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Efficient Utilization of Siderite- and Hematite-Mixed Ore by Suspension Magnetization Roasting: A Pilot-Scale Study

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Abstract: Steel is an important industrial raw material and plays an important role in industrial construction. Studying the efficient utilization of complex refractory iron ore with large reserves is of great significance to ensure the strategic safety of China's iron and steel industry. Focusing on the typical mixed iron ore of siderite ($FeCO_3$) and hematite (Fe_2O_3), this paper analyzes the possible ways of magnetization roasting from the perspective of thermodynamics and highlights that oxidation–reduction roasting is an easy way to realize industrial application. On this basis, a pilot-scale test of suspension magnetization roasting followed by low-intensity magnetic separation is carried out by using a newly developed suspension magnetization roasting furnace. The effects of roasting temperature, CO consumption, and N_2 consumption on the magnetization roasting process are investigated, and 24 h continuous tests are carried out. Here, we find continuous suspension magnetization roasting followed by low-intensity magnetic separation can obtain a total iron grade of over 56% with an average value of 57.18% and a total iron recovery rate of over 91% with an average value of 92.22%. Product analysis shows that after SMR, iron minerals such as siderite and hematite transform into magnetite, with a substantial increase in magnetism, which is conducive to the separation of iron ore. The results provide a reference for the development and utilization of siderite- and hematite-mixed iron ore.

Keywords: siderite; suspension magnetization roasting; magnetic separation; pilot tests

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1. Introduction

Mineral resources are an important material basis for national economic construction and social development. According to statistics, about 80% of the raw materials, 95% of the energy, 70% of the means of agricultural production, and more than 30% of the drinking water required by social production come from mineral resources. Among the many mineral resources, iron ore resources were the earliest discovered, and they are the most widely used and most consumed mineral resources [1,2]. In recent years, the rapid development of the iron and steel industry has increased demand for a large amount of iron ore. China is a country with many poor mines and few rich mines. According to the data provided by the United States Geological Survey (USGS), the average grade of iron ore in the world is 46.67%, while the average grade of iron ore in China is only about 30%, far below the world average. Therefore, in China, a large number of experts study the efficient utilization of complex and refractory iron ore resources.

Siderite is an important iron ore resource in China, with proven reserves of 1.83 billion tons, accounting for 3.4% of the proven reserves of iron ore [3]. Because the maximum grade of siderite is low (about 48%) and often coexists with calcium, magnesium, and manganese, it is difficult to obtain good beneficiation indexes by physical beneficiation methods [4]. Low-intensity magnetic separation (LIMS) after magnetization roasting is an effective method to treat siderite [5–13]. In this process, weakly magnetic siderite and mixed hematite or limonite can be chemically converted into magnetite or magneto hematite, which is suitable for recovery using LIMS [14–16]. Common magnetization roasting methods include shaft furnace magnetization roasting, rotary kiln magnetization roasting, and fluidization magnetization roasting. The grain size of shaft furnace magnetization roasting is 75~15 mm, whereas that of rotary kiln magnetization roasting is 25~0 mm. Shaft furnace magnetization roasting is associated with high energy consumption and low roasting efficiency, and the roasting products are prone to under-roasting or over-roasting, resulting in a low roasting economy [17–19]. Although rotary kiln magnetization roasting can handle full-size samples, it has the problem of ring formation, which leads to low equipment operation rates [20,21]. At the same time, the roasting energy consumption of rotary kiln magnetization roasting is also relatively high, which reduces its application. The smaller the particle size of the ore block, the larger its specific surface area, the less time required for magnetization roasting reaction, and the more uniform the reduction ore. Fluidized magnetization roasting of powder particles is an efficient roasting method, and it has the following advantages [22,23]: (1) particles are mostly suspended in the gas phase in a good dispersion state, which can facilitate full contact between a gas and solid, and the product quality is uniform and stable; (2) the reaction speed is fast, the heat and mass transfer effect in the reaction process is good, and the heat consumption is low; (3) the temperature and air flow are evenly distributed, easy to control, and possess a high automation level; (4) the equipment has few moving parts, low maintenance costs, and can be easily adjusted. In recent years, fluidized magnetization roasting has been widely studied. In particular, the innovative suspension magnetization roasting (SMR) technology and equipment developed by the Han research team of Northeast University of China have been applied in the Jiuquan Iron and Steel Group, realizing significant technological progress in the efficient utilization of refractory iron ore [24–28]. This process can not only convert the refractory weak magnetic separation iron ore into easily dressed strong magnetic iron ore but also further improve the quality of iron concentrate and iron recovery.

