kinetics were analyzed and this leaching process followed a shrinking core model controlled by a combination of interfacial transfer and diffusion across the solid film. Leaching data fitted to the kinetic equation perfectly, and the apparent activation energies for the leaching of nickel, cobalt, and iron were calculated to be 4.01 kJ/mol, 3.43 kJ/mol, and 1.87 kJ/mol, respectively. The Arrhenius constants for Ni, Co, and Fe were 204.38, 16.65, and 7.12 × 10$^{-3}$, respectively, with reaction orders of Ni (a 1.32, b 0.85, c 1.53), Co (a 1.74, b 1.12, c 1.22), and Fe (a 2.52, b −0.11, c 0.94).

Keywords: laterite ore; leaching kinetics; mineral dissolution; chloride leaching; kinetic models

1. Introduction

Nickel ranks as the fifth most abundant element after Fe, O, Si, and Mg in the Earth’s crust, and nickel usually exists as oxides, sulphides, deep-sea nodules, and silicates [1]. Nickel resources with regards to land reserves are 0.47 billion tons and are present in two principal ore deposit types: 60% of sulfide ore and 40% of laterite ore [2]. Due to its special metallurgical advantages, nickel is a very important metal that is widely used in many industries, especially for the preparation of stainless steel and nonferrous alloys helping to increase corrosion resistance and impact strength [3]. Globally, nearly 85% of nickel is consumed by the alloy and stainless steel industry to impart various materials with certain physical and chemical properties [4,5]. The global demand and the price of nickel and cobalt has increased continuously over recent years, and the sulfide ores have gradually been depleted. Attention has thus been directed to the development of selective leaching from laterite ores [6–8]. In contrast to sulfide ores, laterite cannot be readily concentrated because of its low nickel content and the isomorphic goethite or serpentine crystalline structure. The production of nickel from laterites, therefore, usually requires more processing and consumes more energy, leading to greater extraction of nickel from sulfide [9–11].

In recent years, much attention has been directed toward the development of novel methods to exploit nickel laterite ores, such as hydrometallurgy, pyrometallurgy, and biometallurgy [12]. Due to economic reasons, sulfuric acid has been predominantly used. For example, the pressure leaching
(PAL) process and high-pressure leaching (HPAL) processes are the preferred methods used to leach nickel and cobalt from limonite laterites, the merit of these processes being that iron can be precipitated as hematite [13,14].

However, the PAL and HPAL processes have relatively high operational costs, require significant investment, and have high energy consumption at the commercial level compared with atmospheric pressure leaching (AL) [15]. The atmospheric pressure hydrometallurgical process uses sulfuric acid, hydrochloric acid, and citric acid to leach various types of nickel laterite ores [16,17]. Although the atmospheric pressure leaching processes successfully extract Ni and Co, these processes usually consume much more acid and leach a higher number of Fe and Mg purities, which impede their further application [18,19].

In contrast with other conventional hydrometallurgical and pyrometallurgical processes, chloride metallurgy processes have several advantages, such as the comparatively higher leaching from mixed ores, higher leaching selectivity, and easier regeneration of leaching reagents [20–23]. In order to determine and optimize the best processing conditions, kinetic analysis must be comprehensively conducted [4,24,25]. Thus, kinetic studies should be conducted to further determine the mechanism of leaching with ammonium chloride-hydrochloric acid solution, which has been presented in previous work, in order to provide a theoretical reference for its industrial application. In this paper, factors such as leaching time, acid concentration, and leaching temperature are taken into account for kinetic analysis. Mineralogical analyses of the raw ore and the different leaching residues with regards to leaching time, acid concentration, and leaching temperature were conducted to better elucidate the leaching behaviors of various metals. The leaching order of minerals is beneficial to the investigation of mineral stability and the influence of Ni and Co dissolution under the proposed leaching conditions.

2. Experimental and Analytical Methods

2.1. Materials

The tested samples were obtained from Yuanjiang Laterite deposit, which consists of various mixed ores obtained at different surface depths. The ores should be dried and crushed before the leaching. In this paper, the tested samples were sieved through 150 mesh and a particle size fraction of 0.074–0.15 mm was used as materials for leaching experiments. The content of the different elements in the laterite sample are shown in Table 1.

| Elements | Ni  | Co  | Mn  | Fe  | Cu  | Ca  | Mg  | Al  | Si  | Na  |
|----------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Content% | 1.15| 0.08| 0.35| 14.06| 0.07| 0.12| 29.35| 0.34| 23.13| 0.26|

Mineralogical analysis (Figure 1) showed that the main minerals were serpentine \( \text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4 \), silica \( \text{SiO}_2 \), and some iron oxides including magnetite (\( \text{Fe}_3\text{O}_4 \)), hematite (\( \text{Fe}_2\text{O}_3 \)), and goethite (\( \text{FeO(OH)} \)). Although there is not a single nickel mineral, nickel usually exists through surface adsorption and lattice replacement in laterite ore [4].
was determined via potassium permanganate titration methods. Hydrochloric acid solution at different concentrations was used as lixiviant with addition of ammonium chloride. The mineral samples were added into the solution and heated to a certain temperature. After some time, the residue and leaching solution were collected, respectively, through filtration. The leaching of Ni, Co, Mn, Mg, and Fe was determined under different leaching conditions.

