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Study on Preparation and Properties of Industrial Lignin Metallized Pellets for Ironmaking

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Abstract

The pulp and paper industry produces a large amount of industrial lignin as biomass waste every year and it is not effectively utilized, resulting in a waste of resources. In this paper, by exploring the influencing factors of the strength of industrial lignin green pellets and metallized pellets, it is expected that industrial lignin can be effectively applied to the ironmaking industry to achieve the purpose of energy saving and emission reduction. The results show that when the molar ratio of carbon to oxygen is 1.2, the falling strength and compressive strength of industrial lignin pellets can reach 6.8 times/0.5 m and 26.8 N, respectively. When the calcination temperature is 1100 °C, the compressive strength of industrial lignin metallized pellets reaches 78.5 N; when the roasting time is 60 min, the compressive strength of the industrial lignin metallized pellets is 865 N, and the metallization rate is 99.72%. It can meet the requirements of industrial production.

Keywords: industrial lignin; falling strength; metallized pellets; compressive strength; iron joined crystal

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1. Introduction

The annual CO_2 emissions of the steel industry account for about 6.7% of the global total emissions, and the energy consumption and emissions of the ironmaking system account for about 70% of the total energy consumption and emissions of the whole steel process [1–6]. With the implementation of carbon peaking and carbon neutralization goals and requirements, ironmaking technology in the new century should be able to comprehensively deal with resource, energy, and environmental problems.

At present, the main processes of ironmaking include blast furnace ironmaking, smelting reduction, and direct reduction, as shown in Figure 1. In 2023, the world's crude steel production was 1.892 billion tons, of which 71.1% was produced by the traditional blast furnace–converter long process [7]. It can be seen that the blast furnace ironmaking process is still the main process of producing pig iron. According to the limitations of China's resources and energy conditions, China's ironmaking production process will still be dominated by blast furnaces for a long period of time. The blast furnace ironmaking process needs to consume a large amount of coal-based fossil energy,

so how to minimize the energy consumption of the blast furnace ironmaking process is a problem that must be solved to ensure the sustainable development of the ironmaking process in the future.

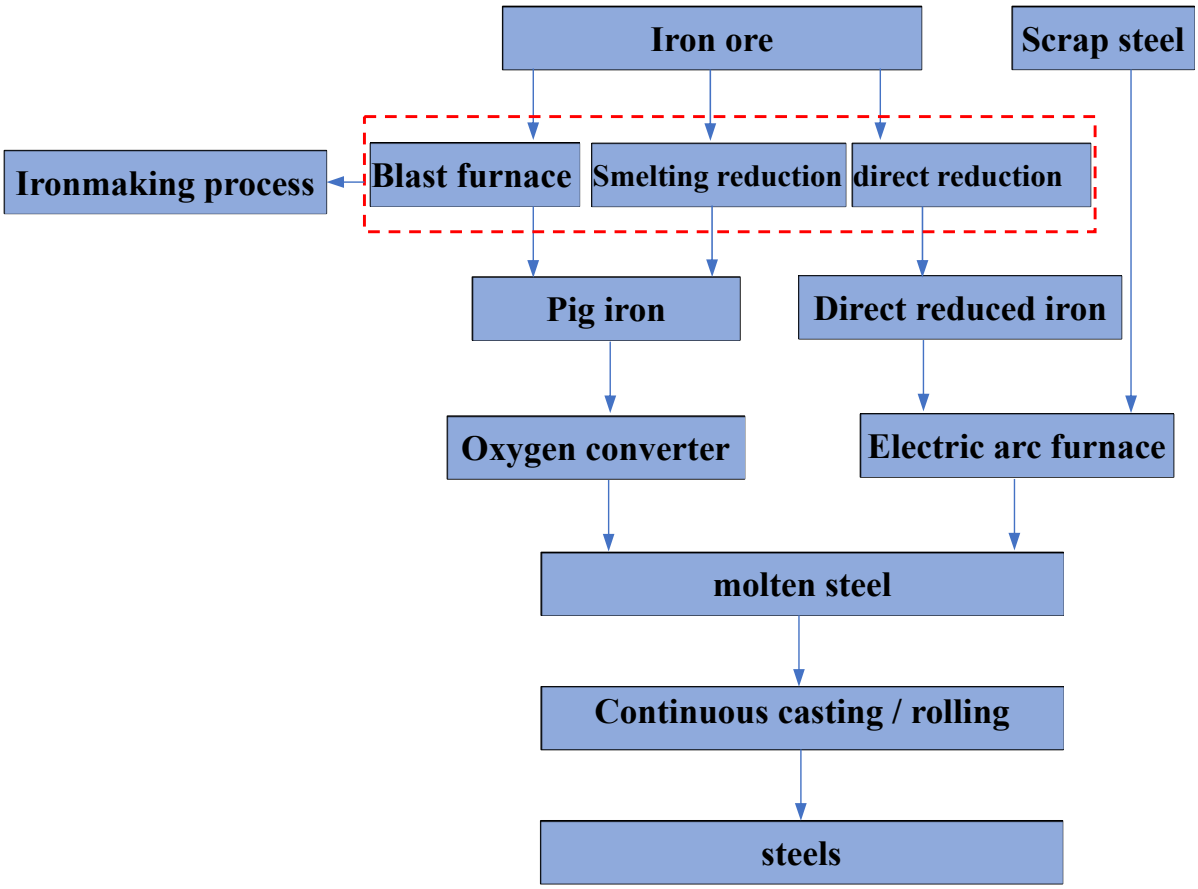

Figure 1. Main process route of iron and steel production.

