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Pilot Study on a New Conveyor Bed Magnetization Roasting Process for Efficient Iron Extraction from Low-Grade Siderite

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Abstract: Realizing the large-scale development and utilization of siderite, a difficult iron ore reserve, has great practical significance in ensuring the supply of iron ore resources. Therefore, a new in-house conveyor bed magnetization roasting–dry cooling process was pilot-tested using low-grade siderite from the Daxigou iron ore mine. A two-stage weak magnetic separation method was used for a beneficiation test to investigate the influence of temperature and CO content on the magnetization of siderite. At 600 °C and 800 °C under suspension, iron minerals were converted into magnetite with an effective 3–5 s residence time. Furthermore, at 600 °C and 750 °C, increasing the calcination temperature increased the iron grade and the concentrate recovery rate. However, calcination at temperatures >750 °C resulted in a slight decrease in the iron grade and recovery rate of the concentrate. 61.50% Fe grade and 80.30% concentrate recovery rate were obtained under 750 °C from magnetization roasting. Magnetization roasting in a reducing atmosphere provides mainly magnetite as the roasted ore, and increased CO content can efficiently promote this effect. At 700–780 °C and when the CO content was increased to more than 3 wt.%, the improvement of the roasting effect was very limited. Rapid cooling of the roasted ore using a mixture of circulating exhaust gas and air could prevent considerable oxidation of the magnetic ferrous material. Therefore, the preferred process conditions are 700–780 °C with a CO content range of 1–3%. It provided a concentrate iron grade of 59.27–61.50% and a recovery rate of 78.32–80.30%. The results of this study provide a reference for the development of conveyor bed magnetization technology, process design, and production control.

Keywords: siderite; conveyor bed; magnetization roasting; dry cooling method; magnetic separation

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1. Introduction

Siderite reserves in China are as high as 1.8 billion tons; however, they have not been utilized on a large scale owing to technological and production cost constraints [1]. The global supply of high-quality iron ore resources is becoming insufficient with the increasing demand for iron and steel to realize economic and social development; hence, developing new technologies for the comprehensive utilization of hard-to-elect iron ores is of great practical importance, particularly in China [2–4]. The Daxigou iron ore mine, located in Shangluo, Shaanxi Province, China, is the largest iron ore mine in China, with a reserve of more than 300 million tons. Extensive technical research and development confirmed that the Daxigou iron ore has a considerable development value; however, successful large-scale industrial production has not yet been achieved [5,6]. A process system was developed for the magnetization roasting of siderite based on suspended-state calcination technology, and a 10,000-ton pilot plant was built for this purpose [7]. As this technology is crucial to the comprehensive utilization of siderite, conducting research on pilot-scale process technology is necessary to apply it industrially [8–10].

The magnetization roasting of siderite includes the efficient decomposition and conversion of $FeCO_3$ into magnetite and avoiding the oxidation of roasted ore during cooling so that the magnetic separation effect is not affected. The iron mineral transformation pattern, reaction kinetics, and magnetic separation of siderite by magnetization roasting have been systematically investigated in previous studies [2–5]. These results confirmed that siderite could be efficiently magnetized by calcination at 600–800 °C for 3–10 s in a reducing atmosphere, and magnetite could be effectively collected by the multiprocess weak magnetic separation method [7,11–13]. Existing studies have confirmed the feasibility and advantages of suspended-state magnetization roasting technology (e.g., fast reaction, good magnetization effect, mature process equipment, etc.). However, owing to the complexity of the reaction and control system, the operational stability of the process in a 10,000-ton pilot system and the influence law of calcination conditions for the calcination effect need to be verified [14–16]. Furthermore, necessary foundations for further industrial design parameters also need to be provided.

Additionally, existing magnetization roasting technology usually adopts the water-cooling method to prevent the oxidation of roasted ore; this is considered the most effective cooling method. However, water cooling has many disadvantages when used in large-scale industrial production. First, it requires a large amount of water and hence is severely limited in areas where water is scarce. Second, the large amount of heat energy that the cooling water removes is not effectively used, which leads to increased energy consumption and higher production costs. Moreover, the excessive high-temperature water vapor generated during the cooling process is very difficult to dispose of. Additional devices and processes must be introduced, increasing construction investment and production costs. Therefore, a reasonable approach is to use dry cooling to avoid these problems [17]. For powdered materials, a suitable medium for efficient dry cooling is gas; however, the introduction of air cooling that leads to magnetite oxidation is an issue [2,4,7,11,13]. Recently, much research has been carried out on dry cooling technology; their results have confirmed that if the dry cooling rate is fast enough, the magnetite can directly cross the oxidation reaction stage, and their oxidation can be prevented [18]. This finding provided the foundation for the development of the dry cooling process, which led to the introduction of multi-stage suspension cooling units that recirculate most of the very low oxygen content exhaust gas into the cooling unit, thereby reducing oxidation. Although the conveyor bed magnetization roasting-dry cooling process system is theoretically and fundamentally feasible, its intrinsic laws and practical application in large-scale production have to be studied and demonstrated in-depth [7,11,13].

