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SEPARAÇÃO MAGNÉTICA DE USTULADO DE MINÉRIO DE FERRO HEMATÍTICO

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RESUMO

A ustulação objetivou o aumento da eficiência da separação magnética de um minério de ferro hematitico. O percentual de coque, o tempo de ustulação e a temperatura foram analisados em vários níveis baseados na literatura prévia. A caracterização da amostra foi feita para avaliar a densidade da amostra e o tamanho tanto da partícula de minério como de coque. A análise termogravimétrica indicou transformação de hematita para magnetita por picos característicos de perda de massa. A transformação hematita-magnetita fornece forte contraste na suscetibilidade magnética entre minerais portadores de ferro e ganga, esta composta basicamente por quartzo. Os resultados demonstraram o incremento no magnetismo e fotografias comprovaram o caráter magnético da amostra ustulada. A melhor condição obtida foi com 35 minutos, 10 % de coque (em massa) e 1,023 K (750 ºC). A eficiência foi aumentada em 5.3 % sob campo de 0.93 T e 240.3 % sob campo de 0.06 T, aumentando a seletividade da separação magnética até sob baixo gradiente magnético. Esta rota poderia ser aplicada com sucesso aos rejeitos de minério de ferro em campo de baixo gradiente, naturalmente após análise econômica cuidadosa, na qual a quantificação de passivos ambientais (principalmente resultante de sua não implementação) é incluída.

PALAVRAS-CHAVE: Hematita, Ustulação Magnetizante, Separação Magnética.

MAGNETIC SEPARATION OF ROASTED HEMATITIC ORE

ABSTRACT

Roasting of hematitic ore has been tested in boosting of magnetic concentration of hematitic iron ore. Coke rate, roasting time, and temperature were analyzed at several levels based on literature. Sample characterization was done to evaluate the density of sample and the size particle of ore and coke. Thermogravimetric analysis has indicated hematite to magnetite transformation by characteristic peaks of mass loss. The hematite-magnetite transformation imparts strong contrast in magnetic susceptibility between iron-bearing minerals and gangue, composed basically of quartz. The results have confirmed the reinforcement of magnetism due to roasting process and photographs proved the magnetic character of roasted sample. The best condition obtained was 35 minutes, 10 % of coke (in mass), and 1,023 K (750 ºC). The yield was increased by 5.3 % under 0.93 T and by 240.3 % under 0.06 T, increasing selectivity of magnetic separation even under low magnetic gradient. This route could be successfully applied to tailings at low gradient field, recommending the application of this technique for iron ore processing, naturally after careful economic analysis, in which the quantification of environmental liabilities (primarily resulting from its non-implementation) is included.

KEYWORDS: Hematite, Iron-bearing Ore, Magnetic Roasting, Magnetic Separation.

1 INTRODUCTION

The discovery of itabirite iron-bearing ores has been on the rise in the past few years, making it possible to maintain the Brazilian ore production and the exploitation of even poorer iron-bearing ores. Hematitic ores are, nowadays, processed by gravity methods, flotation or by high gradient magnetic separation. Gravity separation is applied to situations in which the ore is liberated in coarse fractions. Flotation is the most widely used method nowadays, by its best selectivity, although it is an expensive method. High gradient magnetic separation has a very high initial investment, although with low operational cost. Typically the price of a high gradient separator is about 4 to 5 times the cost of a low/medium gradient separator of the same treatment capacity. Rath et al. (2014) investigated an ore refractory to conventional magnetic methods. Hematite and goethite are converted into magnetite by roasting, allowing separation by low intensity magnetic equipments.

Shao, Veasey and Rowson (1996) measured magnetic susceptibility of iron ores in a wet high intensity magnetic separator. They concluded that hematite is a strongly paramagnetic mineral and can be treated with relatively low fields. In the other hand, goethite and limonite are both weakly paramagnetic minerals and cannot be recovered in the tested conditions. This work aimed at study improvements in magnetic separation of not amenable fine ores, by turning the hematite surface magnetic, through partial reduction by carbon, process called magnetic roasting. This reduction starts by hematite reacting with carbon added previously and with carbon monoxide to form magnetite, wustite and metallic iron depending on temperature roasting. The reaction of carbon is also important and must be pointed that elevated temperatures improved the transformation of carbon dioxide to carbon monoxide. The main reactions in iron reduction are presented below. The enthalpies of formation are taken from literature and shown below (Mazeina & Navrotsky, 2005; Plascencia et al., 2016).