As a clean and renewable green carbon resource, biomass has received widespread attention in the context of low-carbon ironmaking, with its application in blast furnace ironmaking production. At present, the research on biomass ironmaking has been carried out mainly in the following three aspects, as shown in Figure 2: (1) Biomass is applied to the sintering process [8–12]. (2) Biomass is applied to the pelletizing process [13–18]. (3) Biomass is applied to blast furnace injection [19–21]. The staged research results obtained at present show the following: (1) Biomass used in sinter production can effectively reduce the content of CO_2, SO_x, NO_x, and soot in tail gas. (2) The reactivity of biomass char with CO_2 is significantly better than that of pulverized coal. (3) Compared with pulverized coal–iron ore composite burden, biomass–iron ore composite burden can obtain a higher metallization rate at a lower reaction temperature and shorter roasting time. (4) Biomass injection into the blast furnace can effectively reduce the amount of slag and reduce energy consumption. At present, there is only a precedent for the injection of charcoal in small blast furnaces in some countries rich in wood, while industrial or semi-industrial experiments on the application of biomass in sinter production and directly reduced iron production are rarely reported. The main reasons are as follows: (1) The cost of biomass collection and processing is high. Compared with the centralized supply of pulverized coal, the origin of agricultural and forestry waste is relatively scattered, which has obvious regional and seasonal characteristics, thus causing the high costs of biomass resource collection and processing in industrial applications. (2) The carbonization treatment of biomass is

carried out under high temperature and anaerobic conditions and is greatly affected by the moisture content of biomass. A large amount of energy is consumed during the treatment process, which greatly increases the treatment cost. (3) The strength of sinter and metallized pellets produced by adding biomass is low, and cannot meet the requirements of raw material strength for blast furnace ironmaking. (4) Biomass generally has the disadvantages of high moisture and alkali metal content, low fixed carbon content and calorific value, and low energy density. It is difficult to meet the requirements of blast furnace injection technology for fuel process performance without upgrading treatment.

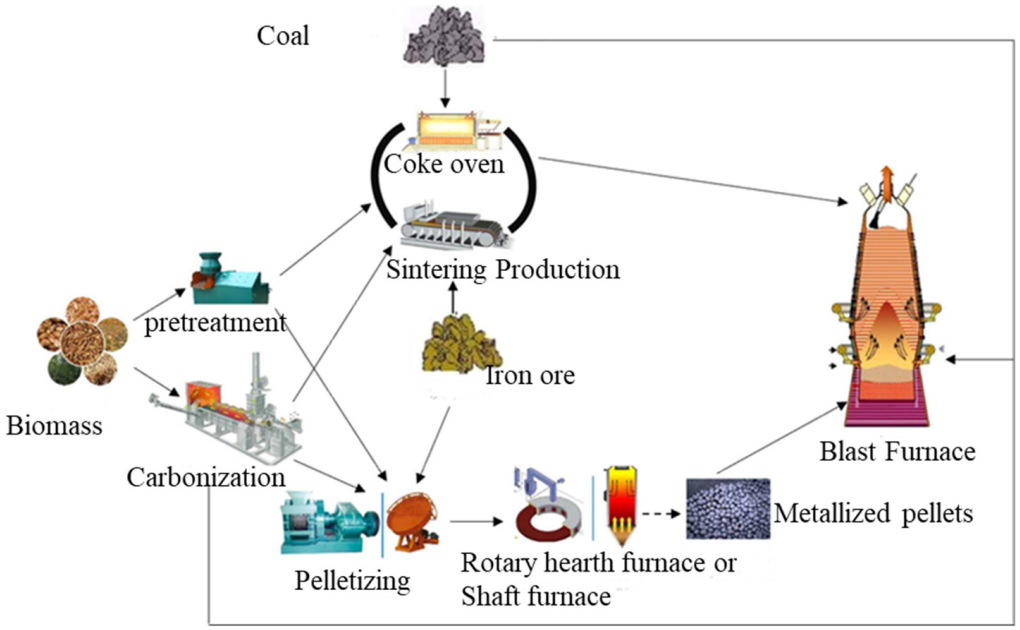

Figure 2. Application route of biomass in blast furnace ironmaking.

Lignin is the second most abundant natural polymer in the plant kingdom. It forms the main structure of plants together with cellulose and hemicellulose, and is regenerated at a rate of 150 billion tons per year [22,23]. Industrial lignin mainly exists in the waste liquid produced by the pulp and paper industry and the wood hydrolysis industry. Industrial lignin with low purity can be obtained by cooking and concentrating the waste liquid. If the waste liquid is treated by acid precipitation, ultrafiltration or flocculation precipitation, relatively pure industrial lignin can be obtained. At present, there is no technical problem in the extraction of industrial lignin, but the chemical treatment in the pulping process leads to a wide range of chemical transformations such as depolymerization, redox, and condensation of the natural structure of lignin [24–26]. These transformations result in a highly complex structure of industrial lignin, which ultimately leads to a low utilization rate. About 20 million tons of industrial lignin is contained in the pulping waste liquor produced by China's paper industry every year [27], but only less than 10% of the total lignin is recycled and made into low value-added products (such as coagulants, lubricants, adhesives, and dispersants). More than 90% of the lignin is still directly discharged into rivers or concentrated and burned as waste liquid, which wastes resources and pollutes the environment [28]. Industrial lignin is difficult to modify due to its complex structure, and it needs special pretreatment, which undoubtedly seriously hinders its application and development. Therefore, if industrial lignin is applied to the production of metallized pellets, it can improve the energy structure of steel production and reduce CO_2 emissions on the one hand, and on the other, it can also absorb a large amount of carbon-containing waste

from the paper industry, reduce the impact of disorderly combustion on the environment, and improve the utilization efficiency of waste.

The application of industrial lignin to the production of metallized pellets is mainly based on the following considerations: (1) The source of industrial lignin is relatively simple and stable, mainly from pulp and paper enterprises. (2) The main components of industrial lignin are C and H, and a large number of reducing gases containing C and H and biomass char are produced during the pyrolysis process, which is very conducive to the reduction of iron oxides. (3) Industrial lignin itself has a certain bonding effect, which is beneficial for improving the strength of cold bonded pellets during production [29]. (4) The production process of industrial lignin metallized pellets can involve a mature rotary hearth furnace or shaft furnace process without the need to develop new equipment, thereby saving on production costs.

