

Article

# Production of Ferronickel Concentrate from Low-Grade Nickel Laterite Ore by Non-Melting Reduction Magnetic Separation Process

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**Abstract:** The production of ferronickel concentrate from low-grade nickel laterite ore containing 1.31% nickel (Ni) was studied by the non-melting reduction magnetic separation process. The sodium chloride was used as additive and coal as a reductant. The effects of roasting temperature, roasting duration, reductant dosage, additive dosage, and grinding time on the grade and recovery were investigated. The optimal reduction conditions are a roasting temperature of 1250 °C, roasting duration of 80 min, reductant dosage of 10%, additive dosage of 5%, and a grinding time of 12 min. The grades of nickel and iron are improved from 2.13% and 51.12% to 8.15% and 64.28%, and the recovery of nickel is improved from 75.40% to 97.76%. The research results show that the additive in favor of the phase changes from lizardite phase to forsterite phase. The additive promotes agglomeration and separation of nickel and iron.

**Keywords:** nickel laterite; non-melting reducing; sodium chloride; magnetic separation

## 1. Introduction

As an important metal, nickel has an important position in industrial production due to its good physicochemical properties [1,2]. Nickel resources mainly exist in the form of nickel sulfide ore and nickel laterite ore. Among them, nickel laterite ore accounts for about 70% of the total nickel reserves in the world [3]. Due to the continuous decline of global nickel sulfide ore reserves, the development and utilization of nickel laterite ore have received increasing attention.

The treatment method of laterite ore can be divided into two types: hydrometallurgy and pyrometallurgy. The pyrometallurgical process has high requirements for the Ni grade, so it is impossible to treat the low-grade nickel laterite [4,5]. At the same time, high temperatures will cause slag and metal rings to form, which is not conducive to the production of ferronickel [6]. The disadvantage of the wet process is that the process is long and the wastewater is difficult to handle [3,7,8]. Therefore, the use of the "low-temperature roast reduction-magnetic separation process" to deal with such resources is attracting everyone's attention [6,9].

The roasted reduction-magnetic separation process is mainly used for the treatment of laterite ore, which requires the laterite ore to be crushed and ground. Then the reductant and additive are mixed with the raw material and roasted at a certain temperature. After that, the roasted products were ground and wet-type magnetic separation was carried out to obtain ferronickel concentrate.

In low-grade nickel laterite ore, the presence of nickel is extremely complex. Therefore, the addition of additives is often used to treat such laterites ore. There are generally two types of additives, one is sulfur and sulfate, and the other is chloride. Valix et al. [10] showed that the addition of elemental S inhibited the formation of forsterite in the reduction process, thus achieving selective separation of nickel and iron. Harris et al. [11] studied the selective vulcanization of nickel laterite at low temperatures, and the results show that nickel oxide can be selectively vulcanized. Although the addition of sulfur and sulfate can contribute to the recovery of nickel, the S content in the product is high [12]. Chloride is characterized by high reactivity of chlorine and the low melting point of metal chloride, which can selectively separate valuable metals [13,14]. Okamoto et al. [15] have shown that with the use of chloride salts as additives to treat nickel laterite, the chlorination reaction occurring during the roasting process can chlorinate and reduce the valuable metals in the ore. Ilic et al. [16,17] studied the selective extraction of nickel from raw materials containing iron and nickel by adding calcium chloride. The results show that it is feasible to selectively extract nickel by adding calcium chloride. Li et al. [18] used calcium chloride as an additive to reduce nickel laterite ore. The results show that calcium chloride can promote the migration of FeO to destroy the structure of the fayalite, thereby increasing the activity of nickel. Fan et al. [19] used ferrous chloride tetrahydrate as a chlorinating agent to selectively chlorinate pre-reduced nickel sulfide laterite, which achieved good results. Compared with other chlorides, sodium chloride has the advantages of wide sources and low prices. In this paper, the non-melting reduction-magnetic separation process of low-grade nickel laterite ore containing 1.13% of nickel was carried out with sodium chloride as an additive. The selective reduction of nickel is achieved by controlling the roasting temperature, the roasting duration, the reductant dosage, additive dosage, and the grinding time. The result is a concentrate with a high nickel grade and high recovery. In addition, the phase change of the ore under the action of the additive was studied.