$2\ FeO(OH) = Fe_2O_3 + H_2O \qquad \Delta Ho_{298} = -561.5\ kJ/mol \qquad (1)$

$3\ Fe_2O_3 + CO = 2\ Fe_3O_4 + CO_2 \qquad \Delta Ho_{298} = -\ 53.1\ kJ/mol \qquad (2)$

$Fe_3O_4 + CO = 3\ FeO + CO_2 \qquad \Delta Ho_{298} = 41.0\ kJ/mol \qquad (3)$

$FeO + CO = Fe + CO_2 \qquad \Delta Ho_{298} = -18.4\ kJ/mol \qquad (4)$

$2\ CO_2 = 2\ CO + O_2 \qquad \Delta Ho_{298} = 566.08\ kJ/mol \qquad (5)$

$C + O_2 = CO_2 \qquad \Delta Ho_{298} = -394.96\ kJ/mol \qquad (6)$

Cui, Liu and Etsell (2002) carried out tests of magnetic roasting with ilmenite, hematite and oilsands. They have concluded that the magnetic susceptibility of hematite can be the same of magnetite after 30 minutes of roasting with 10 % to 20 % of coal and temperatures above 1,073 K (800 ^oC). Li et al. (2010) have found that carbon ratio, roasting temperature and reduction time have a higher impact on recovery of iron-bearing minerals. They have had optimum results, using carbon proportion of 1 %, and roasting at 1,073 K (800 ^oC), for 30 minutes.

As a matter of fact, reductive roasting and magnetic beneficiation of oolitic iron ores from west Hubei province were performed by Peng et al. (2017). Roasting time had significant effect on the iron recovery and increased from 10 to 60 minutes, and was more pronounced for the first 30 minutes. As the thickness of the roasting layer increase from 15 mm to 60 mm, the iron content of concentrate in total changed slightly (Peng et al., 2017).

Sharma and Sharma (2014) have beneficiated iron ores and achieved higher yield and acceptable grades by roasting mineral particles of goethite. Up to 723 K (450 °C), the particles become more magnetic and were converted to magnetite. Yu et al. (2017) studied the magnetization roasting treatment with a low grade iron ore composed by hematite and siderite. Particle magnetic susceptibility could increase due to hematite-magnetite conversion by reduction. The best phase conversion was obtained in 1,073 K (800 °C) for 8 minutes with 8 % of coal. Jang et al. (2014) analyzed the phase transformation of iron-bearing minerals. Goethite is transformed into hematite in temperatures above 673 K (400 °C) and the transformation ends in temperature of 873 K (600 °C), whereas maghemite is formed in temperatures above 973 K (700 °C), being the sample magnetization due to this phase transformation.

Faris et al. (2017) studied magnetizing roasting of an iron-rich rare earths ore and used X-ray diffraction to determine mineralogical properties of roasted products. Temperatures between 873 K (600 °C) and 923 K (650 °C) with mass proportion of coal from 10 % to 20 % l and a roasting time of 90 minutes were the optimal roasting conditions for this ore. The magnetite characteristic peak in about 823 K (550 °C) was not observed, while it can be found in higher temperatures. The highest magnetite peak intensity was observed between 923 K (650 °C) and 1,023 K (750 °C).

2 MATERIALS AND METHODS

2.1 Characterization and Sample Preparation

The density was measured by gas picnometry (Quantachrome Corporation, model Ultrapyc 1200e, version 4.00), allowing to check the mineral proportions as a quick chemical analysis based in Sheldon´s (1964) procedure. Particle size characterization was done by wet screening the material, in order to determine size distribution from the coarsest to the finest fractions.

Coal coke is a high efficiency fuel and reduction agent at steel industry. It is mixed with the blast furnaces load for the production of carbon monoxide (CO), the main reduction agent for iron ores. Here coke preparation consisted in picnometry, particle size characterization and further comminution by successive crushing steps.

