

Article

A Study on the Influence of Drying and Preheating Parameters on the Roasting Properties of Limonite Pellets

Peng Chen ¹ and Xiaolei Zhou ^{2,*} ¹ School of Metallurgical and Energy Engineering, Kunming University of Science and Technology, Kunming 650093, China; chenpeng4629@163.com² Key Laboratory of Clean Metallurgy of Complex Iron Resources, Kunming University of Science and Technology, Kunming 650093, China

* Correspondence: zxl@kust.edu.cn; Tel.: +86-15912182991

Abstract: In this experiment, a pellet preparation method was investigated to study the drying, preheating, and roasting properties of limonitic iron ore from a plant in Yunnan. The aim was to improve the subsequent iron-making process of limonitic iron ore and make it a substitute for sintered ore. This substitution would reduce the amount of blast furnace slag in the iron-making process. Bentonite is commonly used as a primary binder in many pelletizing plant operations. However, its excessive usage leads to a higher risk of slagging and coking in the furnace. In this paper, we aim to decrease the quantity of bentonite added, enhance the iron content in the pellets, and reduce impurities to improve the grade of limonite pellets. The results show that the optimal drying, preheating, and roasting temperatures of limonite pellets are 200 °C, 700 °C, and 1250 °C, respectively, and the optimal roasting time is 20 min, when the diameter of the pellets is 8–13 mm. The compressive strength of limonite pellets with the addition of 1.5% bentonite was the highest, meeting the demands of a general blast furnace, based on which the iron grade of limonite pellet ore was increased by 10.63%.

Keywords: limonite; pellets; bentonite; steel metallurgy

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1. Introduction

The booming development of metallurgical technology and the iron and steel industry has led to the recognition of iron ore pellets as a high-quality charge. These pellets possess excellent metallurgical properties and offer advantages such as the improved permeability of the blast furnace charge column. They are considered essential for optimizing blast furnace charge structure, and their significance in the iron and steel industry is growing [1]. Iron ore pellet drying, preheating, and roasting are important parts of the pellet ore production process; improving pellet drying efficiency and pellet strength is related to the improvement of the quality of the pellet minerals. With the rapid development of the steel industry, China has rapidly become the world's leading steel producer. Zhang Hanquan, of the Wuhan University of Technology, conducted research on the influence of chain grate machine-oxidized pellet preheating process parameters on the finished ball compressive strength [2]. Relevant experimental studies were carried out on the selected raw materials to obtain the production process parameters for high-quality pellets. On the other hand, the continuous optimization of processing systems, improving and perfecting the production equipment, energy saving, and emission reductions are necessary to achieve the production of high-quality pellets. Therefore, to produce high-quality oxidized pellets, a reasonable selection of process parameters is essential [3].

Limonite is characterized by its low content of harmful elements and is a low-grade iron ore. However, low-grade ores often contain high levels of impurities, which can lead to low pellet strength when using limonite. In addition, the pelletized ore undergoes a number of handling, transport, stacking, and movement processes before and after

entering the blast furnace. These processes are subject to a variety of severe mechanical forces such as collision, impact, compression, and friction [4]. These mechanical forces can cause some pellets to break up and produce fines, which can affect furnace operation and production targets. In order to solve these problems, it is possible to increase the density and mechanical strength of the pellets by adding binders to fill the gaps and cracks between the ore particles and bond them together [5–7]. In the steel industry, binders are essential for the pelletization of iron ore concentrates. Bentonite has strong water adsorption capacity, high bonding strength, and a relatively low market price, and is the main binder used in the preparation of pellets [8]. Bentonite inclusions in the impurity content are generally high; every 1% increase in bentonite in the proportion of raw materials will lead to a reduction of about 0.4% to 0.6% of the iron grade of the pellet ore and affect many of its important performance indicators. In particular, it increases the slag load in blast furnace smelting, thus reducing the blast furnace utilization factor. At the same time, the addition of too much bentonite will increase the slagging and coking tendency of the furnace; this coking and slagging material will be attached to the furnace wall or furnace cylinder, and the smelting effect and the blast furnace life will be adversely affected. Compared with foreign countries, the amount of bentonite in domestic pellet plants is higher, generally 2.0% to 3.5% or even higher, with only a few plants having levels below 2.0%. Therefore, the discussion of by how much to reduce the amount of bentonite and improve the grade of limonite ore pellets has become the current focus of many scientific research institutes and plant research directions.

Pellet ore drying, preheating, and roasting are important parts of the pellet ore production process; improving the pellet drying efficiency and pellet strength is related to the improvement of the quality of pellet minerals. Prior to the roasting of the pellets, it is necessary to preheat them [9]. This is because there is still a large amount of crystalline water inside the pellet; if direct roasting leads to rapid evaporation of the internal crystalline water, the pellet shell cannot withstand the phenomenon of cracking or blowing up. Also, the temperature and time of preheating will have an effect on the mechanical strength and properties of the final finished ball.

Zhou Chenggen [10] studied the effect of the preheating temperature of carbon-containing pellets on the compressive strength of finished balls in detail. The results of this study show that the preheating temperature affects the compressive strength and reducibility of the finished balls after roasting. Lu Jianguang [11] studied the effects of Poly-Met Mining Corp (PMC) concentrate preheating temperature on pellet performance. The results of the study showed that the compressive strength of the pellet ore was effectively improved with increasing preheating temperature. In addition, they observed that the porosity of PMC pellet ores decreased and then increased after the preheating temperature was increased, and the porosity reached the lowest value when the preheating temperature was 950 °C. Liu Kai [12] and his team conducted an in-depth study of magnetite pellets using a roasting temperature of 1300 °C. They found that the crystal bridge linkages were further tightened with the increase in roasting time, which enhanced the compressive strength of the pellets. In the study, when the roasting time reached 20 min, it was observed that the distribution of Fe_2O_3 grains became more homogeneous, and the crystal bridge linkages were optimized. This means that the crystal structure of the magnetite pellets was fully adjusted and stabilized during the 20 min roasting time. The compressive strength of the pellets was successfully increased by enhancing the linkage of the crystal bridges. This result suggests that proper time control during the roasting process is essential for the structural optimization and performance enhancement of the pellets. The recrystallization and grain growth processes within the pellet can be carried out smoothly within a suitable temperature range. By reasonably controlling the temperature, the rearrangement of crystals and the growth of grains can be promoted, thus enhancing the mechanical properties and structural stability of the pellet. Therefore, it is crucial to ensure proper temperature control during the roasting process of pellets.