Previous studies have shown that the use of metallized charge in a blast furnace is beneficial for improving the metallization rate of molten iron, increasing the yield of molten iron, and effectively reducing the coke ratio [30–33]. Additionally, the use of clean metallized charge in electric furnace steelmaking can effectively dilute the harmful elements in molten steel, improve the chemical composition of molten steel, and produce high-quality steel and pure steel [34]. In this paper, industrial lignin is used to prepare metallized pellets, and its strength is studied to explore whether it can be applied to blast furnace ironmaking or electric furnace steelmaking. It is hoped that this study will help China to achieve its carbon peak and carbon neutralization targets as soon as possible, and enhance the synergistic emission reduction effect of the iron and steel industry and the pulp and paper industry.

2. Raw Materials and Methods

2.1. Materials

The test raw materials are mainly three reducing agents (Lu'an anthracite, Shenmu bituminous coal, and an industrial lignin) and iron ore powder. The results of the industrial analysis, elemental analysis, and compositional analysis are shown in Tables 1 and 2. Lu'an anthracite, Shenmu bituminous coal, and iron ore powder were provided by an iron and steel company, while IL was provided by Huawei Youbang Chemical Co., Ltd. (Tumen, China). The XRD analysis results of iron ore powder are shown in Figure 3, which shows that the iron ore powder is mainly composed of Fe_3O_4.

Table 1. Proximate analysis and elemental analysis of pulverized coal and industrial lignin (mass, %).

Samples	Proximate Analysis (ad.)				Elemental Analysis (ad.)				
	Fixed Carbon	Ash	Volatile	Moisture	C	H	O	N	S
IL	26.35	8.31	60.44	4.89	59.56	6.82	29.67	0.20	1.82
LA	75.90	10.01	13.38	0.71	82.00	3.41	8.50	1.79	0.20
SM	62.41	7.46	28.28	1.85	75.90	4.14	16.50	1.38	0.63

ad.: air-dried basis.

Table 2. Composition analysis of iron ore powder (wt%).

TFe	FeO	SiO_2	CaO	Al_2O_3	MgO	Others
68.04	24.88	5.50	0.14	0.28	0.36	0.8

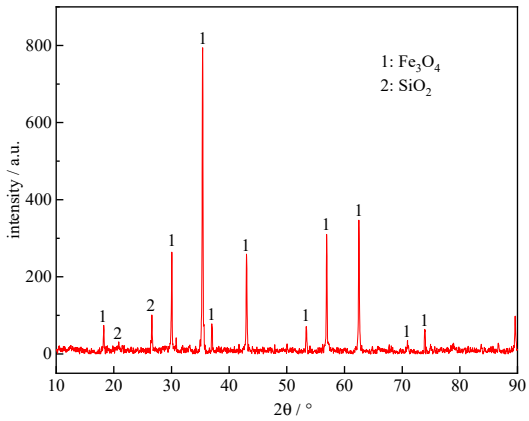

Figure 3. XRD analysis of iron ore powder.

2.2. Methods

2.2.1. Element and Component Analysis

The industrial analysis of industrial lignin was based on GB/T 212-2008 [35]. The O, N, and H elements were analyzed using the ONH836 analyzer (LECO, Laboratory Equipment Corporation, San Jose, CA, USA). The S and C elements were analyzed using an SC-144DR analyzer (Laboratory Equipment Corporation, Hayward, CA, USA). The composition analysis of iron ore powder was provided by the steel company. The phase of iron ore powder was analyzed using the D8ADVANCE X-ray diffractometer (XRD, Bruker, Bremen, Germany), the scanning range was 10–90 degrees, and the scanning rate was 5 degrees/min.

2.2.2. The Preparation of Green Pellets

According to previous research results [36], when n_c/n_o = 1.2 (n_c is the mole fraction of total carbon in the reductant and n_o is the mole fraction of oxygen in Fe_3O_4 in iron ore powder), the reduction rate of iron ore powder reduced by reductant is the highest. Therefore, in this study, the mass ratio of reducing agent to iron ore powder was determined according to n_c/n_o = 1.2. The mixed samples were pelleted with a disk pelletizer (produced by Northeastern University), as shown in Figure 4. The pelletizing time was 40 min, and the compaction time was 3 min. The moisture content of the green pellets was 10~12%, and the diameter of the green pellets was 10~12.5 mm. The diameter of the disk pelletizer is 1000 mm, the depth is 200 mm, the horizontal inclination angle is 45°, and the rotation speed is 20 r/min.

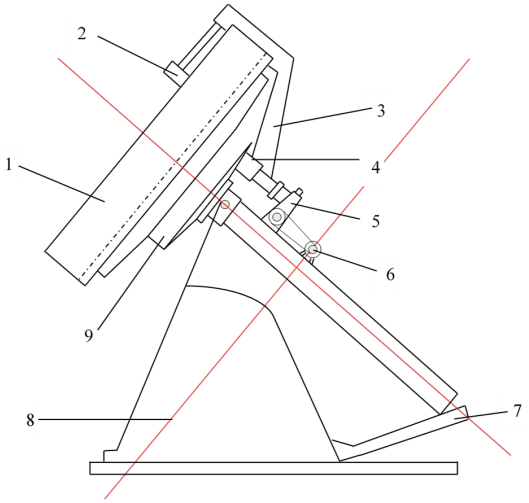

Figure 4. Schematic diagram of disk balling machine. 1—disk; 2—scraper; 3—scraper frame; 4—pinion; 5—reducer; 6—motor; 7—Angle spiral; 8—base; 9—Internal gear ring.