## 2. Materials and Methods

### 2.1. Analysis of Nickel Laterite

The X-ray diffraction (XRD) analysis and chemical analysis of the nickel laterite ore sample is shown in Figure 1 and Table 1. The results in Table 1 show that the nickel content in the nickel laterite ore sample is only 1.13%, and the iron content is 35.79%. The XRD (Rigaku Corporation, Tokyo, Japan) results in Figure 1 show that the main phases in the sample are lizardite ( $\text{Mg}_3[(\text{Si,Fe})_2\text{O}_5](\text{OH})_4$ ), goethite ( $\text{FeO}(\text{OH})$ ), and maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ). The nickel is usually found in serpentine. Due to the low nickel content, the diffraction peaks of the corresponding nickel-containing phases could not be detected.

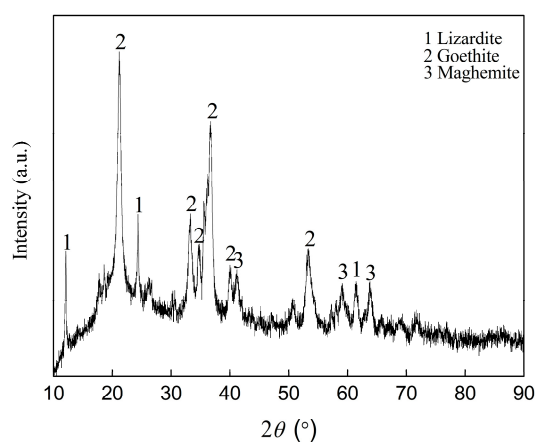


Figure 1. XRD pattern of nickel laterite ore.

**Table 1.** Chemical analysis of the nickel laterite ore sample.

Component	TFe(total)	Ni	Co	MgO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>
Content weight (wt. %)	35.79	1.13	0.097	3.65	10.38	10.31

## 2.2. Reductants and Additives

The experiment used anthracite as a reductant and analyzed pure sodium chloride as an additive. Industrial analysis of anthracite in Table 2 shows a fixed carbon content of 76.43%

**Table 2.** Industry analysis of the reductant.

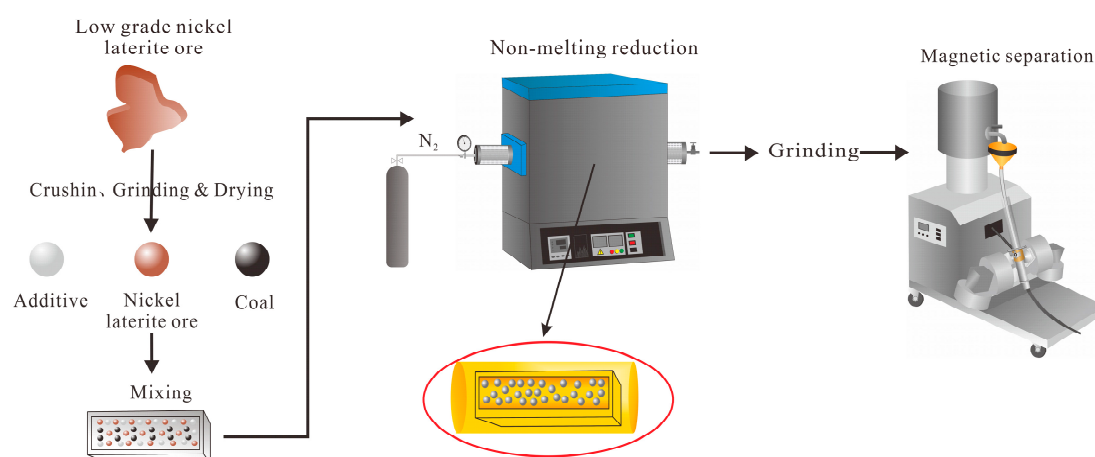
Component	Fixed Carbon	Volatile Matter	Ash	Moisture
Content wt. %	76.43	7.78	15.29	1.02

