1. Introduction

High basicity iron ore sinter and acid iron ore pellet burdens have been successfully used in blast furnaces in the P. R. China for ironmaking, to achieve higher productivity and lower coke rate. However, the pellets here mainly have been made of magnetite concentrate, where lower strength of green pellets occurs due to its coarser size and poor pelletability. In order to increase the strength of green pellets, a higher dosage of bentonite is added, resulting in a lower iron grade of the product pellets. Green pellet strength can be improved by using a damp mill to pretreat magnetite concentrate before balling while the dosage of bentonite can be reduced, but higher consumption of steel ball and electricity is required. Therefore, the damp milling process has to be improved or some new processes, such as high pressure roll grinding (HPRG), need to be incorporated into the pelletizing process to increase green pellet strength at lower energy and bentonite consumption.

Since the introduction of the HPRG in 1984, as a grinding application in the cement industry, the technology is now gradually finding wider acceptance in mineral processing industry. The processing of ores and minerals downstream of the grinding plant might be improved by the application of the HPRG process for energy saving. In some particular cases the recovery and the grade of the product can be improved, and in another, capital and operating costs were reduced in the extraction of gold from gold ores with a higher gold recovery. In iron ore industry, the application of the HPRG process ranges from coarse grinding, e.g. the grinding of excess pebbles in autogenous grinding (AG) circulation loops and crushing ironstone on the spot in mining, to fine grinding to increase the Blaine values in the preparation of pellet feed. However, few pellet plants have been using the HPRG process to pretreat pellet feed except for the Steel Belt References Plant and no articles on the mechanism of the HPRG process to improve pelletizing have been issued so far.

In this paper a study of the pretreatment of magnetite concentrate by using high pressure roll grinding (HPRG) is presented, which aims to improve the pelletability, reduce bentonite dosage and increase the iron grade of pellets. The effect of the HPRG parameters on the green pellet strength was investigated. The mechanism of the pretreatment of magnetite concentrate by HPRG was revealed by determining the surface activity by means of BET nitrogen adsorption, laser size analysis, scanning electron microscopy (SEM), X-ray diffraction (XRD), micro calorimeter and pelletability index. It is demonstrated that the surface activity is improved by increases in specific surface area and the amount of micron size fractions with favored plate and flake morphologies, and higher lattice deformation and defects, resulting in more wetting heat and higher pelletability index. The effect of the HPRG is concluded as mechano-chemical activation of the magnetite concentrate. All of which is further supported by the fact that the drop number of the green pellets increased to 4.9 times by pretreating the concentrate using the HPRG compared to 3.3 times without pretreating, and bentonite reduced by 50%.

KEY WORDS: magnetite concentrate; mechano-chemical activation; pelletability; high pressure roll grinding; surface activity; green pellet strength.

2. Experimental

2.1. Raw Materials

The raw materials used include magnetite concentrate and bentonite. Their chemical compositions, size distributions and physical properties of the iron concentrates are documented in Tables 1, 2 and 3. The size distributions of
iron concentrates are coarse, and the amount of –0.075 mm only amounts to 58.55% and 76.70%, respectively, resulting in a poorer pelletability index of 0.279–0.312. In order to improve pellet productivity and quality it is essential to pretreat iron ore concentrates and improve their pelletability before balling. Pellets were made by using the blends of the two kinds of iron ore concentrates at the ratio of 1 : 1.

The physical properties of bentonite used is presented in Table 4. The content of montmorillonite in the bentonite is about 77.26%, and is fine, with 96% of –0.075 mm in size. It was activated with sodium carbonate in a pellet plant.

<p>| Table 1. Chemical compositions of the iron ore concentrates (mass%). |
|--------------------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|</p>
<table>
<thead>
<tr>
<th>Elements</th>
<th>TFe</th>
<th>FeO</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>P</th>
<th>S</th>
<th>LOI*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conc.1</td>
<td>64.19</td>
<td>20.31</td>
<td>3.73</td>
<td>0.96</td>
<td>1.18</td>
<td>1.81</td>
<td>0.023</td>
<td>0.37</td>
<td>2.25</td>
</tr>
<tr>
<td>Conc.2</td>
<td>60.52</td>
<td>19.33</td>
<td>6.92</td>
<td>1.41</td>
<td>2.01</td>
<td>1.79</td>
<td>0.046</td>
<td>0.60</td>
<td>2.12</td>
</tr>
</tbody>
</table>

