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Strengthening Granulating and Sintering Performance of Refractory Iron Concentrate by Pre-Pelletizing

Fengguang Han, Yongbin Yang *, Lin Wang *, Peiyao Tang, Qiang Zhong, Qian Li and Tao Jiang

School of Minerals Processing and Bioengineering, Central South University, Changsha 410083, China; 630364@baosteel.com (F.H.); 18031081861@163.com (P.T.); zhongqiang@csu.edu.cn (Q.Z.); csuliqian208018@csu.edu.cn (Q.L.); jiangtao@csu.edu.cn (T.J.)

* Correspondence: ybyangcsu@126.com (Y.Y.); wlincsu@163.com (L.W.)

Abstract: May Concentrate Iron ore, as a refractory iron concentrate, deteriorates the permeability of the sintered layer during the sintering process due to its fine particle size and poor granulating performance. Therefore, it cannot be widely used in iron ore sintering. In this study, the strengthening granulation of May Concentrate Iron ore using pre-pelletizing to expand its utilization ratio was proposed. The experiments of conventional granulation, pre-pelletizing granulation and sinter pot test were carried out. Increasing May Concentrate Iron ore (a refractory iron concentrate) proportion was detrimental to conventional granulation, reducing the quasi-particle particle size and strength. May Concentrate Iron granulating and sintering performances were improved by pre-pelletizing. The quasi-particle average size at 36% May Concentrate Iron proportion jumped to 4.92 mm of pre-pelletizing granulation from 3.22 mm of conventional granulation. Meanwhile, the permeability index rose to 0.33 from 0.11, while the falling and drying pulverization ratio fell to 7.05% and 6.11% from 22.59% and 15.88%, respectively. The consolidation mode of matrix materials was liquid phase consolidation, while that of May Concentrate Iron pellets was solid phase consolidation, forming the structure of the pellets embedded in the matrix materials. Furthermore, the partial alkalinity of the matrix materials was increased because of the separation of May Concentrate Iron, generating a large amount of acicular calcium ferrite with better consolidation strength than conventional granulation sintering.

Keywords: granulation; refractory iron concentrates; sintering; pre-pelletizing; composite agglomeration process; metal resource

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1. Introduction

Iron ore is one of the most critical metal resources in the world [1]. However, high-quality iron ore resources are being depleted, so poor-quality iron ores have become a focus of world research [2–4]. Some refractory iron concentrate, with fine particle size, high content of harmful impurities and poor granulating performance, is one of them [5,6]. Sintering is the most common method (75% of the total processing capacity) to agglomerate iron ores into a size suitable for blast furnace ironmaking. However, large-scale use of these iron ore concentrates in sintering will affect the granulation effect and deteriorate the permeability of the sintered layer [7,8]. Therefore, it has become a significant problem in iron and steel metallurgy to utilize refractory iron concentrates in the sintering process on a large scale.

Some scholars have proposed strengthening the granulation of refractory iron concentrate. Intensive mixers were proposed to replace drum mixers for strengthened granulation of fine-particle iron ore concentrates. It improves the granulation by making the added water and quicklime more uniformly dispersed [9]. Meanwhile, adding hot water (about 90 °C) promotes its dispersion and the digestion of quicklime, improving the granulation [10]. Furthermore, various binders are added to the materials to increase the cohesion between the fine particles and improve granulation [8]. However, they all have limited effects and cannot be used exclusively or in high proportion to iron ore concentrates. Some scholars propose to pretreat iron ore concentrate separately to improve its granulation performance before granulating. Wet grinding and high-pressure roller grinding are the most frequently used pretreatment methods [11,12]. A pre-wetting technique for iron ore concentrates was proposed, which involves advanced wetting and grinding of iron ore concentrates with constant total moisture. It can increase the average particle size of the granulated quasi-particles and improve the permeability of the sintered material layer. However, its pretreatment is too tiny to achieve large-scale production [11]. Meanwhile, some studies have improved the sintering process of iron concentrate by compressing it to a certain shape and strength through pre-pressing [13]. However, its effectiveness is insufficient, and the production cost is significantly higher. Furthermore, the separating granulation, MEBIOS, coating granulation, pellet sintering and other granulating methods also played a different role in promoting the granulation of different properties of iron concentrate [14–16]. However, these pre-treatment and new granulation methods were usually limited by production capacity and process cost and were not widely used in industry.