## 2.3. Methods

The 10 g nickel laterite ore sample is crushed and ground to below 250 µm, mixed with anthracite and additives according to their certain mass ratio, and put into a crucible. The crucible was then placed in a tube furnace and roasted under a nitrogen atmosphere. After the reduction, the furnace was cooled to room temperature. The roasted product was ground and wet magnetic separation was able to be used in order to obtain the concentrate. The experimental procedure and detailed experimental conditions are shown in Figure 2 and Table 3. Roasting reduction equipment GSL-1500X tube type high-temperature sintering furnace (Hefei Ke Jing Materials Technology CO.,LTD, Hefei, China) as well as XZM-100 vibration grinding machine (Wuhan Exploration Machinery Factory, Wuhan, China) and DTCXG-ZN50 type magnetic separation tube (Tangshan DT Electrical Equipment CO.,LTD, Tangshan, China) (magnetic field strength 200 mT) were used. The recoveries of Ni and Fe were calculated by the following formula:

$$X = \frac{m \cdot w}{M \cdot W} \cdot 100\%$$

where  $X$  is the recovery of nickel or iron,  $m$  is the weight of concentrate,  $w$  is the nickel or iron content in the concentrates,  $M$  is the weight of raw ore, and  $W$  is the nickel or iron content in the raw ore.

**Figure 2.** Experimental flowsheet.

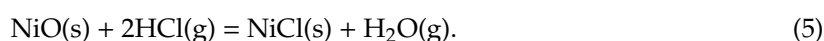
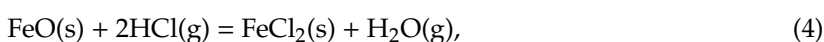
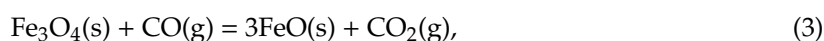
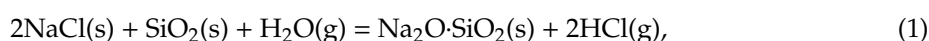
**Table 3.** List of process parameters studied during experiments.

Studied Parameters	Range
Roasting temperature/°C	900, 1000, 1100, 1200, 1250, 1300
Roasting duration/min	10, 30, 60, 80, 100
Reductant dosage/wt. %	2, 5, 10, 13, 15
NaCl dosage/wt. %	0, 5, 8, 10, 15
Grinding time/min	2, 4, 8, 12, 16

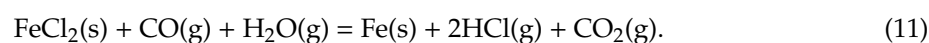
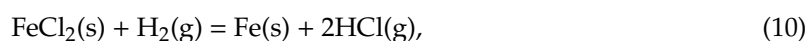
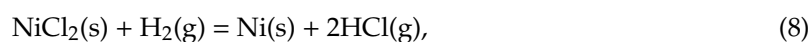
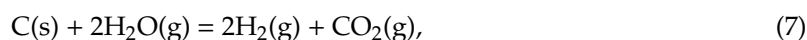
### 3. Results and Discussion

#### 3.1. Thermodynamic Analysis of Selective Reduction

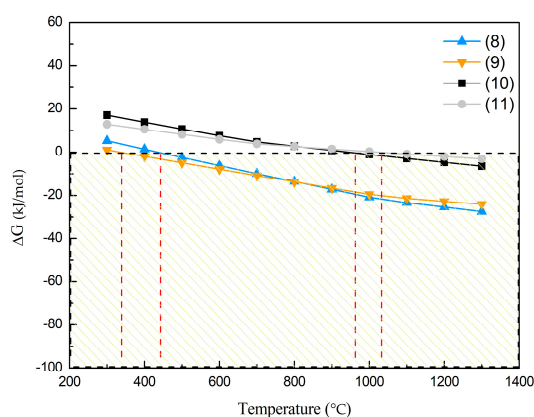
First, sodium chloride is hydrolyzed to generate hydrogen chloride gas. Second, the hydrogen chloride gas can chlorinate valuable metals in ore. In addition, iron oxide is difficult to be reduced by hydrogen chloride. Therefore, iron oxide is first reduced to FeO and then chlorinated. The relevant equations are as follows:



