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Effects of Specularite on the Preheating and Roasting Characteristics of Fluorine-Bearing Iron Concentrate Pellets

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Abstract: Fluorine-bearing iron ore is unique and complex. Serious preheating and roasting problems of fluorine-bearing iron concentrate pellets appear during the industrial production by the grate-kiln process. Besides, specularite has low hydrophilicity, undesirable particle size and shape, dense and smooth particle surface and poor assimilation performance. Thus, it has not yet been widely applied in production. This study applied the specularite to improve the preheating and roasting characteristics of the fluorine-bearing iron concentrate pellets. The experiment results indicated that the roasting properties of fluorine-bearing iron concentrate pellets were improved and the compression strength of roasted pellets increased with the addition of 10% specularite. The suitable roasting temperature range was expanded to more than 140 °C. Compared to other addition, the total iron of pellets was also increased. In addition, the improvement mechanism of adding specularite on the properties of fluorine-bearing iron concentrate pellets was discussed.

Keywords: pellet; fluorine-bearing iron ore; specularite; compression strength; roasting temperature

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1. Introduction

The rapid development of the economy led to increasing demand for iron and steel production. Pelletizing is one of the agglomeration methods for iron ore concentrate. The iron ore pellets have a high compression strength, high reducibility and superior softening-melting properties [1–3]. The grate-kiln process is an industrial applied process to produce pellet production in China [4–6]. With the expense of high-quality iron ore resources, many researchers focus on utilizing low-quality and complex iron ore resources [7–10]. Fluorine-bearing iron is a unique iron ore found in the Baiyun'Ebo region in Inner Mongolia, China [11,12]. The local iron steel company mainly uses fluorine-bearing iron ore to produce steel productions. However, there are also some severe weaknesses in the industrial production of fluorine-bearing iron pellets by the grate-kiln process. Problems include a narrow range of roasting temperature, low compression strength, easy adhesions and bad reducibility [13,14]. Furthermore, severe problems include fluorine pollution, fluorine corrosion on the linings, wide softening-melting zone and poor permeability being found in the blast furnace.

Previous researchers [15–18] reported the effects of dolomite on the roasting and metallurgical properties of fluorine-bearing iron concentrate pellets. However, the compression strength of fluorine-bearing iron concentrate pellets has not improved significantly with the addition of dolomite. In addition, the effects of titanium slag on the properties of fluorine-bearing iron ore pellets were investigated in our previous paper [19]. The results showed that the compression strength of fluorine-bearing iron ore pellets improved up to 3700 N/P, and the suitable roasting temperatures range for producing roasted pellets expanded above 150 °C. As we know, the compression strength of pellets must achieve the requirements of blast furnace smelting. According to the Chinese standard of acidity iron ore pellet for blast furnace (GB/T 27692-2011) [20], the compression strength of iron ore pellets should be higher than 2500 N/P for the first-grade iron ore pellets production. A suitable roasting temperature is necessary for the production of high-quality pellets. Industrial pellet production requires a wide roasting temperature range for smooth operation. The properties of fluorine-bearing iron concentrate pellets could be improved by adding dolomite and titanium slag. However, there are also some shortcomings with the additions of dolomite and titanium slag. Problems include decreasing the total iron content of pellets and a slight increase in the energy consumption in both the grinding and roasting parts.

Some researchers believed that fluorine and alkali metals were the primary reason for the problems in pellets production [11,14]. Our previous paper [14] systematically reported the fundamental solidification mechanism of fluorine-bearing iron ore pellets. The excessive amounts of liquid phases in the pellets generated with alkali metal and fluorine is the main reason for the poor roasting and metallurgical properties of fluorine-bearing iron concentrate pellets. In addition, specularite is also a particular iron ore, which usually has high total iron content and a low gangue amount [21]. However, the specularite is hard to roast while using it for pelletizing, due to the specularite being well-crystallized [22]. Besides, compared to the titanium slag, the total iron content of the specularite ore is higher [23]. Furthermore, the addition of specularite has no negative influence on the subsequent blast-furnace smelting.

In this study, the effects of specularite on the preheating and roasting properties of fluorine-bearing iron concentrate pellet were experimentally investigated. The roasting experiments of fluorine-bearing iron concentrate pellets were conducted in a tube furnace. The microstructures of roasted pellets were observed using SEM-EDS (Scanning Electron Microscope-Energy Dispersive Spectrometer). Finally, the improvement mechanism of adding specularite was also discussed.

2. Materials and Methods

2.1. Materials

The chemical compositions of (FIC) fluorine-bearing iron concentrate, bentonite and specularite are listed in Table 1. As shown in Table 1, the fluorine-bearing iron concentrate belongs to magnetite, containing 1.6 wt.% fluorine. The total iron content of the specularite ore is up to 65.9 wt.% and its SiO_2 content is 2.7 wt.%. The Al_2O_3, CaO and MgO contents are pretty low.

Table 1. Chemical compositions of fluorine-bearing iron concentrate, bentonite and specularite/wt.%.

Kind	TFe	FeO	SiO_2	Al_2O_3	CaO	MgO	F	K_2O	Na_2O	S
FIC	61.9	26.4	4.9	1.7	3.0	0.5	1.6	0.3	0.2	0.1
Bentonite	2.4	0.2	54.6	9.9	5.2	2.7	-	0.9	1.7	0.1
Specularite	65.9	-	2.7	0.2	0.1	-	-	-	-	-

The mass fraction of fluorine-bearing iron concentrate and bentonite size of less than 0.074 mm account for 85.2% and 26.8%, respectively [14]. The specific surface area of fluorine-bearing iron concentrate is 1683 $cm^2.g^{-1}$ [14]. Table 2 shows the particle distribution of specularite. Specularite has a relatively thick grain size, less than 0.074 mm, which only accounts for 2.6%. Its specific surface area is only 865 $cm^2.g^{-1}$.

