Investigation on the garnierite and limonite mixed laterite ore for recovery nickel

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Abstract
Laterite, as an important resource of nickel, has become the focus of development and utilization. This study adopted the method of mixing ore (garnierite and limonite) to increase the recovery rate of nickel in garnierite ore. The phase transformation of the two ores were investigated during the heating process, dehydroxylation and recrystallization were observed and the iron oxides phase was transformed into iron-containing spinel finally. By changing the proportion of the two ores, increasing the iron content in the sample was beneficial to the reduction of nickel. Then, Fe₂O₃, Fe₃O₄ and Fe were used as the iron source to study the mechanism of the iron-bearing minerals in promoting the reduction of nickel. The results indicated that hematite and/or magnetite would react with amorphous silicate minerals to generate magnesioferrite and enstatite, thereby avoiding additional forsterite generation; moreover, the trevorite phase would be formed, which strengthens the aggregation of nickel and iron.

Keywords: Garnierite ore; Limonite ore; Reduction; Phase transformation

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1 Introduction

Nickel resources mainly involve two types of ore, including nickel sulphide ore and laterite ore. As nickel sulphide ore resources were mined continuously, and its reserves have decreased sharply. Plentiful laterite ores have attracted interest in recent, and treatment process has led to greater efforts to improve recovery of nickel from laterite ore.\(^1\) Laterite nickel ore, also known as nickel oxide ore, is formed by nickel, iron and magnesium silicate olivine bedrock through long-term weathering and enrichment.\(^2\) The top lateritic layer is commonly of a high laterisation degree of the limonite type, the lower portion is a garnierite type with lower laterisation degree and the intermediate transition zone is a saprolitic type.\(^3\)

The laterite nickel ore is rich in reserves, but the complex ore phase and structure make it difficult for laterite to efficiently enrich nickel. Thus, many scholars have studied the mechanism of laterite nickel ore dehydration and reduction of iron and nickel oxide under isothermal and non-isothermal conditions.\(^4\)\(^-\)\(^8\) The phase transformation of laterite minerals (saprolite and limonite) reduced at temperatures up to 800 °C and the phase reversibility with cooling were investigated,\(^9\) indicating that dehydroxylation could release nickel, which embedded in main gangue minerals. An Australian garnieritic-type ore demonstrated that chlorite is converted into forsterite and enstatite at 700-800 °C with recrystallization between 800 and 850 °C.\(^10\) However, the temperature of talc transformed to forsterite and enstatite was reached 1000 °C. The microstructure and phase characterisations of laterite ore were investigated and the results shows the nickel is mainly associated with goethite and serpentine particles, which altered factors that affect nickel recovery from a single phase (goethite or serpentine).\(^11\),\(^12\) Nickel is difficult to be extracted from olivine, and nickel recovery is better for single-phase olivine with an Mg/Fe ratio <8. Kawahara et al.\(^13\) studied the reducibility of several types of laterite nickel ore and reported that the reducibility of nickel in ore increased with increasing iron concentration; poor reducibility of nickel in low-iron and/or high-magnesia laterites ore is attributed to magnesium replacement by nickel in the silicate lattice.

Aiming at the characteristic of difficult reduction and enrichment of the dispersive nickel phase in the ore, the low-nickel and low-iron garnierite was taken as the research object in present. The method of mixing ore (limonite-type laterite ore) was adopted to increase the iron content in the raw material for promoting the reduction of nickel. Based on the understanding of the phase transformation of the two ores during the high-temperature roasting process, a detailed investigation on the reduction roasting-magnetic separation of the mixed ore was carried out, and the interaction between the iron phase and nickel in the ore was clarified. The results obtained can clarify the mechanism of nickel phase reduction and enrichment in mixed ore.
2 Experimental

2.1 Materials

Two types of low-grade nickel laterite ore used in the study were supplied by the Yunnan province of China. The chemical compositions of the two laterite ores are listed in Table 1.

