Original Article

Reduction disintegration mechanism of cold briquettes from blast furnace dust and sludge

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\textbf{A B S T R A C T}

It is important to understand the reduction disintegration mechanism in ferriferous burden that is used in blast furnaces. The behavior of this burden in the granular zone of this metallurgical reactor is important for smooth operation. The objective of this work was to prepare cold self-reducing briquettes using blast furnace dust and sludge and binders and compare the reduction disintegration index (RDI) of these agglomerates with conventional ferriferous burdens such as pellets, sinter and iron ore. In the present work, 25 different mixtures were prepared to produce briquettes in two geometries: pillow and cylindrical. The RDI value was determined for the briquettes that passed the tumbling test.

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1. \textbf{Introduction}

Blast furnaces require ferriferous raw materials with adequate reducibility, high cold strength, low reduction disintegration index (RDI), small variations in chemical composition and appropriate particle size.

In the upper regions of the blast furnace, physical degradation in the ferriferous burden occurs between 300 °C and 700 °C because of the reduction of hematite to porous magnetite. During this process, volume expansion and stress relief occur because of the formation and propagation of cracks \cite{1–3}. This expansion is associated with the crystallographic orientation between these minerals and porosity formation \cite{4}, although the crystal lattice contracts during this transformation \cite{5,6}.

Shrinkage occurs during the reduction from hematite (trigonal–hexagonal) to magnetite (isometric–hexoctahedral), with a value of 1.93%.

Self-reducing briquettes are agglomerates that contain mainly iron oxide and carbonaceous material and that require energy input for the reduction reactions to occur.

The Boudouard reaction has a strong effect on the overall carbothermic reduction of iron oxide. The overall rate of the self-reduction reaction is observed to increase with increasing carbon content and surface area and in the presence of catalysts of Boudouard reaction \cite{7–9}. 

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\end{itemize}
The cold agglomeration process has been used in some steel plants with up to 5% of ferriferous burden, as a source of iron and carbon for the blast furnace. However, the disadvantage is the disintegration in the granular zone in this reactor. This is due to the destruction of the binder phase during the temperature increase [10].

Use of the appropriate binder is important to provide cohesion between the particles of the briquette and thereby increase the mechanical strength. Organic and inorganic binders have been used by many researchers in an attempt to manufacture briquettes with an adequate RDI value for use in blast furnaces.

Organic binders decompose at high temperatures (>400 °C) and degrade the briquette, reducing the viscoelasticity observed at room temperature [11–13].

Rheology phenomena may occur during the briquetting process depending on the force applied to the briquette. Granular materials interact by contact forces in this process and resist shearing stress to a certain level, maintaining the static configuration of the particles [14,15].

This study proposes to use blast furnace dust and sludge, and organic and inorganic binders, to produce cold briquettes with appropriate RDIs for use in blast furnaces. Two briquetting processes were used: roller-press and piston briquetting.

### 2. Methodology

The sampling and sample preparation, manufacture of the briquettes and tumbling tests were performed at Blackballs Technologies GmbH in Baesweiler, Germany. The RDI tests were performed in a laboratory at a steel company.

The following steps were performed: sample preparation, manufacture of mixtures and briquettes, curing, tumbling and RDI tests.

Water (W), binder BBTL, magnesium oxide (M0), refractory cement (RC) and one organic and inorganic binder called binder BBTX were used for the preparation of 25 mixtures on a dry basis; for details, see Table 1. Each mixture was comminuted in a ball mill (0.35 m³; 4 h).

Normalization was used in the naming of the briquettes: the letter M indicates a mixture of substances; the letter T indicates pillow briquettes and the letter C indicates cylindrical briquettes; the number inside the parentheses indicates the force/pressure applied. Chemical analyses were performed on the dust, sludge and mixture M05, with the results presented in Table 2.

The major crystalline phases of the blast furnace dust and sludge were hematite, magnetite, graphite, wollastonite and calcite.