Also, pellet diameter is a key parameter, and the effect of pellet diameter on the performance of pelletized ores involves further key parameters such as mass and heat transfer rates, diffusion rate, and reaction surface area. Therefore, when designing and operating a pelletizing process, the diameter of the pellets needs to be reasonably controlled to achieve the desired ore properties. Zhang Pan [13] conducted a roasting process study on high-sulfur magnetite at a site, and some important conclusions were drawn by comparing the results of pellets with different particle sizes under natural ventilation and blast-roasting conditions. For the same pellet quality, the larger the grain size of the pellet, the higher the pass rate, which means that larger grain sizes are more likely to meet the desired quality standards during the roasting process. At the same time, the strength of the roasted pellets was also better, indicating that larger-grain-size pellets have better mechanical properties. Zhang Guocheng [14] conducted a study to investigate the effect of added Mongolian concentrates on the performance of pelletized ores. Valuable conclusions were drawn from this study. It was found that when the pellet grain size reached 10–12.5 mm, the kinetic conditions of the magnetite oxidation reaction improved, which led to an increase in the oxidation of hematite in the pellet ore.

As iron ore grades decrease over time, there is a need to utilize more methods to challenge this. Therefore, limonite has received much attention as a widely distributed iron oxide resource containing crystalline water [15–19]. The drying, preheating, and roasting properties of limonite pellets were investigated in the laboratory, providing an experimental environment to address the existing research status. The importance of this study for improving the efficiency of steel production, optimizing the pellet preparation process, and reducing energy consumption provides a theoretical reference for the use of limonite pellets in production.

2. Materials and Methods

2.1. Test Material

The sample material included in the experiment was Yunnan limonite iron ore, and the main chemical composition of limonite is shown in Table 1. The total iron content of limonite is 54.67%, with a relatively low grade. The veinstone composition is mainly SiO_2 , with a mass fraction of 4.04%, and contains more types of impurities, but the content is small, with a loss on ignition of 14.82%. Yunnan limonite is shown in XRD in Figure 1, where XRD analyses were carried out to determine the mineral composition of the raw material. As shown in Figure 1, the major mineral forms of limonite are $\text{FeO}(\text{OH})$ and Fe_2O_3 .

Table 1. Main chemical composition of limonite (%).

Ingredient	TFe	FeO	SiO_2	S	MnO	TiO_2	Pb	Zn
Content	54.67	0.29	4.04	0.048	3.47	0.27	0.009	0.018
Ingredient	K_2O	Na_2O	As	Cu	Sn	V_2O_5	LOI	
Content	0.095	0.001	0.007	0.009	0.001	0.040	14.82	

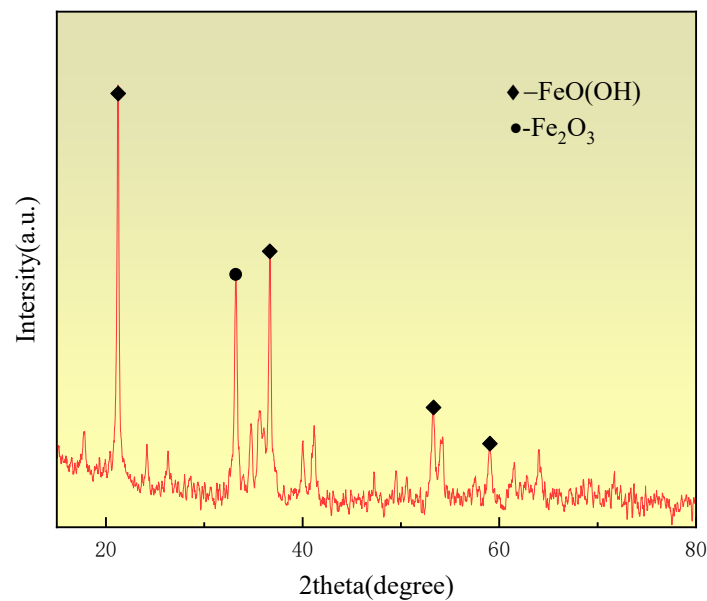


Figure 1. XRD pattern of limonite.

The SEM images of limonite are shown in Figure 2 [20]. It is known that the limonite particles from plants in Yunnan become granular, with a rough surface and loose and porous organization. Strong water absorption, a shape similar to coral on the seabed, and the large wet capacity of the powder show the typical limonite crystallization state. This means that limonite has a strong surface hydrophilicity during the pelletizing process, forming more liquid film and tending to bond to more iron ore particles, which is favorable for the growth of pellets [21].

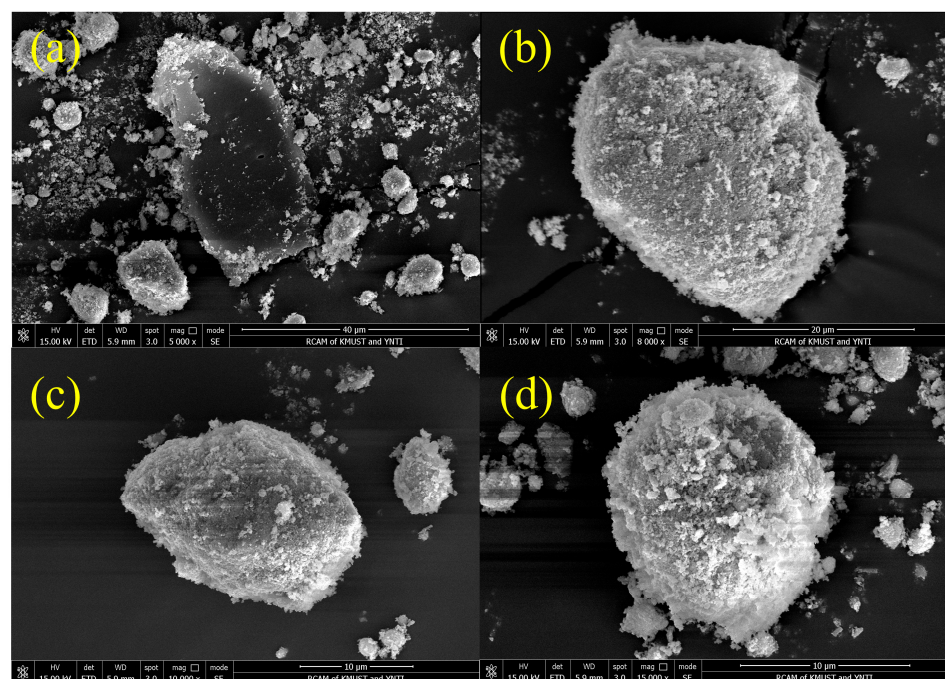


Figure 2. SEM maps of limonite: (a) 5000 $\times$, (b) 8000 $\times$, (c) 10,000 $\times$, (d) 15,000 $\times$.

Table 2 lists the chemical properties of bentonite: bentonite as a pellet binder actually plays a role in montmorillonite, gel price reflects the montmorillonite dispersion performance indicators, and the water absorption rate indicates the bentonite water-absorbing capacity of the indicators [22–24]. The montmorillonite mass fraction of the bentonite used

was as high as 85.97%, which indicated that the bentonite had good physical property indexes.

Table 2. Composition and properties of bentonite.