Table 2. Grain size distribution of specularite/wt.%.

<0.074 mm	0.074–0.125 mm	0.125–0.25 mm	0.25–0.5 mm	0.5–1 mm	1–3 mm
2.6	48.4	5.4	30.3	8.7	2.1

The microstructure images of fluorine-bearing iron concentrate and specularite are illustrated in Figure 1. There are many fine particles in the fluorine-bearing iron concentrate and on the surface of large particles. However, fine partials can be rarely seen in the specularite sample. The specularite particles have a smooth surface and regular shape.

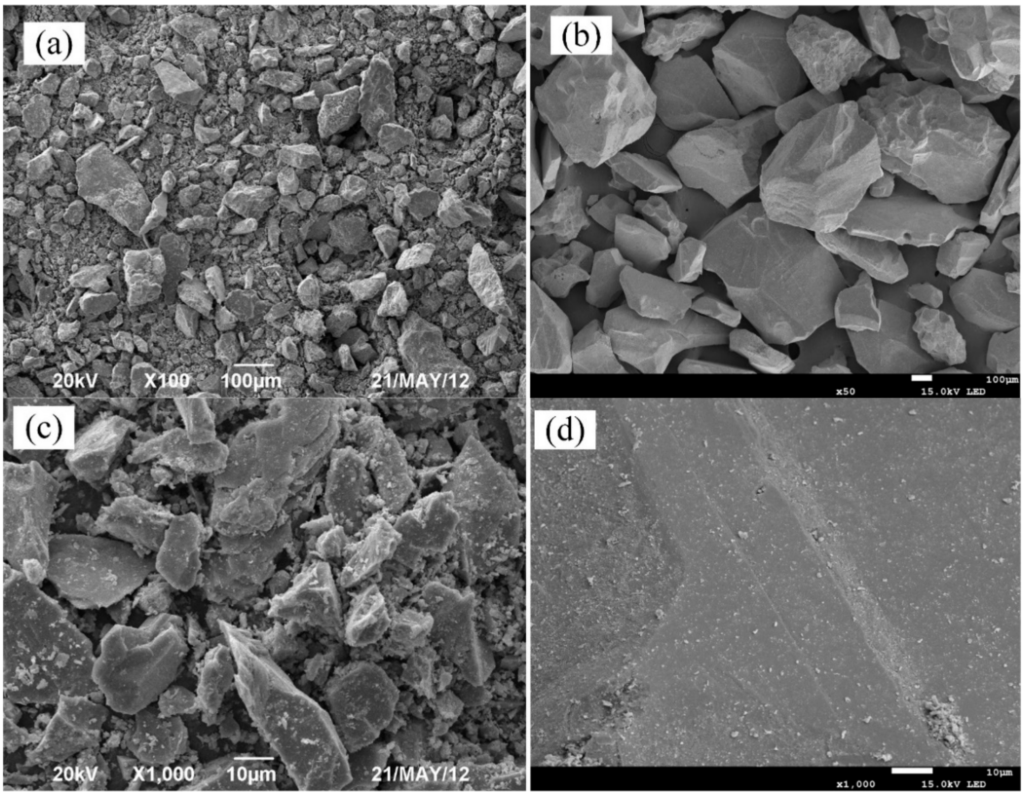

Figure 1. Microstructure of fluorine-bearing iron concentrate (**a**,**c**) and specularite (**b**,**d**).

The mineralogical phase components of specularite are shown in Figure 2. The XRD pattern of the specularite sample indicates that the main mineral phases of specularite are hematite (Fe_2O_3) and minor SiO_2.

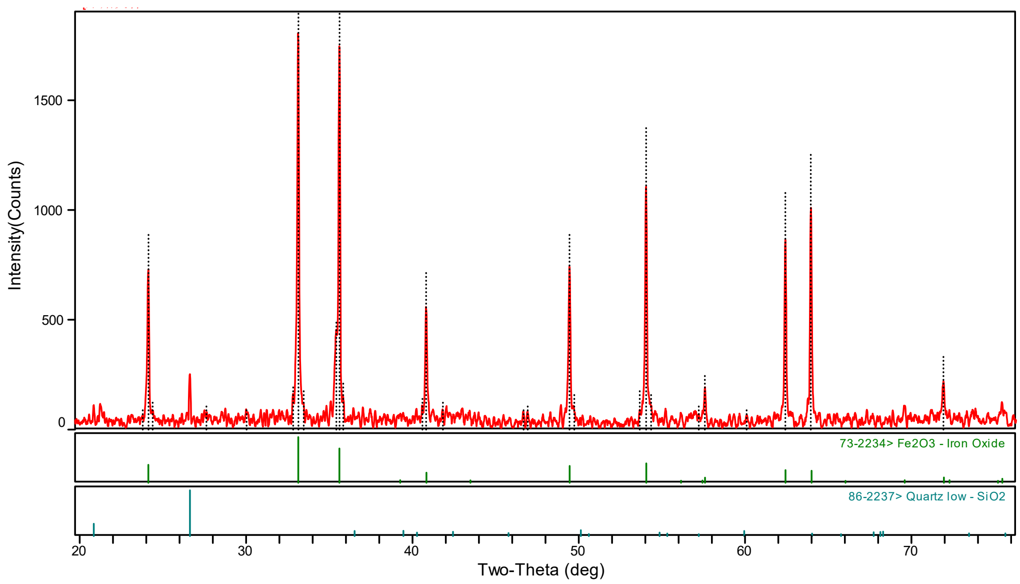

Figure 2. XRD patterns of the specularite.

