Abstract

The demand for sustainable materials in the steel industry and the search for alternatives for the traditional ferriferous burdens utilized in siderurgy stimulated the development of this research. The briquetting process is a usual measure taken for recycling materials such as ironmaking waste (dust and sludge from blast furnaces), which can be reutilized in steel plants. The objective of this article is to investigate the influence of the use of organic and inorganic binders, blast furnace dust and sludge on the mechanical strength of briquettes for use in blast furnaces. The mechanical analysis was performed with the results from tumbling tests, executed to obtain the strength of each sample composition. The results exhibited adequate values of mechanical strength for samples that contained BBTL and BBTX binder, indicating that these materials acted as good binders in the briquetting process.

Keywords: briquetting, tumbling test, blast furnace, mechanical strength.

1. Introduction

A blast furnace is a metallurgical reactor that operates in counter-flow with the iron load and coke descending as gases rise. Particles are transported by the gases and generate blast furnace dust. The sludge formation occurs in the process of cleaning the blast furnace gas, where the material is submitted to a wet process, resulting in a product containing about 45 to 50 % of iron oxides (Lopéz-delgado, 1998). The physical and chemical quality of the blast furnace’s burdens are extremely important parameters for the proper functioning of this reactor. Physical and metallurgical tests, such as tumbling, shatter, reduction disintegration index and reducibility tests are typically used in integrated steel plants.

The mechanical strength of numerous ferriferous burdens is analyzed through tumbling tests. The tests are utilized to simulate the degradation of the ferriferous burden caused by the impact and abrasion that occurs during the loading, handling and transport of these materials. These tests should also be performed on agglomerates, such as self-reducing briquettes. There is not an international standard specified for tumbling tests that implicates the use of briquettes. The ISO 3271 standard is particular to the conventional ferriferous burden utilized in blast furnaces (coke, sinter,
pellets and iron ore). Agglomeration can be described as the union of particulate solids with short range physical forces and nature’s chemical forces, together with the physical and chemical changes in the solid state owing to the briquetting process conditions (Pietsch, 2003). Many co-products can be briquetted, e.g., dust and sludge from blast furnaces and LD converters, mill scale, continuous casting scale, coke breeze and biomass.

The agglomerates should possess sufficient strength for the handling, storing and feeding processes used in a blast furnace. Many factors affect this resistance, including the briquetting pressure, binder content, moisture and temperature

2. Materials and methods

Sampling and sample preparation

The blast furnace dust and sludge samples were collected from global samples provided by a steel plant using standard ABNT NBR 10007-2004 (Sampling of Solid Waste). The dust was obtained in the cyclone or collector equipment. The sludge was originated in the process of cleaning in the Venturi equipment. The dust is usually applied as a sintering burden and the sludge is commonly used in the ceramic and cement industry.

To prepare the mixtures for briquetting, the following binders were used: water (W), BBTX, BBTL, magnesium oxide (MO), refractory cement (RC). The BBTL is a lime-based binder and the BBTX is a mixture of an organic binder and magnesium oxide. In total, 25 different mixtures were prepared with dust, sludge and binders on a dry basis, as indicated in Table 1. All 25 mixtures were comminuted in a ball mill (0.35 m³; 4 h).

To organize the distinct samples, differentiating each one, the naming of the briquettes was normalized as follows: mixture of substances (M); pillow briquettes (T), cylindrical briquettes (C) and the last term indicates the force/pressure applied (in kN) during the sample preparation.

Size classification was executed by passing these mixtures through a 3.0 mm sieve; only the materials passing through this sieve were used for briquetting. The samples were produced utilizing the processes of roller and piston briquetting.

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

Sample characterization

Elemental chemical analysis was performed using inductively coupled plasma optical emission spectrometry and a Leco equipment present in the laboratory of chemical analysis in DEMET/UFMG. These tests were realized on the dust,
sludge, binders and on the mixture M05. This mixture was selected as a reference for the particle size distribution of all 25 manufactured mixtures.

Size analyses were also performed on dust, sludge and the M05 mixture. These materials were submitted to a study of granulometric distribution using laser diffraction analysis equipment present in the laboratory of aqueous processing and characterization of particulate solids of DEMET/UFMG.

The dust and sludge were submitted to semi-quantitative analyzes by X-ray diffraction (XRD). The analysis utilized a diffractometer present in the laboratory of X-Ray diffraction of DEMET/UFMG. An analyzer of adsorption surface area and distribution of pore size, present in the laboratory of aqueous processing and characterization of particulate solids of DEMET/UFMG, was used to obtain the values of the specific surface area, average pore diameter and specific pore volume for 5 samples of pillow briquettes (M03T(90), M13T(30), M14T(20), M16T(60), M23T(20)).

Bulk density
The bulk density was obtained for a set of 3 briquettes, for each sample produced, achieving an average between the 3 series. For the pillow briquettes, the bulk density was calculated through a relationship between the variations of volume due to the submersion of the briquette in a beaker containing water. For the cylindrical briquettes, the bulk density was determined through an association between the mass, measured using a scale, and the volume calculated with the values of diameter and height of each briquette.