Moisture/%	Colloid Index/(%·(3 g) ^{−1})	Swelling Capacity (mL·g ^{−1})	Water Absorption/%	Methylene Blue Index/(g·(100 g) ^{−1})	Montmorillonite Mass Fraction/%
9.38	20.0	34.5	408	38.0	85.97

The composition and properties of the bentonite used in the experiment are shown in Table 2.

2.2. Instrumentation and Test Methods

Instruments and equipment: All brands used in the experiment were made in China. The experimental disc ball-making machine model 6GU-15K had a disc diameter 500 mm, and external dimensions of 600 × 550 × 1000 mm. Also used were a new ball mill QHQM-100; electronic balance PR224ZH and ZN-C10002; ball compressive strength tester DS2; muffle furnace roasting instrument STM-12-14; and digital pressure tester SKE-10KN.

Test Methods:

- (1) **Pellet production:** The experimental process is shown in Figure 3, including the grinding of limonite powder using a ball mill, screening, mixing of additives, molding of raw balls, and determination of strength properties of finished pellets. Before the experiment, a new type of ball mill was used to grind limonite ore from a plant in Yunnan for 72 h, a 100-mesh automatic sieving machine was used to screen the mineral powder for the experiment, 200 g of 100-mesh limonite iron ore powder for the experiment was mixed with bentonite clay according to the certain proportions of the ball in a disc ball-making machine, 3% of distilled water was added in equal amounts to the mineral powder for wetting, and 7% of distilled water was sprayed in order to make a pellet. After stopping the addition of water and material, the raw pellets continued to roll in the disc ball-making machine for about 2 min until the pellets were dense. They were then taken out with a spoon and then a vernier caliper was used to separate the pellets; the particles with diameters of 8–15 mm were left for spare parts, and the rest of the unqualified pellets were discarded. The pellets were dried in a desiccator at 110 °C for 1 h to remove moisture and volatiles for subsequent use.
- (2) **Pellet roasting:** The raw pellets were transferred to the crucible in batches. The crucibles with the pellets were baked in a muffle furnace. The roasting temperature was set as follows, as shown in Figure 4: ramped up to 200 °C over 20 min and held at 200 °C for 20 min; ramped up to 700 °C over 20 min and held at 700 °C for 20 min; and increased to 1250 °C over 40 min and held at 1250 °C for 30 min. Subsequently, the pellets were gradually cooled to room temperature and removed from the furnace.
- (3) **Measurement and testing:** The diameter and weight of raw and roasted pellets were measured using an electronic balance. The compressive strength of raw pellets and roasted pellets was determined by using the raw pellet compressive strength tester DS2 and digital display pressure tester SKE-10KN. Using a vernier caliper to select pellets with a diameter of 8–15 mm, the average compressive strength of raw and roasted limonite pellets was recorded in accordance with YB/T 4848-2020 Physical Test Methods for Roasted Pellets.

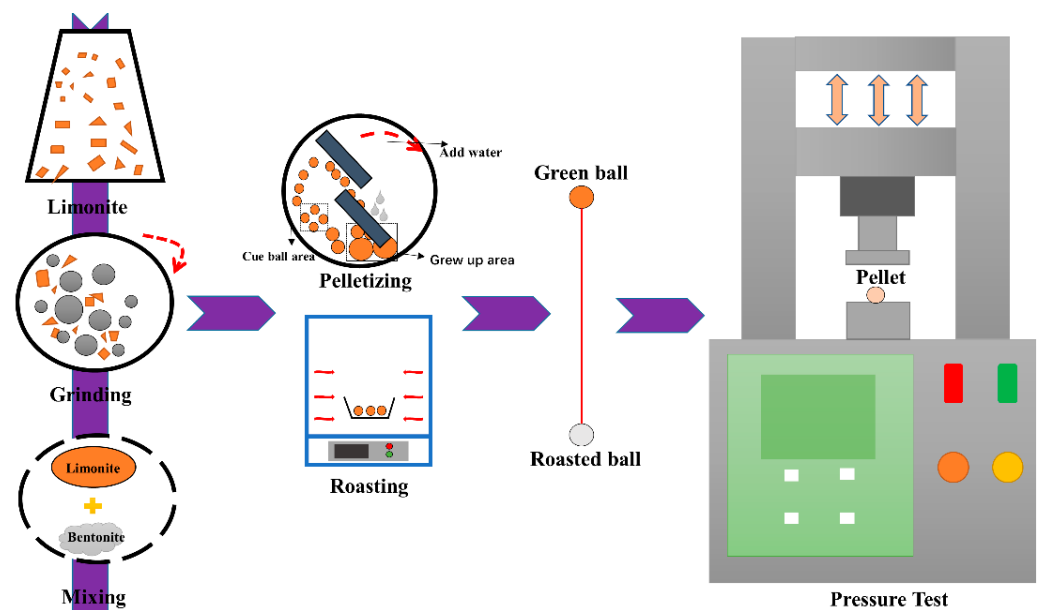


Figure 3. Limonite pellet preparation process.

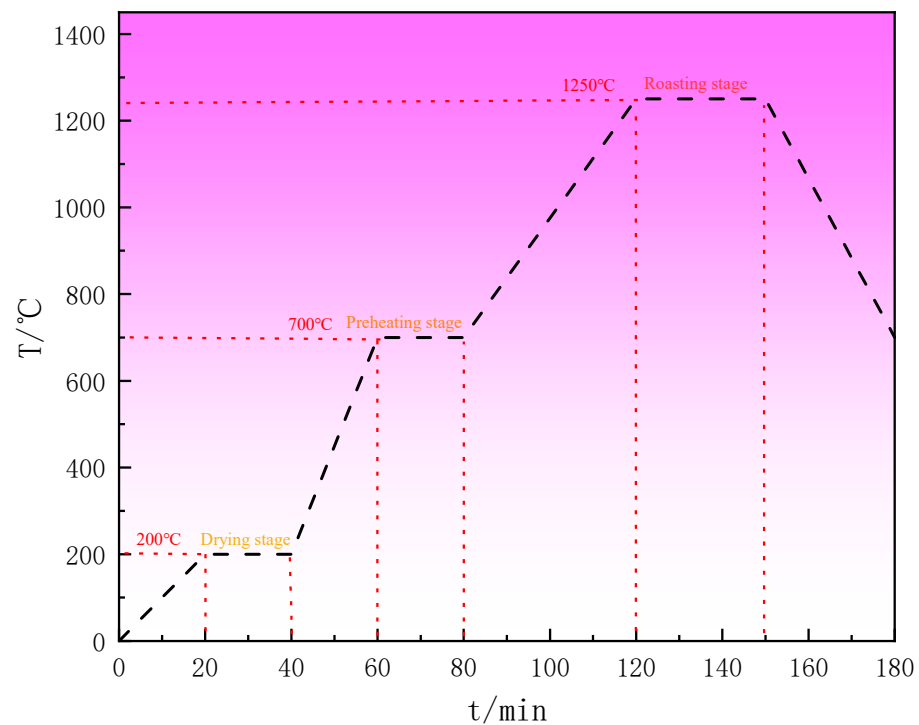


Figure 4. Stepwise heating temperature profile of limonite pellets.