Table 1. Chemical analysis of the laterite ore sample (mass%).

| Sample   | TFe | Ni  | Co  | MgO | SiO₂ | Al₂O₃ | CaO |
|----------|-----|-----|-----|-----|------|-------|-----|
| Garnierite | 9.67| 0.82| 0.033| 31.49| 37.37| 1.89  | 0.033|
| Limonite  | 35.71| 1.17| 0.047| 4.54 | 16.75| 11.17 | 0.053|

The garnierite contains low Ni (0.82 %) and Fe (9.67 %), and high SiO₂ (37.49 %) and MgO (31.49 %). The Ni and Fe contents in the limonite reach 1.17 and 35.71 %, respectively. The anthracite was used as a reductant, and the proximate analysis results list in Table 2, in which shows the fixed carbon is 76.43 %.

Table 2. Proximate analysis of anthracite (mass%).

| Properties | Fixed carbon | Volatile | Ash | Moisture |
|------------|--------------|----------|-----|----------|
| Content    | 76.43        | 7.78     | 15.29| 1.02     |

2.2 Reduction roasting

Laterite ores were dried at 60 °C for 24 h and ground using an XZM-100 laboratory vibratory mill to 95 % passing 0.25 mm. The dried laterite ore was first mixed with reductant, and then the sample was placed in a horizontal tube furnace and heated to the required temperature for a given duration under nitrogen atmosphere, which was introduced at a flow rate of 30 mL/min. After reduction, the roasted sample was cooled in the tube furnace to prevent re-oxidation. The cooled roasted ore was wet-ground in the above-mentioned vibratory mill at a pulp density of 50 % solids, and then the slurry was subjected to magnetic separation (DTCXG-ZN50 Magnetic Tube, magnetic-field intensity of 200 mT) to obtain the ferronickel concentrate.

2.3 Equipment

The X-ray diffraction (XRD) was adopted to analysis phase of the laterite ores and roasted samples, which were obtained using a Japan Science D/max-R diffractometer with Cu Kα radiation (λ=1.5406 Å), operating at 40 kV and 30 mA. Thermogravimetric (TG) and differential scanning calorimetry (DSC) was performed on a NETZSCH STA 449F3 unit under a nitrogen atmosphere, and the samples were heated from 20 to 1200 °C at a constant rate of 10 °C/min. The Scanning Electron Microscopy coupled with Energy Dispersive Spectrometer (SEM-EDS, HITACHI-S3400N) was used to analysis the microstructure of the roasted sample.

3 Results and Discussion

3.1 Nickel distribution

The distribution form of nickel in the ore determines the difficulty degree of its extraction. Low grade and complex embedding form are the fundamental reasons for
the difficult treatment of laterite nickel ore. The distribution ratio of nickel in Ni-bearing minerals is listed in Table 3, the analysis methods was described in a previous report.[14] Nickel is mainly associated with silicates and oxides in two types of ores and accounted for 95.13 % of the garnierite and 96.75 % of the limonite. For limonite, approximately 69.35 % of Ni element is distributed in silicates, and this ratio reaches 84.15 % in garnierite ore. Compared with oxides, nickel is more difficult to reduce in silicates.[12,15]

| Sample        | Existential Phases | Oxides | Silicates | Sulfides | Sulfates | Total |
|---------------|--------------------|--------|-----------|----------|----------|-------|
| Garnierite    | Grade              | 0.09   | 0.69      | 0.03     | 0.01     | 0.82  |
|                | Distribution ratio | 10.98  | 84.15     | 3.65     | 1.22     | 100   |
| Limonite      | Grade              | 0.32   | 0.81      | 0.022    | 0.016    | 1.17  |
|                | Distribution ratio | 27.40  | 69.35     | 1.88     | 1.37     | 100   |

3.2 Phase transformation of laterite ore during roasting

3.2.1 Phase transformation of garnierite ore

The TG/DSC curves of the raw garnierite ore are shown in Fig. 1, which indicated a weakly endothermic peak below 120 °C, corresponding to the removal of adsorbed water from the sample. An obvious endothermic peak located at 610 °C is attributed to dehydroxylation of serpentine, with a weight loss of 9.5 %. With the increasing of temperature, serpentine undergoes recrystallisation and transforms into forsterite and enstatite with a strong exothermic peak at approximately 820 °C.