The purpose of this study is to consider the use of new suspension magnetization roasting technology to recover iron from the mixed ore of siderite and hematite. The roasting temperature, CO, and N_2 flow rates were considered and optimized. In addition, a 24 h continuous stable suspension magnetization roasting test was carried out under optimal roasting conditions. Finally, the roasting products were analyzed to further verify the roasting theory and test results.

2. Materials and Experimental Procedure

2.1. Materials

The main chemical composition and iron phase composition of the samples were analyzed by means of chemical analysis, shown in Tables 1 and 2, respectively. The results show that the total iron (TFe) grade was 35.68%, mainly in the forms of siderite, accounting for 71.16%, followed by hematite, accounting for 25.27%, and a small amount of iron in the form of magnetite, ferric sulfide, and ferric silicate existed. SiO_2 was the main impurity element, and the contents of harmful elements P and S were 0.10% and 0.36%, respectively. $(CaO + MgO)/(SiO_2 + Al_2O_3) < 0.5$, indicating that the ore was acidic.

Table 1. Multi-component analysis of iron ore sample (mass fraction, %).

Component	TFe	FeO	P	S	SiO_2	Al_2O_3	CaO
Counts	35.68	33.78	0.10	0.36	20.73	3.23	1.22
Component	MgO	K_2O	Na_2O	H_2O^+	total C	burning loss	
Counts	1.21	0.14	0.060	1.61	6.51	21.05	

Table 2. Iron phase analysis of iron ore sample (mass fraction, %).

Iron Phase	Fe in Magnetite	Fe in Hematite	Fe in Carbonate	Fe in Sulfide	Fe in Silicate	Total
Content	0.87	8.99	25.32	0.263	0.137	35.58
Percentage	2.45	25.27	71.16	0.74	0.39	100.00

The mineral phase composition and content were analyzed using the Mineral Liberation Analyzer (MLA, developed by the JK mineralogy research center, University of Queensland, Australia), as shown in Table 3. The results show that siderite and hematite were the main recycled minerals, with contents of 51.20% and 9.60%, respectively. The gangue minerals were quartz or chalcedony, mica, chlorite, and kaolinite, with contents of 12.56%, 5.30%, 4.45% and 2.38%, respectively. There was also a small amount of apatite, calcite, pyrite and dolomite.

Table 3. Mineral composition and contents of the ore (mass fraction, %).

Minerals	Counts	Minerals	Counts
Siderite	51.20	Maolinite	2.38
Hematite	9.60	Pyrite	0.43
Mixture of iron ore and chlorite	13.38	Apatite	0.30
Quartz or chalcedony	12.56	Calcite	0.20
Mica	5.30	Dolomite	0.18
Chlorite	4.45	Others	0.02

In suspension magnetization roasting, the ore is heated by burning liquefied gas. CO is the reducing gas, and N_2 is the fluidizing gas.

2.2. Experimental Method

The pilot-scale test system (Figure 1) is composed of a feeding system, preheating system, dust collector, reduction system, cooling system, and a central control system. The raw material is preheated using a cyclone preheating (P02) system. Then, the ore enters the heating system (P04), which contains a strong oxidizing atmosphere. After reaching the predetermined temperature, the ore travels under the action of gravity into the reduction system (P05), where there is a strong reducing atmosphere. The material then stays in the reduction system for a certain amount of time before entering the fluidization cooling system (C01). The cooled material is mixed and condensed for magnetic separation. Power for the system comes from the roots blower, and the heat source mainly comes from liquefied gas combustion. The reaction temperature is adjustable between 500 °C and 650 °C. The feeding speed is fixed at 110 kg/h. The reductant is high-purity CO, and the CO concentration is adjusted with N_2 gas flow.