3. Test Results and Analysis

3.1. Effect of Drying Temperature on Pellet Strength

During the experiment, the proportion of bentonite binder was fixed at 1% for balling, and pellets of 8–13 mm particle size were selected for dry strength testing. The drying time was 20 min, and five drying temperature intervals of 150 °C, 200 °C, 250 °C, 300 °C and 350 °C were set for the experiment. The purpose of using 50 °C as a temperature range was to scale down the experimental error. The strength was tested on a raw ball compressive strength apparatus at the end of the test. As shown in Figure 5, the drying intensity increased and then decreased with the increase in drying temperature. Among them,

the drying intensity at 200 °C showed the best performance, indicating that the drying temperature of 200 °C is the optimal temperature for the roasting process of limonite pellets. The 300 °C and 350 °C pellet performance strength was relatively poor because in the pellet muffle furnace heating process, due to the fixed period of time, the temperature rises too quickly, leading to the raw ball burst phenomenon. In summary, the unfavorable conditions throughout the process ultimately contribute to the poor strength of the pellet.

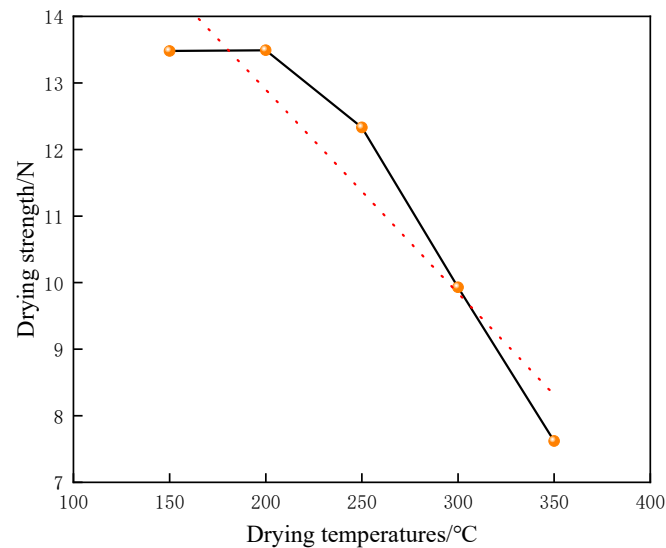


Figure 5. Effect of drying temperature on drying strength.

After exploring the effect of drying temperature on drying strength, the drying temperature of 200 °C was found to be optimal. The experimental conditions were controlled unchanged, and the experimental data of 200 °C pellets were collated as shown in Figure 6: the drying strength increased with the increase in pellet diameter. The reason for this is that larger-diameter pellets have a higher drying strength due to their larger volume, better uniformity of volume contraction, and more uniform distribution of internal stresses. Smaller pellets, on the other hand, may shrink unevenly in volume due to uneven moisture loss during drying, and the pellets are more prone to rupture.

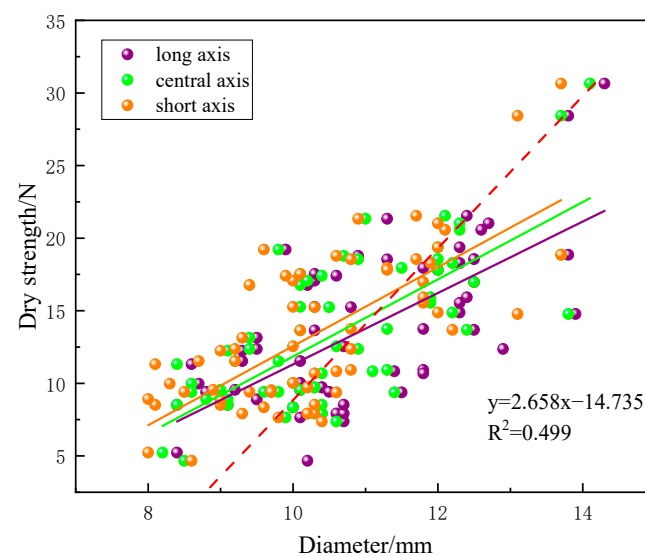


Figure 6. Effect of diameter on drying strength.

3.2. Effect of Preheating Temperature on the Compressive Strength of Pelletized Ores

In this experiment, the control of bentonite addition was 1% as the basis to study the effect of preheating temperature on the compressive strength of pellet ore, and compressive strength tests were performed on pellets with sizes of 8–13 mm. The experimental results are shown in Figure 7, and the highest compressive strength was 1825.95 N at a preheating temperature of 700 °C. This indicates that the preheating temperature of 700 °C just promotes the oxidation of limonite pellets, and the oxidation exotherm is the more active Fe_2O_3 , which is favorable for the formation of crystal bonds between mineral powder particles. The experimental results at preheating temperatures between 700 °C and 900 °C showed that the compressive strength of the pellets showed a decreasing trend as the preheating temperature increased. When the compressive strength of the pellet decreases, this indicates that the preheating process is taking place. During this process, there are transformations in the mineral phase, volatilization of crystalline water, decomposition of certain compounds, and other reactions. Insufficient reaction occurs when the temperature is too low. When the preheating temperature is 900 °C, the temperature is too high and will destroy the internal structure of the pellet; and as a result, it is too late to preheat the pellet directly into the roasting state, which ultimately leads to the compressive strength of the pellet being very low. Therefore, too low or too high a preheating temperature will lead to changes in the compressive strength of the pellet.

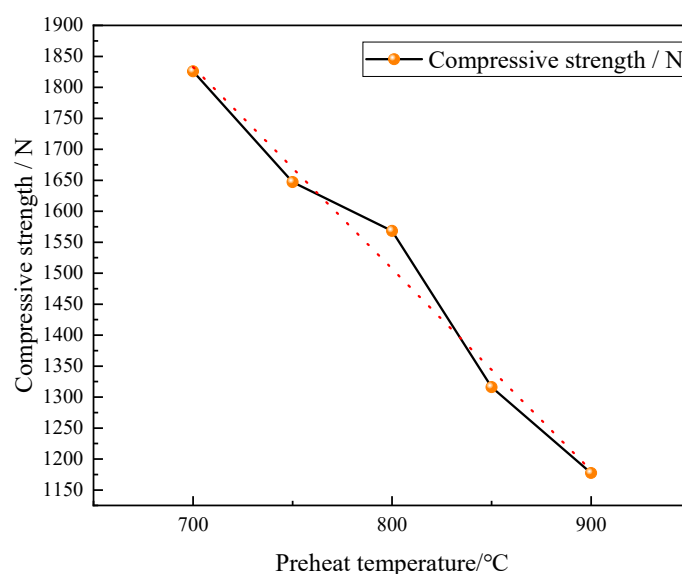


Figure 7. Effect of preheating temperature on compressive strength.