![Fig. 1. TG/DSC curves of the raw garnierite ore.](image)

To study the phase transformation of the nickel laterite ore during the roasting process, the ore samples were roasted for 60 minutes at setting temperature. The XRD patterns of garnierite samples roasted at various temperatures are shown in Fig. 2.
Fig. 2. XRD patterns of the garnierite ore after roasting at different temperatures.

The main minerals, which are evident from the XRD patterns of the garnierite raw ore, are lizardite [(Mg,Fe)\(_3\)Si\(_2\)O\(_5\)(OH)\(_4\)], quartz (SiO\(_2\)) and maghemite (\(\gamma\)-Fe\(_2\)O\(_3\)). At 300 and 500 °C, part of the maghemite is transformed, generating a new phase hematite. At a roasting temperature up to 610 °C, the peak for lizardite completely disappeared, the peak intensity of hematite (\(\alpha\)-Fe\(_2\)O\(_3\)) increased gradually, without the appearance of the silicate mineral phase. In the combined thermogravimetric curves, serpentine underwent dehydroxylation and the crystal structure was broken. The serpentine was converted into amorphous silicate minerals and therefore, there is no peak of silicate minerals in the XRD patterns. At 700, 800 and 900 °C, the structure of silicate minerals transformed from non-crystalline to crystalline, and the new phases forsterite and enstatite were found. TG curve (Fig. 1) depicts that a strong exothermic peak located at 820 °C, so the transformation from non-crystalline silicate minerals to forsterite (Mg\(_2\)SiO\(_4\)) and enstatite (MgSiO\(_3\)) is an exothermic process in accordance with Reaction (1).\(^{16,17}\)

\[
\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4 \rightarrow \text{Mg}_2\text{SiO}_4 + \text{MgSiO}_3 + 2\text{H}_2\text{O}
\]  

As temperature increased to 1000 °C, hematite diffraction peak intensity is significantly reduced, and the new spinel phases are formed: trevorite (NiFe\(_2\)O\(_4\)) and magnesioferrite (MgFe\(_2\)O\(_4\)). Further increasing temperature above 1200 °C, the diffraction peaks for hematite completely disappeared, and the trevorite peak intensity has an increase as a result.

### 3.2.2 Phase transformation of limonite ore

Similar tests were conducted for limonite ore, the TG/DSC curves of the raw limonite ore are presented in Fig. 3. Three main thermal effects are observed. The first endothermic peak at approximately 100 °C corresponds to the elimination of absorbed moisture. The second endothermic peaks as shown at approximately 280 °C represent the dehydroxylation of goethite to form hematite, with a weight loss of 4.7 %, in accordance with Reaction (2).\(^{16,18}\)

\[
2\text{FeOOH} \rightarrow \text{Fe}_2\text{O}_3 + \text{H}_2\text{O}
\]  

This decomposition temperature of goethite is low compared to the report, which decomposition temperature is 310 °C\(^{19}\) or 330 °C,\(^{20}\) this phenomenon may be attributed to the poorly crystalline structure of goethite in the ore. At approximately 850 °C, the third exothermic peak mainly represents the sintering of particles, which
generate $\alpha$-Fe$_2$O$_3$ particles with irregular shape.[21] This peak indicates a recrystallisation phenomenon with no associated weight loss.

