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Quantitative Characterization of the Blast Furnace Pellet Phases with Different Degrees of Reduction

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Abstract: ***The present work proposes a methodology for the quantitative microstructural characterization of blast furnace pellets. Samples with different degrees of reduction, about 14mm in diameter were embedded in epoxy resin, grounded and polished. For each polished section, images were acquired using optical microscope with objective lens of 100x. The images were analyzed one by one through the free ImageJ software, where the area fractions of each phase including pores were measured. Results of hematite, magnetite and wustite fractions were obtained in commercial pellets with different degrees of reduction due to different thermal cycles and atmospheres to which they were subjected.***

Keywords: blast furnace pellets, hematite, magnetite, wustite**.**

1. Introduction

Iron ore is described as a natural material that can possess various compositions, degrees and physical qualities. It can be mined and processed but is economically exploitable depending on the amount of iron present or the concentration of minerals in the rock. Therefore only as classes of oxides, carbonates, sulfides and silicates, classified according to the chemical composition, are explored [1].

Among the main iron ore is the class of oxides, composed of hematite, magnetite and wustite, for example.

Hematite (Fe_2O_3) is the most commonly found iron ore and occurs in large quantities. It is considered the most important iron mineral, occurring in several types of rocks such as granites, andesites, originating from magmatic crystallization, metamorphic rocks, as hematite quartzites and also as a result of magnetite alteration [1]. Contains about 70.0% iron and in hardened pellets is the final stage of oxidation. In addition to the hematite, there is the secondary mineral, known as porous hematite/Martite that occurs due to the weathering of primary magnetite [2].

Magnetite (Fe_3O_4) is commonly found in magnetic and metamorphic rocks, in meteorites and also in beach sand (FONSECA, 2004). It has 72.36% of iron and 27.64% of oxygen, with an average density of 4.9 to 5.2 g/cm^3. It is a very magnetic mineral that can have natural magneto behavior and by this property can be exploited by magnetic methods, and separated from the gang by magnetic separation producing high quality concentrate [1].

The wustite has a true composition that varies from $Fe_{0.9}O$ to $Fe_{0.96}O$ and occurs in nature as a mineral, and can also be obtained synthetically by thermal decomposition of iron oxalate II. It has black coloration, density around 5.7 g /cm^3 and is formed by cubic crystals. Because it has a small amount of Fe^{3+}, it also has semiconductive properties [2]. Ore processing arose from the difficulty in providing ideal iron ore of the granulated type for direct use in blast furnace. The fine ores that were considered as tailings go through a process of agglomeration so that the granulometry reaches acceptable levels for use in the blast furnace and become profitable. The agglomeration can be done by sintering, using ores of granulometry between 0.15 and 12.5mm, or pelletizing, using ores below 0.15mm [1].

Pellets, together with sinter and lump ore, are the main sources of iron to be extracted by blast furnace reduction process. They are obtained through pelletizing, where the process of agglomeration of the ore fines that were generated during the mining and processing of iron ore occurs and could not be used in the reduction processes due to the complexity of the handling, transport and reduction the permeability of the reducing gases inside the blast furnaces [1].

The use and production of iron ore pellets have been stimulated and have become increasingly important due to the trend of increase of fines in the process of iron ore extraction, the generation of pellet feed that uses pelletizing as the most appropriate technology for agglomeration of these ores, environmental restrictions on sintering expansion, since the emission of CO_2 in the pelletizing (100 kg/ton of pellet) is lower than the sintering emission (230 kg/ton of sinter) and the further expansion of the reduction technology which demands pellets for this application [3].

In this context, the quantitative analysis of the pellets becomes a focus of great attention for the evaluation of their performance in the steel processes. The deepening of the knowledge of the quality of the product can be realized through the evaluation of specific characteristics, such as mineral constitution, microstructure and porosity [4].