Thermogravimetric analyses have indicated ore mass losses by varying temperature. A typical thermogram of iron ore tailings sample shows a peak between 373 K (100 °C) and 403 K (130 °C) referring to the loss of water adsorbed on minerals. The peaks between 448 K (175 °C) and 583 K (310 °C) are due to the dehydroxylation of goethite to hematite. Around 1,023 K (750 °C), there is a peak due to magnetite transformation as found by Dauce and coworkers (2019). This technique allows identifying the mineral composition, comparing unroasted and roasted samples and determining the magnetite and hematite composition for each condition.

2.2 Roasting Tests

The experiments were carried out in a muffle furnace, under the conditions presented in Table 1. The products used in the tests (iron ore and coal coke) were prepared in a particle size lower than 106 µm. The roasting tests variables and levels were selected based on literature shown previously in introduction. Just to clarify, the conditions labeled with time 0.0 min and coke 0.0 %

are related to magnetic separation of the raw material. Actually, there was no roasting experiment without coke in mixture.

Table 1 – Test variables in roasting step.

Temperature		Coke proportion		Time	
Levels	Values [ºC]	Levels	Values [%]	Levels	Values [min]
1	450	1	0.0	1	0.0
2	500	2	6.0	2	15.0
3	550	3	8.0	3	25.0
4	650	4	10.0	4	35.0
5	750				

2.3 Magnetic Separation Tests

A magnetic separation campaign was performed using a wet high-intensity magnetic separator Model 3X4L, from Carpco (Figure 1), under four magnetic field intensities. The separation chamber employed, seen in Figure 1, has these effective dimensions: 50 mm width, 60 mm length and 100 mm depth. The matrix consisted of randomly packed 35 mm steel balls. The magnetized ore was retained on the matrix balls (while solenoid electrical current is kept turned on in a pre-set value), whereas the non-magnetic portion passed through the separation chamber and left it by its bottom grid. In sequence the field was turned off and the magnetic ore was taken in a separate recipient, after a previous water washing step (still under magnetic field) to remove the mechanically entrapped gangue. This process was done for increasing field intensities. After each step the tailings were returned for a subsequent separation step under increasing field strength. Therefore, each sample has resulted five products, four of them magnetic under specific field intensity, and the last one non-magnetic. No cleaning step was carried out.

Figure 1 – High gradient magnetic separator (Carpco's); legend: (1) hopper, (2)solenoid, (3)separation chamber (filled by steel balls as matrix), (4)discharge trough.

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In order to control the experimental conditions, firstly the magnetic field strength inside the separation chamber was measured against intensity of the coils' electrical current. Figure 2 displays the effect of electrical current on magnetic field intensity.

For this survey, a portable Hall effect gaussmeter (model TLMP-Hall-15 of Globalmag) was employed with its standard probe (section of 5 mm x 1.5 mm) measuring fields from 0 to 1.5 teslas. This gaussmeter has accuracy of ± 0.0010 tesla. The gaussmeter probe was positioned in the transverse (horizontal) plane of the separation chamber during measurement.

Magnetic field intensity in the transverse plane of symmetry of the separation chamber was also surveyed, in order to characterize the isovalues lines of the magnetic field inside the equipment. Figure 2 displays this spatial distribution in case of electric current of 1.0 A and tension of 12 V. As can be seen, in this case only a small zone resulted in magnetic field values greater than 0.1 T.

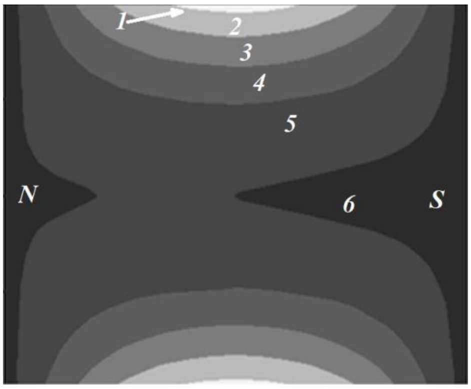

Figure 2 – Visualization of magnetic field in the transverse plane of symmetry. Legend: 1 — below 0.02 T; 2 — between 0.02 T and 0.04 T; 3 — between 0.04 T and 0.06 T; 4 — between 0.06 T and 0.08 T; 5 — between 0.08 T and 0.10 T; 6 — above 0.10 T.