Two types of briquetting process were used: roller and piston briquetting. Then, pillow and cylindrical briquettes were prepared using these processes, as shown in Figs. 1 and 2.

The following mixtures were used in the roller briquetting: M01, M02, M03, M04, M05, M06, M07, M09, M10, M11, M12, M13, M14, M15, M16, M17, M18, M22, M23, M24 and M25, totaling 21 mixtures. A BBT-BP20 machine was used in these experiments. The total volume of a briquette was 3.4 cm³, the roller velocity was 1.7 rpm and the screw feeder rotational velocity depended on the force applied to the briquettes. The strength ranged from 10 kN to 100 kN.

The following mixtures were used in the piston briquetting: M01, M02, M03, M04, M05, M06, M07, M12, M13,

### Table 1 – Mixtures prepared using the briquetting process.

<table>
<thead>
<tr>
<th>Mixture</th>
<th>Dust (%)</th>
<th>Sludge (%)</th>
<th>Binders (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>M01</td>
<td>80</td>
<td>–</td>
<td>BBTX: 20</td>
</tr>
<tr>
<td>M02</td>
<td>70</td>
<td>–</td>
<td>BBTX: 30</td>
</tr>
<tr>
<td>M03</td>
<td>60</td>
<td>–</td>
<td>BBTX: 40</td>
</tr>
<tr>
<td>M04</td>
<td>–</td>
<td>80</td>
<td>BBTX: 20</td>
</tr>
<tr>
<td>M05</td>
<td>–</td>
<td>70</td>
<td>BBTX: 30</td>
</tr>
<tr>
<td>M06</td>
<td>–</td>
<td>60</td>
<td>BBTX: 40</td>
</tr>
<tr>
<td>M07</td>
<td>–</td>
<td>90</td>
<td>BBTX: 10</td>
</tr>
<tr>
<td>M08</td>
<td>–</td>
<td>83 (wet basis)</td>
<td>BBTL: 17</td>
</tr>
<tr>
<td>M09</td>
<td>80 (wet basis)</td>
<td>–</td>
<td>BBTX: 20</td>
</tr>
<tr>
<td>M10</td>
<td>80</td>
<td>–</td>
<td>BBTX: 20</td>
</tr>
<tr>
<td>M11</td>
<td>76</td>
<td>–</td>
<td>BBTX: 24</td>
</tr>
<tr>
<td>M12</td>
<td>35</td>
<td>35</td>
<td>BBTX: 30</td>
</tr>
<tr>
<td>M13</td>
<td>40</td>
<td>40</td>
<td>BBTX: 20</td>
</tr>
<tr>
<td>M14</td>
<td>45</td>
<td>45</td>
<td>BBTX: 10</td>
</tr>
<tr>
<td>M15</td>
<td>60</td>
<td>30</td>
<td>BBTX: 10</td>
</tr>
<tr>
<td>M16</td>
<td>60</td>
<td>20</td>
<td>BBTX: 20</td>
</tr>
<tr>
<td>M17</td>
<td>–</td>
<td>80</td>
<td>MO: 20</td>
</tr>
<tr>
<td>M18</td>
<td>–</td>
<td>76</td>
<td>MO: 24</td>
</tr>
<tr>
<td>M19</td>
<td>–</td>
<td>80</td>
<td>BBTL: 20</td>
</tr>
<tr>
<td>M20</td>
<td>–</td>
<td>76</td>
<td>BBTL: 24</td>
</tr>
<tr>
<td>M21</td>
<td>–</td>
<td>71</td>
<td>RC: 24; W: 5</td>
</tr>
<tr>
<td>M22</td>
<td>–</td>
<td>70</td>
<td>BBTL: 30</td>
</tr>
<tr>
<td>M23</td>
<td>–</td>
<td>66</td>
<td>BBTL: 34</td>
</tr>
<tr>
<td>M24</td>
<td>33</td>
<td>33</td>
<td>BBTL: 34</td>
</tr>
<tr>
<td>M25</td>
<td>49</td>
<td>19</td>
<td>RC: 19; W: 13</td>
</tr>
</tbody>
</table>