The composite agglomeration process developed by Central South University divided fine and coarse materials into pelletizing and matrix materials. After pelletizing, the average particle size of the fine materials was significantly increased to improve the granulating effect and permeability. This process had been applied to sintering vanadium titanium magnetite, dust removal ash, high silicon and low iron ore and so on [17,18]. In addition to expanding the scope of resource utilization, the composite agglomeration process can also solve the problem of the shortage of acid charge in industrial production. However, the particle size and weight of the pellets are more significant than those of conventional quasi-particles; if the traditional sintering distribution system consisting of a shuttle distributor, round roller distributor and multi-roller distributor is adopted, the pellets are easy to be broken due to excessive falling impact and extrusion of a roller. Therefore, to cope with these disadvantages, a pre-pelletizing process was proposed on its basis in this study. The pellet size is reduced from 8–15 mm to 4–6 mm. It is similar to the quasi-particles made of matrix materials, which can effectively solve the problem of segregation in the distribution system caused by significant differences in size. The smaller-weight particles are more resistant to falling impact during distribution. Furthermore, the large particle size returns can support the material bed, preventing the particles from breaking due to crushing. Therefore, pre-pelletizing is more suitable for the existing sintering distribution system and effectively reduces industrial transformation costs.

In this study, the strengthening granulation of the refractory iron concentrate using pre-pelletizing to expand its utilization ratio was investigated. The experiments of conventional granulation and pre-pelletizing granulation were carried out, and their effect on granulation and sintering indexes were studied. Furthermore, the strengthening mechanism of pre-pelletizing granulation on the performance of granulation and sintering was analyzed. This method is expected to enable the large-scale utilization of refractory iron ore concentrates to alleviate global iron ore resource shortages.

2. Materials and Methods

2.1. Materials

All raw materials in this study were provided by a local steel industry in Jiangsu province. The iron ores used in this experiment were blended ore (conventional iron ore) and May concentrate iron ore (a refractory iron concentrate). Other raw materials included dolomite, quicklime, limestone, coke breeze and bentonite. Their chemical compositions are shown in Table 1. May concentrate iron ore (MCI) is a kind of refractory iron concentrate in China, with low cost. In actual industrial production, to ensure the sintering effect, the MCI proportion should not exceed 12%. The blended ore was obtained by mixing various imported rich ore with relatively high iron grades. Among the raw materials, dolomite, quicklime and limestone were used to adjust the content of CaO, SiO_2 and MgO in the sintering products, coke breeze was used as the fuel in the sintering process, and bentonite was used as the binder for the pre-pelletizing.

Table 1. Chemical compositions of raw materials (wt.%).

Minerals	TFe	SiO_2	Al_2O_3	CaO	MgO	P	S	LOI
May concentrate	57.55	5.21	1.11	2.82	0.97	0.092	0.32	6.86
Blended ore	58.63	5.32	1.76	0.30	0.14	0.068	0.057	6.18
Dolomite	0.37	2.23	0.59	30.42	19.56	0.026	0.007	44.69
Coke breeze	1.35	7.10	/	1.35	0.20	0.039	0.75	83.07
Limestone	0.50	2.58	0.77	51.95	1.15	0.014	0.13	38.05
Lime	0.16	1.08	0.18	87.57	1.26	0.018	0.004	7.80
Bentonite	5.27	54.01	14.96	5.22	3.18	0.092	0.26	10.59

The particle size compositions of MCI and Blended ore are shown in Tables 2 and 3. The content of −0.074 mm particle size in MCI is 84.86%, which is fine and suitable for pelletizing. If MCI was directly mixed with other materials, the small particles scattered in the mixture will fill the space formed by the interdependence of large particles and block the airflow channel, which reduced the permeability of the sintered layer and affect the sintering velocity and heat transfer in the sintering process. The particle size of blended ore is relatively coarse and the content of +3 mm is 56.54%, which is suitable for direct use as sintering raw materials. The XRD patterns of the two iron ores are shown in Figure 1. The main components in the blended ore are hematite, limonite and gangue. MCI is mainly composed of magnetite, hematite, siderite and gangue.

Table 2. Particle size composition of May concentrate iron.

Minerals	Particle Size Composition (wt.%)					Specific Surface Area ($cm^2 \cdot g^{-1}$)
	+0.15 mm	0.10~0.15 mm	0.10~0.074 mm	0.074~0.045 mm	−0.045 mm	
May concentrate	0.72	5.47	8.95	13.01	71.85	1337

Table 3. Particle size composition of Blended ore.

Minerals	Particle Size Composition (wt.%)							Specific Surface Area ($cm^2 \cdot g^{-1}$)
	+8 mm	5~8 mm	3~5 mm	1~3 mm	0.5–1 mm	0.15–0.5 mm	−0.15 mm	
Blended ore	10.31	26.10	20.13	23.42	3.98	8.02	8.04	708

2.2. Experimental Method

2.2.1. Experimental Procedures

The flow chart of pre-pelletizing granulation sintering is shown in Figure 2. In conventional granulation sintering, MCI was directly mixed with other raw materials. In the pre-pelletizing granulation sintering, all MCI was mixed with an appropriate amount of bentonite (binder) and then pre-pelletized. Other raw materials were granulated according to the conventional granulation process, and two kinds of particles were mixed evenly for 0.5 min to obtain the final quasi-particles in the secondary mixing.