This paper reports a pilot study on a 10,000-ton conveyor bed magnetization roasting–dry cooling process system using Daxigou siderite as the raw material. A two-stage weak magnetic separation method was adopted to recover the magnetite, and the magnetization effect was characterized by the improvement in iron grade and recovery rate of the concentrate. In addition, the effect of the magnetization roasting temperature and CO content on the magnetization of the siderite was investigated to optimize the roasting parameters. This paper also analyzes the power consumption required for grinding and the energy consumption of the magnetization roasting process, and it discusses the economics of this new process. The results of this paper will provide a reference for the development of conveyor bed magnetization technology, process design, and production control.

2. Materials and Methods

2.1. Raw Materials

The test raw materials were collected from the Daxigou iron ore mine in Shangluo City, Shaanxi Province, China. The raw ore was crushed by a crusher into lumps with a particle size <5 cm and then ground into powder using a vertical mill. The particle size of the finished product was a −200-mesh standard sieve, accounting for 98.47% of raw ore powder (d50 = 41.68 μm). The finished material, after grinding, was mixed thoroughly and then loaded into a silo for the conveyor bed magnetization roasting test.

X-ray diffraction (XRD; D/MAX-2200, RIKEN, Wako City, Japan) was used to analyze the mineral phases of the siderite. The test conditions were as follows: Cu target Kα rays, a

45 kV tube voltage, a 40-mA tube current, and a scanning angle 2θ, ranging from 5° to 80°. The XRD pattern of the raw material is shown in Figure 1.

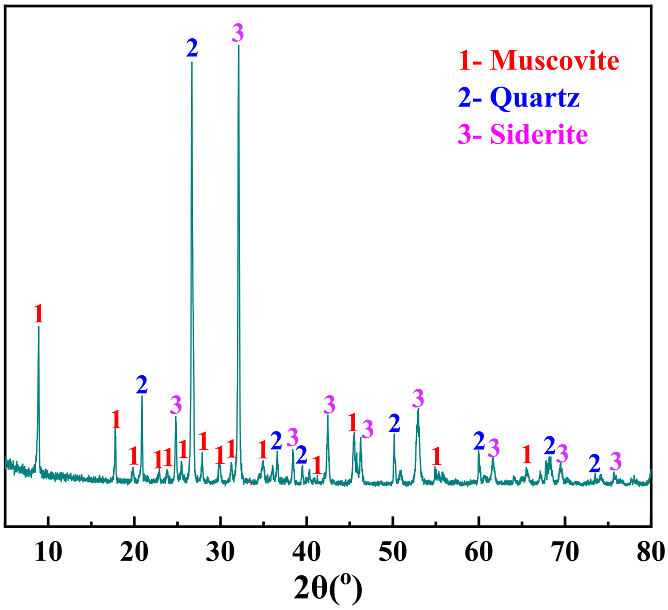


Figure 1. X-ray diffraction pattern of Daxigou siderite.

The chemical elements of the test siderite were analyzed using an X-ray fluorescence spectrometer (S4PIONEER, Bruker, Karlsruhe, Germany). The X-ray tube parameters were as follows: 4.2 kW, 60 kV (Max), and 140 mA (Max). The iron grade was analyzed using the chemical analysis method, and the results of the chemical elemental analysis are shown in Table 1. The main iron mineral in the raw material was $FeCO_3$, and quartz and mica are the impurities present in the ore. Furthermore, siderite also contains Mn, Mg, S, and other elements. Daxigou siderite is a sedimentary, metamorphic type with an average iron grade of 21.38 wt%, typical of low-grade difficult iron ore.

Table 1. Chemical composition of siderite (wt%).

Al_2O_3	SiO_2	Fe_2O_3	CaO	MgO	MnO	K_2O	SO_3	T_{Fe}
11.07	36.85	39.45	0.81	1.96	0.64	2.63	0.39	21.38

2.2. Backscattered Electron and Energy Dispersive Spectroscopy (BSE-EDS)Analysis

The backscattering method was used to analyze the micromorphology of the raw siderite ore. The image showing the backscattering is shown in Figure 2, and the analysis of areas 1 and 2 are shown in Table 2. The results of the analysis showed that the minerals in the white areas (e.g., area A) are mainly iron-bearing minerals (primarily $FeCO_3$), and the gray areas (e.g., area B) are mainly oxides of silica and aluminum (quartz and mica). $FeCO_3$ and veinlets in the Daxigou siderite show a complex intercalation distribution pattern with $FeCO_3$ distributed in large continuous masses that can be well dissociated on a scale of 50–80 μm. On the other hand, iron ore distributed as microfine particles is interspersed with veinlets and need to be dissociated on a scale of 10–20 μm. This is the reason for grinding this material to 200 mesh size.

