Note

Effect of Test Condition on Decrepitation Index and Test Repeatability for Lump Iron Ore

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1. Introduction

Lump iron ore used as blast furnace feedstock is natural products and its properties substantially vary. As one of the properties specific to lump iron ore, the decrepitation index (hereinafter referred to as “DI”) is controlled in accordance with a standardized test method specified in ISO 8371.¹⁾ However, it has been pointed out through an international joint experiment project under ISO TC102/SC3/WG7 (decrepitation) that the test method has several weak points relating to test repeatability or reproducibility.²⁾ We then conducted a study on the effect of test method and test condition on measurement value and test repeatability.

2. Standardized Test Method

As a standardized test method, ISO 8371 requires to dry lump iron ore already screened to a grain size range of $-25\text{ mm} +20\text{ mm}$, and heat 500 g of them in a furnace temperature of 700°C rapidly in 30 min. Cracking induced during the rapid heating is defined as “decrepitation”. These decrepitated test portions are further screened to different grain size ranges to determine the DI based on the mass ratio. The ISO standard requires to use a 6.3 mm mesh screen to evaluate the test portions obtained from 10 repeated tests.

This ISO standard provides indefinite test condition. There is no specific requirement for the type of furnaces or the geometry of test portion holders. Japanese laboratories mainly use two types of test equipment: a combination of a muffle furnace and a box type test portion holder shown in Fig. 1 and a combination of a reduction furnace and a cylindrical type test portion holder shown in Fig. 2. They conduct the testing according to their original detailed test condition.

This report uses the relative standard deviation (hereinafter referred to as “RSD”) as a measure to evaluate test repeatability. As shown in Eq. (1), the RSD indicates the standard deviation of measurements divided by their average value expressed as a percent. In other words, the RSD represents relative variation of measurements.

$$\text{RSD}(\%) = \frac{\text{SD}}{\bar{X}} \times 100 \quad \dots\dots\dots(1)$$

Fig. 1. Muffle furnace and portion holder (box type).

Fig. 2. Reduction furnace and portion holder (cylindrical).

Table 1. Testing condition.

Case	Furnace	Test portion holder	Test portion mass	Other condition
1	Muffle furnace	Box type	500g	—
2	Muffle furnace (Small type)	Box type	500g	—
3	Reduction test furnace	Cylindrical type	500g	—
4	Muffle furnace	Box type	500g	Fixed Sample
5	Muffle furnace	Box type	1,000g	—

3. Experimental Method

Tests were conducted in accordance with ISO 8371 as the basic test condition. Three types of heating furnaces were used: muffle furnace (capacity 21.0L-15kVA), small-type muffle furnace (7.5L-2kVA) and reduction furnace (27.6L-20kVA). The furnace temperature was measured on the furnace wall shown in Fig. 1 and Fig. 2. Two types of test portion holders were used: box type holder (W 150×L 100×H 80×t 2 mm, weight 1.5 kg) and cylindrical type holder (φ96×H 110×t 1 mm, weight 0.6 kg). The both holders were made of SUS304 material (stainless steel). These heating furnaces and test portion holders were used in two different combinations. One is a combination of a muffle furnace and a box holder, and the other is a combination of a reduction furnace and a cylindrical holder. Lump iron ore produced in Australia was prepared as the test sample. For the use of 500 g sample as specified in the ISO standard, the test portions were put in the box holder in a single layer or in the cylindrical holder superimposed to form two layers. The temperature of the test portion was measured in the center of the test portion holder or layers. To determine the DI, the mass ratio of the test portions screened with a mesh size of 4.76 mm was used to calculate the DI (hereinafter referred to as “DI_{4.76}”). The average DI for each test condition was used for comparison.

There are mainly two possible factors affecting the DI and RSD. One is heating factors such as furnace type, heating temperature, heating time and test portion holder. The other is test portion factors such as mass. To determine the effect of these factors, the following comparative experiments were conducted. The test conditions used in individual cases are shown in Table 1.

Fig. 3. Effect of furnace type on heat pattern.

Fig. 4. Effect of furnace type and portion holder on DI and RSD.

Fig. 5. Effect of portion heating time on DI.

4. Experimental Result

4.1. Heating Furnace and Test Portion Holder

The heat pattern of test portion was identified for different combinations of heating furnace and test portion holder, and the effect of these different test equipments on the DI and RSD was examined. **Figure 3** shows the heat pattern of test portions for Cases 1 to 3. The test equipment combination of a muffle furnace and a box holder (Case 1) shows the fastest increase in test portion temperature while the combination of a reduction furnace and a cylindrical holder (Case 3) shows the slowest increase. The combination of a small muffle furnace and a box holder (Case 2) is found to have a result between Cases 1 and 3. **Figure 4** indicates the DI and RSD for the individual Cases. According to the figure, these values change by the test equipment combination. With considering the results of the heat pattern, a test equipment combination with the fastest increase in test portion temperature shows higher DI and better RSD. These results imply that the heating rate affects the DI and RSD.

4.2. Test Portion Heating Time

Through comparative experiments with various additional heating times to the standard time of 30 min, the effect of heating time on the DI and RSD was examined. **Figure 5** shows the effect of test portion heating time between Cases 1 and 3. Decrepitation increase for 30 min from the start of heating, which corresponds to the heating time specified by the ISO standard. After that, decrepitation does not increase so much. For Case 1, the test portions reach the specified heating temperature of 700°C in 15 min as shown in Fig. 3 and decrepitation became 70 to 80% of the value after 30 min. For the next 15 min, the temperature is maintained at 700°C and the rest 20 to 30% decrepitated. A similar tendency can be found in Case 3. These results imply that much of decrepitation occurs during the initial temperature increase period and decrepitation is unlikely to increase develop after the 30-min heating. Therefore, the heating time of 30 min stated in ISO standard is considered to be appropriate.