The fixed carbon is hydrolyzed at a high temperature to form hydrogen and carbon monoxide or carbon dioxide. The metal chloride is reduced by these educing gases. The relevant equations are as follows:



The Gibbs free energy of the (8)–(11) reaction formula was calculated using the HSC Chemistry 6.0 software (Outotec Oyj, Espoo, Finland) as shown in Figure 3. As can be seen from the figure, the temperatures at which NiCl<sub>2</sub> is reduced by hydrogen and carbon monoxide are 435 °C and 335 °C. The temperatures at which FeCl<sub>2</sub> is reduced by hydrogen and carbon monoxide are 944 °C and 1022 °C. The above mentioned temperature only represents the reaction spontaneous temperature. The higher the temperature, the more easily the chemical reaction is to occur, the more sufficient the reaction. Compared to the temperature at which FeCl<sub>2</sub> was reduced, the temperature at which NiCl<sub>2</sub> was reduced is extremely low. Therefore, selective reduction can be achieved by controlling the temperature.



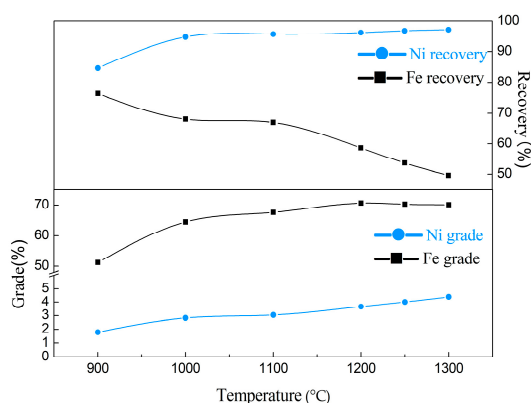
**Figure 3.** Gibbs free energy change curves of reactions with the change of temperature.

### 3.2. Effect of Different Influencing Factors on the Grade and Recovery of Ni and Fe

The recovery of nickel and iron in untreated concentrate (just grinding and magnetic separation) are 37% and 47.12%. In order to further improve the recovery of nickel and iron, the different factors were investigated.

#### 3.2.1. Effect of Reduction Roasting Temperature

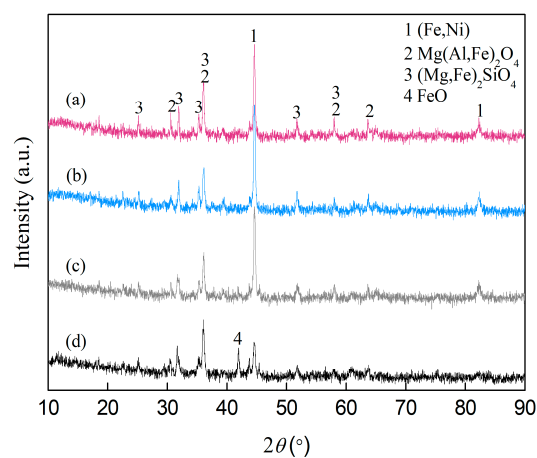
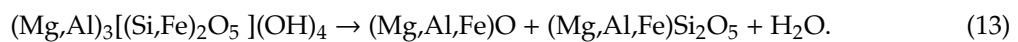
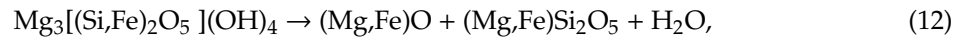
Under the condition of a roasting duration of 60 min, reductant dosage and additive dosage of 10%, and a grinding time of 12 min, the effects of reduction roasting temperature on nickel, iron grade, and recovery in the experimental process were investigated. It can be seen from Figure 4 that the grade and recovery of Ni increase with the increase of the roasting temperature. At 900 °C, the Ni grade and recovery are only 1.79% and 84.59%. When the temperature is raised from 1100 °C to 1250 °C, the Ni grade is increased from 3.04% to 4.01%, and the recovery is increased from 94.67% to 96.75%. The Ni grade increase was obvious. When the temperature is further increased to 1300 °C, the Ni grade reaches 4.39%, and the recovery does not change much. It can be seen that increasing the roasting temperature is beneficial to improve the grade of Ni. However, the roasted samples were found to be partially melted at 1300 °C. The phenomenon is not conducive to reduction roasting operation and increase of the grinding energy consumption. In this experiment, the nickel was enriched by the non-melting reduction as the main research purpose. Therefore, 1250 °C was selected as the subsequent experimental temperature.