The roasted products are ground to less than 45 μm (pass rate 90%) using a small mill and then separated using a low-intensity magnetic separator. The 24 h continuous stability test is carried out under the optimal conditions. Before the test is started, the fluidized bed temperature should reach the optimal temperature. The roasting products are sampled every 2 h, and then magnetic separation is carried out using a magnetic field intensity of 119.37 kA/m.

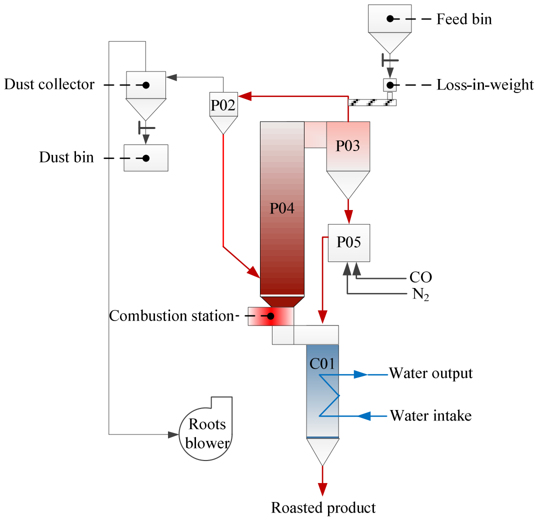

Figure 1. Structure diagram of suspension magnetization roasting furnace.

3. Thermodynamic Basis for SMR

Magnetite has good magnetism, which makes it easy to obtain high-grade iron concentrate through magnetic separation. Hematite, siderite, limonite, and other weakly magnetic minerals usually need other means to improve their iron concentrate grade, such as flotation or magnetization roasting [29,30]. Magnetization roasting is a process of transforming weak magnetic iron minerals into strong magnetic iron minerals. Limonite is transformed into hematite after dehydration, and hematite reacts in a reducing atmosphere to form strongly magnetic magnetite. The main chemical reactions are as follows:

$$Fe_2O_3{\cdot}H_2O \rightarrow Fe_2O_3 + H_2O \; \Delta G^{\theta}_{T} = 49,171 - 152.40T \tag{1}$$

$$3\alpha\text{-}Fe_2O_3 + CO \rightarrow 2Fe_3O_4 + CO_2 \; \Delta G^{\theta}_{T} = -52,130 - 41.0T \tag{2}$$

There are three magnetization roasting methods for siderite: neutral roasting, weak oxidation roasting, and oxidation-reduction roasting. The main chemical reactions of siderite during suspension roasting are as follows:

(1) Neutral roasting

Heated siderite decomposes in a neutral atmosphere; Reaction (3) is as follows:

$$FeCO_3 \rightarrow FeO + CO_2 \; \Delta G^{\theta}_{T} = 74,893 - 180.77T \tag{3}$$

The initial temperature of Reaction (3) is 141 °C, and FeO is oxidized at temperatures above 570 °C due to the instability of FeO.

When the temperature is below 570 °C, Reaction (4) occurs:

$$4FeO \rightarrow Fe + Fe_3O_4 \; \Delta G^{\theta}_{T} = -48,540 + 57.37 \tag{4}$$

Joint Reaction (3) and Reaction (4), give Reaction (5):

$$4FeCO_3 \rightarrow Fe + Fe_3O_4 + 4CO_2 \; \Delta G^{\theta}_{T} = 251,032 - 665.71T \tag{5}$$

When the temperature is above 570 °C, Reaction (6) occurs:

$$3FeO + CO_2 \rightarrow Fe_3O_4 + CO \; \Delta G^{\theta}_{T} = -35,380 + 40.16T \tag{6}$$

Joint Reaction (3) and Reaction (6) give Reaction (7):

$$3FeCO_3 \rightarrow Fe_3O_4 + CO + 2CO_2 \; \Delta G^{\theta}_{T} = 39,513 - 140.61T \tag{7}$$

(2) Weak oxidation roasting

It can be seen from Reaction (7) that the chemical equilibrium shifts to the right when the CO content is lower. Therefore, the introduction of a small amount of O_2 can reduce the CO content and increase the CO_2 content.

$$2CO + O_2 \rightarrow 2CO_2 \ \Delta G_T^\theta = -561,900 + 170.46T \tag{8}$$

At the same time, oxygen can also react with FeO to give Reaction (9):

$$6FeO + O_2 \rightarrow 2Fe_3O_4 \ \Delta G_T^\theta = -774,180 + 411.42T \tag{9}$$