### Table 2 – Chemical analysis of dust, sludge and mixture M05.

<table>
<thead>
<tr>
<th>Elements (%)</th>
<th>Dust</th>
<th>Sludge</th>
<th>Mixture M05</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>52.58</td>
<td>21.59</td>
<td>41.75</td>
</tr>
<tr>
<td>K</td>
<td>0.13</td>
<td>0.19</td>
<td>0.30</td>
</tr>
<tr>
<td>Na</td>
<td>0.10</td>
<td>0.22</td>
<td>0.17</td>
</tr>
<tr>
<td>Zn</td>
<td>0.11</td>
<td>3.05</td>
<td>0.77</td>
</tr>
<tr>
<td>P</td>
<td>0.07</td>
<td>0.12</td>
<td>0.09</td>
</tr>
<tr>
<td>Mg</td>
<td>0.20</td>
<td>0.53</td>
<td>0.30</td>
</tr>
<tr>
<td>Ca</td>
<td>0.77</td>
<td>1.3</td>
<td>1.86</td>
</tr>
<tr>
<td>Mn</td>
<td>0.46</td>
<td>0.09</td>
<td>0.43</td>
</tr>
<tr>
<td>Al</td>
<td>0.67</td>
<td>1.21</td>
<td>1.24</td>
</tr>
<tr>
<td>C</td>
<td>32.53</td>
<td>49.56</td>
<td>19.65</td>
</tr>
<tr>
<td>S</td>
<td>0.31</td>
<td>1.59</td>
<td>0.46</td>
</tr>
</tbody>
</table>
M14, M15, M16, M21 and M22, totaling 14 mixtures. One cylindrical matrix, piston and a hydraulic press (20 t capacity) was used to prepare the cylindrical briquettes. Each mixture was placed inside the matrix with a mass of approximately 35 g and subjected to seven different pressures (10, 20, 50, 100, 150, 200, and 300 MPa). Three cylindrical briquettes were produced with each mixture for the tumbling tests.

Non-standard tumbling tests were performed on all the manufactured cylindrical and pillow briquettes samples. For each mixture and applied force, a tumbling test was performed. Before performing the tumbling test, sieving of each briquette sample using a 7.5 mm sieve was performed.

The non-standard tumbler drum used had the following specifications: diameter = 252 mm; width = 158 mm; two internal fins of 145 mm length and 25 mm width. These tests were performed with a sample mass of 100 g, drum rotation velocity of 25 rpm and 50 rotations. For comparison, tumbling tests were performed on four samples of sinter used in a coke blast furnace, and a cumulative value of 90% retained on the 7.5 mm sieve was taken as the standard. The curing time used in the tumbling tests was 6 days.

The RDI tests were performed according to standard ISO4696-1 (Iron Ores–Static Test for Low-Temperature Reduction Disintegration) in the pillow and cylindrical briquettes samples that had cumulative values greater than 90% retained on 7.5 mm sieve (tumbling test). The curing time for the RDI tests was 5 months.

In total, 17 samples were selected for the RDI tests, and values higher than 90% were obtained for all these samples in the tumbling tests: M03T (90 kN), M05T (40 kN), M11T (50 kN), M12T (60 kN), M13T (30 kN), M14T (20 kN), M16T (60 kN), M23T (20 kN), M24T (15 kN), M04C (100 MPa), M05C (200 MPa), M07C (150 MPa), M13C (150 MPa), M15C (300 MPa), M16C (300 MPa) and M22C (50 MPa).

X-ray diffraction analyses were performed on sample M13T (30 kN) after the RDI test – core and periphery – to investigate the major phases present. This mixture contained hematite in its composition.

To ensure the smooth operation of blast furnaces the value of RDI-1,3,15,mm < 30% is taken as the standard for use of briquettes in blast furnaces. For values of this index greater than 35–40% the blast furnaces’ performance is affected [16–18]. RDI-1,3,15,mm values less than 27% are acceptable for a smooth operation of blast furnaces [19].