After exploring the effect of preheating temperature on compressive strength, Figure 8A represents a three-dimensional plot of the average weight of pellets against preheating strength at different preheating temperatures. As can be seen from Figure 8A, 700 °C corresponds to the best pellet strength. B, C, D, E, and F in Figure 8 show the mechanism of the weight effect on the compressive strength of pellets at the different preheating temperatures, 700 °C, 750 °C, 800 °C, 850 °C, and 900 °C, respectively, which were investigated in this experiment. It can be concluded that the compressive strength of the pellet increases with an increase in the weight of the pellet under different preheating temperature conditions. It can be seen that the highest number of pellets was accounted for by the 2 g mean compressive strength above 2000 N and at 700 °C, while 900 °C accounted for the lowest number of pellets, and with an increase in preheating temperature, the strength of the pellets gradually decreases, which further proves that 700 °C is the optimal preheating temperature for limonitic iron ore pellets.

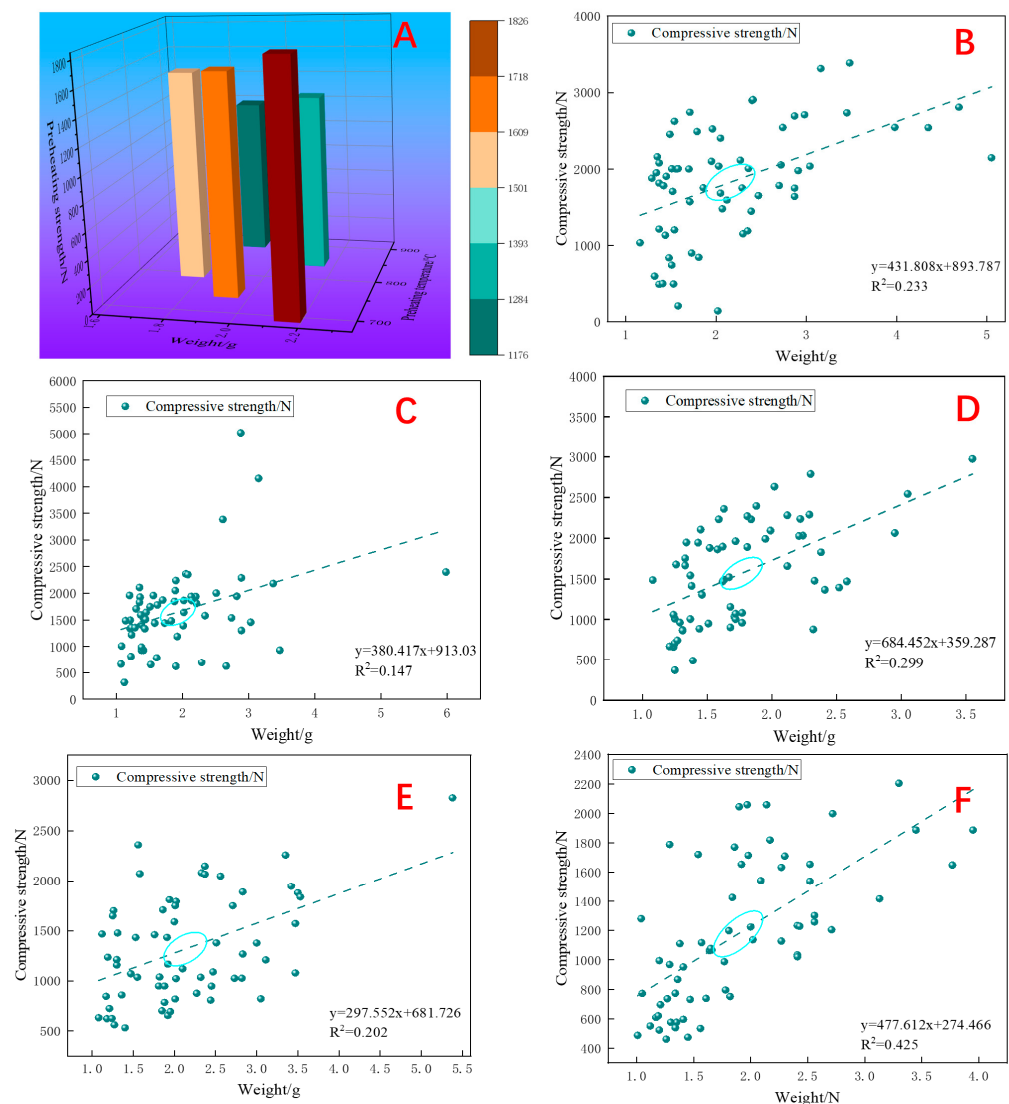


Figure 8. Effect of weight on compressive strength at different preheating temperatures. (A) Three-dimensional map of different preheating temperatures; (B) 700 °C; (C) 750 °C; (D) 800 °C; (E) 850 °C; (F) 900 °C.

Figure 9A represents a three-dimensional plot of the average diameter of the pellets against the preheating strength at different preheating temperatures. It can be seen from Figure 9A that 700 °C corresponds to the best pellet strength. Figure 9B–F show the laws for investigating the effect of diameter on compressive strength at preheating temperatures of 700 °C, 750 °C, 800 °C, 850 °C, and 900 °C. The proportion of bentonite was fixed at 1% and pellets with 8–13 mm grain sizes were screened for compressive strength testing, and the long, middle, and short axes of each pellet were recorded. The collated experimental data are shown in Figure 9C–E below: at preheating temperatures of 750 °C, 800 °C and 850 °C, it can be seen that the compressive strength of the pellets gradually increases as the long, middle, and short axes of the pellets continue to increase. A relationship between pellet diameter and compressive strength cannot be seen in Figure 9F, and the compressive strength is also relatively small. Compared with the compressive strength of pellets at the 700 °C preheating temperature, although the strength of some pellets at the 750 °C preheating temperature is high, it is not concentrated. It can therefore be concluded that the optimal preheating temperature of limonite pellets is 700 °C.