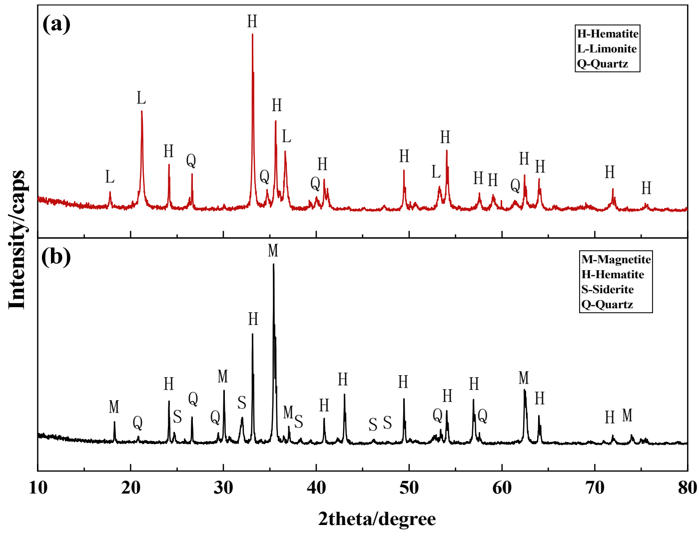

Figure 1. XRD pattern of Blended ore (**a**) and MCI (**b**).

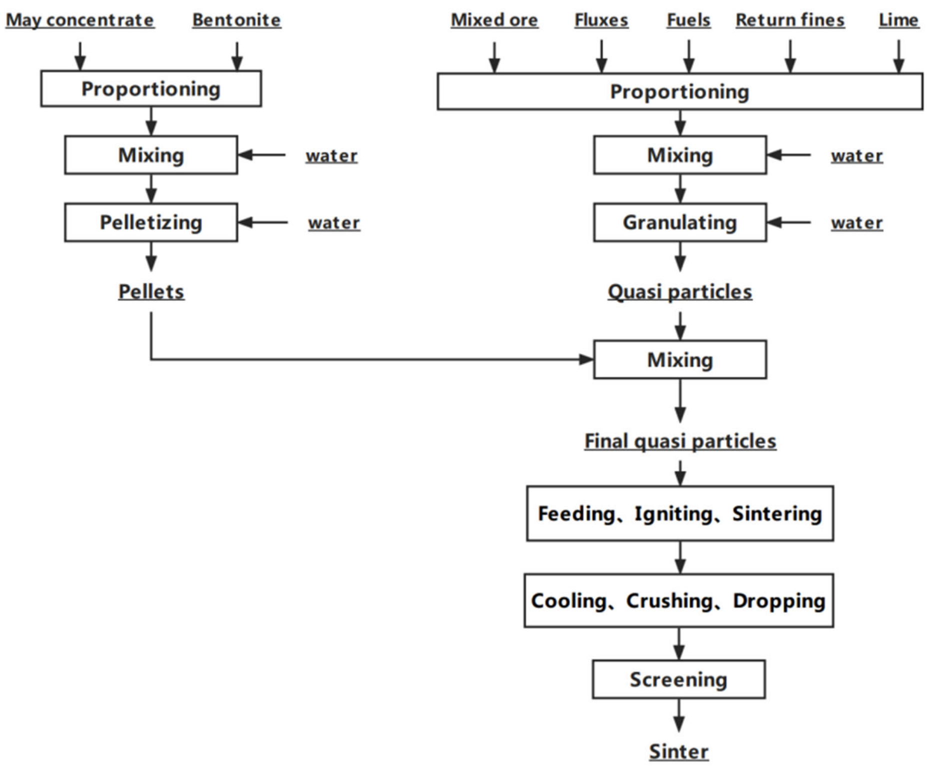

Figure 2. The process of pre-pelletizing granulation sintering.

2.2.2. Pre-Pelleting Experiment

The pre-pelletizing experiment was conducted in a φ1000 mm disc pelletizer. A certain amount of water and 2.0% bentonite by mass fraction were added and mixed into the MCI. The raw material moisture was 6.0% after adding water. During pelleting, the rotating velocity of the disc was 25 r/min, and the pelleting time was 15 min. The particle size of the pellets was 4–6 mm, and the moisture content was 8.0–8.5%.

2.2.3. Sintering Pot Experiment

The sinter pot test was carried out in a φ180 mm × 1000 mm sinter pot. Four groups of experiments were set up with the proportion of 12%, 20%, 28% and 36% of May concentrate.