3. Results and discussion

3.1. Pillow briquettes

The selection criterion for the RDI tests among the pillow briquettes samples that obtained cumulative values retained on a 7.5 mm sieve >90% was random to obtain 10 samples of mixtures of different proportions.

Fig. 3 presents the RDI results for the pillow briquettes. In total, 70% of the samples had RDI-3,15,mm values below 30% (the reference value used in the blast furnace). The best results were obtained for M11T (50 kN), M23 (20 kN), M24 (15 kN) and M24T (20 kN), which contained dust, sludge and BBTL.

The best results for the briquettes containing dust, sludge and BBTX binder were achieved for M12T (60 kN) and M13T (30 kN). For briquettes M14T (20 kN) and M16T (60 kN), which contained more than 40% dust, the RDI results were greater than 30%.

The optimal combination of sludge and BBTX binder was M05T (40 kN). Sample M03T (90 kN), which contained dust and BBTX binder did not exhibit adequate RDI values for use in a blast furnace. The presence of dust in the briquette composition appears to be unfavorable with respect to RDI.

3.2. Cylindrical briquettes

The selection criterion for the RDI tests for the cylindrical briquettes that obtained cumulative values retained on a 7.5 mm sieve >90% was random to obtain seven samples of mixtures of different proportions. For the same mixture, the briquetting pressure near the maximum obtained was selected.

Fig. 4 presents the RDI results for cylindrical briquettes. In total, 71.4% of these samples exhibited RDI-3,15,mm values below 30% (the reference value).

The briquettes containing mixtures of sludge and BBTX binder, M04C (100 MPa), M05C (200 MPa) and M07C (150 MPa), with the sludge content representing more than 70% of the content, exhibited RDI values below 30%. The presence of sludge and BBTX binder in the cylindrical briquettes improved the RDI results.
Fig. 4 – RDI values of cylindrical briquettes.

In the mixtures containing dust, sludge and BBTX binder, values greater than 40% for dust in this mixture resulted in RDI values greater than 30%, such as for M15C (300 MPa) and M16C (300 MPa).

The presence of sludge and BBTL, i.e., in M22C (50 MPa) resulted in RDI values below 8%.

Fig. 5 presents secondary electron images of samples M13T (30 kN) – core and periphery – after the RDI test. Fig. 5(a) shows the porous magnetite particle in the briquette periphery, and this morphology is similar to that reported in the literature. Fig. 5(b) shows the porous magnetite and fracture that occurred during the transformation of hematite to magnetite, similar to that reported in the literature [20–24]. The majority mineral phases identified by X-ray diffraction, as shown in Figs. 6 and 7, indicated that all the hematite phase was transformed into magnetite because of the absence of hematite.

Fig. 5 – Secondary electron image of briquette M13T (30 kN) – core (a) and periphery (b) – magnification 5000 x.

Fig. 6 – X-ray diffraction pattern of M13T (30 kN) – core.

Fig. 7 – X-ray diffraction pattern of M13T (30 kN) – periphery.
4. Conclusions

The conclusions that were drawn from the RDI experiments in cold self-reducing briquettes are:

- The presence of sludge and BBTX binder in the briquettes generated adequate RDI results (<30%);
- Dust, sludge and BBTX binder, with dust contents greater than 40%, in the briquettes generated RDI values greater than 30%. For dust contents greater than 40% the physical integrity of the briquettes appears to worsen in RDI tests;
- In total, 70% of the pillow briquette samples and 71.4% of the cylindrical briquettes samples exhibited RDI values less than 30%;
- The physical and chemical composition of the raw materials of the mixture and the pressure applied to the mixture in preparing the briquettes are important factors in attaining adequate RDI values for use in blast furnaces;
- BBTL is an adequate binder for the mixtures because of the low RDI values;
- The mechanical strength of the briquettes is related to the effectiveness of the contact between the particles and binders, fracture toughness of the phases present and homogeneous distribution of the binder in the mixture.

Conflicts of interest

The authors declare no conflicts of interest.

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