* weight loss on ignition

| Table 2. Physical properties of the iron ore concentrates. |
|--------------------------|----------------|----------------|
| Items | Pelletability index | Bulk density(g/m³) | Specific density (g/m³) | moisture (%) |
| Conc.1 | 0.312 | 2.61 | 4.69 | 8.45 |
| Conc.2 | 0.279 | 2.49 | 4.61 | 7.32 |

| Table 3. Size distributions of the iron ore concentrates (mass%). |
|--------------------------|----------------|----------------|----------------|----------------|
| Size(mm) | +0.425 | -0.425 | -0.18 | -0.15 | -0.106 | -0.075 | -0.045 |
| Conc.1. | 2.50 | 0.25 | 4.90 | 10.95 | 4.70 | 12.95 | 63.75 |
| Conc.2 | 2.10 | 2.30 | 10.60 | 17.40 | 9.05 | 20.10 | 38.45 |

| Table 4. Physical properties of bentonite. |
|--------------------------|----------------|----------------|----------------|----------------|----------------|----------------|
| colloid | Blae adsorption | montmorillonite | Moisture adsorption | Swelling | Size |
| (%) | (g/100g) | (%) | (2 hrs, %) | (ml/g) | (<0.075mm, %) |
| 51.00 | 34.15 | 77.26 | 136.95 | 8.00 | 96.0 |

2.2. Experimental Procedure

2.2.1. Characterisation of Concentrate Particles

Particle characterisation included size distribution, specific surface area and morphology of magnetite concentrate particles, and was determined by using Laser Size Analyzer, BET Nitrogen Absorption method and SEM, respectively.

2.2.2. Surface Activity

Surface activity was assessed by the measurement of wetting heat of magnetite concentrate particles in water and the degree of lattice deformation and defects of the concentrate. The former was measured by utilising a micro-calorimeter, and the parameters of which include 0.1–10°C/h temperature scanning rate, 2°C/min maximum temperature ascending rate, and 1 milli J utmost instantaneous detection energy. The latter was theoretically derived by the calculation from the XRD data. The X-ray diffractor has the main parameters of 12 kW rating power, and small angle diffractor with Kratky slit.

2.2.3. Pelletability Index

The pelletability index is used to evaluate whether a sample is easily pelletized, and is usually defined as the following:

\[ K_p = \frac{W_m}{W_c} \left( W_s - W_m \right) \] ..........................(1)

Where, \( K_p \) is the pelletability index, \( W_m \) and \( W_c \) are the saturated molecular moisture (%) and capillary moisture (%) of the magnetite concentrate, respectively. \( K_p \) is the function of size distribution and surface hydrophility, on which the pelletizing behaviour depends greatly. When the value of \( K_p \) is less than 0.3, the pelletability is poorer, whilst super pelletability indicates when \( K_p \) is more than 0.6. The saturated molecular and capillary moisture were determined by using a press filtering and capillary water ascending methods, respectively.¹⁹

2.2.4. Pelletization Procedure

The moist magnetite concentrate was pretreated by the
HPRG with double rolls of 200 mm in diameter and 75 mm width to improve its pelletability. The investigated parameters of HPRG process cover roll pressure, rotation speed of rolls, pretreating times for the concentrate to pass through rolls and dosage of bentonite. The moist magnetite concentrate was pretreated by HPRG at a normal feed rate of 30 kg/min, then mixed with bentonite in a kneader-mixer at 350 rpm with a 150 rpm orbital motion for 5 min.

Pellets were made from the mix of iron ore concentrate and bentonite in a disc pelletizer of 1 m in diameter and 0.2 m rim depth, and inclined at 45° to the horizontal. A small amount of the mixed material was added to the pelletizer disc, rotating at 35 rpm, to make pellet “seeds”. The seeds were moistened with water mist to retain moisture content while adding additional material to enlarge them into pellets. The time required for pelletization was 15 min, and 5 kg of mixed material was added. Another 2 min was needed to compact the green pellets after finishing adding the mixed material and water. The diameter of finished green pellets was controlled at 12–16 mm by screening.

The drop number and compressive strength of the finished green pellets were measured, and used to evaluate the ability of the green pellets to remain intact and retain their shape during handling, respectively. The procedure for the measurement of drop number is as follows: a single freshly made green pellet is dropped repeatedly from a height of 0.5 m onto a steel plate. The number of drops required for fracture is recorded for 20 pellets and the results averaged.

3. Results and Discussion

3.1. The Effect of HPRG Parameters on the Strength of Green Pellets

The effect of roll pressure of HPRG on the green pellet strength is illustrated in Fig. 1. The strength of the green pellets sharply increases with an increase in rolls pressure. Under the conditions of pretreating the concentrate twice at 120 rpm rotation speed of the rolls, and with 1 mass% bentonite and 8 mass% moisture for balling, the highest strength of green pellets occurs at 2.33 MPa rolls pressure and the drop number and compressive strength reach 4.9 times and 25.7 Newton per pellet, respectively, in the tested pressure range. The compressive strength of pellet hardly changes when roll pressure increases from 0 to 0.96 MPa, due to little increase in micron size fractions and almost no lattice deformation. Drop number is going up abruptly because of obvious increase in micron size fractions and lattice deformation as shown in Figs. 5 and 7.