2.3. Analytical Methods

All leaching experiments were performed in a three-necked flask, which was set in a water bath, and the stirring speed was fixed at 300 rpm. Hydrochloric acid solution was collected, respectively, through filtration. The leaching of Ni, Co, Mn, Mg, and Fe was determined using atomic absorption spectrophotometry. X-ray diffraction analyzer (Rint-2000, Bruker corporation, Karlsruhe, Germany) was used to determine the structure of raw ores and leaching residues. Atomic absorption spectrophotometry (Ruli-160) was used to determine the content of Ni, Co, and Mn in the leaching solution. The Fe content was determined via potassium permanganate titration methods.

3. Results and Discussion

3.1. Leaching Mechanism

On the basis of previous work, the optimal leaching conditions consisted of a temperature of 90 °C, ammonium chloride concentration of 3 mol/L, a hydrochloric acid concentration of 2 mol/L, a liquid–solid ratio of 6:1, and leaching time of 1.5 h [22]. The following experiments were based on this optimization condition. According to ore phase analysis (Figure 1), the majority of valuable metals in this laterite ore exists as composites of ferrite, silicate, or oxide in certain kinds of mineralogical phases such as goethite, layered montmorillonite, and serpentine. Therefore, it was essential to study the dissolution mechanism of these minerals to optimize the selective leaching process of laterite. Possible reactions concerning these valuable metals are as follows:

\[ \text{NiFe}_2\text{O}_4(s) + 2H^+ = \text{Ni}^{2+} + \text{Fe}_2\text{O}_3(s) + H_2O(l) \]  \hspace{1cm} (1)

\[ \text{CoFe}_2\text{O}_4(s) + 2H^+ = \text{Co}^{2+} + \text{Fe}_2\text{O}_3(s) + H_2O(l) \]  \hspace{1cm} (2)

\[ \text{MnFe}_2\text{O}_4(s) + 2H^+ = \text{Mn}^{2+} + \text{Fe}_2\text{O}_3(s) + H_2O(l) \]  \hspace{1cm} (3)

\[ 2\text{NiO} \cdot \text{SiO}_2(s) + 4H^+ = 2\text{Ni}^{2+} + \text{SiO}_2(s) + 2H_2O(l) \]  \hspace{1cm} (4)

\[ 2\text{CoO} \cdot \text{SiO}_2(s) + 4H^+ = 2\text{Co}^{2+} + \text{SiO}_2(s) + 2H_2O(l) \]  \hspace{1cm} (5)

\[ 2\text{MnO} \cdot \text{SiO}_2(s) + 4H^+ = 2\text{Mn}^{2+} + \text{SiO}_2(s) + 2H_2O \]  \hspace{1cm} (6)

![Figure 1. XRD pattern of the laterite sample.](image-url)
The leaching mechanism of laterite nickel ore in acidic solution was studied by comparing the XRD patterns of different leaching residues and raw ore to observe any changes in characteristic peaks. The leaching temperature, acid concentration, and leaching time as important parameters for the leaching process were studied comprehensively.

In Figure 2, the XRD diagram of different leaching residues (raw ore pattern included) shows that most characteristic goethite peaks at 2θ are 22.1°, 33.6°, 36.8°, 42.8°, and 54.1°, which changed following an increase in the concentration of hydrochloric acid. They diminished gradually and eventually disappeared at an acid concentration of 2 mol/L. The characteristic serpentine peaks became gradually weaker and even disappeared at high concentrations of HCl at 2θ angles of 12.1°, 24.2°, and 60.1°, which is similar to that of goethite. The characteristic 12.1° serpentine peaks were still present at an acid concentration of 4 mol/L, which means that serpentine is more stable than goethite under these leaching conditions. Magnetite and hematite were still present in the residues, even when the HCl concentration was increased to 4 mol/L. The disappearance of the goethite peaks suggests that goethite dissolves more readily than hematite and magnetite, which signifies that the iron in the solution originated mostly from the goethite. The order of dissolution of these minerals with increasing HCl concentration are goethite > serpentine > magnetite ≈ hematite.