Nonlinear regression analysis of magnetic field intensity (in teslas) versus electric current (in amperes) data using the EasyPlot package has revealed excellent data adherence, with a sigmoidal curve showed below.

$$H = k \times \left(1 - \exp\left(\ln\left(\frac{1}{2}\right) \times \left(\frac{i}{i_{50}}\right)^{n} \right) \right) + H_0 \quad (7)$$

Table 2 summarizes the regression values for the curves in the absence and in the presence of matrix.

Table 2 – Calibration curves for the magnetic separator.

Parameter [unit]	Without matrix	With matrix
Standard error [T]	0.0037	0.0193
Maximum deviation [T]	0.0062	0.0273
Coefficient of determination (R^2) [-]	0.9996	0.9966
Scaling coefficient: k [-]	0.581	0.978
Sharpness index: n [-]	1.358	1.390
Median current: i_{50} [A]	2.77	2.22
Residual field intensity: H_0 [T]	0.0207	0.0074

The magnetic separator feed was slurry with solid mass concentration of 30.0 %. The solids were both raw mix and products of previous roasting stages, in order to assess the effect of reductive roasting at five different temperatures. Washing was done spraying 250 mL of water from a wash bottle during the course of each experiment, in order to get rid of non-magnetic entrapped impurities.

3 RESULTS AND DISCUSSION

3.1 Sieving, Chemical and Mineralogical Characterization

As the raw samples were in coarse particle size range, they were comminuted to simulate difficult-to-treat fines for magnetic separation process. The samples were fragmented 100 % below 106 µm. Comminution had encompassed three stages using jaw crusher, roll crusher and disc grinding mill (the latter in closed circuit). The procedure was carried out in duplicate. The average size distributions for iron ore and coke after comminution are presented in figure 3.

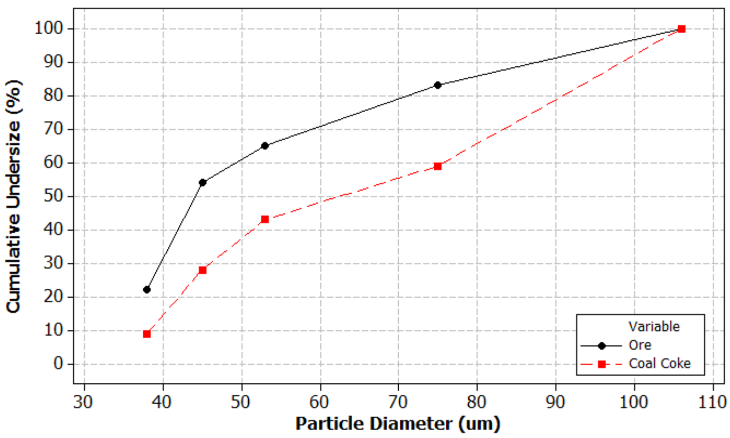

Figure 3 – Size determination by sieving.

Sample densities for both raw iron-bearing ore and coal coke were determined employing a helium pycnometer model Ultrapyc 1200e (from Quantachrome Instrument). The theoretical overall density of the mineral sample was calculated taking into account the volumetric fractions and experimental densities for each size range, in order to check the measurement accuracy.

The thermogravimetric analysis has indicated mass loss of 0.183 percentage point between 473 K (200 °C) and 573 K (300 °C) due to goethite-hematite phase transformation. The release of water in the reaction of pure goethite represents 10.14 % of mass loss. The unroasted sample has a goethite content of 1.8 %. The thermogram of roasted and unroasted samples is presented in figure 10 ahead.

Table 3 – Gas picnometry.