The amount of coke breeze was 5.0%, the alkalinity of the sinter was 1.8, and the content of MgO was 1.4%. In the conventional granulation process, all raw materials were manually mixed at first, and then granulated in a drum mixer with φ600 mm × 1000 mm for 6 min. In the pre-pelletizing sintering process, the raw materials except MCI were manually mixed. The MCI was pre-pelletized according to the above conditions. Other raw materials were granulated in the drum mixer for 5.5 min, and then the pellets were evenly added into the mixer. We mixed them for 0.5 min to obtain the final quasi-particles. After uniform sampling, the moisture, average particle size, permeability, and strength indexes were tested. The final quasi-particles were put into the sintering pot with a material layer height of 760 mm. After ignition at 1050 °C and 8.0 kpa negative pressure for 1.5 min and heat preservation for 1 min, the suction negative pressure was adjusted to 15.5 kpa to accelerate sintering. It was the sintering endpoint when the temperature of the sintering flue gas reached the highest and started to drop. After sintering, the suction negative pressure was adjusted to 8.0 kpa to start cooling. The sinter cake was poured out and crushed by a roller crusher as the temperature of the vacuum chamber dropped below 150 °C. The crushed ore was dropped and screened to the finished sinter, and its sintering indexes were tested.

2.2.4. Evaluation Indexes

The evaluation indexes of the granulation included particle size composition, average particle size, permeability index, the falling pulverization ratio (FPR) and drying pulverization ratio (DPR) [19,20]. Among these indexes, FPR was used to simulate and evaluate the pulverization rate of the quasi-particles when they were subjected to falling impact in the process flow. 500 g of material was dropped three times at the height of 2 m (simulated the process of material from shuttle feeders to feed bin, feed bin to round roller distributor, and round roller distributor to the reflector in industrial production), and the particle sizes composition before and after falling were measured. The percentage reduction of +3 mm before and after falling was the FPR. DPR was used to simulate and evaluate the pulverization rate of the quasi-particles when they were subjected to drying impact in the sintering process flow. 100 g of quasi-particle was dried at 300 °C and 1.4 m/s airflow for 3 min, and the percentage reduction of +3 mm before and after drying was the DPR. The increase in DPR and FPR represented the decrease in quasi-particle strength. The evaluation indexes of sintering mainly included vertical sintering velocity (VSV), yield, tumbler index (TI), solid fuel consumption (SFC) and productivity. These indexes were used to evaluate the output and quality of sintering.

In the experiments, the permeability index (J) of the quasi-particles was calculated as:

$$J = Q \times (H/P)^{0.6}/\mathrm{A} \tag{1}$$

where Q (m^3/min) is air volume through the quasi-particle material layer, H (m) is the quasi-particle layer height, P (Pa) is the suction negative pressure, A (m^2) is the bottom area of the filling cup. The falling pulverization ratio (FPR) was calculated as:

$$FPR = [m_{0+3}/m_0 - m_{+3}/m] \times 100\% \tag{2}$$

where m_0 (g) is the total weight of the quasi-particle material before falling, m_{0+3} (g) is the weight of particles larger than 3 mm in the quasi-particle material before falling, m (g) is the total weight of the quasi-particle material after falling, m_{+3} (g) is the weight of particles larger than 3 mm in the quasi-particle material after falling. The drying pulverization ratio (DPR) was calculated as:

$$\mathrm{DPR} = [n_{0+3}/n_0 - n_{+3}/n] \times 100\% \tag{3}$$

where n_0 (g) is the total weight of the quasi-particle material before drying, n_{0+3} (g) is the weight of particles larger than 3 mm in the quasi-particle material before drying, n (g) is the total weight of the quasi-particle material after drying, n_{+3} (g) is the weight of particles larger than 3 mm in the quasi-particle material after drying.

3. Results and Discussion

3.1. Granulation and Sintering of Mixed Iron Ore

3.1.1. Conventional and Pre-Pelletizing Granulation

Figure 3 shows the effects of MCI proportion on the conventional (a~b) and pre-pelletizing (c~d) granulation. As shown in Figure 3a, with an increase in MCI proportion to 36% from 12%, the +3 mm quasi-particles percentage reduced to 29.47% from 63.29%, while that of 1–3 mm and −1 mm rose to 58.28% and 12.25% from 33.82% and 2.89%, respectively. And the average particle size decreased to 3.22 mm from 3.87 mm, as given in Figure 3b. This meant that increasing MCI dosage was detrimental to conventional granulation. Meanwhile, the quasi-particles permeability index dropped to 0.11 from 0.22, while the FPR and DPR climbed to 22.59% and 15.88% from 14.85% and 8.19%, respectively. This indicated that the increase in the MCI dosage reduced the quasi-particles particle size and strength, which was not conducive to subsequent sintering. Therefore, it is necessary to strictly control the MCI proportion in conventional granulation sintering. The maximum dosage in actual production is 12%.