Figure 2 shows the effect of the rotation speed of rolls on green pellet strength with the concentrate pretreated twice under 2.33 MPa roll pressure, adding 1 mass% bentonite and 8 mass% moisture for balling. With an increase in rotation speed of the rolls, the strength of green pellets decreases. When the rotation speed of the rolls increased from 100 to 160 rpm, the drop number and compressive strength decreased from 5.1 times and 26.9 Newton per pellet to 2.6 times and 25.3 Newton per pellet. It may be explained that the retaining time between the high pressure rolls decreases with an increase in the rotation speed of rolls.

As is depicted in Fig. 3, the green pellet strength is greatly affected by the times for iron ore concentrate to pass through pressure rolls for pretreatment under 2.33 MPa roll pressure, 120 rpm roll speed, adding 1 mass% bentonite and 8 mass% moisture for balling. With more pretreatment times, the strength of green pellets is improved. When the concentrate was pretreated twice by passing through pressure rolls, the drop numbers of green pellets reached 4.8 times compared to 0.8 times without pretreatment. However, the more the pretreatment times, the higher capital and production cost due to more HPRG machines to be installed and longer flowsheet. According to the requirement set for the drop number of green pellet strength ≥ 3 times, 19 twice are enough for iron concentrate to be pretreated by the HPRG.

The effect of pretreatment of iron ore concentrate twice under 2.33 MPa pressure and at 120 rpm roll speed on the dosage of bentonite is presented in Fig. 4. Bentonite dosage
is reduced markedly by using HPRG to pretreat the concentrate. It also can be noticed that the drop number of green pellets is increased from 3.3 times to 14.3 times by pretreating iron ore concentrate using the HPRG compared to that without pretreating where 2 mass% bentonite was added, and similar strength of green pellets can be achieved under the conditions: 1 mass% bentonite and pretreating the concentrate twice under 2.33 MPa pressure and at 120 rpm roll speed. It is further suggested that pelletizing can be improved by using HPRG to pretreat magnetite concentrate with a lower dosage of bentonite, and the strength of green pellets can reach the requirement set for the industrial standard.

It may be concluded that not only the strength of pellets is increased, but also the dosage of bentonite is decreased by using HPRG to pretreat magnetite concentrate before pelletizing. The compressive strength of green pellets is always much higher than the required value of \( \approx 15 \) Newton per pellet, with or without the iron ore concentrate pretreated by HPRG.

3.2. Characterisation of Concentrate Particles Pretreated by HPRG

The size distributions of the magnetite concentrate pretreated twice by HPRG under 2.33 MPa pressure and at 120 rpm roll speed are shown in Figure 5, with the size fractions of \(-10\) micron, \(-40\) micron and \(-75\) micron increased from 15.0%, 55.0% and 76.7% to 26.0%, 67.0% and 88.3%, respectively, by pretreating the concentrate using HPRG under the above mentioned optimum conditions. Therefore, pretreatment of magnetite concentrate not only reduces the average size, but also increases the amount of micron size fractions, which favours the pelletising.

Figure 6 is the particle morphology of the magnetite concentrate under SEM. It can be seen that the particles of the magnetite concentrate occur as cubic shapes before pretreating by HPRG. However, the particles of the magnetite concentrate pretreated twice by HPRG under 2.33 MPa pressure and at 120 rpm roll speed occur as plate and flake shapes, as well as have visible cracks and flaws. Particles occurring in plate, flake and columnar shapes possess higher pelletability than those in cubic and spheric shapes, so it can be concluded that the pretreatment of magnetite concentrates by HPRG can improve their pelletability by modifying particle shapes, as is put forward during mechanical activation of millerite by Mulak et al. The specific surface area of the magnetite concentrate is \( 1170 \text{ cm}^2/\text{g} \) without pretreatment (Table 5), which presents poorer pelletability because the required minimum specific surface area is usually \( 1500 \text{ cm}^2/\text{g} \) for obtaining super pelletability. However, the specific surface area is raised to \( 1590 \text{ cm}^2/\text{g} \) by HPRG due to the increase in the amount of micron size fractions and modification of particle morphology, which is much higher than that without pretreating.