2.2. Experimental Methods

2.2.1. Pelletization, Preheating and Roasting Process

The pelletization experiments were taken as follows. Firstly, 5 kg of fluorine-bearing iron concentrate was blended with a given amount of bentonite and a designed amount of specularite. It should be noted that the adding proportion of bentonite is 1.75 wt.%, which is based on the properties of green pellets, including the compression strength of above 10 N/P and drop strength of above 3.0 time/0.5 m. After being thoroughly mixed, a small part of the mixture was placed into the balling drum and handfeed concentrate over 10 min, spraying water as necessary; then 2 min of balling was allowed without water addition. As shown in Figure 3, the rotary speed and the duration for pelletization were 22 r/min and 12 min, respectively. The green pellets with a 10–14 mm diameter were dried in an oven at 110 °C for more than 4 h to obtain dry pellets for the following preheating and roasting experiments.

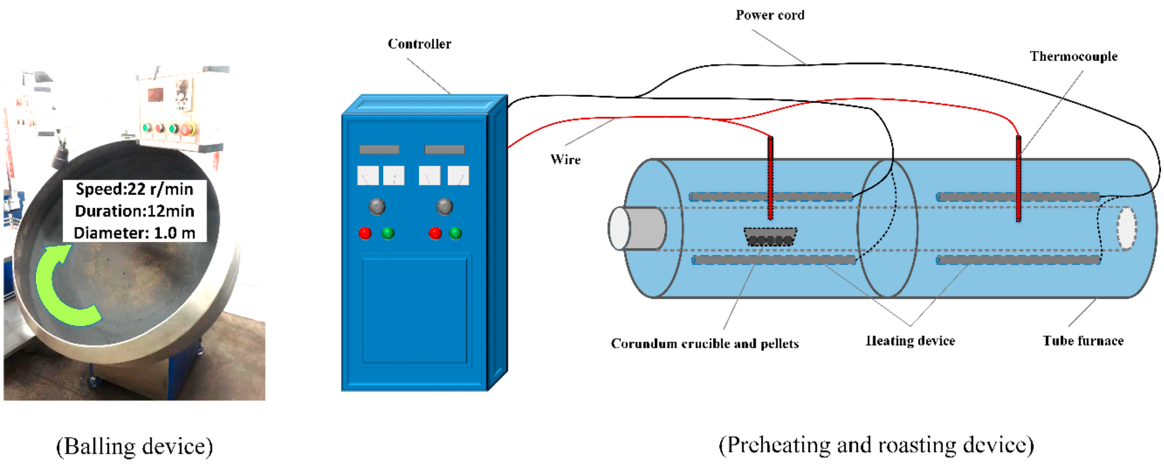

Figure 3. Schematic diagram of balling, preheating and roasting devices.

The preheating and roasting tests were conducted in a horizontal tube furnace (HF-Kejing, Hefei, China). In general, the production of iron ore pellets includes five processes: drying, preheating, roasting, homogenizing and cooling. During the preheating process, the small amount of moisture that has not been eliminated during the drying process is further eliminated here. The main reactions in the preheating process are oxidation of magnetite to hematite, decomposition of carbonate minerals, decomposition and oxidation of sulfides and specific solid-phase reactions. As illustrated in Figure 3, the tube furnace contained two parts: the preheating and coterminous roasting. The outer diameter and inner diameter of the tube are 60 mm and 50 mm, respectively. The preheating and roasting experiments were conducted in the air atmosphere. The green pellets were placed in the corundum boat and moved to the heating zone from room temperature step by step for 5 min. The compression strengths of preheated and roasted pellets were measured by a LJ-1000 material experimental machine. More details about the balling and roasting experiments could be found in our previous papers [14,24]. In addition, a part of the roasted pellets was stochastically chosen for the following analysis.

2.2.2. Analysis and Characterization

The chemical compositions of samples were analyzed by chemical methods [25], which are shown in Table 3. The roasted pellets were mounted on the epoxy resin and then polished for SEM-EDS analysis. The phase compositions of the samples were analyzed by the X-ray powder diffraction (XRD) technique (RIGAKUD/Max 2500, Tokyo, Japan), operating at 40 kV and 4 mA with Cu Kα radiation, step 4°/ min and range of angles analyzed from 10 to 80°. The microstructure and phase compositions of the samples were determined by SEM (Scanning Electron Microscope) (TESCAN, MIRA3-LMH, Brno, Czech) with EDS (Energy Dispersive Spectrometer) (Oxford X-MAX20, Oxford, UK). The EDS results were calibrated by the XPP ((eXtended Pouchou and Pichoir)) method [26]. The relative deviation is 5% for elements with content higher than 20%, 10% for those with 3–20%, 20% for those with 1–3% and 50% for those with 1% or less. The thermodynamic software FactSage 8.0 [27] was used to calculate the phase diagram. The used module was "Phase Diagram", with the corresponding databases "FToxid" and "FactPS."

Table 3. The chemical methods used to analyze the chemical compositions of samples.