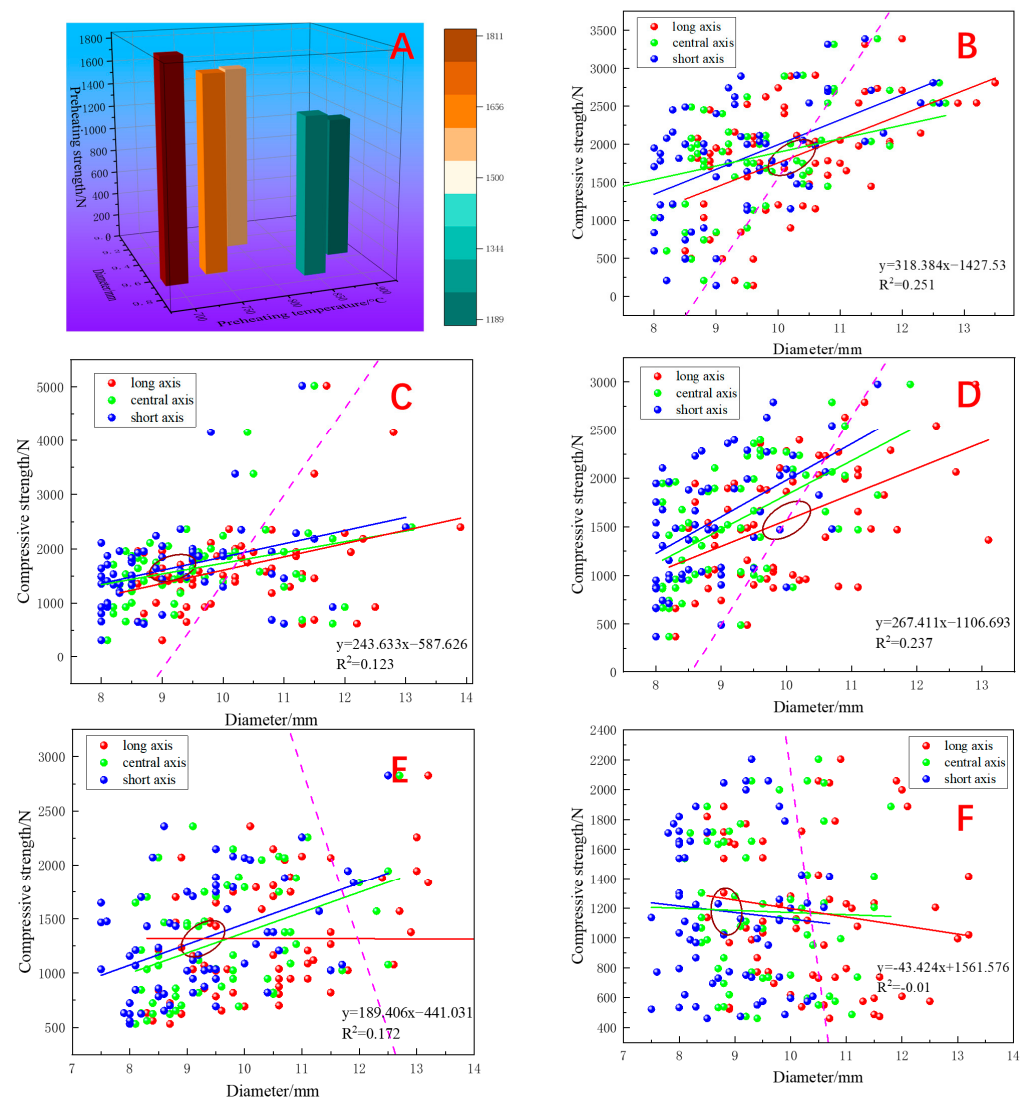


Figure 9. Effect of diameter on compressive strength at different preheating temperatures. (A) Three-dimensional map of different preheating temperatures; (B) 700 °C; (C) 750 °C; (D) 800 °C; (E) 850 °C; (F) 900 °C.

3.3. Effect of Roasting Temperature on the Strength of Pelletized Ores

The proportion of bentonite was fixed at 1%, and the preheating temperature was 700 °C. Pellets with 8–13 mm grain sizes were screened to explore the effect of roasting temperature on the compressive strength of pellet ores. According to the experimental results, the effect of roasting temperature on the compressive strength of pellets is very significant, and the compressive strength gradually increases with the increase in roasting temperature. As shown in Figure 10, the compressive strength of the pellets reaches its highest value when the roasting temperature is 1250 °C, and the compressive strength of limonite pellets improves by 1600 N when the roasting temperature is increased to 1250 °C compared to 1050 °C. Reason for analysis: The recrystallisation and grain growth processes within the pellet require sufficient temperature. A temperature that is too low is not conducive to these two processes, leading to a looser internal structure, increased porosity, and insufficient crystallization. As a result, the compressive strength of the pellets increased substantially when the roasting temperature was raised.

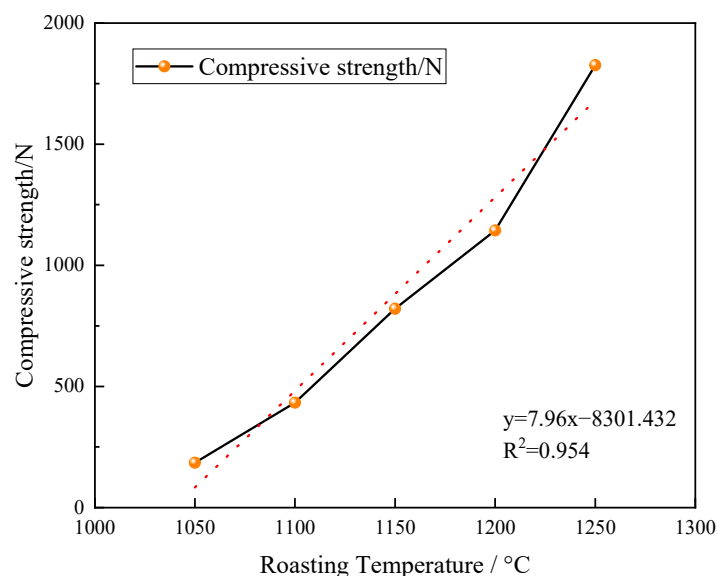


Figure 10. Effect of roasting temperature on compressive strength of pellets.

3.4. Effect of Roasting Time on the Strength of Pelletized Ores

The experimental conditions were controlled unchanged to investigate the effect of roasting time on compressive strength. The experimental results are shown in Figure 11A, and the highest compressive strength was seen for the roasting time of 20 min. The compressive strength of the pellets increased gradually with the increase in time when the roasting time was between 10 min and 20 min; in contrast, between 25 min and 30 min, the pellet compressive strength decreased gradually with time. Recrystallisation and grain growth within a pellet takes time, and prolonged roasting allows these processes to be completed gradually, resulting in an increase in the compressive strength of the pellet. However, after a certain period of time, the compressive strength no longer improves. This is because the degree of crystallization within the pellet ore increases, leading to an increased expansion rate. As a result, the pellet may experience internal rupture or even melting, resulting in a reduction in its compressive strength.

In order to investigate the influence law of the strength of the pellets of suitable size particle classes by collating the experimental data, B, C, D, E, and F in Figure 11 were used to represent the relationship between the diameter of limonitic iron ore pellets and their compressive strength under the roasting times of 10 min, 15 min, 20 min, 25 min, and 30 min. From the graph, it can be concluded that the strength increases with increasing diameter, where it is optimal at a roasting time of 20 min. Analyzing the reasons, larger-diameter pellets have more particle connection points, resulting in closer internal mechanical connections and a more stable structure. It can also be seen in the figure that some of the small-grain-size pellets also perform better in terms of compressive strength. The reason may be that the small-grained pellet particles have a more uniform shape, which makes the particles have a strong bonding effect between them, and the close connection between the grains inside the pellet reduces the existence of pores and crevices, which enhances the overall structural stability and compressive strength. The conclusion shows that a pellet diameter of 8–13 mm is the most suitable size, and the pellet strength is also good and uniform.