**Figure 4.** Effect of roasting temperature on the grades and recoveries of Ni and Fe.

In order to further clarify the influence of temperature, the XRD analysis was performed on samples at different roasting temperatures. The results are shown in Figure 5. It can be seen from the XRD pattern that the main phase of the roasting product is forsterite. The existence of the phase is

mainly attributed to the following: (1) forsterite is formed by recrystallization of silicate in minerals [20]; (2) serpentine is transformed into forsterite during roasting (Equations (12) and (13)) [21]. No matter how the forsterite is formed, Ni and Fe are released during the phase transformation, which is beneficial to reduction. The intensity of the Fe–Ni diffraction peak in the roasting product was weak at 950 °C, and the FeO diffraction peak was detected. This indicates that Fe and Ni were not fully reduced at the temperature. When the temperature rises to 1050 °C, the intensity of the Fe–Ni diffraction peak obviously increases. Moreover, when the temperature rises to 1250 °C, the Fe–Ni diffraction peak further enhances. The results indicate that the increase of temperature is beneficial to the reduction of Fe and Ni.

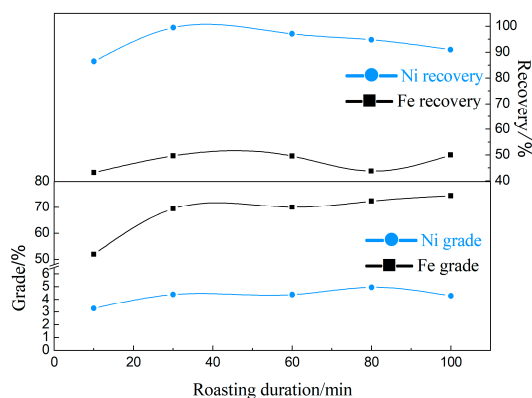


**Figure 5.** XRD patterns of laterite ore after chloridization and reduction roasting at various temperatures: (a) 1250 °C; (b) 1150 °C; (c) 1050 °C; (d) 950 °C.

In conclusion, the roasting can transform the phase of the ore and the transformation is beneficial to the reduction of nickel and iron. Secondly, the high temperature provides the necessary thermodynamic conditions for the chlorination and reduction reactions.

### 3.2.2. Effect of Roasting Duration

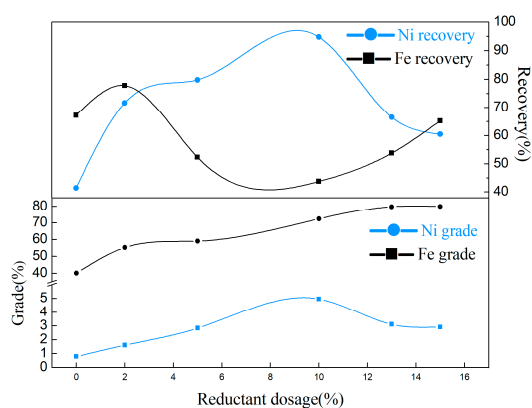
The experiment was conducted at a roasting temperature of 1250 °C, reductant dosage and additive dosage of 10%, and a grinding time of 12 min. The results are shown in Figure 6. When the roasting duration was 10 min, the Ni grade and recovery were 3.28% and 86.50%. When the roasting duration was extended to 30 min, the Ni recovery reached 99.52% and the Ni grade was increased to 4.40%. When the roasting duration was extended to 80 min, the Ni grade reached 4.96%. In addition, iron recovery was at a low level. Kinetic studies have shown that the reduction of iron is slow and difficult compared to the reduction of nickel, resulting in a higher partial pressure of FeCl<sub>2</sub> in the system to inhibit the chlorination of iron oxides [22,23]. In conclusion, the appropriate extension of the roasting duration is beneficial to the recovery of nickel. Considering the nickel grade and recovery, a roasting duration of 80 min is appropriate.