Roller briquetting
The mixtures from M01 to M07, M09 to M18 and M22 to M25 (totaling 21 mixtures) were used in the roller briquetting process. It was not possible to realize this process with all of the samples because some mixtures got adhered to the rollers making it impossible to create a pillow briquette.

Each briquette produced in the machine, with a roller velocity of 1.7 rpm, had a total volume of 3.4 cm³. The screw feeder’s rotational velocity depended on the force applied to the briquettes. The pressure applied in the process ranged from 10 to 100 kN and depended on the following parameters: visual analysis of the briquettes (absence of cracks and geometrical uniformity) and rheology phenomena.

Piston briquetting
The mixtures from M01 to M07, M12 to M16, M21 and M22 (totaling 14 mixtures) were used in the piston briquetting. A hydraulic press (with a maximum capacity of 20 t), one cylindrical matrix and a piston were utilized in the process. Approximately 35 g of each mixture were placed inside the matrix. The material was subjected to seven different pressures (10, 20, 50, 100, 150, 200 and 300 MPa), and three cylindrical briquettes were produced with each mixture. It was not possible to realize this process with all of the mixtures, since the pressure band utilized caused undesirable effects with some samples, where the process reaches the yield point between the piston and the matrix. The pressure exerted by the piston on the mixture increased, and the maximum limit was dictated by the maximum selected pressure (300 MPa) or when the rheology phenomena occurred.

Tumbling tests
The tumbling test did not follow the ISO 3271 standard due the amount of samples utilized and the different dimensions of the rotary tumbler. A normalization was settled beyond the international standards to perform this test with a conventional ferriferous burden (sinter and pellets).

Every pillow and cylindrical briquette sample produced passed through the tumbling test, performing a non-standard test for each mixture and for each force applied (during briquetting process). The tumbling test followed the parameters described by Lemos (2015).

The samples that exhibited cumulative values of 90% or more on the 7.5 mm sieve were selected for the reduction disintegration index (RDI) test, which will be not covered by this article.

3. Results and discussion
Sample characterization
The chemical composition of the dust, sludge and mixture M05 (used as reference) are presented in Table 2. The percentage of zinc obtained in the analysis is close to the results found in literature according to Machado (2004). The carbon and iron contents detected were high compared to the values exhibited in researches done by Rocha (2007) and Kurunov (2012). That inconformity can be associated to the differences in efficiency of the cleaning process of the waste gas between ironmaking industries. The carbon content in the dust normally has a greater particle size, compared to the particles in the sludge. The first step in the gas cleaning process should remove those larger particles within the cyclone equipment. The values shown in Table 2 exhibited the inefficiency of the equipment in the industry. The size distribution, percentage of passing material (10, 50 and 90 %) and the diameter (μm), is present in Table 3.

The x-ray diffraction analysis exhibits that in both dust and sludge materials, the presence of hematite, magnetite and graphite was identified. There are some differences between the analyses; the dust contained the wollastonite, whereas the sludge exhibited calcite.

The calcite phase (CaCO₃) comes from the degradation of the fluxing agent utilized in the blast furnace to adjust the basicity of the slag and as a desulfurizing element. The wollastonite, a calcium silicate (CaSiO₃), derives from the rupture of the slag present in the sinter and pellet during the descent of these agglomerates in the blast furnace.
Table 2
Elemental analysis of dust, sludge and mixture M05.

| Elements (%) | Dust | Sludge | Mixture M05 |
|--------------|------|--------|-------------|
| Fe           | 52.58| 21.59  | 41.75       |
| K            | 0.13 | 0.19   | 0.30        |
| Na           | 0.10 | 0.22   | 0.17        |
| Zn           | 0.11 | 3.05   | 0.77        |
| P            | 0.07 | 0.12   | 0.09        |
| Mg           | 0.20 | 0.53   | 0.30        |
| Ca           | 0.77 | 1.3    | 1.86        |
| Mn           | 0.46 | 0.09   | 0.43        |
| Al           | 0.67 | 1.21   | 1.24        |
| C            | 32.53| 49.56  | 19.65       |
| S            | 0.31 | 1.59   | 0.46        |

Table 3
Size distribution of dust, sludge and mixture M05.

| % Passing | Diameter (μm) |
|-----------|---------------|
|           | Dust | Sludge | Mixture M05 |
| 10        | 2.46 | 1.01   | 1.11        |
| 50        | 21.88| 5.05   | 6.27        |
| 90        | 68.18| 14.58  | 25.81       |
| Average diameter | 29.27 | 6.52 | 10.08 |

**Tumbling tests**

The samples of pillow briquettes that had values above 90% were: M03T(90), M05T(40), M06T(35), M11T(20), M11T(40), M11T(50), M12T(60), M13T(30), M14T(20), M14T(30), M15T(40), M15T(60), M16T(60), M23T(20), M24T(15) and M24T(20). Figure 1 shows the cumulative amount retained using the 7.5 mm sieve (%) as a function of each pillow briquette analyzed.