2.2.3. The Falling Strength of Green Pellets

Ten green pellets with uniform particle size were selected and dropped naturally from a height of 0.5 m to a steel plate with a thickness of 2 cm until cracks or fractures appeared in the pellets. The number of drops was recorded as N times, and the number of drops for each green pellet was (N − 1) times. The average value of 10 green pellet drops is the strength index of green pellet drops.

2.2.4. The Compressive Strength of Green Pellets

The compressive strength of the pellets was measured using the KQ-2 particle strength tester (Figure 5), with a maximum range of 100 N and an accuracy of 0.01 N. Ten green pellets with uniform particle size were selected for testing on the tester, and the average value was obtained as the compressive strength index of green pellets.

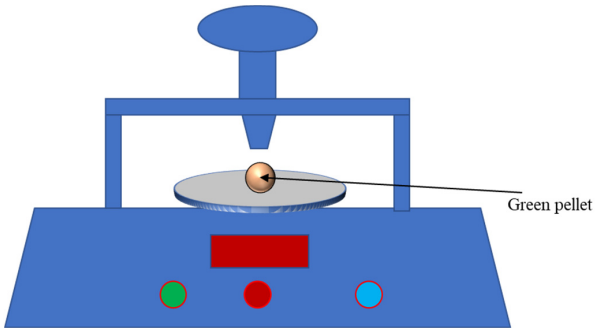

Figure 5. Compression strength tester for green pellets.

2.2.5. The Roasting of Green Pellets

The previous research [37] results show that the reduction of iron ore powder by industrial lignin has been basically completed when the carbon–oxygen molar ratio of industrial lignin to iron ore powder is 1.2 and the calcination temperature is 900 °C. Therefore, the green pellets were placed in a roasting furnace and heated from room temperature to 900 °C, 1000 °C, and 1100 °C at a heating rate of 10 °C/min under nitrogen atmosphere and then cooled to room temperature to obtain metallized pellets.

2.2.6. The Compressive Strength of Green Pellets After Roasting

After roasting, ten pellets were selected and their compressive strength was measured by using the metallurgical raw material compressive strength detection device (maximum range of 5000 N, accuracy of 1 N) manufactured by Northeastern University, as shown in Figure 6. The average compressive strength of the 10 roasted pellets was calculated as the compressive strength index of the roasted pellets.

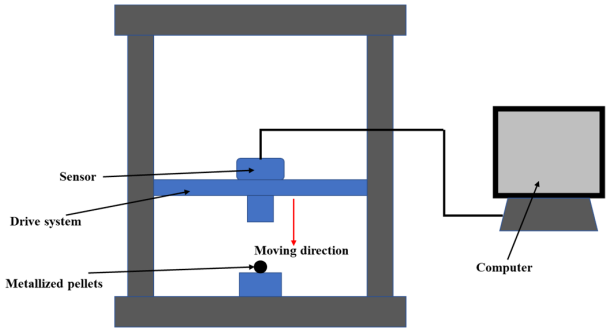

Figure 6. Compression strength tester for product pellets.

3. Results and Discussion

3.1. The Strength of Green Pellets

The appearance of the prepared green pellets is shown in Figure 7. The falling strength and compressive strength of the three green pellets are shown in Figures 8 and 9, respectively. It can be seen that the falling strength and compressive strength of industrial lignin green pellets are 6.8 times/0.5 m and 26.8 N, respectively, which are higher than those of the other two kinds of pulverized coal green pellets. According to the previous production practice experience, under laboratory conditions, when the falling strength of green pellets is greater than 3.0 times/0.5 m, and the compressive strength of green pellets is greater than 10 N, industrial production requirements can be met [38]. Therefore, only the industrial lignin green pellets meet the standard, while the carbon-containing green pellets produced by Lu'an anthracite and Shenmu bituminous coal cannot meet the production requirements. This is because industrial lignin contains a certain amount of hydroxyl groups, and hydroxyl groups are hydrophilic groups, which can form hydrogen bonds with water molecules, which is conducive to strengthening the interaction between iron ore particles in the process of pelletizing, thereby improving the falling strength and compressive strength of green pellets.

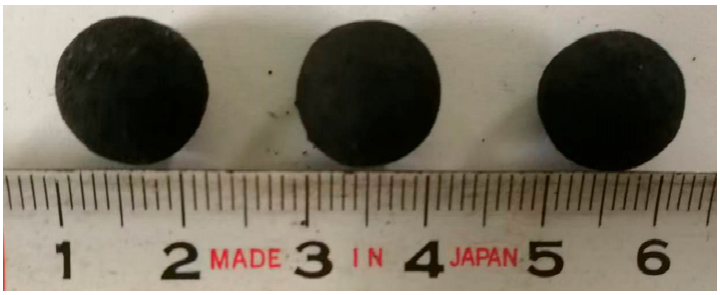

Figure 7. Appearance of green pellets.

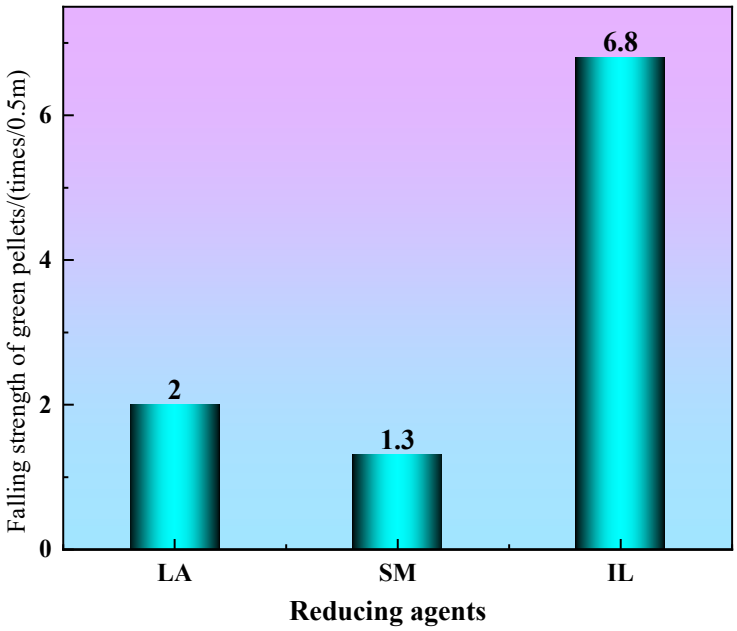

Figure 8. The falling strength of three green pellets.