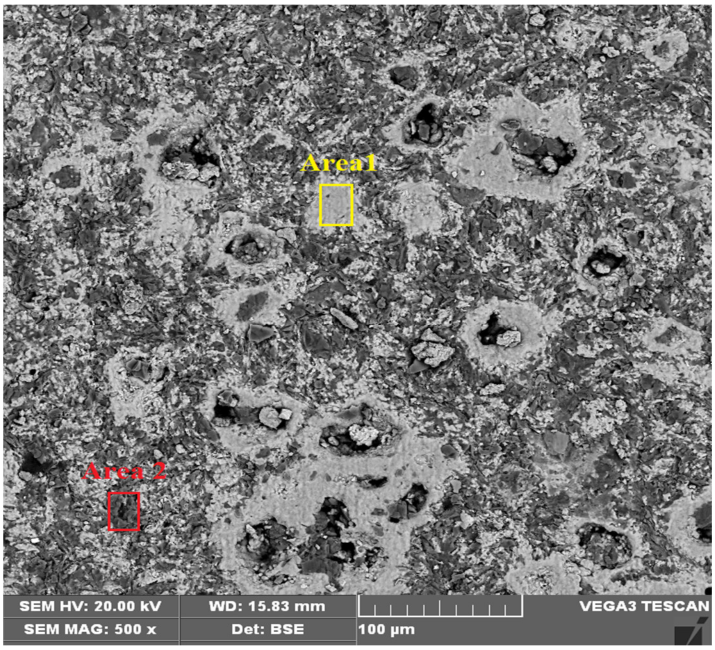


Figure 2. Backscattering image of siderite.

Table 2. Energy spectrum analysis of Areas 1 and 2.

Area 1			Area 2		
Element	Mass Percentage (%)	Atomic Percentage (%)	Element	Mass Percentage (%)	Atomic Percentage (%)
O	28.97	56.37	O	42.58	56.23
Fe	63.65	35.48	Si	48.37	37.12
Si	5.45	6.15	Fe	3.18	1.22
Mg	1.01	1.30	K	2.14	1.35
Ca	0.28	0.22	Al	3.56	2.93

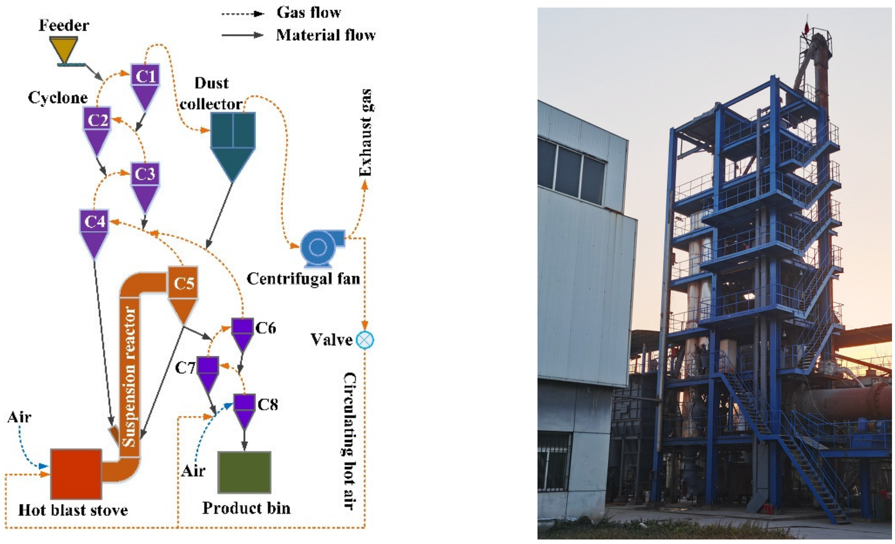
2.3. Analysis of Grinding Work

The new conveyor bed magnetization roasting–the dry cooling process uses powdered material with tens of micron scale, which must be sufficiently grounded from the raw iron ore. In the traditional rotary kiln process, lumpy materials several centimeters in size are taken, magnetized, and roasted before wet grinding. Calcination is generally considered useful as it reduces the grinding power consumption; hence, the grinding power of the new process is a parameter of interest. In this paper, the grinding work index of Daxigou siderite is analyzed concerning the "Test method for easy grinding of cement raw materials" [19]. The test equipment is a φ305 mm × 305 mm standard test ball mill, and a 200-mesh product sieve is used as the product size control standard.