Above 400 °C, γ-Fe_2O_3 is transformed to α-Fe_2O_3 [31]. In an oxidation environment with temperatures higher than 400 °C, Fe_3O_4 will also be oxidized, and Reaction (10) occurs:

$$4Fe_3O_4 + O_2 \rightarrow 6\alpha\text{-}Fe_2O_3 \ \Delta G_T^\theta = -457,640 + 252.46T \tag{10}$$

When the temperature is below 400 °C, Reaction (11) occurs:

$$4Fe_3O_4 + O_2 \rightarrow 6\gamma\text{-}Fe_2O_3 \ \Delta G_T^\theta = -453,626 + 256.67T \tag{11}$$

After introducing a small amount of oxygen to a neutral atmosphere, the main reaction of Reactions (8)–(11) can be identified from the Ellingham diagram of these reactions (Figure 2).

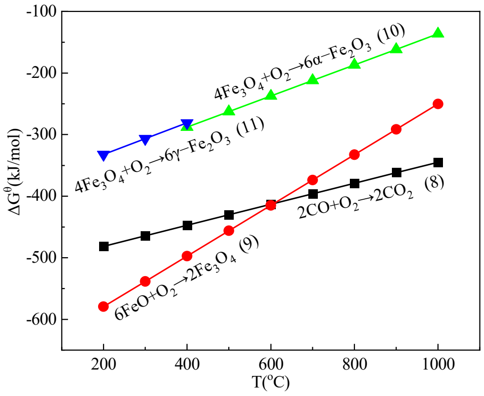

Figure 2. Ellingham diagram of siderite oxidation roasting intermediate product.

The lower the position of the reaction in the Ellingham diagram, the more easily the reaction occurs. As can be seen from Figure 2, Reactions (8) and (9) intersect at 608 °C, so when $t < 608$ °C, Reaction (9) is the dominant reaction, and when $t > 608$ °C, Reaction (8) is the dominant reaction. Reactions (8) and (9) do not intersect at 400 °C, which may be due to the fitting of Gibbs free energy to ΔG_T^θ = A+BT form, but this does not affect our judgment on the reaction trend. Reactions (8) and (9) are lower than Reactions (10) and (11), indicating that Fe_3O_4 is not easily oxidized without excessive oxygen.

Therefore, the total reaction equation of $FeCO_3$ roasting in a weak oxidizing atmosphere is:

$$6FeCO_3 + O_2 \rightarrow 2Fe_3O_4 + 6CO_2 \ \Delta G_T^\theta = -54,137 - 112.20T \tag{12}$$

(3) Oxidation–reduction roasting

The siderite is oxidized to weak magnetic α-Fe_2O_3 in a strong oxidizing atmosphere. α-Fe_2O_3 is then reduced to Fe_3O_4 in a reductive environment. Reaction (13) is shown below followed by Reaction (2):

$$4FeCO_3 + O_2 \rightarrow 2\alpha\text{-}Fe_2O_3 + 4CO_2 \ \Delta G_T^\theta = -369,095 - 364.65T \tag{13}$$

The roasting of siderite-containing refractory iron ore in a neutral or weak oxidizing atmosphere needs to be strictly controlled and may change with variations in hematite content. However, the oxidation–reduction roasting of siderite can easily be realized in industry due to the ease of performing oxidation followed by reduction.

A feasible path is as follows: combined with the characteristics of the new suspension magnetization roasting furnace, siderite–hematite-mixed ore is first heated in a strong oxidizing atmosphere, where it is convenient to control the full combustion of fuel. It is then placed in a reducing atmosphere so that it can be unified into strongly magnetic magnetite, and, then, low-intensity magnetic separation can be utilized to obtain high-quality iron concentrate.

4. Results and Discussion

4.1. Effect of the Roasting Temperature

Roast temperature is a key factor influencing roasting efficiency. The higher the roasting temperature, the faster the reaction rate. The suspension roasting temperature is adjusted by controlling the liquefied gas supply to the combustion station. Figure 3 shows the effect of roasting temperature on suspension roasting performance at a CO gas flow rate of 4.5 Nm^3/h and a N_2 gas flow rate of 0 Nm^3/h.