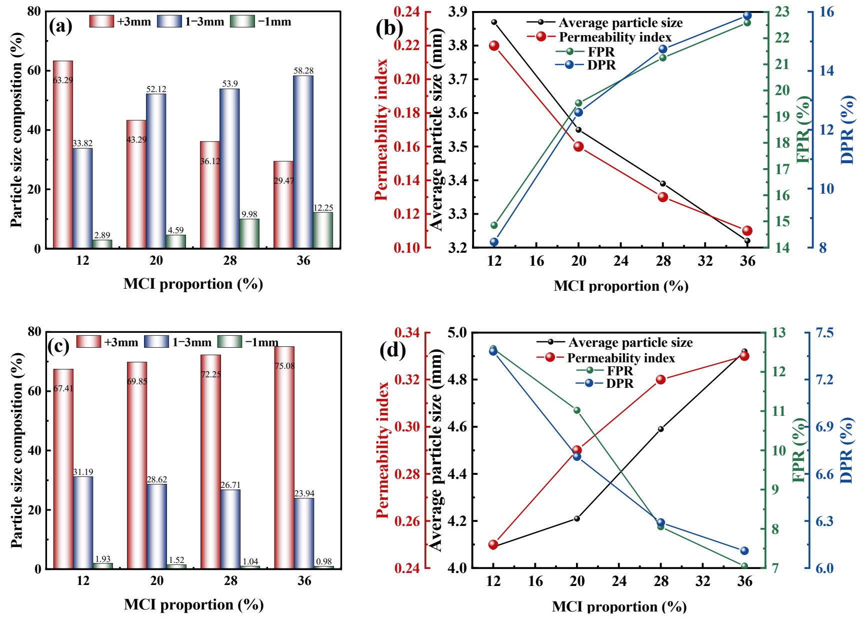

Figure 3. Effect of MCI proportion on granulation. Conventional granulation: (**a**,**b**); Pre-pelletizing granulation: (**c**,**d**).

In this study, pre-pelletizing was used to strengthen the granulation of the MCI to increase its dosage share. As shown in Figure 3c, the quasi-particle particle size by pre-pelletizing was improved, compared to the conventional granulation. With 12% MCI dosage, the +3 mm particle percentage of pre-pelletizing granulation increased to 67.41%, while that of conventional granulation was just 63.29%. And the average particle size of pre-pelletizing granulation increased to 4.09 mm from 3.87 mm of conventional granulation. Meanwhile, the permeability index, FPR and DPR of pre-pelletizing granulation at 12% MCI dosage were significantly better than those of conventional granulation, as given in Figure 3b,d. Furthermore, more noteworthy was that increasing the percentage of pre-pelletized MCI improved the particle size composition even further, while increasing the MCI percentage in conventional granulation was detrimental to the quasi-particle particle size. Thus, pre-pelletizing can be used not only to improve granulation but also as a method to effectively increase MCI dosage. With the pre-pelletized MCI dosage increased to 36%

from 12%, the average particle size jumped to 4.92 mm from 4.09 mm. Meanwhile, the permeability index rose to 0.33 from 0.25, while the FPR and DPR fell to 7.05% and 6.11% from 12.59% and 7.38%, respectively. Therefore, the granulation performance of the mixed iron ore can be improved by pre-pelletizing MCI, and the improved effectiveness was enhanced by increasing the pre-pelletized MCI dosage. Figure 4 shows the morphology of quasi-particles at 28% MCI proportion. It is easy to spot visually that the quasi-particles from pre-pelletizing granulation were larger than conventional granulation. This confirms the test results in Figure 2.

Figure 4. The morphology of quasi-particles of conventional granulation (**right**) and pre-pelletizing granulation (**left**) at 28% MCI proportion.

3.1.2. Sintering Index

The main purpose of the granulation process studied above is to mix the raw material and form quasi-particles in preparation for sintering. Therefore, a series of sinter pot experiments were carried out using quasi-particles from conventional granulation and pre-pelletizing granulation. The related sintering indexes are shown in Figure 5. The SFC using the quasi-particles from conventional granulation jumped to 78.69 Kg/t from 68.12 Kg/t with the MCI proportion rising to 36% from 12%, while the VSV, TI, yield and productivity dropped to 11.11 mm/min, 61.42%, 73.98% and 0.49 $t \cdot m^{-2} \cdot h^{-1}$ from 23.21 mm/min, 63.18%, 79.96% and 1.34 $t \cdot m^{-2} \cdot h^{-1}$, respectively. This indicated that the increase in MCI dosage was detrimental to the sintering production, and it should not be higher than 12%. The deterioration of each index was due to the deterioration of the conventional granulation by increasing MCI proportion, as shown in Figure 3a,b of Section 3.1.1.