**Figure 6.** Effect of roasting duration on the grades and recoveries of Ni and Fe.

### 3.2.3. Effect of Reductant Dosage

Figure 7 shows the effect of the reductant dosage on Ni, Fe grade, and recovery. As can be seen from Figure 7, when the reductant dosage is increased from 2% to 10%, the Ni grade and recovery are increased from 1.61% and 71.67% to 4.96% and 94.81%. Subsequently, with the increase in the amount of the reductant dosage, the Ni grade and recovery showed a downward trend. The added reductant dosage increased the reducing atmosphere. The strong reducing atmosphere is not conducive to the reduction of nickel [24]. In contrast, for the reduction of iron, more reductant dosages are advantageous. Therefore, as the amount of the reductant dosage increased, the Fe grade showed an upward trend. For the sake of comprehensive consideration, the optimum amount of the reductant dosage is determined to be 10%.

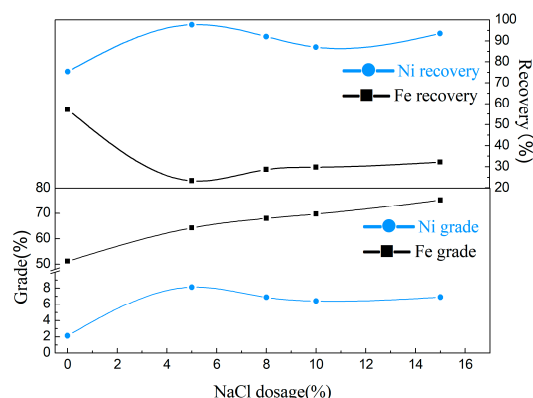


**Figure 7.** Effect of reductant dosage on the grades and recoveries of Ni and Fe.

### 3.2.4. Effect of Additive Dosage

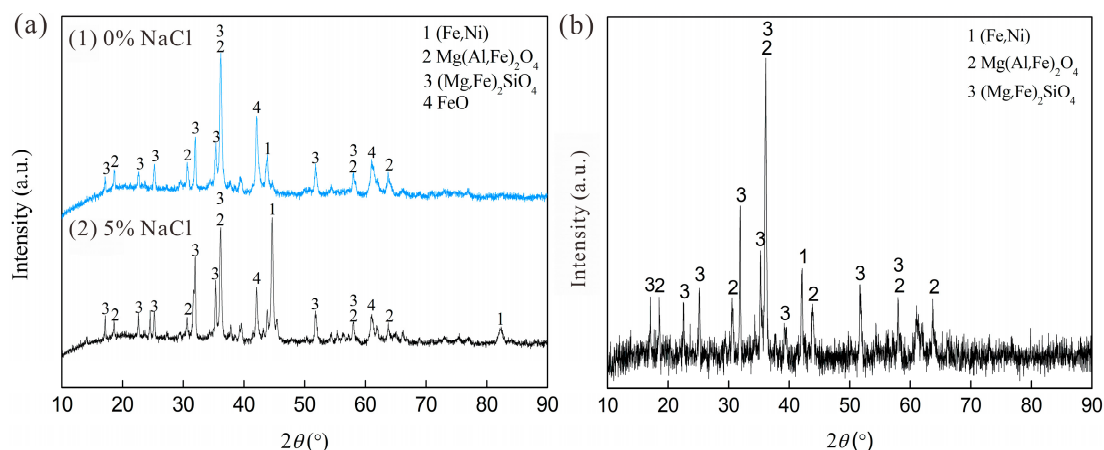
In order to study the effect of different additive dosages on the grade and recovery of Ni and Fe, the experiment was carried out under the conditions of a roasting temperature of 1250 °C, a roasting duration of 80 min, a reductant dosage of 10%, and a grinding time of 12 min. The results are shown in Figure 8. In the condition without additive, the Ni grade and recovery were low, only 2.13% and 75.40%. The Fe grade and recovery were 51.12% and 57.13%. With the addition of additives, the Ni grade and recovery increased rapidly. The Ni grade and recovery at 5% of the additive reached 8.15% and 97.76%. This indicates that the addition of appropriate additives promotes the reduction of Ni. Since this study mainly considered the improvement of nickel grade and recovery, the optimum additive dosage was determined to be 5%.