The phases most commonly present in blast furnace pellets during the reduction process are hematite, magnetite and wustite. Because the reflectance of these minerals is very distinct, the light microscopy of reflected light can be used for the qualitative characterization, since they can be visually identified by the optical microscope through their colors [4].
The present work presented a methodology based on digital optical microscopy and image analysis, aiming at helping the

quantification of phases in commercially produced iron ore pellets. To assist in the quantification of the phases, X-ray diffractometer was used.

2. Methodology

2.1. Materials

For the quantification of the phases present in the pellets with different degrees of reduction, pellets called PBF produced commercially were submitted to thermal treatment in reducing atmosphere and in inert atmosphere. For this, a SPECTRO muffle furnace with a nominal maximum temperature of 1200ºC was used. The samples were submitted to thermal cycles with maximum temperature of 600ºC, 900ºC, 1000ºC and 1050ºC with isotherms of 75 minutes.

In addition, a pellet from the same batch was used to serve as a reference in relation to the pre-existing phases.

2.2. Sample preparation

The samples, after the heat treatment, were cold embedded in epoxy resin. The samples were then cut in half, grounded and polished in an automatic polishing machine.

A sample taken from the same batch of the other pellets was also used.

2.3. Image Acquisition and Analysis

The acquisition of images was done by NIS Elements software through a Nikon DS-Fi1 video camera attached to the Nikon Eclipse LV150 optical microscope. Microscope lighting was always kept constant by direct digital control of lamp voltage. The processing and analysis of the images obtained were done using the free ImageJ software.

2.4. X-ray diffractometer

The X-ray diffraction (XRD) method was used with the objective of assisting in the verification of the phases present in the samples. For the use of XRD, the sample was manually disaggregated in a agate mortar with pistil to granulometry of less than 45 μm and then the powder samples were compacted in a specific sample holder. X-ray diffraction tests were performed In a LABX diffractometer model XRD6100 (SHIMADZU), with copper radiation K (λ=1,542 Å), power of 40mV and 30mA, goniometer speed of 2°/min and measurement range of 10º to 90º in 2θ.

3. Results and Discussions

3.1. X-ray diffractometer

The analysis of the samples by X-ray diffraction was used with the objective of identifying the phases present in the pellets that underwent reduction by means of reducing atmosphere and samples submitted in an inert atmosphere (non-reduced pellets).

The results of X-ray diffraction of the phases found in the non-reduced pellets and in the reduced pellets subjected to thermal cycles at temperatures of 600°C, 900°C, 1000°C and 1050°C for 75 minutes in an inert atmosphere are shown in Figures 1 and 2 .