Item	Chinese Standard	Method
Total Fe	GB/T6730.5-2007	Titanium(III) chloride reduction
FeO	GB/T6730.8-1986	Potassium dichromate volumetris method
Si	GB/T6730.10-1986	Gravimetric method
Al	GB/T6730.11-2007	EDTA titrimetric method
S	GB/T6730.61-2005	High-frequency combustion with infrared absorption method
F	GB/T6730.28-2006	Ion selective electrode method
K and Na	GB/T6730.49-1986	Flame atomic absorption spectrophotometric method
Mg and Ca	GB/T6730.13-2007	EGTA-CyDTA titrimetric method

3. Results and Discussion

3.1. Preheating and Roasting Properties

In general, specularite contains a high total iron grade. However, the specularite has low hydrophilicity, undesirable particle size and shape, dense and smooth particle surface and poor assimilation performance. Thus, it has not yet been widely applied in production. The preparation of specularite pellets needs a high roasting temperature (about 1300 °C), and the formation of liquid phases is hard during the roasting process [23]. Furthermore, a large amount of liquid phase (more than 10 wt%) is formed at a low roasting temperature (about 1200 °C) for the fluorine-bearing iron concentrate [14]. According to the previous investigation, the suitable proportion of liquid phases in the roasting pellets should be about 5–7% for achieving high compression strength [1]. Therefore, the suitable range for roasting temperature is narrow and the compression strength of the roasted pellets is low. It can be explained that the volume of the high formed content of liquid phases in the pellets shrinks during the cooling process, and causes cracks in the roasted pellets. Previous results [19] indicated that the control of liquid phase amount is critical to improving the properties of fluorine-bearing iron concentrate pellets. Thus, it can be inferred that the roasting properties of fluorine-bearing iron concentrate pellets would probably be improved with the addition of specular hematite.

This part investigated the effects of specularite addition on the preheating and roasting properties of fluorine-bearing iron concentrate pellets under constant preheating and roasting conditions. As shown in Table 4, the compression strength of roasted pellets reaches a high level with the addition of specularite.

Table 4. Effect of specularite content on compression strength of pellets.

Specularite/%	Preheating Temperature/°C	Preheating Time/min	Roasting Temperature/°C	Roasting Time/min	Compression Strength/(N/P)
0	920	16	1080	10	2463
5%	900	14	1080	10	2470
10%	900	14	1080	10	2955
15%	900	14	1080	10	2471

The effects of specularite on the compression strength of roasted fluorine-bearing iron concentrate pellets are shown in Figure 4. The addition proportion of specularite was 10 wt.%. It can be seen that the compression strength of roasted pellets with and without specularite increases following a decrease with the increasing preheating temperature. The compression strength of roasted fluorine-bearing iron pellets with 10 wt.% specularite is higher than that of the fluorine-bearing iron pellet without an additive [14]. The highest compression strength is up to 2800 N/P, at the preheating temperature of 900 °C.

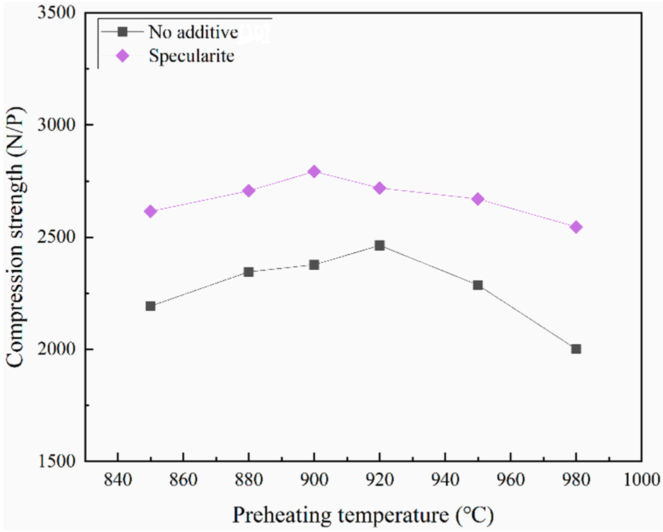

Figure 4. Effects of preheating temperature on compression strength of roasted pellets (preheating time 16 min, roasting temperature 1080 °C, roasting time 10 min).

Figure 5 shows the effects of preheating time on the compression strength of roasted pellets. The compression strength of roasted pellets has an increasing trend as the preheating time increases from below 16 min. Nevertheless, the compression strength decreases with the further increase of the preheating time. The compression strength of roasted pellets with specularite is more than 3000 N/P when the preheating time is 14 min.

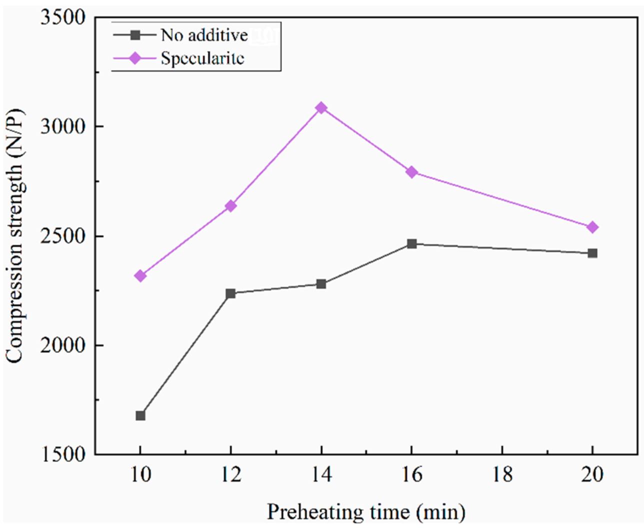

Figure 5. Effects of preheating time on compression strength of roasted pellets (preheating temperature 950 °C, roasting temperature 1080 °C, roasting time 10 min).