The grinding power index of the Daxigou iron ore is 18.12 $kW \cdot h \cdot t^{-1}$, indicating that the power consumption of grinding 1 t of Daxigou iron ore to −200 mesh is 18.12 kW·h. This is because the magnetite content of the Daxigou iron ore is very low; moreover, the ore is brittle, and the grinding power index is close to that of limestone, which is a medium-difficult material.

2.4. Conveyor Bed Magnetization Roasting Pilot

A schematic of the new conveyor bed magnetization roasting-dry cooling process, including outlets C1 to C8 and an image of the plant is shown in Figure 3. The preheating unit of the pilot system comprises a four-stage cyclone; the conveyor bed reactor is a ducted structure, and the cooling unit contains a three-stage cyclone. The hot air furnace is fueled by pulverized coal; a portion of the exhaust gas discharged from the bag collector circulates through the hot air furnace and cooling unit to reduce the oxygen content in the system. In addition, each unit is equipped with temperature and pressure measurement points at the inlet and outlet pipelines and gas composition measurement points at the outlet of the conveyor bed reactor (C5 inlet; Figure 3) for the O_2 and CO content analysis.



(**a**) (**b**)

Figure 3. (**a**) Conveyor bed magnetization roasting process system flowchart and (**b**) image of the plant.

During the pilot test, the roasting temperature was controlled between 600 °C and 800 °C (middle temperature of the furnace), and the CO content in the C5 outlet gas was 0–5%. The residence time of the material in the suspension reactor was about 3–5 s. The temperature range of the material discharged from C8 is 160–230 °C. The charge was about 600–800 kg/h, and the continuous operation time was 72 h. Temperature and CO, content influence tests were carried out under stable operation for 2–3 h using each set of parameters.

2.5. Analysis of Roasted Ore, Iron Ore Concentrate, and Tailings

XRD analysis was conducted on the roasted ore, concentrate, and tailings to estimate the composition of the iron minerals. Chemical titration was utilized to analyze the composition of the iron compounds in various treated iron ores. The effect of the process parameters (i.e., temperature and CO content) on the magnetization effect was investigated using iron grade (the mass percent of iron in the sample) and recovery rate (the percentage of iron in the concentrate to the total iron, including that in the concentrate and tailings).

2.6. Magnetic Separation Analysis

Magnetic separation involves one rough and one selected two-stage weak magnetic separation process, beneficiation parameters, and processes, as shown in Figure 4. Based on the BSE-EDS analysis results shown in Figure 2, the grinding size of the ore was strictly controlled during the selection, which is crucial to ensuring the beneficiation effect. XRD and chemical titration analyses were performed on the concentrated iron ore and tailings to determine the iron grade and estimate the iron yield and recovery rate.

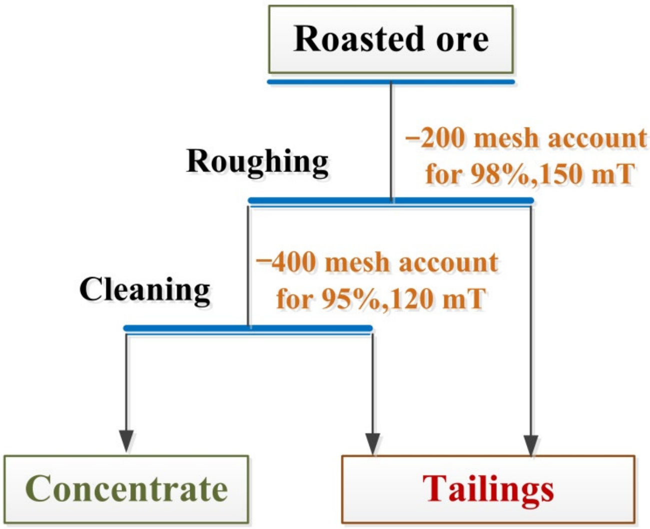


Figure 4. Flow chart of magnetic separation.