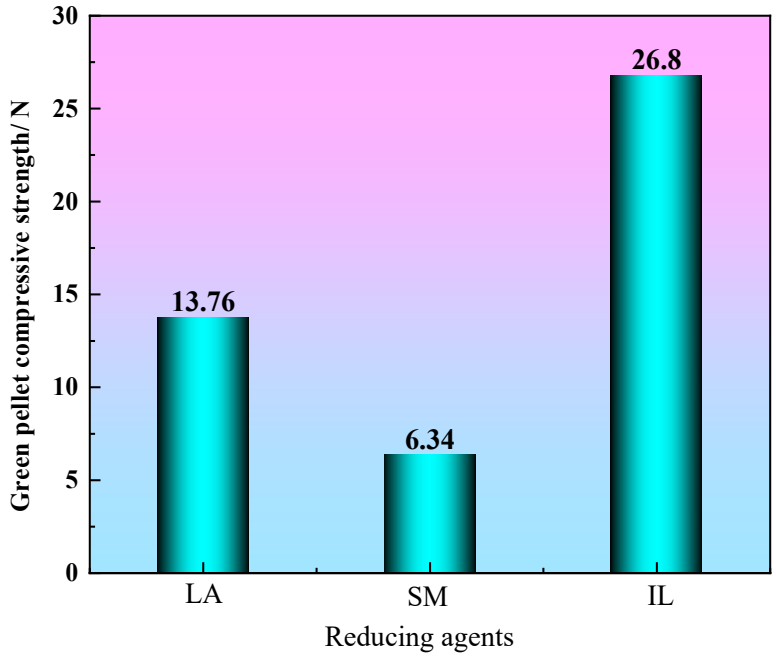

Figure 9. The compressive strength of three green pellets.

3.2. The Strength of Metallized Pellets

3.2.1. The Effect of Roasting Temperature on the Compressive Strength of Metallized Pellets

Whether the strength of metallized pellets can meet the requirements of blast furnace production is one of the key issues for the application of industrial lignin pellets in blast furnaces. The test results are shown in Figure 10. It can be seen from the figure that the compressive strength of the metallized pellets increases with the rise in the calcination temperature. When the calcination temperature increased from 900 °C to 1000 °C, the compressive strength increased from 8 N to 24.5 N, which is an increase of 206.25%. When the calcination temperature increased from 1000 °C to 1100 °C, the compressive strength increased from 24.5 N to 78.5 N, which is an increase of 220.41%. Han et al. [39] studied the effects of bamboo charcoal, charcoal, and straw fiber on the compressive strength of carbon-containing pellets. The results showed that the compressive strength of carbon-containing pellets with biomass was at a low level of 80~500 N in the low temperature zone below 1000 °C, but they could resist the pressure between the layers, and there would be no large amount of damage of pellets during multi-layer distribution. But their pellets are prepared by pressing under high pressure, so the compressive strength will be high. Zhao et al. [40] used biomass pyrolytic carbon as a reductant when preparing carbon-containing pellets (pellets with a size of **Φ** 20 mm × 15 mm) and studied the effect of calcination temperature on the compressive strength of carbon-containing pellets. The results showed that with the increase in reduction temperature, the strength of biomass carbon-containing pellets decreased first and then increased. Due to lattice distortion in the temperature range of 900–1000 °C, volume expansion was caused, and the resulting strength was the worst (the maximum was only 107 N).

The overall morphology and microstructure of industrial lignin metallized pellets after calcination and cooling at different temperatures are shown in Figures 11 and 12, respectively. Figure 11a,b shows that when the calcination temperature is 900 °C and 1000 °C, there are many cracks on the surface of the pellets after calcination. When the calcination temperature is 900 °C, the internal microstructure of the pellets (Figure 12a) shows that there are many pores and no obvious iron joined crystal; When the calcination temperature is 1000 °C, the internal microstructure of the pellets (Figure 12b) shows that the

number of pores in the pellets is significantly reduced, and a small area of iron joined crystal region begins to appear. Therefore, the compressive strength of pellets calcined at 900 °C and 1000 °C is very poor. The appearance of pores is due to the continuous precipitation of volatiles in industrial lignin during the heating process, and the appearance of iron joined crystal is due to the continuous reduction of iron ore powder by the reducing gas and the coke generated by the pyrolysis of industrial lignin. When the calcination temperature continues to rise to 1100 °C, Figure 12c shows that the iron oxides inside the pellets are basically reduced to metallic iron, and the iron joined crystal with a large area is formed. The macroscopic morphology of the pellets at this time (Figure 11c) also shows that the cracks basically disappear, so the compressive strength of the pellets is further improved.