In comparison, the sintering indexes using quasi-particles from pre-pelletizing granulation were better at all MCI proportions, especially the VSV and productivity. Furthermore, It was noteworthy that the VSV and TI were improved with increasing MCI proportion by pre-pelletizing granulation. They climbed to 24.85 mm/min and 65.06% from 23.73 mm/min and 63.59% with increasing MCI proportion to 36% from 12%. Therefore, pre-pelletizing granulation is an effective method to improve the sintering index and increase the MCI proportion.

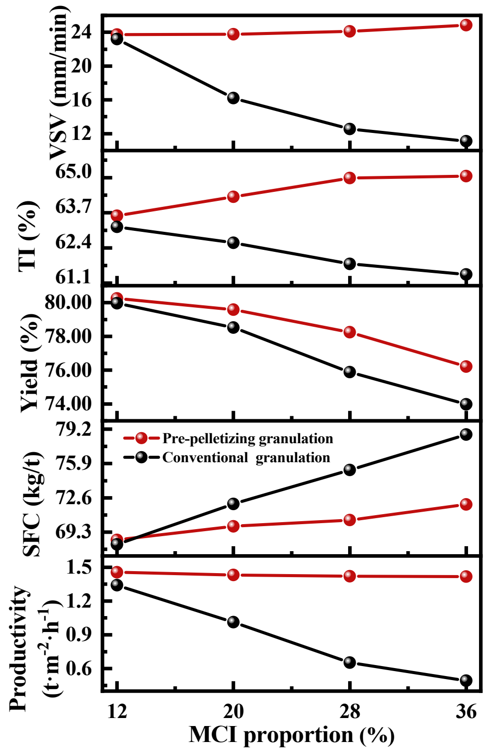

Figure 5. Sintering indexes of different MCI proportions using conventional and pre-pelletizing granulation.

3.2. Strengthening Mechanism of Pre-Pelletizing

3.2.1. Microstructure of Finished Sinter

For the sintering process, the permeability improvement of the material layer is the fundamental reason for the improvement of sintering output indexes. And it is conducive to reducing gas resistance, ensuring fuel combustion and thermal convection, which make VSV and productivity increase significantly. Furthermore, this improvement is also conducive to accelerating the diffusion of ions and the flow of the liquid phase in the sintering process, accelerating the formation of the bonding phase [21,22]. The microstructure of the sinter of conventional granulation sintering at 28% MCI proportion is shown in Figure 6a. The microstructure of the sinter was very uneven, and large pores and thin walls were formed in the sinter. It might be due to the siderite decomposition of MCI. The content of calcium ferrite in the bonding phase was low, while the contents of calcium ferruginous olivine and calcium silicate were relatively high. This was the main reason for the poor yield and strength of the finished sinter. MCI contains 22% siderite, and $FeCO_3$ in the siderite will be decomposed during sintering, as shown in Equation (4). $FeCO_3$ usually starts to decompose at about 400 °C, and completely decompose at about 560 °C, generating FeO and CO_2 [23,24]. While increasing fuel consumption, the escape of carbon dioxide also causes an explosion and serious shrinkage of the sinter bed, which produces large holes and thin walls structure inside the sinter, affecting the yield and TI. Besides, the longitudinal shrinkage of the sintered layer will lead to a decrease in the height of the sintered layer, reducing the area of convection heat transfer and the heat storage effect of the sintered layer. The shrinkage of the sintered layer in the transverse direction makes the wind speed in the edge increase significantly, too fast combustion makes the raw materials burn insufficiently and the yield decrease, while the wind speed in the middle decreases and the VSV slows down [25,26].

$$FeCO_3(s) = FeO(s) + CO_2(g)\ \Delta_r G^{\theta}{}_m = 75.34 - 0.170\,T\ (J/mol) \quad (4)$$