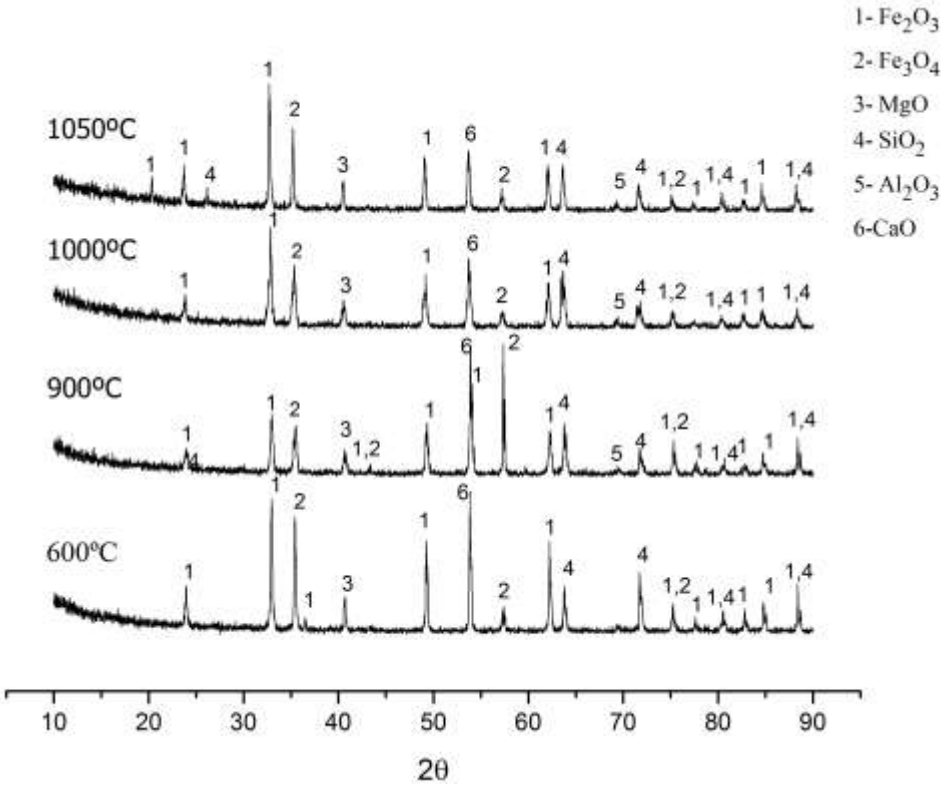

Figure 1: X-ray diffraction of non-reduced pellets subjected to thermal cycling at isotherm temperatures of 600ºC, 900ºC, 1000ºC and 1050ºC during 75 minutes in an inert atmosphere

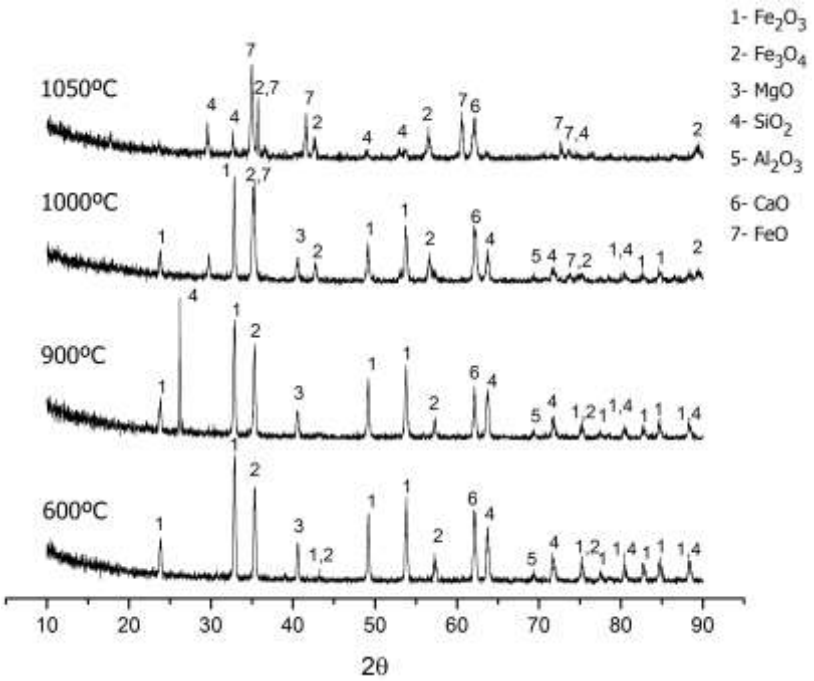

Figure 2: X-ray diffraction of reduced pellets subjected to thermal cycles at isotherm temperatures of 600ºC, 900ºC, 1000ºC and 1050ºC for 75 minutes.

It was possible to observe a great similarity between the peaks obtained in the pellet called PBF removed from the same batch of the other samples, and of the non-reduced pellets submitted to a thermal cycle, thus justifying a great equivalence in the chemical compositions of these samples. It was still possible to affirm with this result, that the thermal cycles to which the samples were submitted did not provide conditions necessary for structural changes in the pellets.

According to Geerdes et al. (2015), the reduction of magnetite to wustite occurs in the temperature range between 600 and 900 ° C in blast furnaces. When analyzing the data obtained by XRD, the presence of wustita is observed from a temperature of 1000ºC in reduced pellets (Figure 2). This result was possibly obtained by the heating rates used.