\[
\begin{align*}
\text{NiO}(s) + 2H^+ &= Ni^{2+} + H_2O(l) \\
\text{CoO}(s) + 2H^+ &= Co^{2+} + H_2O(l) \\
\text{FeO(OH)}(s) + 3H^+ &= Fe^{3+} + 2H_2O(l) \\
\text{Fe}_2\text{O}_4(s) + 6H^+ &= 2Fe^{3+} + 3H_2O(l) \\
\text{FeOOH}(s) + HCl &\rightarrow FeOCl(s) + H_2O(l)
\end{align*}
\]

Figure 2 shows the XRD (raw ore pattern included) spectra obtained from leaching experiments at different acid concentration. Figure 3 shows the XRD (raw ore pattern included) spectra obtained from leaching experiments at different temperatures. It shows that between temperatures of 50 °C and 90 °C, certain characteristic peaks corresponding to goethite and serpentine diminished and even disappeared. The residue did not contain any goethite at a temperature of 90 °C. This illustrates that the leaching temperature had a greater influence on goethite than serpentine. As shown in Figure 3, the magnetite mineral was affected by the leaching temperature similarly to the hematite mineral, and nearly all the characteristic peaks corresponding to hematite and magnetite were present at this temperature. The fact that most of the serpentine dissolved can be concluded from Figure 3 due to a decrease in the amount of serpentine and an increase in the amount of SiO₂.
Figure 3. Leaching residues’ XRD diagram at different leaching temperatures.

The main changes in the content of different minerals according to leaching time is shown in Figure 4. The characteristic Goethite peaks nearly disappeared, and the characteristic serpentine peaks diminished significantly after the laterite was leached for 60 min. The characteristic peaks corresponding to goethite disappeared prior to serpentine, indicating that goethite can be leached more readily than serpentine. The characteristic peaks corresponding to magnetite and hematite were still present in the leaching residue at a leaching time of 120 min (Figure 4), suggesting that hematite and magnetite are relatively stable.

Figure 4. Leaching residues’ XRD diagram at different leaching times.

3.2. Leaching Kinetics

The process of laterite leaching with ammonium chloride hydrochloric acid solution is a typical solid–liquid reaction. However, the curves for leaching rate vs. time for Ni, Co, and Fe did not fit traditional kinetic models, and so traditional kinetic models were not appropriate to describe the dynamics of the process in this research. A new shrinking core model was studied by Dickinson et al. [26] and Dehghand et al. [27], which was applied to simulate and calculate the process as follows:

$$\frac{1}{3} \ln(1 - w) + [(1 - w)^{-1/3} - 1] = k_m t$$  \hspace{1cm} (12)

where $w$, $k_m$, and $t$ represent the leaching fraction, the apparent reaction rate constant, and leaching time, respectively.
This model shows that the reaction rate can be affected by interfacial transfer and diffusion across the product layer. The leaching rates of Ni, Co, and Fe vs. time were calculated using Equation (12), and the relationship between the values of $\frac{1}{3} \ln(1 - w) + [(1 - w)^{-1/3} - 1]$ and leaching time for Ni, Co, and Fe at different temperatures are presented in Figures 5–7.

Figure 5. The $\frac{1}{3} \ln(1 - w) + [(1 - w)^{-1/3} - 1]$ of nickel leaching.

Figure 6. The $\frac{1}{3} \ln(1 - w) + [(1 - w)^{-1/3} - 1]$ of cobalt leaching.

Figure 7. The $\frac{1}{3} \ln(1 - w) + [(1 - w)^{-1/3} - 1]$ of iron leaching.

High fitting degrees (the minimal R$^2$ of 0.92131) indicate that these values are linear and verify the suitability of this shrinking core model to the process.

Temperature can affect the rate constant. The Arrhenius equation was used to express the relationship between the rate constant ($k$) and temperature ($T$):

$$k = A \exp\left[-\frac{E_a}{RT}\right]$$

(13)
In Equation (13), \( A \) and \( E_a \) are the frequency factor and apparent activation energy, respectively. Equation (13) can be expressed as the following:

\[
\ln k = \ln A - \frac{E_a}{(2.303RT)}
\]  

(14)

The Arrhenius plots are shown in Figures 8–10, which describe the relationship between the rate constant and temperature and the data presented in Figures 5–7. They show that plots in these figures of \( \ln k \) against \( 1/T \) give a straight line.