Figures 2 and 3 demonstrate the cumulative amount retained using the 7.5 mm sieve (%) as a function of briquetting pressure for each cylindrical briquette analyzed.

The mechanical strength of the briquette can be associated with contact effectiveness between the particles (interlocking, molecular forces, capillary forces) and binders, fracture toughness of the phases and the homogeneous distribution of the binders.

The application of sludge and BBTX binder induced higher cumulative values retained, using the 7.5 mm sieve, in relation to the use of dust and BBTX binder. The interactions between the components of the sludge and BBTX binder appear to be more effective.

Considering the cylindrical briquettes without BBTL and refractory cement, there is a positive correlation between the bulk density and the percentage of cumulative amount retained using the 7.5 mm sieve for the pressure band utilized in the process. Usually the increase of the bulk density leads to a raise in the values obtained in the tumbling test above 90%. The values remain constant until the maximum pressure is pre-established. Raising the content of BBTX and reducing the percentage of dust in the mixtures demonstrated a greater cumulative amount retained. This tendency is similar to the pillow briquettes fabricated utilizing the same mixture.

The mixtures containing a considerable amount of sludge and binders or mixtures made of dust, sludge and
binders have a high mechanical strength due the good effective contact between the particles.

Considering only the briquettes containing BBTL and refractory cement, the percentage of cumulative retained had an increase followed by a reduction in value, when the pressure was raised. There was not verified any correlation between the rise of the pressure applied with the increase and stabilization of the mechanical strength of the briquettes. The binder became quite brittle for briquettes produced with pressures above 100 MPa.

**Porosity and bulk density**

Table 4 shows values for the specific surface area, average pore diameter, total pore volume and porosity for five selected samples of pillow briquettes. Observing the pillow briquette samples analyzed, the M23T(20) exhibited an adequate value for porosity, being relatively close to the values obtained in the literature for traditional ferriferous burden utilized in blast furnaces. The value found was approximately 29.31% of porosity; it is superior to the value stated for the pellet presented in the Gandra (2012) research (23%) and the maximum value found for sinter (21.46%); this latter study realized by Pimenta (1992). The value was also equivalent to the one stated by Henriques (2012) for goethite samples (29.42%).

The bulk density was calculated for all pillow and cylindrical briquettes. The values range from 1.1 to 2.4 g/cm³. Considering the pillow briquettes, the average bulk density determined was around 1.54 g/cm³ (maximum of 2.07 and minimum of 1.07 g/cm³). For the cylindrical briquettes, the amount of pressure utilized during the briquetting process was directly proportional to the bulk density of the sample. As expected, considering all the briquettes evaluated, the increase of pressure applied in the process also increased the bulk density of the sample.

According to Gandra (2012), the average density of the sinter was around 4.0 g/cm³ and about 3.5 g/cm³ for pellets. The average density of an iron ore containing hematite was close to 3.5 g/cm³, stated by Henriques (2012). Comparing the utilization of briquettes in relation to the use of convectional ferriferous burden in blast furnaces, it is observed that the residence time of the briquettes in the metallurgical reactor is inferior. That fact can be explained by the lower values of density for the briquettes analyzed in comparison to the values found for sinter, pellets and iron ore. Increasing the pressure in the process leads to low values of porosity (as shown in Table 4) and high values of density; these changes lead to the retardation of the reducibility of the briquettes.

**Table 4**

| Sample   | Specific surface area (m²/g) | Average pore diameter (nm) | Total pore volume (cm³/g) | Porosity (%) |
|----------|-------------------------------|---------------------------|---------------------------|--------------|
| M03T(90kN) | 6.181                         | 14.77                     | 0.02282                   | 3.62         |
| M13T(30kN) | 17.813                        | 14.59                     | 0.06496                   | 10.13        |
| M14T(20kN) | 15.985                        | 10.07                     | 0.04025                   | 7.97         |
| M16T(60kN) | 5.986                         | 14.65                     | 0.02193                   | 4.28         |
| M23T(20kN) | 79.571                        | 10.45                     | 0.20790                   | 29.31        |
4. Conclusions

- In total, 48.5% of the pillow and 67.5% of the cylindrical briquette samples had values higher than 90% in the tumbling tests.
- The physical and chemical composition of the raw materials, interlocking and pressure applied on the briquettes are important factors in establishing good mechanical strength.
- The refractory cement, which is a hydraulic binder, did not produce good results in the tumbling tests because of the volume expansion and weak bond strength between the particles contained in the briquette.
- The increase of the bulk density for cylindrical briquettes without hydraulic binders raises the values of the tumbling test, in which 90% of the resultant material was larger than the aperture of the 7.5 sieve.
- The BBTL produced good results in the tumbling tests for the pillow briquettes and for some pressures in the cylindrical briquettes.
- The samples containing sludge and BBTX binder and the samples with dust, sludge and BBTX binder produced good results in the tumbling tests. The cold strength increased with increasing pressure for these mixtures.

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