3.3. Surface Activity Improvement by HPRG

Green pellet strength depends considerably on the capillary force among particles inside pellets, which is affected mainly by such parameters as size distribution and surface properties of the concentrate. There is a direct relationship between surface activity and crystal structure, therefore the
measurement of XRD of magnetite concentrate was performed and the results are documented in Table 6. The half width of peaks widens and relative intensity of diffraction decreases with the pretreatment of the magnetite concentrate, which means lattice defects or lattice deformation occurred to some extent.

In order to quantitatively reveal the degree of lattice deformation, the relationship of grain size of crystallite and the degree of lattice deformation with the half width of peaks are expressed as Scherrer and Bragg equations, \( K, l \), respectively:

\[
\beta_d = K \lambda / (D \cos \theta) \quad \text{(2)}
\]

\[
\beta_s = \varepsilon \tan \theta \quad \text{(3)}
\]

Where: \( K \), shape coefficient; \( \lambda \), the wavelength of X-ray source; \( D \), size of crystal grains; \( \theta \), diffraction angle; \( \varepsilon \), the degree of lattice deformation; \( \beta_d \) and \( \beta_s \) are the half width of diffraction peaks due to the variation of grain size of crystallite and the lattice deformation. According to the principle of diffraction intensity, there is the following relationship between \( \beta_d \) and \( \beta_s \):

\[
\beta = \beta_d + \beta_s \quad \text{.........................(4)}
\]

where, \( \beta \) is the total half width of diffraction peaks. The following formula can be derived by combining the above Eqs. (2)-(4):

\[
\beta \cos \theta = \varepsilon \sin \theta + K \lambda / D \quad \text{.........................(5)}
\]

The values of \( \sin \theta \) and \( \beta \cos \theta \) are replaced by \( x \) and \( y \), re-
respectively, and $x$ is plotted against $y$ in Fig. 7. The variation of lattice deformation degree due to mechano-chemical activation can be derived from the slope of the regressed line in Fig. 7 as follows:

$$\Delta \varepsilon = \varepsilon_a - \varepsilon_b = 0.1192 \quad \text{.........(6)}$$

The degree of lattice deformation of the magnetite particles increased by 11.92% by using HPRG compared to that without pretreatment of the iron ore concentrate.

That the surface is activated by the mechanical action of the pressure rolls can be further proved by the wetting heat and pelletability index (Table 5). It can be observed that wetting heat increases from 0.28 to 0.67 $J/g$ by pretreating magnetite concentrate using HPRG. The higher surface free energy of the particles is thought to be due to the lattice deformation and defects by HPRG, with more wetting heat released when these particles come in contact with water molecules.

It can also be noticed from Table 5 that the pelletability index is increased from poor (0.30) to super (0.64) by pretreating the magnetite concentrate. There is agreement among results for the degree of lattice deformation, wetting heat, green pellet strength and strength of the green pellets.

The improved surface activity of concentrate particles is ascribed to the mechano-chemical activation of the HPRG, in which mechanical energy is transferred into chemical energy and stored in the magnetite crystal grains, resulting in a higher degree of lattice deformation and defects.13,14)

4. Conclusions

(1) The operation parameters of HPRG for pretreating the magnetite concentrate are optimised as follows: 2.33 MPa pressure, 120 rpm rotation speed of pressure rolls and pretreating the concentrate twice by passing it through the rolls. The drop number of the green pellets increased from 3.3 times to 4.9 times by HPRG, but the dosage of bentonite is reduced from 2 to 1%.

(2) Pelletability is improved by the increases in micron size fractions and specific surface area, modification of particle morphology and enhanced surface activity. Surface activity enhancement of magnetite concentrate is ascribed to mechano-chemical activation of HPRG, in which mechanical energy is transferred into chemical energy and stored in the magnetite crystal grains, resulting in a higher degree of lattice deformation and defects.

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Nomenclature

\[ K_p: \quad \text{The pelletability index} \]
\[ W_m: \quad \text{The saturated molecular moisture} \ (%\) \]
\[ W_c: \quad \text{The saturated capillary moisture} \ (%\) \]
\[ K: \quad \text{Shape coefficient} \]
\[ \lambda: \quad \text{The wavelength of X-ray source} \]
\[ D: \quad \text{Size of crystal grains} \]
\[ \theta: \quad \text{Diffraction angle} \]
\[ \varepsilon: \quad \text{The degree of lattice deformation} \]
\[ \beta_d: \quad \text{Half width of diffraction peaks due to the variations of grain size of crystallite} \]
\[ \beta_l: \quad \text{Half width of diffraction peaks due to the lattice deformation} \]
\[ \beta_0: \quad \text{The total half width of diffraction peaks} \]
\[ I_r: \quad \text{Relative intensity of diffraction} \]

REFERENCES