4.3. Test Portion Heating Temperature

Using the test equipment combination of the reduction furnace and the cylindrical holder in Case 3, the test portions were heated to different furnace temperature levels. **Figure 6** shows the test portion heat pattern for various furnace temperature levels under the test condition of Case 3. **Figure 7** shows the DI and RSD for various furnace temperature levels. Changes of furnace temperature resulted in different heat patterns and different heating rates. However, there was almost no difference in the DI value between

Fig. 6. Effect of portion heating temperature on heat pattern.

Fig. 7. Effect of portion heating temperature on DI and RSD.

700°C, which is the standard furnace temperature, and 800°C, which involves a faster heating. For the furnace temperature of 600°C with slower heating, a lower DI value was obtained. It should be noted that heating to 800°C brought the worst RSD among all.

From these results, it is believed that raising the furnace temperature to 800°C compared with it to 700°C will hardly affect the DI. In turn, the furnace temperature below 700°C must affect the DI value. Therefore, the ISO standard furnace temperature of 700°C is considered to be appropriate.

4.4. Box Holder Size (Fixed Sample)

Vacant space inside the test portion holder depends on the size of the holder. The effect of the presence of such space on the DI and RSD was examined. **Figure 8** shows the results of the comparative experiment to determine the effect of holder size. The figure indicates the resultant DI and RSD for two hypothetical test conditions: Case 1 where there is space inside the holder and Case 4 (**Fig. 9**) where there is no space inside the holder with test portion pressed down with the wire netting. Compared to Case 1, Case 4 shows lower DI values by about 30% and worse RSD values for the all iron ore brands. Since there is no difference in test portion heat pattern between these Cases, the lower DI values and the worse RSD values for Case 4 can be attributed by the holder size and/or holder internal space. When the test portion was put in the heating furnace and heated to around 300 to 500°C, decrepitation sound was heard as if the test portions cracked. At the same time, metallic sound was also heard. It can be guessed that the

Fig. 8. Effect of fixed sample on DI and test repeatability.

Fig. 10. Effect of portion mass on heat pattern.

Fig. 11. Effect of portion mass on DI and RSD.

metallic sound was originated by the collision of the test portion with the internal walls of the holder. When the test portion was pressed down with the wire netting, this metallic sound was not heard and a lower DI value was obtained. From these facts, decrepitation probably occurred when the test portions bumped against the internal walls of the holder. Therefore, it is believed that the holder size and internal space affect the movement and heat transfer of the test portion, and thereby they affect the DI and RSD.

4.5. Test Portion Mass

Using the test equipment combination of a muffle furnace and a box holder, the test portions of different weights were heated. Then, the heat pattern of these test portions was determined and the effect of test portion mass on the DI and RSD was examined. **Figure 10** shows the heat pattern of the test portions of different weights (Cases 1 and 5). For 1 000 g test portions (Case 5), which is double to the ISO standard mass, the test portion was found eventually heated at a slower heating rate. As shown in **Fig. 11**, Case 5 gave a lower DI value and a worse RSD value than Case 1. The effect of heating rate on the DI and RSD as well as heat pattern of test portions was also identified in Sec. 4.1. Thus, the heating rate of test portion is one of the important test conditions. The experiments above also show that the heating rate changes by furnace specifications, test portion holder and test portion mass. It is believed that the lower DI and worse RSD were caused by the slower heating rate due to the increased mass of the test portions. Therefore, the mass of test portions is also an important factor.

5. Discussion

Figure 12 shows the DI-SD relationship obtained from the experimental tests above using a same sample under various test conditions. According to the figure, the SD does not vary greatly on the whole although it gets slightly larger as the DI gets higher. The RSD decreases (*i.e.*, improves) as the DI increases. Since the RSD is determined using the equation with the average DI as a denominator and the SD as a numerator, a combination of smaller SD variation and higher DI will lower (*i.e.*, improve) the RSD. Therefore, selecting a heating condition for higher DI will limit the RSD or variation to a low level.

6. Conclusion

We conducted a study on the effect of test condition on

Fig. 12. Effect of DI sensitivity on Standard Deviation and Relative Standard Deviation (the iron ore is constant).

test repeatability for the decrepitation test of lump iron ore. From the study, we obtained the following conclusion:

- (1) The DI and the RSD change according to the heating rate of test portion. The factors affecting the heating rate include furnace type, test portion holder and test portion mass.
- (2) The decrepitation progresses by the heating rate faster.
- (3) The major part of the decrepitation occurs during the temperature increase period. Only little decrepitation is observed during the continual temperature retention phase after the temperature of test portion reaches 700°C.
- (4) Comparing with the ISO standard furnace temperature of 700°C, decrepitation decreased for 600°C while 800°C involves almost same decrepitation.
- (5) The DI depends on the size of the test portion holder and the presence of the space in the holder. This is presumed that the test portions move and collide with the inner walls of the holder while they are heated, which causes decrepitation.
- (6) The RSD has been improved with the higher DI.
- (7) Therefore it is expected that selecting appropriate heating conditions including heating rate will stabilize the tests and achieve adequate test repeatability.

REFERENCES

- 1) International Organization for Standardization: ISO 8371, (2007).
- 2) ISO/TC102/SC3 Meeting Document, (3N1024E, Quebec 2007) (Private Document).