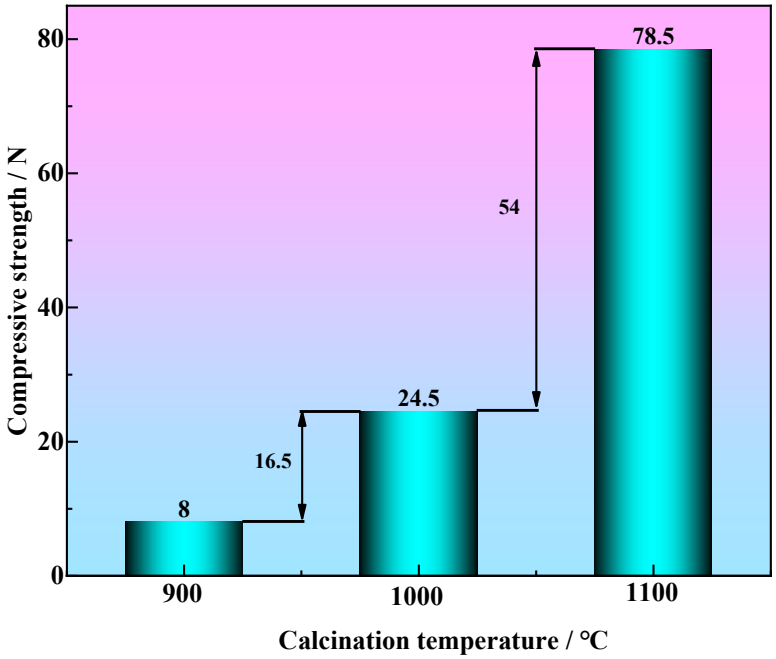

Figure 10. Compressive strength of alkali lignin pellets after roasting.

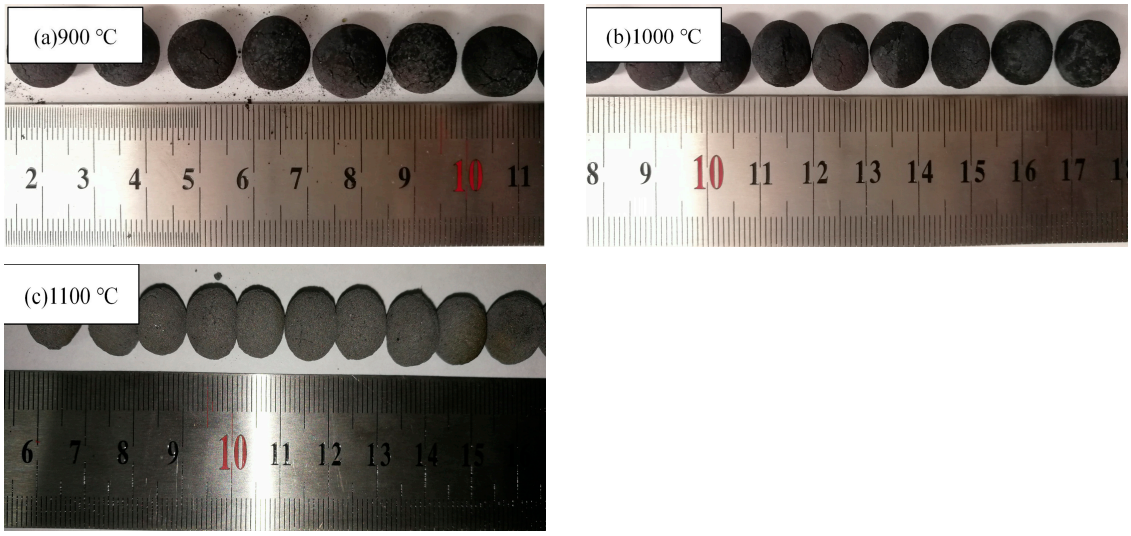

Figure 11. Macroscopic morphology of alkali lignin pellets after roasting at different temperatures: (**a**) 900 °C, (**b**) 1000 °C, and (**c**) 1100 °C.

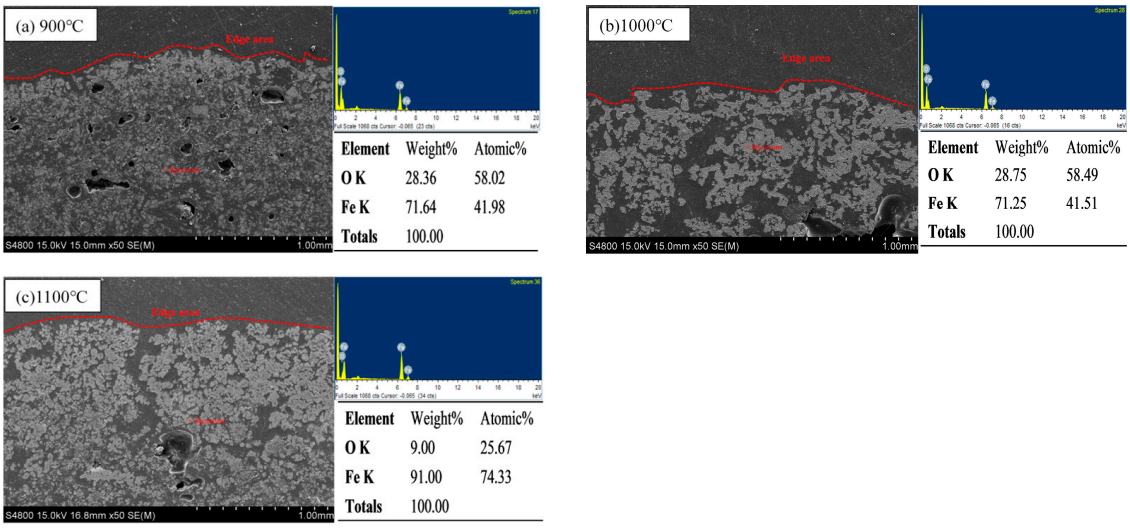

Figure 12. SEM-EDS diagram of alkali lignin pellets after roasting at different temperatures: (**a**) 900 °C, (**b**) 1000 °C, and (**c**) 1100 °C.