It is well accepted that oxidation has a crucial impact on the compression strength of magnetite pellets. According to the unreacted shrinkage model [1], oxidation reactions of magnetite is carried out from the surface to the centre of the pellets in layers. The oxidization process accords with the adsorption-diffusion theory of chemical reaction. The inward diffusion of oxygen plays a vital role in oxidation. Suppose oxidation of magnetite is not complete in the preheating stage; in that case, the Fe^{2+} can react with silica to form ferrosilicate such as Fe_2SiO_4, whose melting point is only 1208 °C. Then, the liquid phases would generate in the roasting process when the temperature is higher than 1208 °C. Lastly, due to different shrinkage rates of solid and liquid phase, concentric cracks are generated in the cooling process of pellets, resulting in uneven pellet structure and decreasing pellet strength [1]. This theory can be used to explain the results in Figures 4 and 5. The higher preheating temperature will accelerate the oxidization of the surface of magnetite pellets to form a tight layer. Then, oxygen diffusion would be prevented, leading to the decrease of compression strength of roasted pellets.

Figure 6 illustrates the effects of roasting temperature on the compression strength of roasted pellets. The compression strength of roasted pellets increases firstly, following decreases with the increasing of roasting temperature. As the roasting temperature increases, a large amount of liquid phase appears at high temperatures and the proportion of liquid-phase consolidation increases [14,16]. In general, consolidation of pellet ore relies mainly on solid-phase consolidation, where the particles are bonded together by the diffusion-reaction of solid phases to form connecting bridges or connecting necks, compounds or solid solutions [1]. A certain quantity of liquid phase can improve the solidification of the pellet after the roasting process. However, an excess amount of liquid phase is detrimental to the solidification of roasted pellets. Thus, the excess amount of liquid-phase consolidation in the roasted pellets becomes the limiting factor of the strength of pellets, leading to a decrease of the compressive strength of pellets.

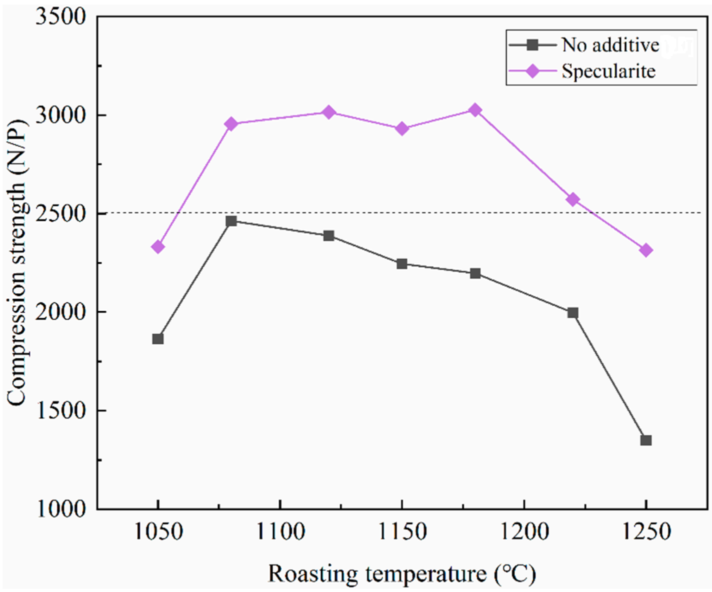

Figure 6. Effects of roasting temperature on compression strength of roasted pellets (preheating temperature 950 °C, preheating time 14 min, roasting time 10 min) (a horizontal dotted line at 2500 N/P is the first-grade qualified standard for blast furnace [20]).

In general, the compression strength of roasted pellet should be higher than 2500 N/P as the qualified standard, according to the modern blast furnace practice. Thus, the horizontal dotted line at 2500 N/P in Figure 6 could clarify whether the compression strength of pellets reaches the requirement. Moreover, the compression strength of fluorine-bearing iron pellet with 10 wt% specularite is higher than the fluorine-bearing iron pellet without additive. The highest compression strength is up to 3100 N/P at 1180 °C. With the addition of specularite, the suitable roasting temperatures with corresponding compression strength high than 2500 N/P are from 1080 °C to 1220 °C. The suitable roasting temperature range expands to 140 °C, which is beneficial for industrial production and operation.

Figure 7 shows the effects of roasting time on compression strength of roasted pellets with 10 wt.% specularite. The compression strength of fluorine-bearing iron concentrate pellets with the addition of specularite slowly increases when the roasting time increases from 8 to 10 min, following a decrease with the further increase of roasting time. Furthermore, the compression strength of fluorine-bearing iron concentrate pellet with 10 wt.% specularite is higher than the fluorine-bearing iron concentrate pellet without additive. However, the prolong of roasting time causes the formation of excessive liquid phases. The pellet volume then shrinks, producing a large cavity, resulting in a decrease in the compression strength of the pellets.

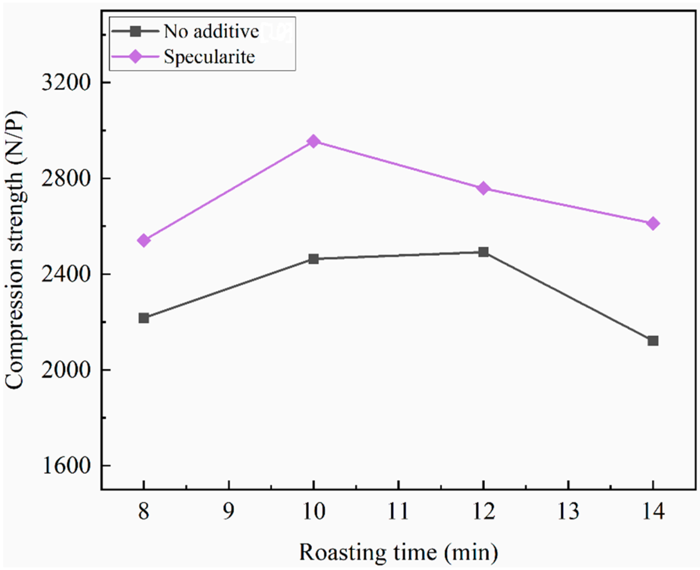

Figure 7. Effects of roasting time on compression strength of roasted pellets (preheating temperature 950 °C, preheating time 14 min, roasting temperature 1080 °C).