3. Results and Discussion

3.1. Effect of Temperature on Iron Extraction from Low-Grade Siderite

The primary mineral components of Daxigou siderite are $FeCO_3$, quartz, and muscovite, and the main reactions that occur during calcination are as follows [1,3,4,20]:

$$FeCO_3 \rightarrow FeO + CO_2\uparrow \tag{1}$$

$$3FeO + CO_2 \rightarrow Fe_3O_4 + CO\uparrow \tag{2}$$

$$KAl_2(Si_3Al)O_{10}(OH)_2 \rightarrow KAl_2(Si_3Al)O_{11} + H_2O_{(g)}\uparrow \tag{3}$$

Owing to the difficulty of separating reactions (1) and (2), they can be written as the following reaction equation:

$$3FeCO_3 \rightarrow Fe_3O_4 + 2CO_2 + CO\uparrow \tag{4}$$

Based on reactions (2) and (4), siderite produces CO during decomposition. CO prevents the oxygen from entering the conveyor bed reactor and thus avoids the oxidation of magnetite to Fe_2O_3. Therefore, continuous production of CO is also essential to maintain a reduced atmosphere in the conveyor bed reactor.

The effect of temperature on the conversion of iron minerals into magnetite was analyzed using the XRD patterns of the roasted ores (with CO content of 1–3%). The XRD patterns of the roasted ores at different temperatures are shown in Figure 5.

As shown in Figure 5, $FeCO_3$ and white mica underwent noticeable decomposition reactions after magnetization roasting, whereas quartz showed no obvious changes. At calcination temperatures of 600 °C and 650 °C, weak $FeCO_3$ diffraction peaks were observed, indicating that a small amount of undecomposed $FeCO_3$ was still present in the roasted ore. However, when the roasting temperature was increased to >700 °C, no $FeCO_3$ diffraction peaks were detected in the roasted ore, indicating its complete decomposition. White mica first underwent a dehydroxylation reaction during the roasting process, followed by a desilication at a high temperature, gradually becoming amorphous. Figure 5 shows that the muscovite diffraction peak decreased as the roasting temperature increased. At 600–800 °C, considerable magnetite diffraction peaks were detected in all the roasted ores. Further study revealed that the height of the magnetite diffraction peaks increased gradually with increasing temperature in the range of 600–750 °C, indicating increasing the magnetite content. When the roasting temperature was >750 °C, an increase in the roasting temperature caused the magnetite diffraction peak in intensity to decrease slightly. When the roasting temperature was 800 °C, FeO was detected in the roasted ore, indicating that the overreduction was responsible for decreased magnetite content [11].

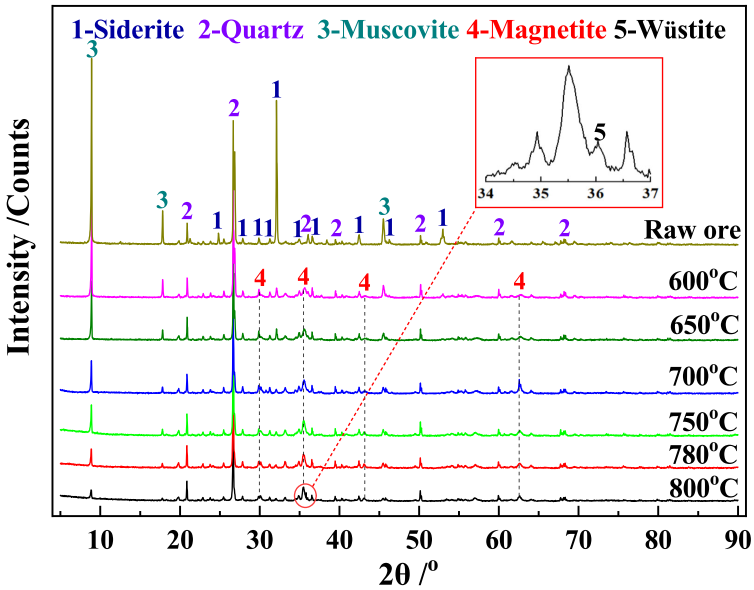
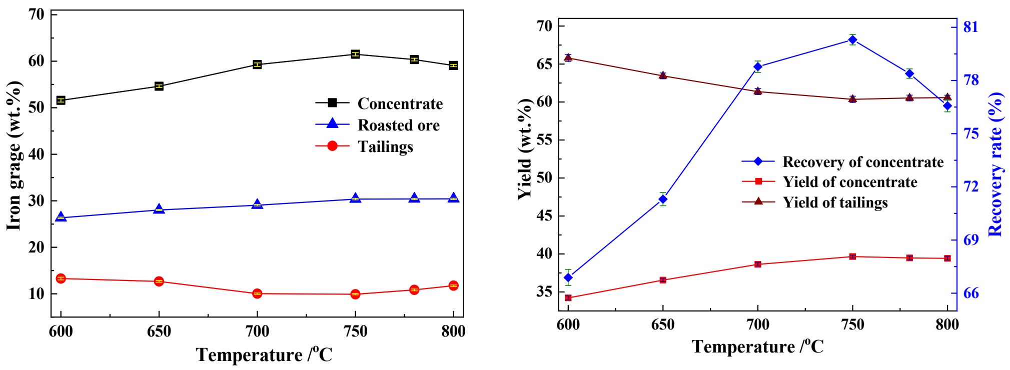


Figure 5. XRD patterns of the roasted ore.