3.2.2. The Effect of Holding Time on the Compressive Strength of Metallized Pellets

The analysis in the previous section shows that when the roasting temperature rises to 1100 °C, metal iron has been formed in the metallized pellets, and there are also iron crystals present. However, the compressive strength of the pellets after roasting is still poor, so the method of prolonging the roasting time is adopted to promote the full growth of iron crystals. Under a roasting temperature of 1100 °C, the roasting time was set to 15, 30, 45 and 60 min, respectively, in order to study the effect of roasting time on the compressive strength of industrial lignin metallized pellets. The experimental process is shown in Figure 13.

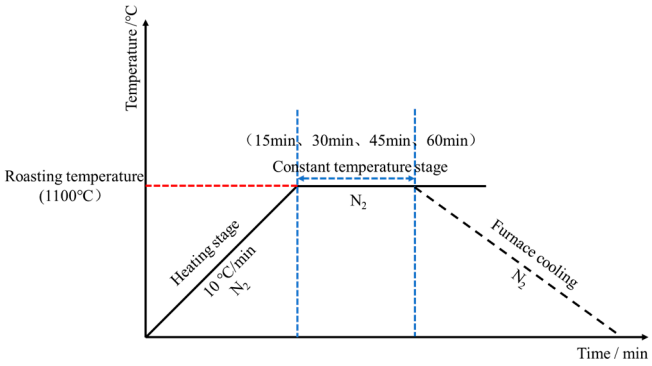

Figure 13. Roasting system of alkali lignin pellets.

The effect of calcining time on the compressive strength of industrial lignin metallized pellets is shown in Figure 14. It can be seen from the figure that after calcining, the compressive strength of the metallized pellets increases with the extension of the calcining time. When the calcination time was extended from 0 min to 15 min, the compressive strength increased from 78.5 N to 314 N, which is an increase of 300%. When the calcination time was extended from 15 min to 30 min, the compressive strength increased from 314 N to 496 N, which is an increase of 57.96%. When the calcination time was extended from 30 min to 45 min, the compressive strength increased from 496 N to 854 N, which is an increase of 72.18%. When the calcination time continues to extend to 60 min, the compressive strength increases from 854 N to 865 N, and the increase is negligible, indicating that the extension of calcining time to this period has no effect on the improvement of compressive strength.

Zhao et al.'s [40] research on the compressive strength of biomass carbon-containing pellets showed that when the reduction temperature was 1100 °C and the reduction time was 40 min, the lateral compressive strength of carbon-containing pellets was only 392 N. This is because the biochar they used was prepared by high temperature pyrolysis, which is not cohesive.

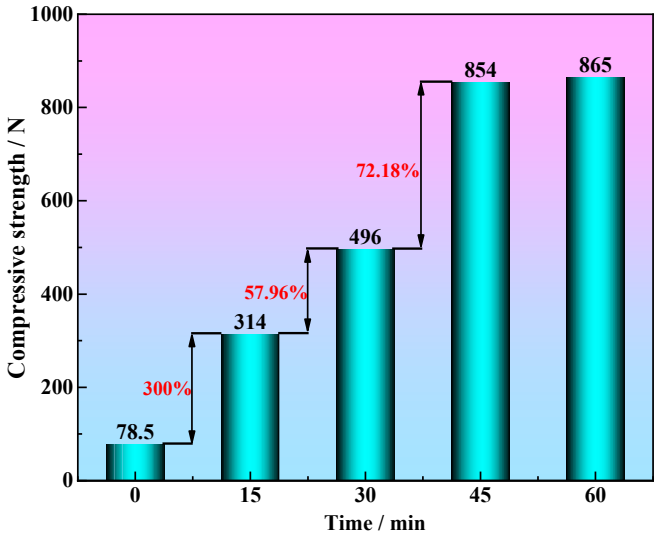

Figure 14. Effect of roasting time on compressive strength of pellets at 1100 °C.

At a calcination temperature of 1100 °C, the effects of different calcination time on the overall morphology and microstructure of industrial lignin metallized pellets are shown in Figures 15 and 16, respectively. Figure 15 shows that with the extension of calcining time, the visible metal iron phase gradually appeared on the surface of industrial lignin metallized pellets. The microstructure of industrial lignin pellets in Figure 16 also shows that with the extension of calcining time, the white areas (metal iron phase) inside the pellets are gradually connected into pieces, and the metal iron phase in the edge area of the pellets is also gradually thickened, which finally makes the compressive strength of the pellets gradually improve after calcining.

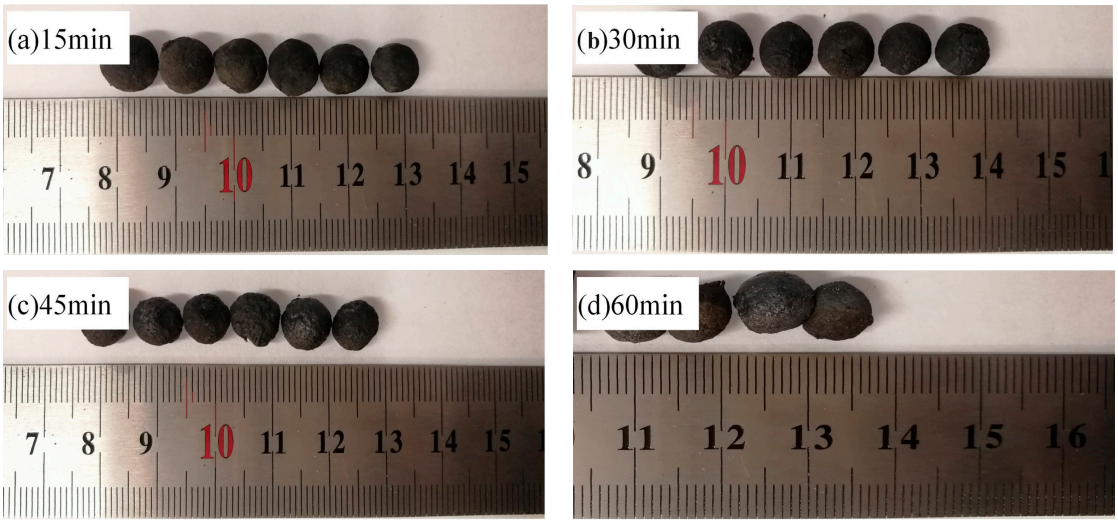

Figure 15. Morphology of alkali lignin pellets after roasting for different timings at 1100 °C: (**a**) 15 min, (**b**) 30 min, (**c**) 45 min, and (**d**) 60 min.