In particular, the compression strength of fluorine-bearing iron concentrate pellet without significant difference and almost up to more than 2500 N/P when the roasting temperature ranges from 1080 °C to 1220 °C. The suitable range of roasting temperature effectively expanded to more than 140 °C with the addition of specularite.

3.2. *Microstructure of Roasted Pellets*

The microstructure images of roasted fluorine-bearing iron concentrate pellets without specularite and with specularite (10 wt.%) are shown in Figure 8a–f, respectively. The samples were preheated at 920 °C for 16 min and roasted at 1080 °C for 10 min. The dark grey area is the silicate phase and the white-grey area is the Fe_2O_3 phase.

Compared to the specularite-free pellets (Figure 8a,b), the area of the silicate phase decreases with the addition of specularite (Figure 8c,d), from which it can be inferred that the formation of the liquid phase decreases. Furthermore, according to the SEM images, after the roasting of the specularite pellets, the solid connected crystals develop better, which may also be the reason for the increase in compressive strength. The microstructures of roasted fluorine-bearing iron concentrate pellets become densified. The dispersed Fe_2O_3 crystals change to interconnected and tightened by bonding and reacting with the silicate phase, which improves the compression strength of pellets [28].

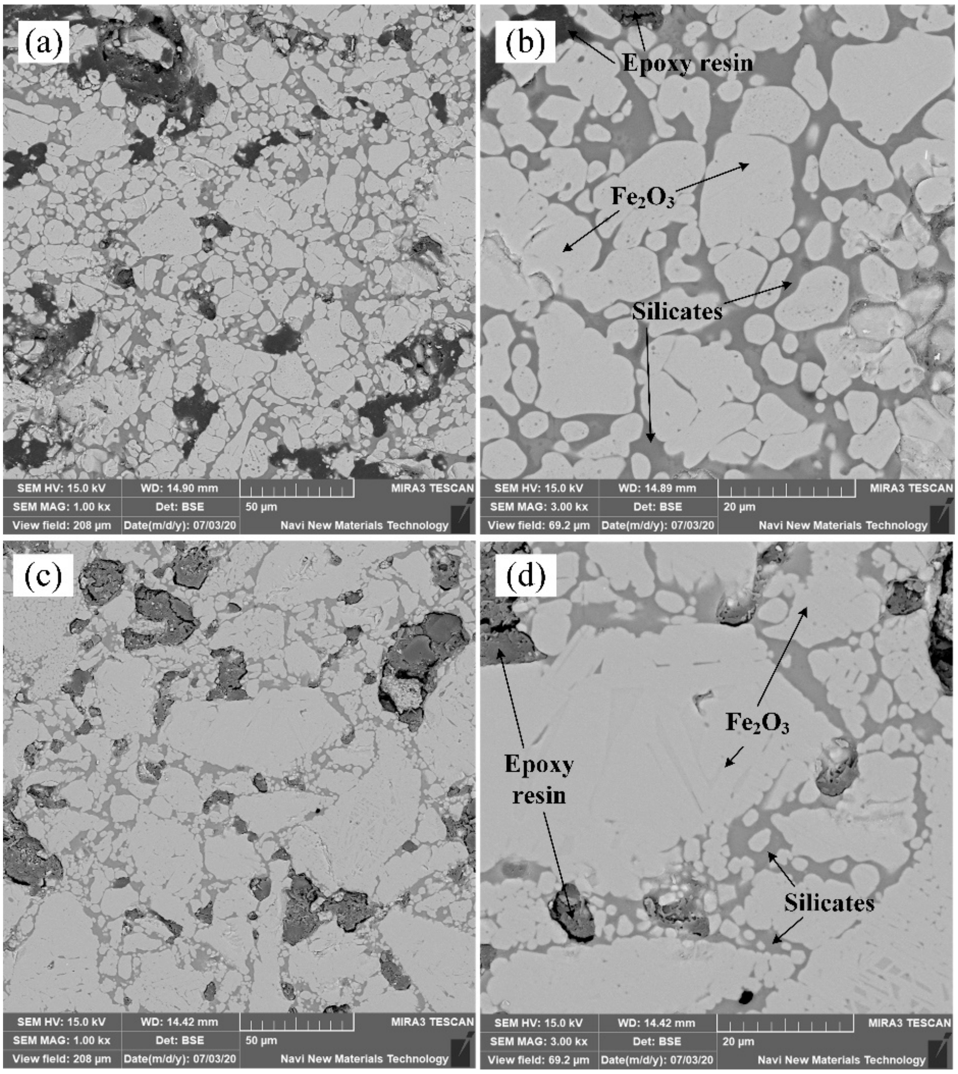

Figure 8. Microstructure of roasted fluorine-bearing iron concentrate pellet without (**a**,**b**) and with specularite (**c**,**d**) (preheating at 920 °C for 16 min and roasting at 1080 °C for 10 min).

3.3. Element Distribution and Phase Composition Analysis

The EDS analysis of the roasted pellets without or adding specularite were carried out to investigate the chemical composition of the main phases of the roasted pellets. The pellets were preheated at 920 °C for 16 min and roasted at 1080 °C for 10 min. Figure 9 shows the distribution of Fe, Si, Ca, Al, Mg, K, Na and F in the roasted pellets. The chemical compositions of the phases are listed in Table 5. The contents of oxides of each phase were collected from more than 10 points, and the deviations were also calculated. It can be observed that the main phases contain hematite and silicates. The chemical composition of silicates in different locations of pellets are various. In addition, the F, Na and K elements are mainly distributed in the silicate phases. The Fe_2O_3 contents of the silicates phases are higher than that in the roasted pellets with no additive. The iron oxides may diffuse into the silicates and improve the properties of silicates, resulting in the improvement of preheating and roasting properties of pellets.