**Figure 8.** Effect of additive dosage on the grades and recoveries of Ni and Fe.

In order to better compare the effect of the additive on the ore, the roasting product was subjected to XRD analysis. Experimental conditions: roasting temperature 1250 °C, roasting duration 80 min, reductant dosage 10%. The XRD results of roasting products are shown in Figure 9a, (1) no additive and (2) additives with a dosage of 5%. The XRD results showed that there was no significant difference in the phase composition of the two groups, and the forsterite was the main phase. The intensity of the Fe–Ni diffraction peak of (1) is remarkably enhanced as compared to (2). This indicates that the addition of the additive can promote the reduction of Ni and Fe. At the same time, the intensity of the forsterite diffraction peak of (1) is enhanced, indicating the additive promotes the formation of forsterite. The formation of forsterite facilitates the selective enrichment of nickel. The Fe element exists in forsterite and spinel, which is discharged into the tailings during the magnetic separation process, thereby increasing the Ni grade in the concentrate. The XRD pattern of the magnetic separation tailings of the groups (1) (Figure 9b) shows that the main phases in the magnetic separation tailings are forsterite and spinel.

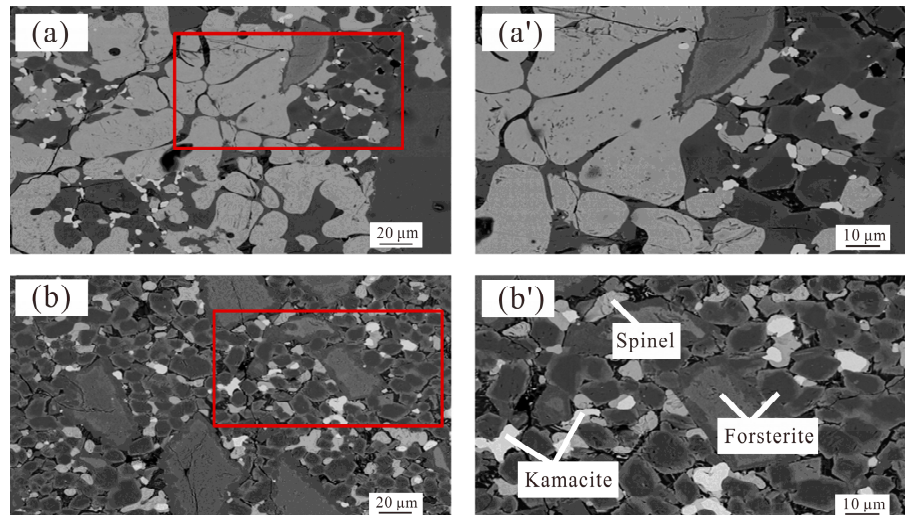


**Figure 9.** XRD patterns of: (a) roasted product; (b) tailing.

The roasting product was subjected to scanning electron microscopy (SEM) (Hitachi, Tokyo, Japan) analysis, and the results are shown in Figure 10: (a) no additive, (b) additives dosage 5%. The bright white phase is ferronickel particles. The light gray phase is the spinel and the dark gray phase is the forsterite. The ferronickel particles in Figure 10a are small and dispersed. The size of the ferronickel particles in Figure 10b is remarkably improved, and the ferronickel particles distributed in the roasting product are large. Related literature reports [25] that the chloride is vaporized at high temperature and adsorbed on the carbon surface to be reduced to metal, and then other chlorides are adsorbed on the metal surface to complete the process of reduction and growth. The spinel phase of Figure 10a is