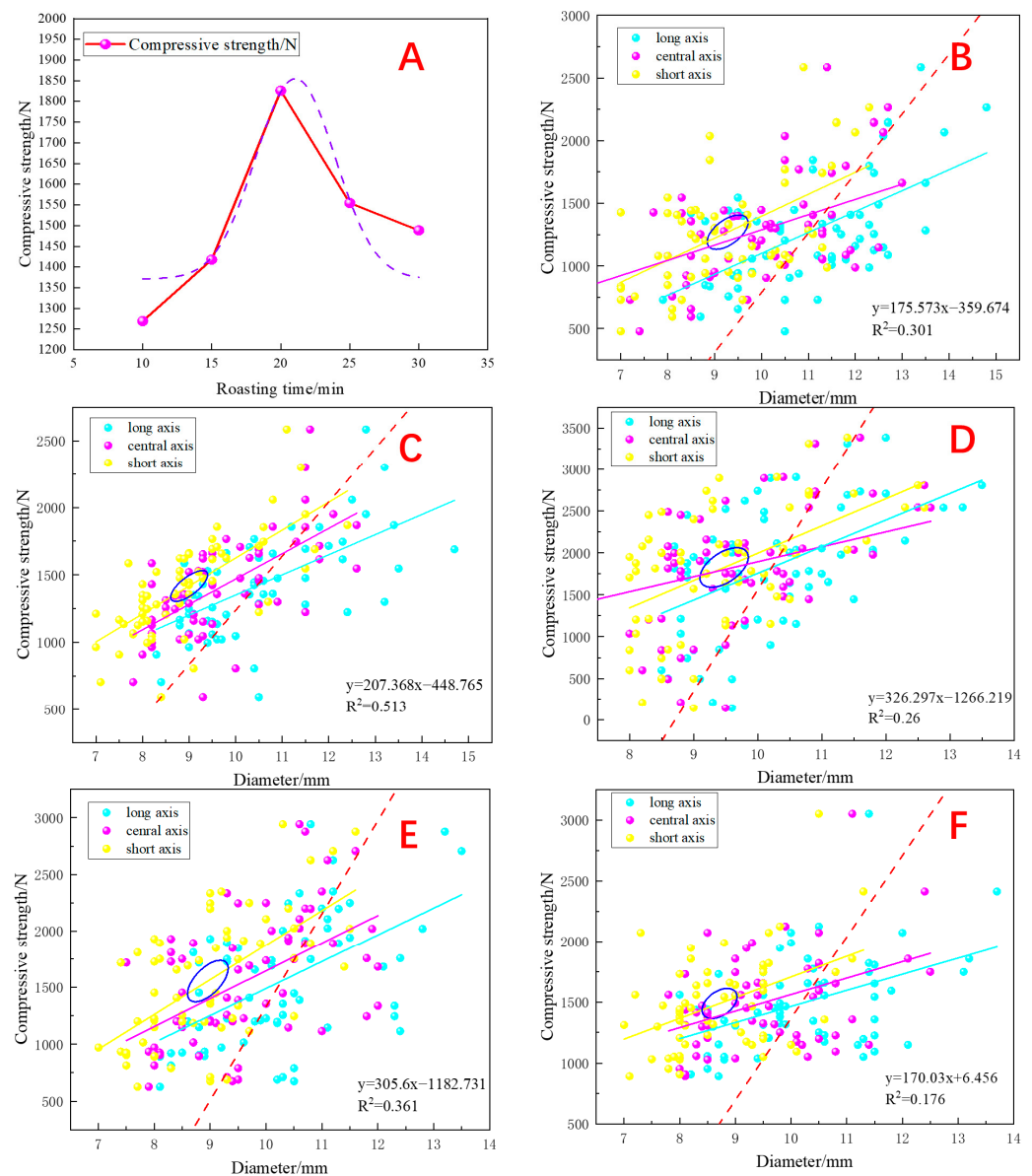


Figure 11. Effect of diameter on compressive strength of pellets at different roasting times. (A) Roasting time; (B) 10 min; (C) 15 min; (D) 20 min; (E) 25 min; (F) 30 min.

To further investigate the effect of roasting time on compressive strength, the effect of the weight of pellets on compressive strength was recorded separately for different roasting times in the experiment. As shown in Figure 12, the pellet compressive strength still increased with increasing mass, where A, B, C, and D represent the roasting times of 10 min, 15 min, 25 min, and 30 min, respectively. At 10 min and 15 min of roasting, the compressive strength of the pellets increased with time. As the roasting time continued to be extended, the volume inside the pellet ore gradually increased due to crystallization changes. This resulted in a higher expansion rate within the pellet and the presence of surface cracks, ultimately leading to the low strength of limonite. As a result, the pellet strength was low and dispersed at 25 min and 30 min of roasting, as seen in the graph.