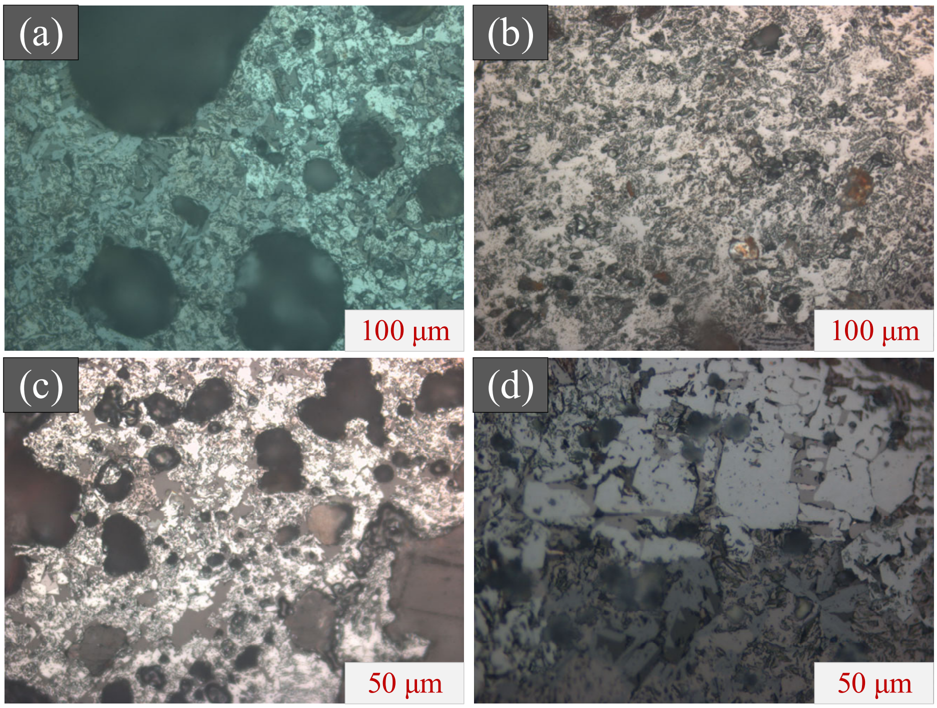

Figure 6. Microstructure of finished sinter from conventional and pre-pelletizing granulation sintering at 28% of MCI proportion. (**a**) conventional granulation sinter; (**b**) pre-pelletizing granulation sinter; (**c**) MCI pellet sinter; (**d**) conventional material and MCI pellet connection.

The microstructure of the sinter of pre-pelletizing granulation sintering at 28% MCI proportion is shown in Figure 6b–d. The sinter alkalinity should be maintained at 1.8–2.2 to ensure that more calcium ferrite is generated in the bonding phase, giving the sinter good mechanical strength. After MCI was separated and pelleted, although the total alkalinity of the final quasi-particles remained unchanged, the alkalinity of the conventional granulation material was improved. Taking the sintering of the proportion of 28% MCI as an example, the calculated alkalinity of the materials was 1.8. After the separation of MCI, the partial alkalinity of the conventional granulation materials reached about 2.5, reducing the content of calcium iron olivine and wollastonite in the bonding phase. As shown in Figure 6b, different from conventional granulation, the content of dicalcium ferrite was significantly increased, forming a large amount of acicular calcium ferrite and magnetite interwoven structure, with better consolidation strength. Furthermore, pores between grains were smaller and more evenly distributed, which was conducive to the improvement of sinter strength.

As shown in Figure 6c, the consolidation mode of pellets was the solid phase consolidation of hematite recrystallization. It can be seen that the grain size of the hematite was large, the edge was lubricated, and the crystallization was in good condition. Similar to the composite agglomeration process, as shown in Figure 6d at the junction of matrix materials and pellets, solid phase consolidation and liquid phase consolidation both coexisted, forming a structure with good strength performance of pellets embedded in matrix materials.

3.2.2. Bulk Density of Granulated Material and Sintering Shrinkage

The shrinkages of sinters were measured after sintering, the results are shown in Figure 7. The transverse and longitudinal shrinkage in conventional granulation sintering rose to 30 mm and 97 mm from 12 mm and 56 mm with MCI increasing to 36% from 12%. On the contrary, the transverse and longitudinal shrinkages in pre-pelletizing sintering were always about 50 mm and 15 mm. This meant pre-pelletizing can effectively solve the shrinkage problem caused by siderite. This was due to the denser structure of the MCI pre-pelleted pellets, compacting the material layer and inhibiting the shrinkage space of the material layer. The increase of the bulk density of the raw material layer by pre-pelletizing in Figure 8 proves this view. This effectively improves the output and quality of sintering.