![TG/DSC curves of the raw limonite ore.](image)

**Fig. 3** TG/DSC curves of the raw limonite ore.

Fig. 4 shows that the main minerals of limonite raw ore are goethite (FeOOH) and maghemite. After roasting at 200 °C, the diffraction peak was basically identical to the raw ore, indicating that the ore phase did not change at this temperature. At a roasting temperature up to 300 °C, the goethite peak completely disappeared, and the hematite peak appeared simultaneously. This is consistent with the results of thermogravimetric analysis, namely, goethite is transformed to hematite at 280 °C. As the temperature increased to 800 °C, the peak intensity of maghemite decreased; while the peak intensity of hematite has an increase. This finding indicates that the maghemite gradually converted into hematite, which is similar to the DSC curve (Fig. 4) through the appearance of the exothermic peak at 850 °C. As increasing temperature to 1000 °C, the maghemite peak disappears and the characteristic peaks of magnesioferrite (MgFe$_2$O$_4$) and trevorite (NiFe$_2$O$_4$) appeared. Further increasing temperature, the hematite phase disappeared, and the magnesioferrite and trevorite peak strengthened.

![XRD patterns of the limonite ore after roasting at different temperatures.](image)

**Fig. 4.** XRD patterns of the limonite ore after roasting at different temperatures. Goethite (FeOOH); Trevorite (NiFe$_2$O$_4$), Magnesioferrite (MgFe$_2$O$_4$), Hematite ($\alpha$-Fe$_2$O$_3$), Maghemite ($\gamma$-Fe$_2$O$_3$), Quartz (SiO$_2$).
3.3 Effect of limonite content on garnierite ore reduction

The garnierite sample with various proportions of limonite ore (ranging from 0 to 80 %) was roasted at 1200 °C for 60 minutes. Coal dosages added in the sample were based on the C/O ratio, namely, the amount of coal addition in terms of the gram-atomic ratio of the fixed carbon in the coal added to the combined oxygen in nickel and iron oxides. Fig. 5 shows the magnetic separation results, indicating that as limonite ore increases to 60 %, nickel recovery gradually improves from 30.52 % to 71.55 %; the corresponding iron recovery increased from 47.23 % to 81.72 %. Thereafter, the recoveries of nickel and iron remain stable. Experimental results demonstrated that the reduction rate of nickel in garnierite is promoted by the addition of limonite ore.

Fig. 5. Effect of limonite ore proportion on the recovery of iron and nickel in mixed ore.

As analysed above, the main mineral phases of garnierite ore roasted at 1200 °C included forsterite, enstatite, magnesioferrite, trevorite and quartz; limonite ore after roasting included hematite, trevorite, magnesioferrite and quartz. Fig. 6 shows the XRD patterns of the mixture of the two ores after roasted 1200 °C.

Fig. 6. XRD patterns of mixed ore after roasting at 1200 °C for 60 minutes with different proportion of limonite ore proportion. Quartz (SiO₂), Trevorite (NiFe₂O₄), Magnesioferrite (MgFe₂O₄), Enstatite (MgSiO₃), Forsterite (Mg₂SiO₄).

The diffraction peak of forsterite in the roasted sample reflects a weak intensity when adding 20 % of limonite. With increasing the limonite proportion, the forsterite peak gradually disappears. This may be attributed to the formation of enstatite, as
expressed in Reaction (3). However, nickel element which embeds in enstatite is easier to extract than nickel in the forsterite, which could be a major reason for improving nickel recovery. In addition, the hematite peak is unobserved and the spinel peak intensity (maghemite and magnesioferrite) strengthened. The Ni-Fe alloy could be generated from the Fe-rich matrix, thus the formation of iron-containing spinel may be beneficial to the reduction of nickel and improves the nickel recovery rate in the later process.

\[
2\text{Mg}_2\text{SiO}_4+\text{SiO}_2 \rightarrow \text{Mg}_2\text{SiO}_4+2\text{MgSiO}_3 \tag{3}
\]