The use of the optical microscope in the analysis of the microstructure of the iron ore pellets was fundamental to identify the phases present for each thermal cycle used, as well as for the quantification performed based on the data obtained by X-ray diffraction.

In order to obtain a homogeneity to observe the behavior of the present phases, the micrographs and quantifications of the phases were made from two regions of the pellet: the center and a section near the edge. All the micrographs were obtained after a 75 minutes isotherm and with a 100X magnification.

Because there were no phase transformations during the thermal cycles, it was possible to observe modifications in the hematite coloration, which in all the micrographs was found in greater quantity. From the observation of the images of the non-reduced samples, it was possible to analyze that, depending on the temperature at which they were submitted, the staining of the hematite varies from light gray, lilac, in the pellet after thermal cycle of 900ºC, purple in the other images. Magnetite was found in these samples generally in the contours of the hematite and can be differentiated by dark purple color (Figure 3).

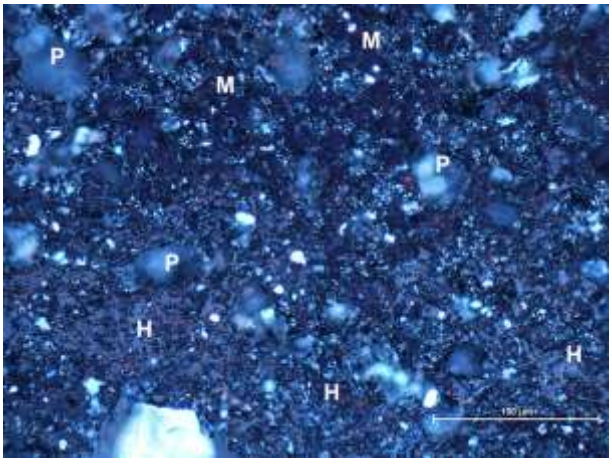

Figure 3: Micrograph of the edge of a sample subjected to a thermal cycle of 900ºC in an inert atmosphere where: H - hematite, M - magnetite and P - pores.

It is also possible to verify the non-homogeneity of the samples, since the pellet used in the thermal cycle of 600ºC presented a higher amount of SiO_2 than in the other samples (Figure 3).

From the thermal cycle of maximum temperature of 1000ºC, it was observed the reduction of magnetite to wustita, found in the images through the gray lead color, which is seen with greater clarity in the micrograph of the thermal cycle ball 1050ºC (Figure 4).

After the determination of the colors of the phases present in the samples, the image treatment and subsequent

quantification of the phases were performed using the Image J software.

The present phases (hematite, magnetite and wustite) present distinct colors in the images, as can be observed in Fig. 3 and Fig. 4. Thus, all the images used in this work were acquired with distinct staining and later transformed into toned images of gray, with quantization of 8 bits.

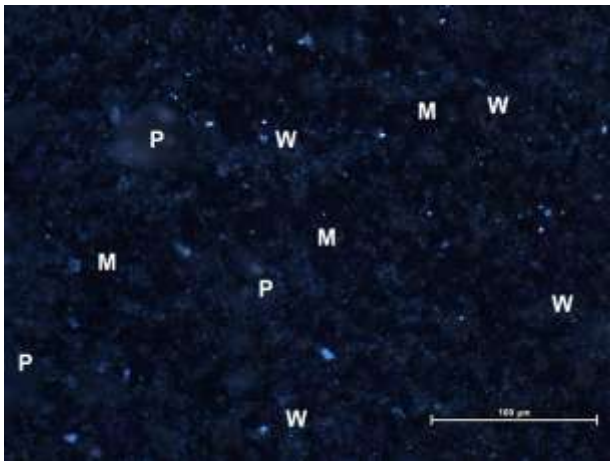

Figure 4: Micrograph of the edge of a sample subjected to a thermal cycle of 1050ºC in an inert atmosphere where: M - magnetite, W - wustite and P - pores.