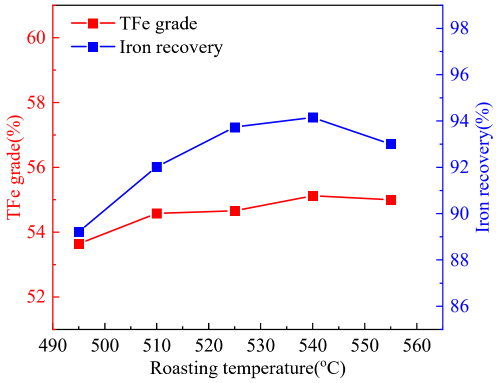

Figure 3. Roasting temperature test results.

As shown in Figure 3, as the roasting temperature increases from 495 °C to 540 °C, the TFe grade of iron concentrate increases from 53.64% to 55.12%, and the iron recovery rate increases from 89.21% to 94.15%. This is because as the temperature increases, the reaction rate accelerates and the reaction becomes more efficient. However, if the roasting temperature continues to rise, the TFe grade and recovery rate of iron concentrate somewhat decrease, which may be attributed to the sample temperature being too high to cool down quickly and then becoming oxidized by air again. Through comprehensive analysis, it is estimated that the most efficient roasting temperature lies between 525 °C and 540 °C.

4.2. Effect of CO Consumption

CO is used as a reduction gas in suspension roasting. CO consumption impacts the reduction effect, where reduction is more efficient when the CO volume is optimal. However, an excessive CO volume will lead to further reduction of magnetite to FeO, which has poor magnetic properties, thus reducing the recovery efficiency of magnetic separation. Figure 4 displays the effect of CO consumption on suspension roasting performance at 525 °C and a N_2 flow rate 0 Nm^3/h.

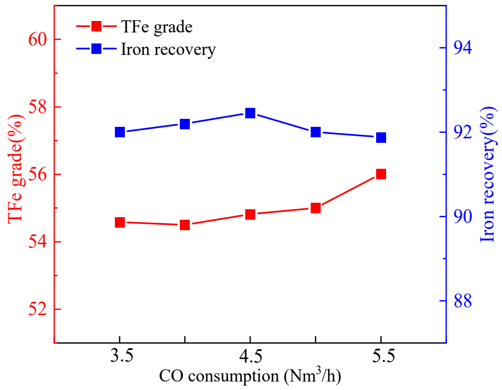

Figure 4. CO consumption test results.

At 110 kg/h, the theoretical dosage of CO required is 3.54 Nm^3/h (calculated according to Reactions (11) and (12)). As shown in Figure 4, with an increase in CO consumption from 3.5 Nm^3/h to 5.5 Nm^3/h, the TFe grade of iron concentrate increased from 54.58% to 56.01%. The iron recovery rate initially increased and then decreased. This may be due to the increase in reducing gas, which led to less residence time of materials in the reactor, thus affecting the iron recovery rate. Hence, the optimal CO consumption was determined to be 4.5 Nm^3/h, with TFe grade and TFe recovery rates of iron concentrate are 54.82% and 92.46%, respectively. Under such conditions, the excess coefficient of CO is 27%, with the excess CO being introduced into the furnace for combustion, ensuring safety, and saving energy.

4.3. Effect of N_2 Consumption

The gas flow rate will affect the fluidization state in the reactor, thus affecting the residence time of particles in the reactor. Under the conditions of higher than the critical fluidization speed and an increased gas flow rate, the fluidization state in the reactor will change from scattered to aggregated fluidization. Following this, a pneumatic conveying system will be formed [32]. In this reactor, the state of the fluidized bed is either a scattered or an agglomerated fluidized bed. N_2 is used to regulate the fluidization state in the reactor to obtain better test results. Figure 5 displays the effect of N_2 consumption on suspension roasting performance under the roasting conditions of 525 °C and CO consumption of 4.5 Nm^3/h.