The variation in iron grade with temperature is plotted in Figure 6a. The yield and recovery rate trends against temperature are plotted in Figure 6b.



(**a**) (**b**)

Figure 6. (**a**) Iron grade and (**b**) yield and recovery of samples.

No iron loss occurred during roasting. Hence it was concluded that the iron grade of the roasted ore was mainly affected by the decomposition of $FeCO_3$ and white mica. The decomposition of $FeCO_3$ and white mica produced a mass loss that increased the relative iron content of the roasted ore, thereby increasing the iron grade. Moreover, the iron grade of the roasted ore increased more slowly as the roasting temperature increased because most of the $FeCO_3$ and muscovite were decomposed at 600 °C. The relatively stable roasted ore grade, the iron grade of the concentrate, and the iron grade of the tailings were correlated, as shown in Figure 6a. The magnetite content increased gradually with an increase in the roasting temperatures (between 600 °C and 750 °C), and the iron grade of the concentrate also increased gradually under similar magnetic separation conditions. Accordingly, the tailing iron grade gradually decreased as more iron minerals entered the concentrate. When the temperature increases above 750 °C, the concentrate iron grade decreases slightly. The tailing iron grade increased slightly due to the appearance of FeO in the roasted ore due to an overreduction phenomenon, causing the weakening of magnetism. The concentrate iron grade reached a maximum of 61.50% at 750 °C and was >59% in the temperature range of 700–800 °C, which meets the requirements of raw material iron grade for iron and steel metallurgy.

As shown in Figure 6b, the yields of the concentrate and the tailings were also correlated. With an increase in the magnetite content of the roasted ore, the yield of the concentrate gradually increases, whereas that of the tailings gradually decreases. Above 750 °C, as the magnetism of the roasted ore decreased slightly, the concentrate yield decreased proportionately, whereas the tailing yield increased slightly.

The recovery rate is an important economic indicator for the utilization technology of iron ore, provided that the iron grade of the concentrate meets the requirements of the application. Furthermore, a high recovery rate implies better economics. The recovery rate is mainly affected by the magnetic mineral content in the roasted ore and the beneficiation process parameters, particularly the magnetic field strength and the dissociation size (i.e., grinding fineness). The beneficiation process used in this paper has been verified. Its beneficiation effect is ideal since the recovery rate depends mainly on the content of magnetic minerals in the roasted ore. As shown in Figure 6b, the recovery trend is positively correlated with the iron grade of the concentrate, but the variation in recovery rate is more than that in iron grade. With an increase in temperature, the magnetite content in the roasted ore increases and the recovery rate also gradually increases when the temperature is between 600 °C and 750 °C. However, above 750 °C, the magnetite magnetism decreases due to overreduction, and hence, the recovery rate also decreases accordingly. The recovery rate reached a maximum of 80.30% at 750 °C and remained above 78% between 700 °C and 780 °C. Considering the concentrate iron grade and recovery indices, the appropriate temperature for the conveyor bed magnetization roasting process is found to be 700–780 °C, antemperature is around 750 °C. In this range, a better index of concentrate iron grade (>59%) and recovery (>78%) can be obtained, which not only meets the quality requirements of the metallurgical industry for concentrate but also provides a better economy.

3.2. Effect of CO Content on Iron Extraction from Low-Grade Siderite

To investigate the effect of CO content on the magnetization roasting of siderite, a total of three atmospheres with low CO (0–1%), medium CO (1–3%), and high CO contents (>3%) in the conveyor bed reactor were investigated. The control objective for changing the CO content at the conveyor bed reactor outlet was achieved by changing the combustion state, adjusting the amount of coal added to the hot air furnace, and changing the ratio of circulating air to air.

The effects of CO content on the magnetization roasting of siderite were characterized by four indicators: the iron grade of the roasted ore, concentrate, and tailings, as well as the recovery rate. These are shown in Figure 7a–d, respectively.