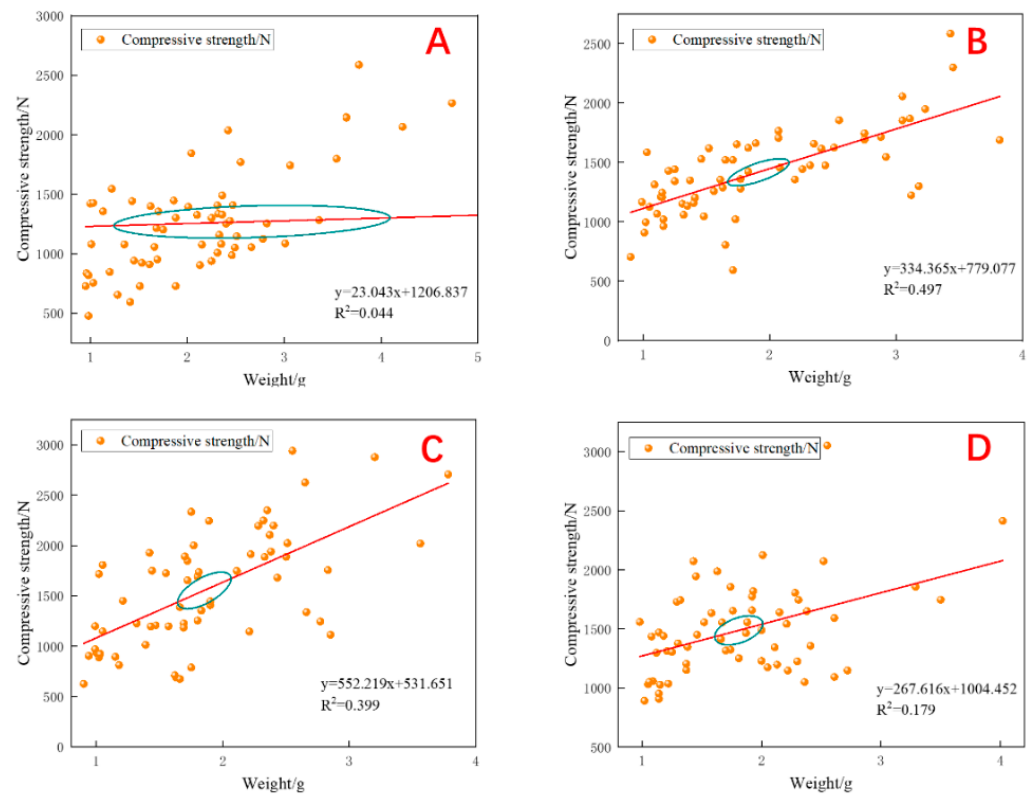


Figure 12. Effect of weight on compressive strength for different roasting times. (A) 10 min; (B) 15 min; (C) 25 min; (D) 30 min.

3.5. Effect of Bentonite Ratio on Compressive Strength

In order to investigate the effect of the bentonite ratio on the compressive strength of finished balls, during the experiment, the drying temperature was set at 200 °C, the roasting temperature was set at 1250 °C, the preheating temperature was set at 700 °C, the roasting time was set at 20 min, and the compressive strength of the roasted balls was tested. The experimental results are shown in Figure 13: the compressive strength of the roasted balls showed a trend of first increasing and then decreasing with the increasing bentonite ratio. Among them, the maximum compressive strength of the roasted balls reached 2042.88 N at a 1.5% bentonite ratio. The ratios increased at 0.6% to 1.5% strength and continued to increase to 1.8% when the strength began to decrease. Bentonite can improve the particles between the iron ore materials, so that after roasting, the internal molecular bonding of the pellet becomes larger, and the strength of the pellet is improved. When the bentonite ratio continued to increase, a large amount of bentonite piled up in the material between the particles, leading to shrinkage, incomplete pore space, and large porosity after pellet roasting, and ultimately a decline in compressive strength.

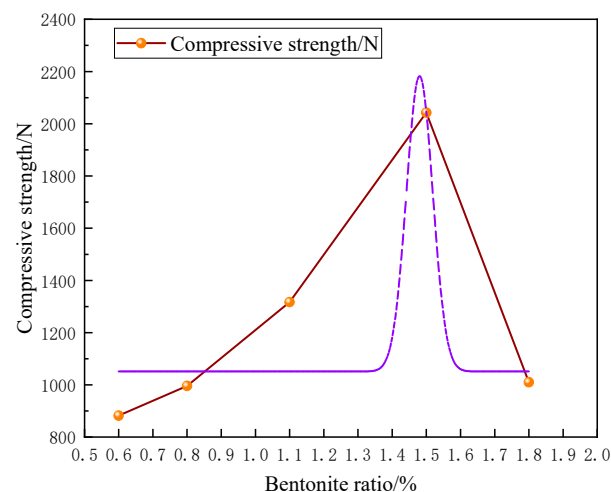


Figure 13. Effect of bentonite ratio on compressive strength.

3.6. SEM-EDS Image Analysis of Limonite Pellets

Figure 14 shows the SEM-EDS images of the bentonite–limonite pellets. From the figure, it can be observed that after roasting under the condition of 1250 °C, the main components of limonite pellets were elemental iron and oxygen, which were uniformly distributed on the surface of the pellets. According to the results of the spectral analysis, the percentage of iron and oxygen was 89.6 per cent. With the addition of the bentonite binder, the pores of limonite pellets were uniformly distributed and interconnected, and such structural characteristics help to improve the physical stability and mechanical properties of the pellets. After the pellets were oxidised and roasted, the lapping of crystal bridges between their grains was easier, thus increasing their compressive strength. In addition, the iron (Fe) content of the bentonite–limonite pellets was 65.3 per cent. In contrast, the total iron content of limonite pellets was only 54.67 per cent, so bentonite increased the iron content of limonite pellets by 10.63 per cent.

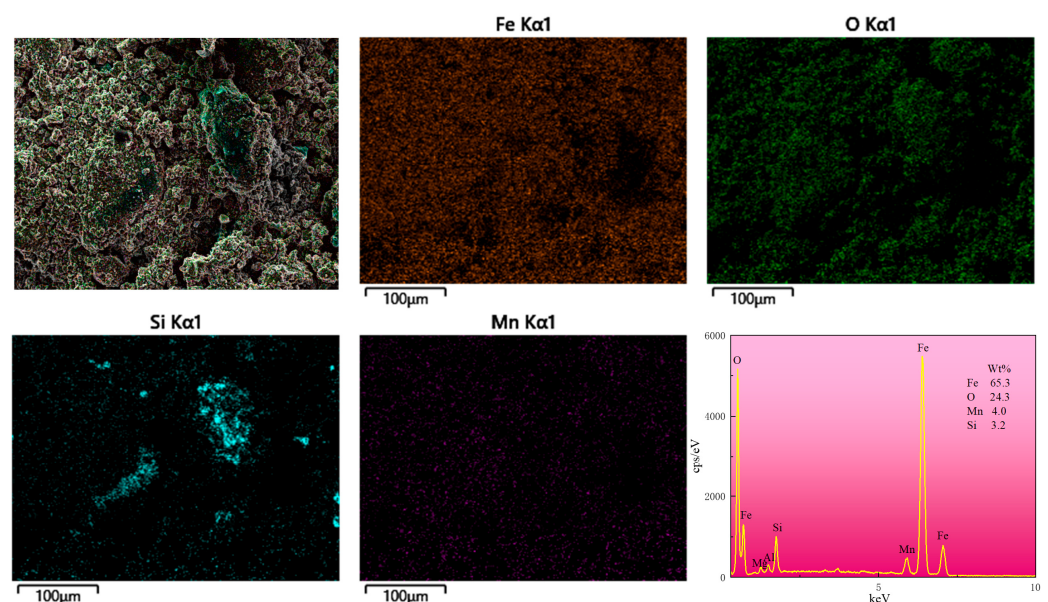


Figure 14. SEM-EDS image of bentonite–limonite pellets at 1250 °C.

4. Conclusions

In this study, based on the multidimensional experimental results, limonite pellets showed the best results under the conditions of an optimal drying temperature of 200 °C, optimal preheating temperature of 700 °C, optimal roasting temperature of 1250 °C, and

optimal roasting time of 20 min; the pellets with 8–13 mm grain diameter had the optimal size and uniform strength. In limonite pellets, when the amount of bentonite added reaches 1.5%, the compressive strength of the pellets reaches the highest value, meeting the needs of a general blast furnace. After oxidizing and roasting treatment, the crystal bridges between the grains of limonite pellets are more conveniently lapped, thus improving their compressive strength.

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