3.4 Reaction mechanism analysis

The iron in the limonite ore is mainly in the form of goethite and maghemite, and they would be converted into hematite as increasing temperature. In order to investigate the effect of iron phase in mixed ore on nickel reduction, Fe\textsubscript{2}O\textsubscript{3}, Fe\textsubscript{3}O\textsubscript{4} and Fe was used as iron source and added to garnierite ore, respectively. The added quantities of Fe\textsubscript{2}O\textsubscript{3}, Fe\textsubscript{3}O\textsubscript{4} and Fe are based on the Fe/Ni ratio, which is calculated from the composition of mixed ore (garnierite mixed with limonite in various proportions). Fig. 7 shows the results of nickel reduction rate in the sample (reduction at 1200 °C for 60 minutes) as a function of Fe/Ni ratio. With the addition of Fe\textsubscript{2}O\textsubscript{3}, the metallization rate of nickel exhibited a gradual improvement from 75.0 to 83.0 % with increasing Fe/Ni ratio from 16.7 to 24.6. With the addition of Fe\textsubscript{3}O\textsubscript{4}, as the Fe/Ni ratio increased from 16.7 to 24.6, the metallization rate of nickel improved from 74.3 to 76.5 % and stabilized thereafter. Fe\textsubscript{2}O\textsubscript{3} and Fe\textsubscript{3}O\textsubscript{4} reflect varying degrees of promoting the nickel oxide reduction in the sample. However, the metallization rate of nickel is stabilized at approximately 71 %, with the addition of metallic Fe, indicating that metallic iron does not obviously influence the nickel reduction.

![Graph](image)

**Fig. 7.** Effect of different Fe/Ni ration on the nickel metallization rate.

The XRD patterns of the mixture (garnierite ore and Fe\textsubscript{2}O\textsubscript{3}) with various Fe/Ni ratios after roasting at 1200 °C are shown in Fig. 8. Without adding Fe\textsubscript{2}O\textsubscript{3}, the pattern (Fig. 8a) exhibits a high intensity of forsterite diffraction peak. As Fe\textsubscript{2}O\textsubscript{3} dosage increased, the forsterite diffraction peak intensity was significantly reduced, and the intensity of the enstatite and spinel (trevorite and magnesioferrite) diffraction peak increased accordingly. The phase for the Fe\textsubscript{2}O\textsubscript{3} were not appear in the XRD analysis results.
Fig. 8. XRD patterns of the mixture (garnierite ore and Fe₂O₃) with different Fe/Ni ratio after roasting at 1200 °C for 60 minutes. a: Fe/Ni=11.79; b: Fe/Ni=16.72; c: Fe/Ni=20.92; d: Fe/Ni=24.56; Quartz (SiO₂), Trevorite (NiFe₂O₄), Magnesioferrite (MgFe₂O₄), Enstatite (MgSiO₃), Forsterite (Mg₂SiO₄).

Combining the XRD analysis results in Fig. 2, for the garnierite roasted at different temperatures, the peak intensity of hematite was significantly reduced and finally disappeared, and the intensity of enstatite and spinel (trevorite and magnesioferrite) diffraction peaks accordingly appeared when roasting temperature was above 900 °C, illustrating that hematite reacts with magnesium-bearing minerals and/or nickel-bearing minerals to generate trevorite and magnesioferrite. Silicate minerals are the nickel and magnesium host phases. From the viewpoint of thermodynamics, it is difficult to generate the spinel phase by the reaction of hematite with forsterite and/or enstatite. The formation of magnesioferrite mainly due to the reaction between hematite and amorphous silicate minerals produced by serpentine dehydroxylation. This process can be expressed by Reaction (4), which avoids the generation of olivine and embeds nickel in enstatite. The newly generated magnesioferrite reacts with nickel silicate to form trevorite, in accordance with Reaction (5), and nickel in trevorite is more easily to reduce. Furthermore, nickel oxide would react with hematite to form trevorite, as in Reaction (6). Figure 7 shows that Fe₃O₄ does influence the nickel metallization rate, but the promotional role of Fe₂O₃ is stronger under the same Fe/Ni ratio conditions. The addition of Fe₃O₄ does not prevent the generation of olivine, but instead generates magnesioferrite, in accordance with Reaction (7) and (8).