For the treatment of the images, in the first moment the micrography was submitted to a filter so that the contours between the phases became more evident. This color image was then transformed into a black and white image and the pores were selected and colored so that they did not interfere in the quantification of the phases, since they could be found in these images in white or black colorations. With this, it was possible to quantify the pores.

For quantification of the phases, the magnified (colored and black and white) images were placed side by side and through the "Threshold" command, the area of each phase was quantified according to the gray tones of the image in black and white related to what was observed in the color image (Figure 5).

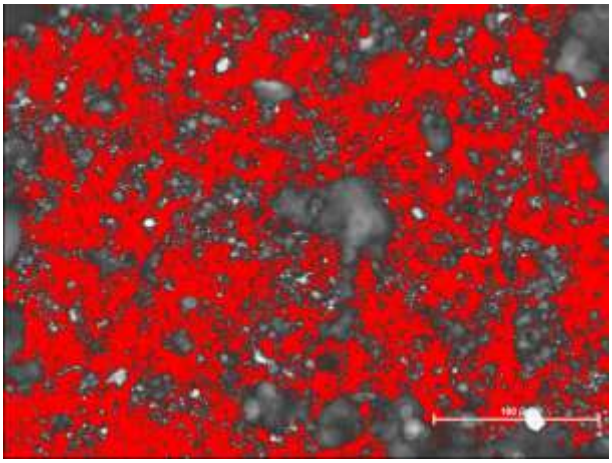

Figure 5: Quantification of magnetite by micrograph analysis using Threshold of a pellet subjected to a thermal cycle with reducing gas at 900ºC.

Due to the similarity in the colors of MgO, Al_2O_3, SiO_2, CaO (white color in the color image) it was not possible to quantify them separately.

In Table 1 it is possible to observe the distribution of the phases present in each analyzed sample, a result obtained through the X-ray diffraction data and the analysis of the images obtained by optical microscopy.

Table 1: Distribution of the phases present in the samples used in the present work

Sample	Temperature (ºC)	Constituents				
		Fe_2O_3	Fe_3O_4	FeO	MgO/ SiO_2/ Al_2O_3/ CaO	Pores
PBF/45	-	67,71%	23,04%	-	3,69%	5,58%
Unreduced	600	66,77%	19,79%	-	4,71%	3,47%
	900	64,97%	21,44%	-	5,64%	7,95%
	1000	66,02%	21,31%	-	4,85%	7,83%
	1050	70,53%	17,68%	-	4,57%	4,53%
Reduced	600	55,73%	37,34%	-	1,64%	5,20%
	900	49,86%	47,37%	-	2,78%	8,58%
	1000	20,74%	52,66%	15,16%	4,84%	5,64%
	1050	-	36,34%	57,16%	1,66%	4,93%

By analyzing the distribution of the phases present in the non-reduced samples (submitted to inert gas atmosphere), it is possible to observe a homogeneity regarding the hematite and magnetite phases present in the pellets submitted to different thermal cycles.

On the observation of the reduced samples, it is possible to verify a quantitative increase of magnetite, in relation to the sample PBF, of 62,1% at 600ºC and of 128,6% until the temperature of 1000ºC, decreasing in 1050ºC due to the transformation of Magnetite in wustite by the reducing atmosphere to which they were submitted.

4. Conclusion

From the use of XRD techniques, Optical Microscopy, it was possible to observe that the thermal cycles to which the samples were submitted in inert atmosphere did not provide conditions for structural changes in the pellets; And in the pellets that were reduced, the presence of wustite was observed only at a temperature of 1000°C, possibly due to the heating rates used.

It was also observed that it is possible to quantify phases such as hematite and magnetite and wustite using only

Optical Microscopy and software for image processing and analysis. However, this method is inefficient to quantify constituents such as MgO, SiO_2, Al_2O_3 and CaO because they have similar optical appearance.

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