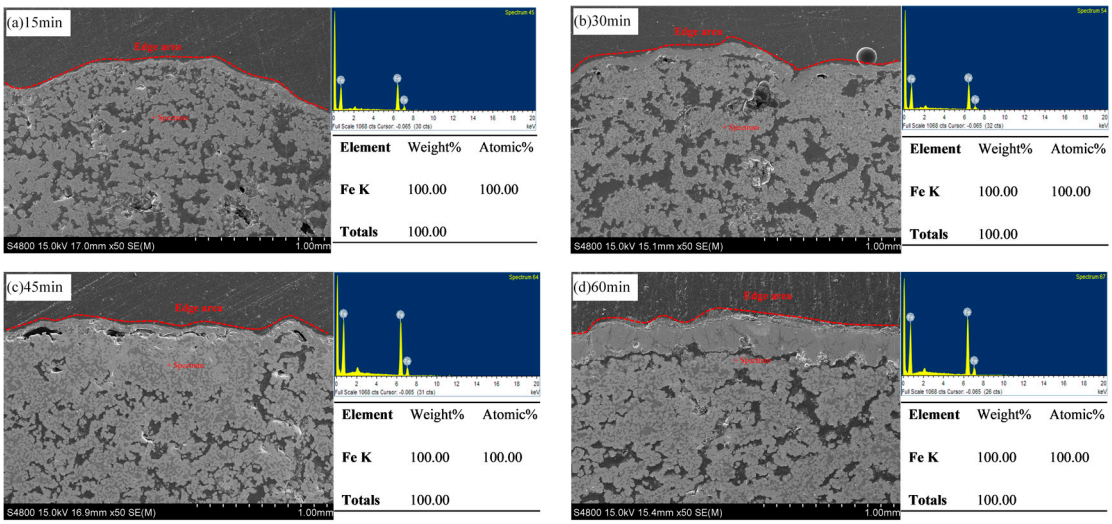

Figure 16. SEM-EDS diagram of alkali lignin pellets after roasting for different time at 1100 °C: (**a**) 15 min, (**b**) 30 min, (**c**) 45 min, and (**d**) 60 min.

The main reactions of industrial lignin pellets during calcining and reduction are as follows [41]:

$$Fe_3O_4 + C = 3FeO + CO \quad \Delta G^{\theta} = 207{,}510 - 217.62T \tag{1}$$

$$Fe_3O_4 + CO = 3FeO + CO_2 \quad \Delta G^{\theta} = 35{,}380 - 40.16T \tag{2}$$

$$FeO + CO = Fe + CO_2 \quad \Delta G^{\theta} = -13{,}160 + 17.21T \tag{3}$$

$$FeO + C = Fe + CO \quad \Delta G^{\theta} = 158{,}970 - 160.25T \tag{4}$$

Reactions (1) and (2) can occur at lower temperatures. In addition, the research of Xiang et al. [42] shows that industrial lignin produces CO at lower temperatures during pyrolysis, which can provide good kinetic conditions for the reduction of iron ore powder. With the continuous increase in calcination temperature, Reactions (3) and (4) began to proceed, and the iron oxide was continuously reduced to metal iron. At this time, the relatively dense metal iron shell was formed on the surface of industrial lignin pellets. The research shows that the shape of the spherical shell is curved evenly and symmetrically. The arched surface of the structure allows the pressure on one part of the spherical shell to be evenly transmitted to the other parts. The spherical shell structure can effectively absorb the energy generated by the external impact and skillfully 'offset' each other [43]. In addition, the formed metal iron shell blocks the spillover of CO gas, which is conducive to the reduction of iron oxides in industrial lignin pellets and is also very beneficial for the improvement of the metallization rate. The metallization rate of calcined products is an important index to measure the quality of metallized pellets, which is directly related to its subsequent application value [44]. Table 3 shows the composition analysis of the pellets after reduction at a calcination temperature of 1100 °C and a calcination time of 60 min. It can be seen that the metallization rate (R = 100% × MFe/TFe) of the pellets after reduction is 99.72%, indicating that the iron oxide inside the pellets has been completely reduced to metallic iron.

Table 3. Composition analysis of alkali lignin pellets after roasting for 60 min at 1100 °C (wt%).

	Al_2O_3	CaO	MgO	SiO_2	TFe	MFe	FeO
Metallized pellets	0.46	1.04	0.36	7.3	87.99	87.74	0.19

4. Conclusions

1. Because industrial lignin contains a certain amount of hydroxyl, the falling strength and compressive strength of industrial lignin green pellets are 6.8 times/0.5 m and 26.8 N, respectively, which can meet the requirements of industrial production.
2. When the molar ratio of industrial lignin to iron ore powder is nc/no = 1.2, the roasting temperature is increased from 900 °C to 1100 °C and the compressive strength of the metallized pellets is increased from 8 N to 78.5 N. This is mainly because the higher reduction temperature can make the pellet structure more compact, with less voids, and the metal iron crystals can be connected into sheets.
3. When the molar ratio of industrial lignin to iron ore powder is nc/no = 1.2 and the roasting time is extended from 0 min to 45 min, the compressive strength of metallized pellets increases from 78.5 N to 854 N, which is an increase of about 988%. This is mainly due to the continuous formation of a relatively dense metal iron shell on the surface of the industrial lignin pellets.

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