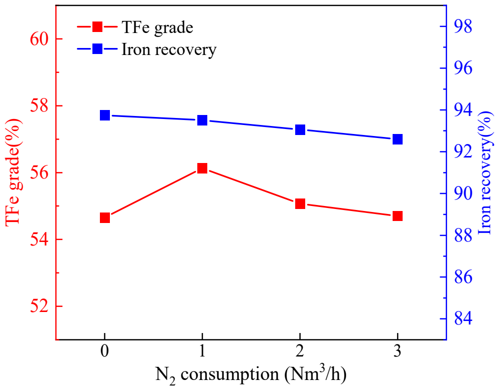

Figure 5. N_2 consumption test results.

The experimental results are shown in Figure 5, where an increase in N_2 consumption leads to an decrease in the recovery rates of iron concentrate from 93.74% to 92.60%. This phenomenon may have resulted from the reduction in residence time due to the increase in N_2 flow. The iron concentrate TFe grade increased from 54.66% to 56.13% and then decreased to 54.7%. Therefore, an appropriate flow rate of nitrogen was determined at

1 m^3/h. In this way, not only can the material maintain a good fluidization state, but it can also ensure a long residence time.

4.4. 24 h Continuous Suspension Magnetization Roasting Test

To verify the quality and stability of products yielded from suspension roasting, a pilot-scale continuous stability test was carried out with the following conditions: reduction temperature of 525~540 °C, CO consumption of 4.5 Nm^3/h, and N_2 consumption of 1 Nm^3/h. Subsequently, a continuous stability operation was performed for 24 h, sampling every 2 h.

The test results are presented in Figure 6. Continuous suspension roasting can obtain an iron concentrate TFe grade of more than 56%, (average value of 57.18%) and an TFe recovery rate of more than 91% (average value of 92.22%), indicating that continuous suspension roasting produces good stability.

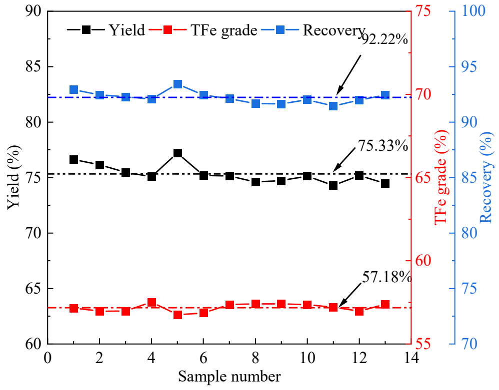

Figure 6. Separation indexes for continuous and stable operation experiment.

4.5. Property Analysis of Roasting Products

4.5.1. Chemical Analysis of Roasting Products

The chemical analysis of iron ore suspension roasting–magnetic separation products is presented in Table 4. Compared with Table 1, TFe increased from 35.68% to 45.10% after roasting, which was attributed to carbon loss in siderite and oxygen loss in hematite. After magnetic separation, the grade of magnetic concentrate was 57.18%, and the grade of tailings was 12.31%, indicating that the iron ore after roasting was effectively separated. This is attributed to the transformation of siderite and hematite into magnetite after suspension roasting.

Table 4. Chemical analysis of suspension roasting–magnetic separation products (%).

Sample	TFe	P	S	SiO_2	Al_2O_3	CaO	MgO	K_2O	Na_2O	MnO_2
Roasted product	45.10	0.15	0.31	26.97	3.97	1.69	1.77	0.13	0.07	1.30
Concentrate	57.18	0.13	0.35	12.42	2.37	2.28	1.97	0.07	0.06	1.66
tailings	12.31	0.16	0.16	69.64	8.71	1.04	0.72	0.45	0.10	0.16

4.5.2. Iron Phase Analysis

The results of iron phase analysis for the suspended roasting–magnetic separation products are shown in Table 5. Compared with Table 2, iron mainly exists in magnetite in roasted products with a distribution rate of 93.72%, followed by hematite and siderite with distribution rates of 3.40% and 2.26%, respectively. This suggests that iron ore is converted into magnetite after suspension roasting. The content of magnetite in magnetic concentrate was 55.13%, with a distribution rate of 97.20%. The content of siderite, hematite, and magnetite in tailings was 4.63%, 3.94% and 3.47%, respectively, further indicating that the phase transformation of iron ore occurs in the process of suspension roasting.

Table 5. Phase analysis results of roasted products (%).