\[
\begin{align*}
\text{Mg}_3\text{Si}_2(\text{OH})_4\text{O}_5+\text{Fe}_2\text{O}_3 & \rightarrow 2\text{MgSiO}_3+\text{MgFe}_2\text{O}_4+2\text{H}_2\text{O} \\
\Delta G^\theta &= 153.667-0.287T \text{ kJ/mol} \\
\text{Ni}_2\text{SiO}_4+2\text{MgFe}_2\text{O}_4 & \rightarrow 2\text{NiFe}_2\text{O}_4+\text{Mg}_2\text{SiO}_4 \\
\Delta G^\theta &= -47.366-0.032T \text{ kJ/mol} \\
\text{NiO}+\text{Fe}_2\text{O}_3 & \rightarrow \text{NiFe}_2\text{O}_4 \\
\Delta G^\theta &= -17.596-0.0025T \text{ kJ/mol} \\
\text{Mg}_3\text{Si}_2(\text{OH})_4\text{O}_5+\text{Fe}_3\text{O}_4 & \rightarrow \text{Mg}_2\text{SiO}_3+\text{MgFe}_2\text{O}_4+\text{FeSiO}_3+2\text{H}_2\text{O} \\
\Delta G^\theta &= 211.029-0.271T \text{ kJ/mol} \\
\text{Mg}_3\text{Si}_2(\text{OH})_4\text{O}_5+2\text{Fe}_3\text{O}_4 & \rightarrow \text{MgSiO}_3+2\text{MgFe}_2\text{O}_4+\text{Fe}_2\text{SiO}_4+2\text{H}_2\text{O}
\end{align*}
\]
\[ \Delta G^\theta = 197.063 - 0.246T \text{ kJ/mol} \] (8)

Fig. 9 presents SEM images and EDS spectra of the reduction roasted mixture (garnierite ore and Fe₂O₃) at 1200 °C for 60 minutes with Fe/Ni=24.6. The SEM-EDS results clearly clarified the metallic iron in the sample (point 1), which is the reduced product of added Fe₂O₃ and attached with ferronickel particles (point 2). The reaction product of trevorite is a continuous series of metallic solid solutions. Therefore, it is demonstrated that nickel-bearing minerals are transformed to NiFe₂O₄ before reduction, and the formation of trevorite strengthens the aggregation of nickel and iron.

Based on the above analyses, the reduction of nickel in the garnierite promoted by hematite [Fe₂O₃] can be described as follow: Fe₂O₃ reacts with amorphous silicate minerals firstly, which produced by serpentine dehydroxylation, to avoid the generation of olivine; and then the newly generated magnesioferrite reacts with nickel silicate (nickel-containing enstatite) to produce trevorite that is easier to reduce.

4. Conclusions

(1) With increasing the temperature, lizardite within garnierite undergoes dehydroxylation and recrystallization at 610 and 820 °C, respectively. Limonite is mainly consisted of goethite and maghemite phase. The dehydroxylation of goethite to form hematite occurs at approximately 280 °C; while maghemite is gradually converted into hematite at roasting temperature below 800 °C. Hematite would begin to transform to iron-containing spinel above 1000 °C.

(2) The addition of limonite is beneficial to the reduction nickel within garnierite. As limonite ore increases from 20 to 60 %, nickel recovery gradually improves from 30.5 to 71.6 %.

(3) Hematite [Fe₂O₃] reacts with amorphous silicate minerals produced by serpentine dehydroxylation to avoid the generation of olivine, embedding nickel in enstatite. The newly generated magnesioferrite also reacts with nickel silicate to produce a more easily reduced trevorite, which strengthens the aggregation of nickel and iron.
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