As shown in Figure 7a–d, when the CO content is low (<1%), the magnetization roasting effect is considerably worse due to insufficient reducing atmosphere in the conveyor bed reactor. It is mainly reflected in the significantly lower iron grade, concentrate iron grade, and recovery rate of the roasted ore compared with the two cases where higher CO contents are present. However, the overall trend remained because the magnetization reaction of the iron minerals slowed down due to insufficient CO content. It reveals that maintaining sufficient CO content is essential for the efficient magnetization of roasting siderite. The variation pattern in the iron grade of the tailings was more complicated. The iron grade of the tailings was relatively high, below 750 °C, implying that more iron had entered the tailings. However, above 780 °C, it was lower than the other two cases with higher CO content. It may be because the overreduction of magnetite is suppressed when relatively insufficient CO content is present, thereby avoiding the loss of magnetite.

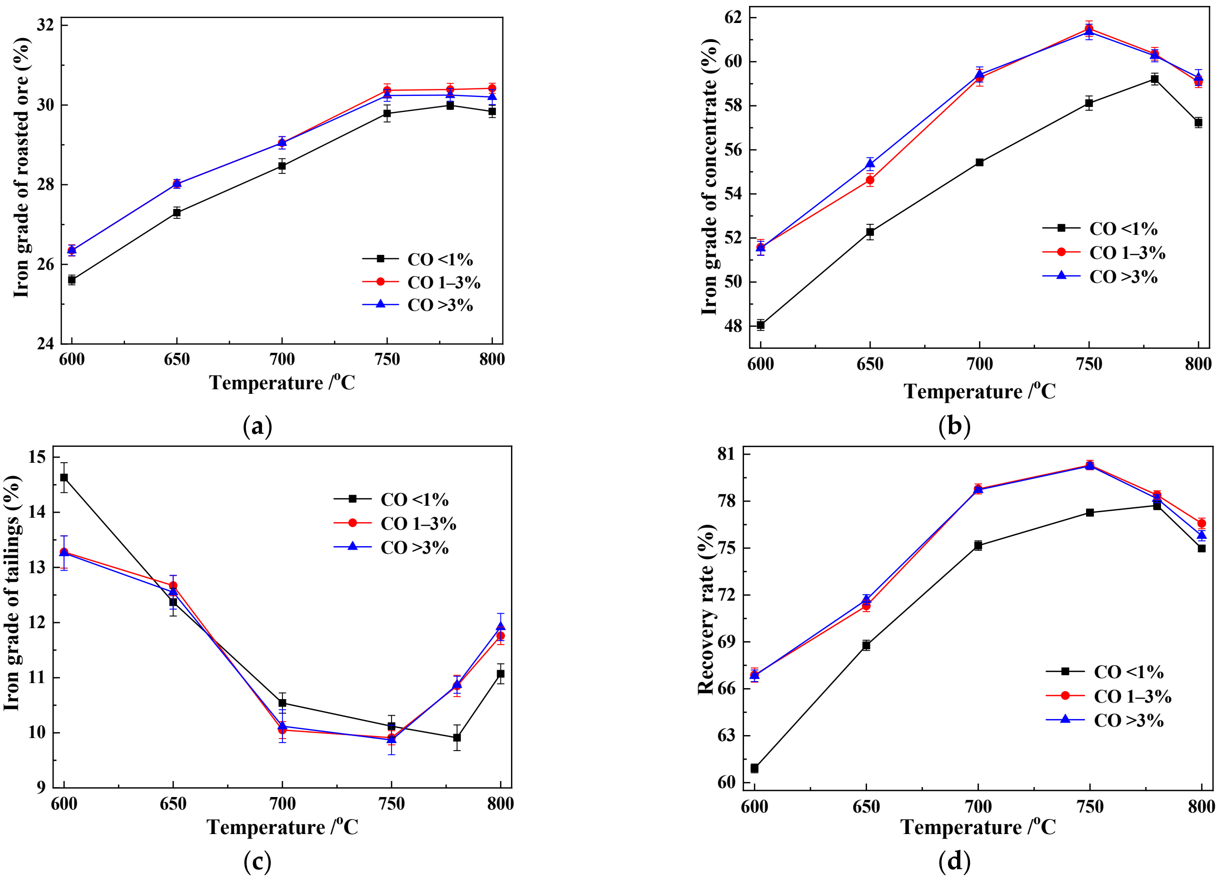


Figure 7. Effect of CO content on magnetization roasting. (**a**) Iron grade of roasted ore; (**b**) Iron grade of concentrate; (**c**) Iron grade of tailings; (**d**) Recovery rate.