	Size range [mm]	Mass Fraction [%]	Density [kg/m³]	Content Hematite (Fe_2O_3) [%]	Content Iron (Fe) [%]	Content Quartz (SiO_2) [%]
Coke	+ 0.00 - 6.35	Overall analyzed	1,510	-	-	-
Iron Ore	+ 0.00 - 6.35	Overall analyzed	4,550	87.00	60.90	13.00
	+ 3,35 - 6.35	25.13	4,430	83.78	58.65	16.22
	+ 2.36 - 3.35	22.20	4,660	89.71	62.80	10.29
	+ 1.70 - 2.36	22.40	4,670	90.08	63.06	9.92
	+ 1.18 - 1.70	8.83	4,680	90.25	63.18	9.75
	+ 0.85 - 1.18	6.13	4,800	93.17	65.22	6.83
	+ 0.30 - 0.85	3.27	4,940	96.50	67.55	3.50
	+ 0.075 - 0.30	6.18	4,600	88.30	61.81	11.70
	+ 0.00 - 0.075	5.87	4,440	83.91	58.74	16.09
	+ 0.000 - 6.350	Overall calculated	4,600	88.45	61.92	11.55

3.2 Roasting Tests and Magnetic Separation

Figure 4 shows that time and coal proportion did not have a great influence on the yield under 0.06 T fields, while temperature has shown a significant effect. Figure 5 illustrates the coke influence on roasting and the influence of roasting time is shown in figure 6.

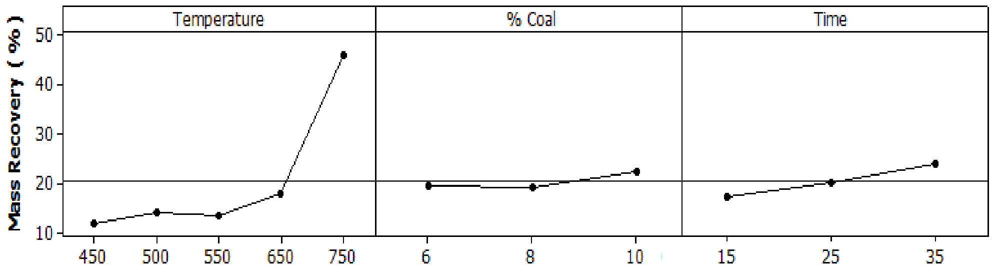

Figure 4 – Influence of parameters at 0.06 T.

The temperature proved to be the main variable in roasting process as can be seen in figure 7. Temperatures below 1,023 K (750 ºC) have not increased magnetic susceptibility (after cooling), while at 1,023 K (750 ºC) there was increase of the sample's magnetism, leading to yield increase. In view of these results, experiments at higher temperatures were not performed, since at 1,230.0 K (750 ºC) the resulting cold material exhibited high magnetic susceptibility, allowing high recovery values even by magnetic separation under low field gradient .

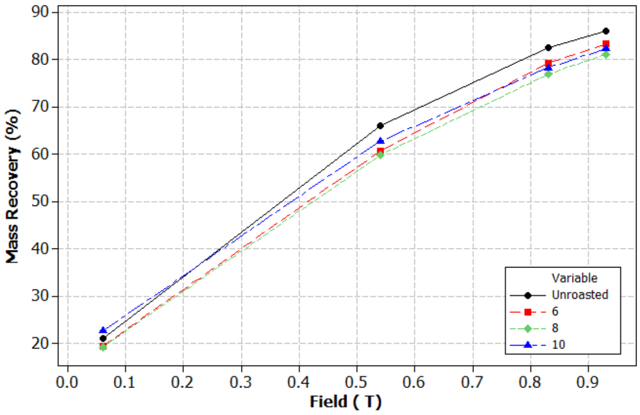

Figure 5 – Influence of coke percentage.

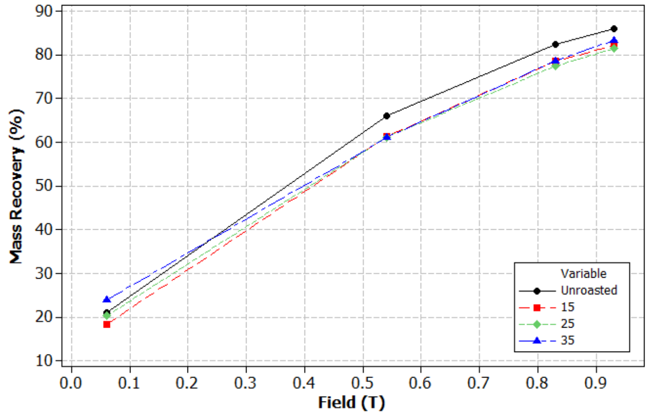

Figure 6 – Influence of time.