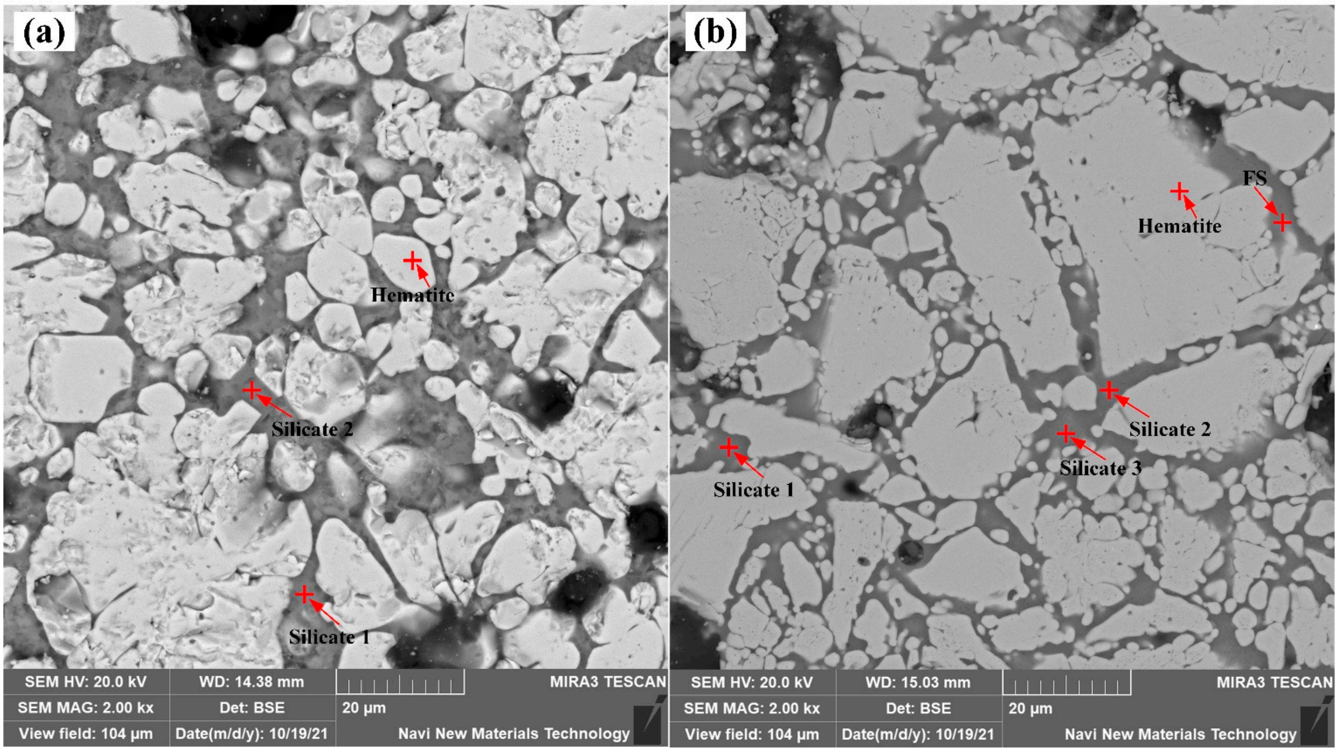

Figure 9. EDS analysis of roasted fluorine-bearing iron concentrate pellets with no additive (**a**) and adding specularite (**b**) (preheating at 920 °C for 16 min and roasting at 1080 °C for 10 min).

Table 5. Chemical compositions of the phases marked in Figure 9 in the roasted pellets.

Roasted Pellets	Phase	Chemical Composition/wt.%							
		Fe_2O_3	CaO	SiO_2	Al_2O_3	MgO	CaF_2	K_2O	Na_2O
No additive	Hematite	98.2 ± 1.3	0.2 ± 0	0.2 ± 0.1	1.1 ± 0.3	0.1 ± 0	0	0	0
	Silicate 1	6.9 ± 1.5	12.5 ± 0.9	41.9 ± 6.9	9.5 ± 1.4	5.0 ± 1.1	21.1 ± 5.9	1.7 ± 0.2	1.3 ± 0.1
	Silicate 2	19.2 ± 5.5	9.2 ± 1.8	40.6 ± 2.6	9.2 ± 0.6	5.1 ± 0.5	11.7 ± 1.5	1.7 ± 0.4	1.3 ± 0.1
Adding specularite	Hematite	97.0 ± 1.1	0.4 ± 0.2	0.6 ± 0.5	0.7 ± 0.2	0.1 ± 0.1	0	0	0.0 ± 0.1
	FS	84.5 ± 4.3	2.7 ± 0.7	7.1 ± 2.1	3.2 ± 1.0	0.8 ± 0.2	0	0.5 ± 0.2	0.4 ± 0.1
	Silicate 1	64.9 ± 4.6	5.2 ± 0.4	17.4 ± 3.9	6.4 ± 1.6	1.6 ± 0.3	1.8 ± 1.8	1.4 ± 0.3	0.8 ± 0.2
	Silicate 2	38.9 ± 7.8	10.9 ± 1.5	29.9 ± 3.9	8.0 ± 1.2	3.3 ± 0.5	5.6 ± 2.5	1.9 ± 0.5	0.9 ± 0.2
	Silicate 3	11.1 ± 3.0	14.1 ± 2.4	37.2 ± 1.8	10.3 ± 0.0	3.6 ± 0.2	14.4 ± 0.6	2.7 ± 0.2	1.3 ± 0.2

3.4. Phase Diagram Analysis

Previous research reported that the main reason for the low compression strength of fluorine-bearing iron concentrate pellets is the excessive amount of generated liquid phases at high temperatures during the roasting process [14,16]. In this part, the phase diagram of the SiO_2-CaO-Fe_2O_3-10wt%Al_2O_3-5wt%MgO system was used to evaluate the formation of the liquid phase in the pellets. The approximate areas of the silicates phases in the diagram were marked in Figure 10, according to the elemental contents of silicates in Table 5. As shown in Figure 10, the areas of the silicates phases with specularite have higher melting points, which means they have high melting points and lower proportions of formed liquid phases during the roasting process.