![Arrhenius plot for nickel leaching.](image)

**Figure 8.** Arrhenius plot for nickel leaching.

![Arrhenius plot for cobalt leaching.](image)

**Figure 9.** Arrhenius plot for cobalt leaching.

![Arrhenius plot for iron leaching.](image)

**Figure 10.** Arrhenius plot for iron leaching.

From the slope of these lines, the activation energy can be calculated. The activation energies for nickel, cobalt, and iron are 4.01 kJ/mol, 3.43 kJ/mol, and 1.87 kJ/mol, respectively. This shows that the leaching of Ni, Co, and Fe are all controlled by solid diffusion as the \( E_d \) rates are in the range of 1–5 kJ/mol, further verifying the suitability of this kinetic model [28].
Equation (15) can be used to express the reaction rate constants. The major factors, including leaching time, liquid–solid ratio, leaching temperature, and the concentration of leaching regent (i.e., ammonium chloride and hydrochloric acid) in the leaching process can all be expressed:

\[ k_m = k_0 [HCl]^a [NH_4Cl]^b (c_{L/S})^c \exp\left[-\frac{E_a}{RT}\right] \]  

(15)

In Equation (15), \( k_0 \) is the Arrhenius constant; \( a, b, \) and \( c \) represent the reaction order with respect to hydrochloric acid concentration, ammonium chloride concentration, and \( c_{L/S} \); and \( c_{L/S} \) is the liquid–solid ratio.

Substituting \( k_m \) from Equation (15) into Equation (12) gives the following:

\[ \frac{1}{3} \ln(1-w) + [(1-w)^{-1/3} - 1] = k_0 [HCl]^a [NH_4Cl]^b (c_{L/S})^c \exp\left[-\frac{E_a}{RT}\right] t. \]  

(16)

The apparent reaction rate constants for Ni, Co, and Fe in the shrinking core model can be determined by different factors affecting the dissolution of Ni, Co, and Fe; a kinetic equation with regards to the leaching of each metal can be established. This will significantly benefit improvements in laterite leaching processes.

The reaction order of each metal \( (a, b, \) and \( c) \) can also be calculated through the relationship between \( k \) and \( c(HCl) \), \( c(NH_4Cl) \), and \( c_{L/S} \) (the slope of \( \ln k - \ln c(HCl) \), \( \ln k - \ln c(NH_4Cl) \), \( \ln k - \ln c_{L/S} \)), shown in Figures 11–13.
The Arrhenius constants ($k_0$) for nickel, cobalt, and iron, calculated through the intercept in Figures 11–13 are 204.38, 16.65, and $7.12 \times 10^{-3}$, respectively.

All data are applied to Equation (16) and so the final kinetic equations can be described as the following:

$$
\text{Ni : } \frac{1}{3} \ln(1-w) + \left[(1-w)^{-\frac{1}{3}} - 1\right] = 204.38[HCl]^{1.32}[NH_4Cl]^{0.85}([L_{/S}]^{1.53} \exp[-4010/(RT)]t \quad (17)
$$

$$
\text{Co : } \frac{1}{3} \ln(1-w) + \left[(1-w)^{-\frac{1}{3}} - 1\right] = 16.65[HCl]^{1.74}[NH_4Cl]^{1.12}([L_{/S}]^{1.22} \exp[-3430/(RT)]t \quad (18)
$$

$$
\text{Fe : } \frac{1}{3} \ln(1-w) + \left[(1-w)^{-\frac{1}{3}} - 1\right] = 7.12 \times 10^{-3}[HCl]^{2.52}[NH_4Cl]^{-0.11}([L_{/S}]^{0.94} \exp[-1870/(RT)]t \quad (19)
$$

4. Conclusions

In summary, the leaching mechanism study showed that all the major leaching conditions can affect the dissolution of these minerals. The dissolution order is: Goethite > serpentine > hematite and magnetite, which is consistent with the results observed for the extraction of metals.

The activation energies of 4.01 kJ/mol for nickel, 3.43 kJ/mol for cobalt, and 1.87 kJ/mol for iron, which proved a solid diffusion-controlled extraction of Ni, Co, and Fe. Reaction orders $(a, b, and c)$ for the metals were determined to be: Nickel (1.32, 0.85, and 1.53), cobalt (1.74, 1.12, and 1.22), and iron (2.52, −0.11, and 0.94). Kinetic equations demonstrated that the leaching of Ni, Co, and Fe during the leaching of laterite using a hydrochloric acid-ammonium chloride system can be separated into two stages. The first stage involves interface exchange on the surface of the solid particle and the second stage concerns diffusion over the solid-doped membrane. In addition, it can be concluded that nickel, cobalt, and iron, which are present in laterite, undergo two types of reactions: Surface adsorption and lattice replacement.

Author Contributions: Preliminary idea, technical guidance, and writing guidance, J.L.; data collection, data analysis, and paper writing, Y.Y.; discussion and proofreading, Y.W. and W.L.; data provided, Y.C.; project supervision, R.W.; Experimental guidance, Z.X. All authors have read and agreed to the published version of the manuscript.

Funding: The project was sponsored by National Natural Science Foundation (51974140), (51564021), and (5176040277), Department of Education of Jiangxi Province (GJJ160593), and Jiangxi Province Postdoctoral Science Fund (2017KY17).

Conflicts of Interest: The authors declare no conflict of Interest.
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