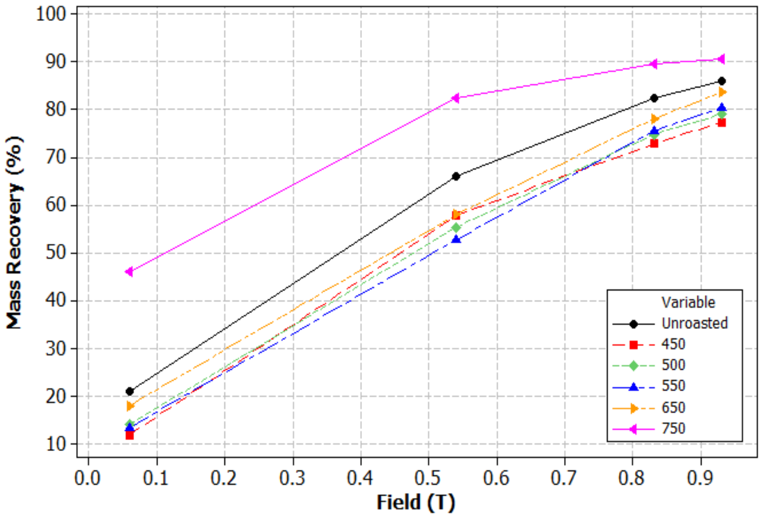

Figure 7 – Influence of temperature on yield.

The tests at 1,023 K (750 °C) are shown in figure 8. The best result (750 °C — 35 minutes — 10 % coke) provided a yield 240.3 % higher than the natural ore under lower field intensity (0.06 T). In turn, the field of 0.93 T has provided corresponding gain of 5.3 %.

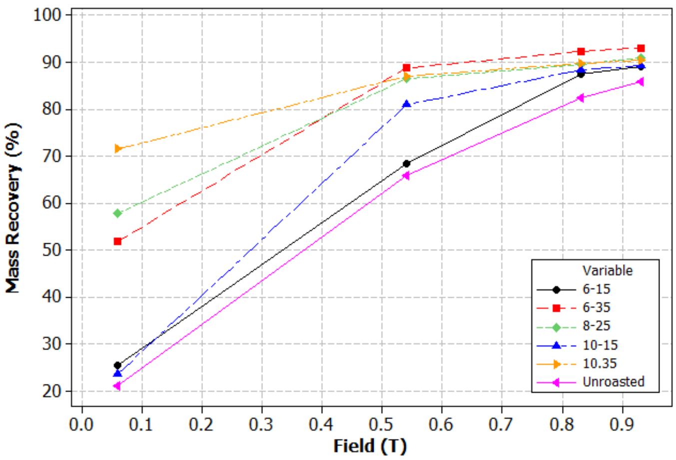

Figure 8 – Effect of coke proportion on yield for 750 °C.

Roasting allows a decrease in the magnetic field needed for the separation process as can be seen in figure 10. To reach 72 % yield in the natural ore, intensity of 0.65 T is required, while this recovery can be reached under field intensity of about 0.06 T for the roasted ore. Recovery relative gains under lower magnetic fields are higher, indicating roasting process (obviously after cooling step) acts as a magnetic booster for iron-bearing minerals.

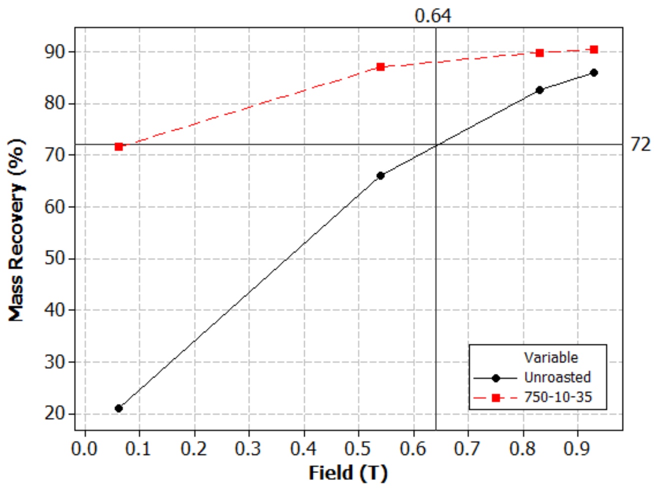

Figure 9 – Mass recovery improvement during roasting.