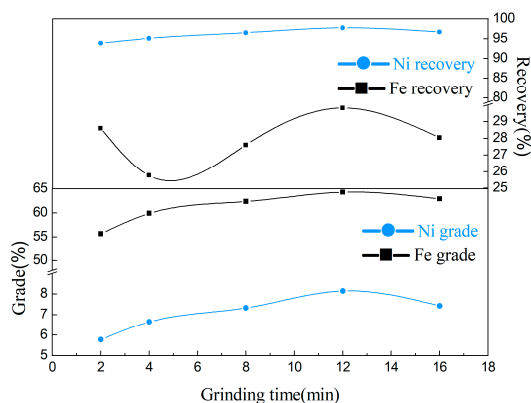
significantly reduced and the forsterite phase is significantly increased, indicating that the additive promotes the formation of forsterite. This result is consistent with the XRD analysis of Figure 9a. In addition, Figure 10b shows a sharp crack in the forsterite phase, which is beneficial to the diffusion of reducing gas and metal chloride, and promotes the reduction of Ni and Fe.



**Figure 10.** SEM images of roasted ore under two conditions: (a) and (a') without NaCl; (b) and (b') 5 wt pct NaCl.

### 3.2.5. Effect of Grinding Time

In order to improve the separation and recovery effect of ferronickel in the magnetic separation process, experimental research on different grinding times was conducted. The experimental conditions were as follows: the roasting temperature was 1250 °C, the roasting duration was 80 min, the reducing agent was 10%, and the additive was 5%. The results are shown in Figure 11. The Ni grade and recovery were 5.78% and 93.89% at 2 min of grinding time. At this time, the roasted product size after grinding is large. The average size of roasted products is  $D_{90} = 45.25 \mu\text{m}$ . The results showed that the ferronickel particles were not well separated from the roasting product. With the prolonged grinding time, the Ni grade increased significantly and reached 8.15% at 12 min, at which time the nickel recovery was 97.76%. The average size of roasted products is  $D_{90} 19.43 \mu\text{m}$ . The results show that prolonging the grinding time is beneficial to the separation of ferronickel particles from roasted products in the magnetic separation process. However, the prolongation of the grinding time continues, and the size of the roasted product after grinding is further reduced. At this point, the Ni grade and recovery decrease, and the average size of roasted products is  $D_{90} = 15.11 \mu\text{m}$ . The reason is that the particle size of some ferronickel particles decreases with the extension of grinding. It is difficult to collect too small ferronickel particles during magnetic separation.



**Figure 11.** Effect of grinding time on the grades and recoveries of Ni and Fe.

#### 4. Conclusions

The higher nickel grade and recovery were obtained by adding sodium chloride. As a cheap and easily available additive, sodium chloride has obvious advantages compared to other additives. The phase transformation in the roasting process was analyzed. The effect of roasting function on the recovery of nickel and iron was explained.

- (1) The addition of sodium chloride as an additive during the reduction process can significantly improve the Ni and Fe grades and recovery in the concentrate. Under the conditions of a roasting temperature of 1250 °C, roasting duration of 80 min, reductant dosage of 10%, additive dosage of 5%, and a grinding time of 12 min, the grade of concentrate Ni and Fe was increased from 1.13% and 51.12% without additives to 8.15% and 64.28%, and the recovery of Ni was increased from 75.40% to 97.76%;
- (2) The addition of additives promotes the transformation of the lizardite phase to the forsterite phase, facilitates the dissociation of nickel from the mineral, and improves the reduction effect of nickel. At the same time, the aggregation and growth behavior of ferronickel particles is improved, and the efficiency of magnetic separation is improved.

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

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