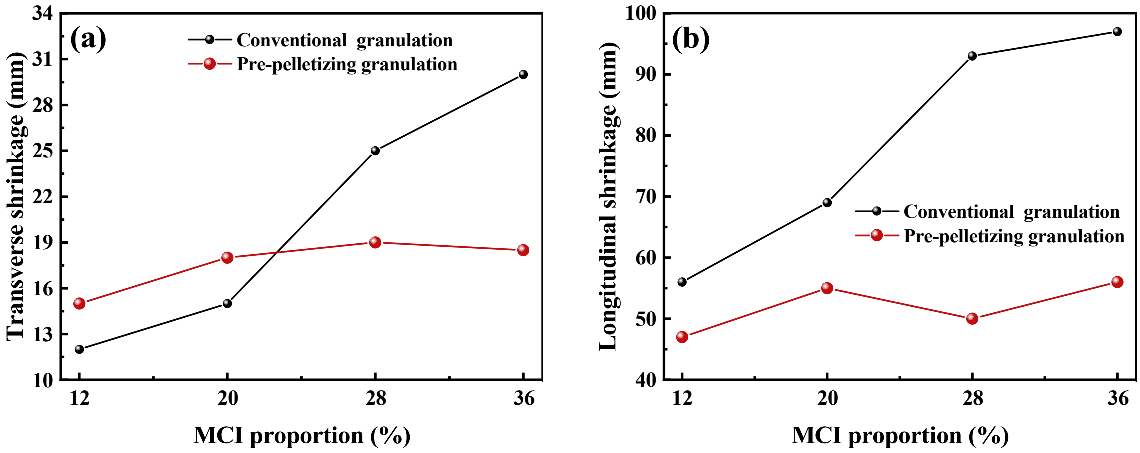

Figure 7. Transverse (**a**) and longitudinal (**b**) shrinkage of sinter.

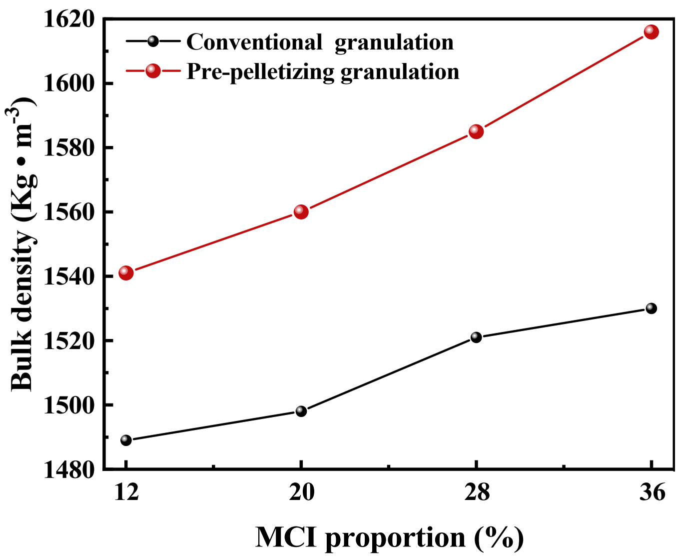

Figure 8. Quasi-particles bulk density from conventional and pre-pelletizing granulation.

4. Conclusions

Refractory iron concentrates cannot be widely used in iron ore sintering due to their fine particle size and poor granulating performance. In this study, the strengthening granulation of the refractory iron concentrate using pre-pelletizing to expand its utilization ratio was proposed. The experiments of conventional granulation, pre-pelletizing granulation and sinter pot test were carried out. The effects of May concentrate iron ore (MCI, a refractory iron concentrate) proportion on granulation and sintering by conventional and pre-pelletizing granulation were investigated. And conventional granulation sintering and pre-pelletizing granulation sintering were compared. The following conclusions were obtained:

Increasing MCI proportion was detrimental to conventional granulation, reducing the quasi-particle particle size and strength. And it further affected the sintering conditions and indicators. It is necessary to strictly control the MCI proportion in conventional granulation sintering. The maximum dosage in actual production is 12%.

The limited MCI proportion was increased from 12% up to 36% or even more by pre-pelletizing. The quasi-particle average size at 36% MCI proportion jumped to 4.92 mm of pre-pelletizing granulation from 3.22 mm of conventional granulation. Meanwhile, the permeability index rose to 0.33 from 0.11, while the FPR and DPR fell to 7.05% and 6.11% from 22.59% and 15.88%, respectively. And the sintering indexes were improved, especially the VSV and productivity.

MCI was densified by pre-pelletizing, restraining the shrinkage of the material layer caused by the decomposition of its siderite. The consolidation mode of matrix materials was liquid phase consolidation, while that of MCI pellets was solid phase consolidation, forming the structure of the pellets embedded in the matrix materials. Furthermore, the partial alkalinity of the matrix materials was increased because of the separation of MCI, generating a large amount of acicular calcium ferrite with better consolidation strength than conventional granulation sintering.

Author Contributions: Conceptualization, T.J.; methodology, Q.Z.; software, Q.Z.; validation, Q.Z., Q.L. and Y.Y.; formal analysis, L.W. and Y.Y.; investigation, F.H.; resources, Q.L.; data curation, F.H., L.W. and P.T.; writing—original draft preparation, F.H., L.W. and P.T.; writing—review and editing, L.W. and Y.Y.; visualization, Y.Y.; supervision, Q.L.; project administration, T.J.; funding acquisition, Y.Y. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The data that supports the findings of this study are available within the paper.

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