The identification of phase transformation is possible comparing the thermogram obtained by thermogravimetric analysis of roasted and unroasted products. The thermogram of Figure 10 shows a peak between 473 K (200 ºC) and 573 K (300 ºC) corresponding to the goethite to hematite transformation. On the other hand, the phase transformation of hematite to magnetite can be seen between 873 K (600 ºC) and 973 K (900 ºC). The characteristic peak of goethite/hematite phase transition can be clearly seen on the curve corresponding to the non-roasted material (dashed curve 2), in contrast to the curve of the roasted sample (dashed curve 1). In turn, there is no peak of magnetite/hematite shift in unroasted sample, but it is very pronounced in roasted sample in the aforementioned range.

The thermogram analysis indicates that mass loss due to hematite transformation into magnetite is 0.754 percentage point and corresponds to a magnetite content of 21.8 %. The increase of magnetic product in the separation process was due to this phase transformation.

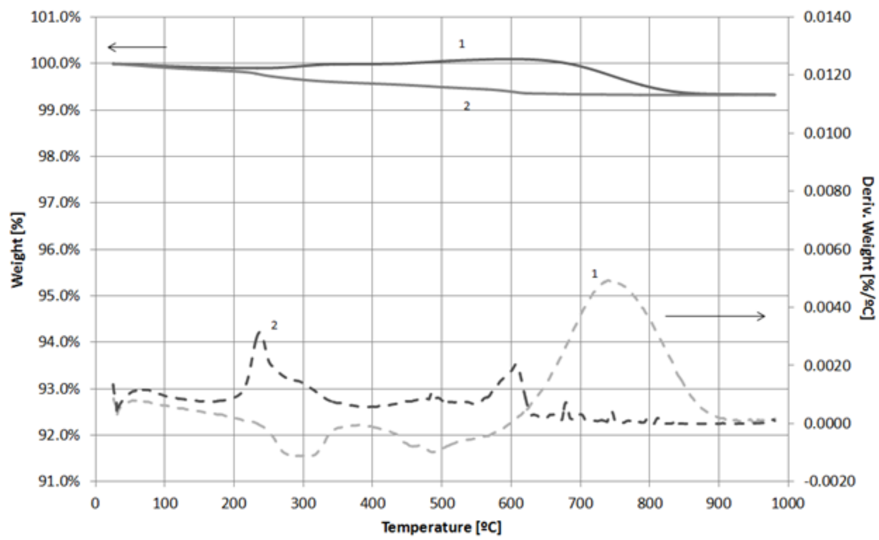

Figure 10 – Thermogram of roasted (1) and unroasted sample (2) (dashed line: derivative of mass loss; solid line: mass loss).

Figure 11 visually illustrates the effect of reductive roasting. The magnetic force lines were materialized by depositing a plastic dish with a few grams of roasted material on a neodymium-based magnet.

Figure 11 – Magnetic roasted fines over a strong magnet (scale divisions in millimeters).

4 CONCLUSION

The characterization pointed a sample with 87.0 % hematite, which makes this sample rich at approximately 60.9 % iron. Particle size analysis showed fine particles in sample. Magnetic separation tests reached satisfying results only for temperatures of 1,023 K (750 ^{o}C). The best result obtained was with 10.0 % of coke in the raw mixture and during the longest roasting time (35 minutes), which shows that the raise of the two variables are highly benefic to the roasting process, provided the temperature is enough to trigger the targeted chemical reactions. The roasting process raised the yield in 5.3 % under 0.93 T and 240.3 % under 0.06 T. Through roasting, it's possible to obtain the same recoveries in fields around 10 times lower, recommending the application of this technique for iron ore processing, naturally after careful economic analysis, in which the quantification of environmental liabilities (primarily resulting from its non-implementation) is included. Thermogravimetric analysis has proved that hematite to magnetite transformation improved the performance of the selective magnetic separation.

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