Sample		Fe in Hematite	Fe in Carbonate	Fe in Sulfide	Fe in Silicate	Fe in Magnetite	Total
Roasted product	Count	1.02	1.54	0.032	0.25	42.39	45.232
	Distribution	2.26	3.4	0.07	0.55	93.72	100.00
Concentrate	Distribution	0.39	0.79	0.018	0.39	55.13	56.718
	Count	0.69	1.39	0.03	0.69	97.20	100.00
tailings	Count	4.63	3.94	0.056	0.014	3.47	12.11
	Distribution	38.23	32.54	0.46	0.12	28.65	100.00

4.5.3. Phase Transformations of Roasted Products

XRD patterns of the products roasted at different temperatures for continuous stability are shown in Figure 7. Compared with the raw ore, it is evident that there is no diffraction peak for siderite in the roasting products, whereas diffraction peaks for both magnetite and hematite are observed. This indicates that the siderite presenting in the raw ore was transformed into magnetite. This is seen in all the XRD curves of roasted products obtained at different roasting temperatures. The largest diffraction peaks of magnetite were observed when roasted at 525 °C. The diffraction peaks for quartz did not change, indicating that there was no phase change of quartz during the roasting process.

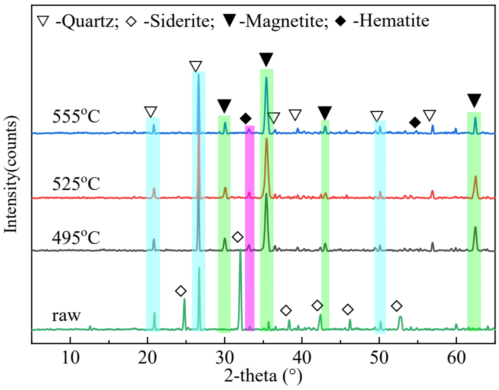

Figure 7. XRD patterns of products roasted at different temperatures.

4.5.4. Magnetic Performance of the Iron Ore before and after SMR

The magnetic properties of raw ore and roasting products were tested, and the results were shown in Figure 8. The specific magnetization of the raw ore is nearly a straight line, indicating that the raw ore is a weak magnetic mineral. The magnetization of roasted products increased significantly with the increase in magnetic field strength. At a magnetic field strength of 30.97 kA/m, the maximum specific magnetization was 4.995×10^{-4} m^3/kg. This demonstrates that suspension roasting enhances the magnetic properties of minerals, which is beneficial to iron ore separation.

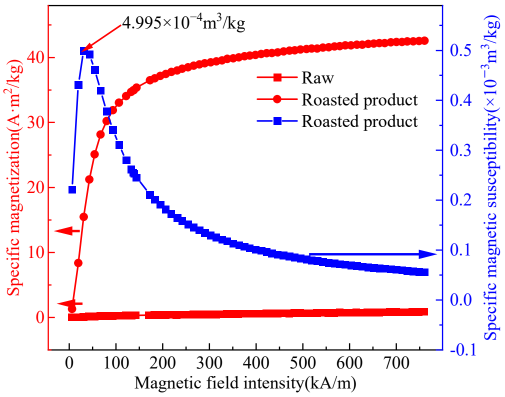

Figure 8. Magnetic analysis of iron ore.

5. Conclusions

(1) After suspension magnetization roasting, the TFe grade of raw material increased from 35.68% to 45.10%. After magnetic separation, iron concentrate with a TFe grade of 57.18% and tailings with a TFe grade of 12.31% were obtained. In total, 93.72% of the iron in the roasted product existed as magnetite, and the value in the iron concentrate was 97.20%.

(2) The new suspension roaster is suitable for processing siderite–hematite-mixed ore. The continuous test results of the 24 h pilot-scale demonstrated that suspension roasting is stable. Under the conditions of a roasting temperature of 525–540 °C, a CO consumption of 4.5 Nm^3/h, and a N_2 consumption 1 Nm^3/h, a TFe grade of over 56% with an average value of 57.18% and an iron recovery rate of over 91% with an average value of 92.22% were obtained.

(3) The analysis of suspension magnetization roasting products demonstrated that after suspension magnetization roasting, magnetism is significantly enhanced, and siderite, hematite, and other weak magnetic minerals are transformed into strong magnetic magnetite.

The test results show that suspension magnetization roasting followed by magnetic separation is an effective method for treating siderite–hematite-mixed iron ore.

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