The magnetization reaction can be completely conducted under atmospheric conditions of higher CO contents (>1%). Although an increase in CO content from 1–3% to >3% further improved the magnetization roasting effect, the overall change was insignificant. Furthermore, as seen in Figure 7a–d, an increase in the CO content at a higher temperature range above 750 °C leads to a more significant overreduction, further reducing the magnetization effect. During the actual synthesis of magnetite, CO is sourced from the magnetization reaction and the anoxic combustion of coal in a hot-blast furnace. Moreover, increasing the CO content requires more coal powder consumption to maintain a relatively stable gas volume in the system. However, the improvement in the magnetization effect is limited due to the increased CO content; therefore, controlling the CO content in the 1–3% range is more suitable to better balance the concentrate quality and the production economy.

3.3. Comparison of Water Cooling and Dry Cooling

Atmospheric conditions with CO content of 1–3% were selected to compare dry and water cooling by setting a sampling tube on the material pipeline from C5 to C6 (as shown in Figure 3), followed by opening the plug of the sampling tube during system operation and discharging the roasted ore into a bucket for water cooling. The roasted ore, concentrate, and tailings were then analyzed, and the results were compared with material analysis data from dry cooling. The analytical results for dry and water cooling are shown in Table 3.

Table 3. Analysis results of dry cooling and water cooling.

Analysis Items	Cooling Method	600 °C	650 °C	700 °C	750 °C	780 °C	800 °C
Iron grade of roasted ore (%)	Dry cooling	25.39	26.05	26.94	27.98	28.09	28.03
	Water cooling	25.42	26.14	26.87	28.11	28.15	28.14
Iron grade of concentrate (%)	Dry cooling	48.05	52.27	55.43	58.12	59.21	57.24
	Water cooling	48.72	52.35	55.50	58.26	59.23	57.32
Recovery (%)	Dry cooling	60.91	68.78	75.16	77.27	77.73	74.97
	Water cooling	61.32	69.01	75.19	77.24	77.65	74.87

As shown in Table 3, water cooling generally improves the effectiveness of magnetization roasting and beneficiation because of its faster cooling rate and rapid isolation from oxygen, thereby avoiding oxidation. However, the experimental results of this paper show that under the condition of dry cooling, which is achieved by circulating waste gases and very low oxygen contents, excellent results close to those obtained when using water cooling are obtained. Therefore, based on the experimental results of this paper, it is feasible to adopt the exhaust-gas-circulation dry cooling method to roast siderite ores. It also has remarkable advantages in terms of saving water, saving energy and being a simple process compared with the water-cooling method.

3.4. Energy Consumption Analysis

To evaluate the energy consumption level of the new conveyor bed magnetization roasting-dry cooling process, the system's parameters under stable operation were measured and combined with theoretical calculations to obtain the mass and heat balance of the process, as shown in Tables 4 and 5, respectively. As a result, for each ton of processed siderite ore, 0.73 t of roasted ore can be obtained, which will consume 42.4 kg of standard coal. As a result, the total thermal efficiency of the system was 49.6%.

Table 4. Mass balance data.

Input	Mass (kg)	Temp. (°C)	Specific Heat (kJ/kg·°C)	Output	Mass (kg)	Temp. (°C)	Specific Heat (kJ/kg·°C)
Siderite	765.0	30	0.72	Roasted ore	555.3	185.7	0.76
Moisture content	35.0	30	4.18	Exhaust gas	2147.1	220.1	1.1
Hot-blast air	1375.9	850	1.1	Fly ash	81.3	220.1	0.72
Circulating air	550.1	80	1.0	-	-	-	-
Air leak	57.8	30	1.0	-	-	-	-
Total	2783.7	-	-	Total	2783.7	-	-

Table 5. Heat balance data.

Input	Sensible Heat (kJ)	Output	Sensible Heat (kJ)
Siderite	0	Roasted ore	65,709.8
Moisture content	0	Exhaust gas	448,758.1
Hot-blast air	124,1106	Fly ash	11,116.4
Circulating air	27,500	Reaction endothermic	616,161.6
Air leak	0	Surface heat dissipation	126,860.6
Total	1,268,606	Total	1,268,606

4. Conclusions

1. A new process for the magnetization roasting–dry cooling of siderite on a conveyor bed at 700–780 °C and >1% CO content for 3–5 s obtained a concentrate iron grade of 59.27–61.50% and a recovery of 78.32–80.30%.
2. An increase in the calcination temperature had a positive effect on improving the iron grade and recovery of the concentrate in the temperature range of 600–750 °C;

however, calcination at temperatures above 750 °C led to a slight decrease in the iron grade and recovery of the concentrate.

3. Increased CO content effectively promoted the magnetization roasting effect. However, when the CO content was increased above 3%, improvement in the magnetization roasting effect in the temperature range of 700–780 °C was very limited.
4. This new conveyor bed magnetization roasting-dry cooling process is highly efficient and saves energy and water; therefore, it could be adapted to the magnetization roasting of low-grade siderites.

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