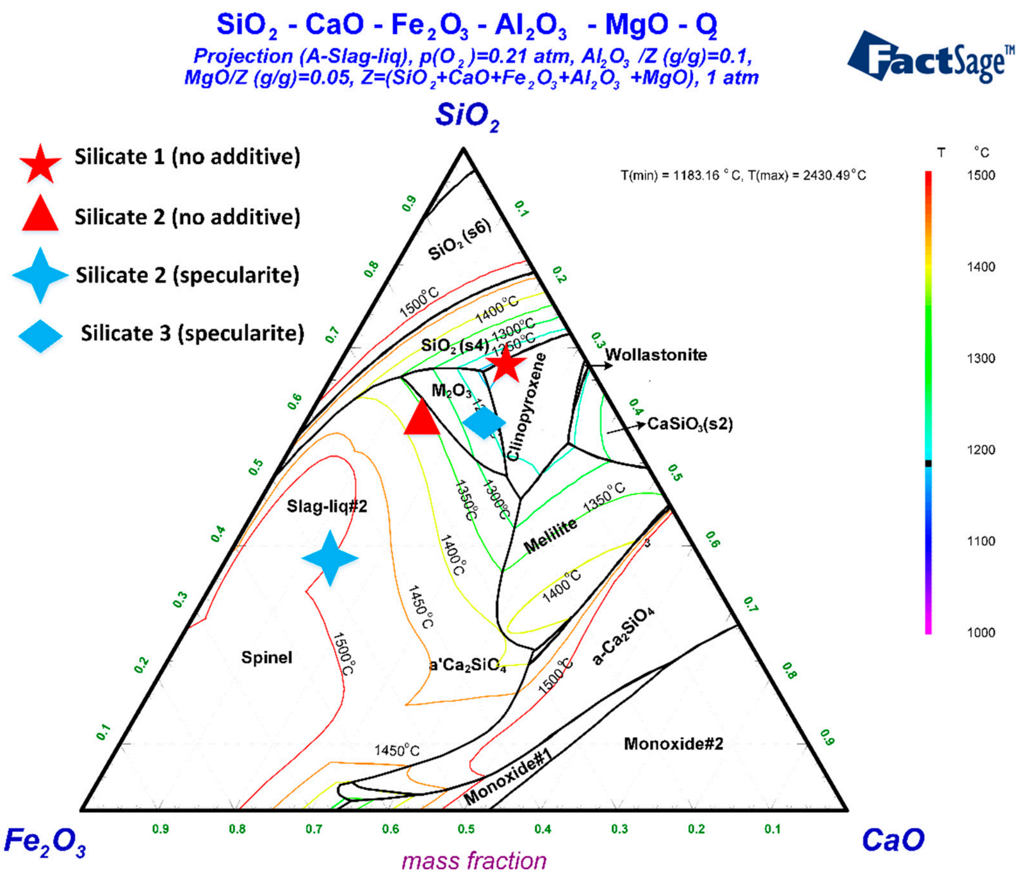

Figure 10. Phase diagram of SiO_2-CaO-Fe_2O_3-10wt%Al_2O_3-5wt%MgO-O_2($P_{(O2)}$ = 0.21) calculated by FactSage 8.0 (marked locations according to Table 5).

3.5. Technical and Economic Evaluation

In general, the fluorine-bearing iron concentrate contains high amounts of fluorine and alkali metals. Thus, the fluorine-containing pellets can form liquid phases during the roasting process [14,16]. The suitable range for roasting temperature is narrow and difficult to operate and run in plant production. The liquid phase formation is potentially suppressed by adding specularite. The suitable roasting temperature range is 1080–1220 °C, up to 140 °C, and the pellet strength can be 2966 N/P at 1080 °C. The highest pellet strength increased from 2463 N/P to 3027 N/P with the addition of specularite.

The main reasons for adding specularite to improve the roasting performance of fluorine-bearing iron ore pellets are as follows. The addition of specularite in the pellets could dilute the harmful influences of fluorine and alkali metals to control the formation of the liquid phase. Thus, the preheating and roasting properties of fluorine-bearing iron ore pellets could be improved. Furthermore, compared to other additives such as dolomite, specularite has high total iron and low gangue, which is beneficial to improve the quality of pellets.

4. Conclusions

Fluorine-containing iron concentrate and specularite both belong to the particular iron ore. The productions have poor properties, and production is complicated when they are singly used to produce the pellets. This study improved the roasting properties of fluorine-bearing iron concentrate pellets. The compression strength of roasted pellets increased with specularite. The suitable roasting temperature range is 1080~1220 °C, up to 140 °C, and the pellet strength can be 2966 N/P under the condition of the lowest roasting temperature of 1080 °C. The highest pellet strength increased from 2463 N/P to 3027 N/P with the addition of specularite. In addition, the specularite has high total iron and low gangue, which is beneficial to improve the quality of pellets.

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