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Bench-scale calcination and sintering of a goethite iron ore sample

M.S.S. Silva [a], M.M.F. Lima [b], L.M. Graça [c], R.M.F. Lima [a,*]

[a] Department of Mining Engineering, Federal University of Ouro Preto, Campus Morro do Cruzeiro, CEP: 35400-000, Ouro Preto, Minas Gerais, Brazil
[b] Department of Control and Automation Engineering and Fundamental Techniques, Federal University of Ouro Preto, Campus Morro do Cruzeiro, CEP: 35400-000, Ouro Preto, Minas Gerais, Brazil
[c] School of Mines, Federal University of Ouro Preto, Campus Morro do Cruzeiro, CEP: 35400-000, Ouro Preto, Minas Gerais, Brazil

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ABSTRACT

This work presents the results of bench calcination and sintering studies conducted on an iron ore sample from Iron Quadrangle, Brazil with high goethite content. The natural samples and the products of calcination and sintering were characterised by inductively coupled plasma optical emission spectroscopy (OES/ICP), X-ray spectrometry and gravimetric methods. Optical microscopy, X-ray diffraction (XRD) and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDS) were used to identify the phases in the studied samples. Density, specific surface area, specific pore volume and pore diameter were determined through gas pycnometry. All of the natural sinter feed samples had a Fe grade >64% and a very high phosphorus grade (~0.17%). After calcination, a 3.1–3.4% increase was observed in the Fe grade compared to that of the natural sinter feed samples. The average Fe grade of the sintered samples was 59.1%. The identified phases with XRD in the raw materials were hematite and goethite. In the calcined samples, only hematite was identified because of the thermal decomposition of goethite. The density, specific surface area, specific pore volume and pore diameter of the calcined samples increased compared to those of the natural sinter feed samples. Hematite, brownmillerite, anorthite, and gehlenite were identified in the sintered samples. The sites for phosphorus occurrence were calcium silicates and apatite. The sintered samples exhibited specific surface areas lower than those of the calcined samples. This result was ascribed to the destruction of the pore structure by the sintering process. No relationship between the proportions of nucleate, intermediate and agglomerate particles used in mixture of sinter tests with the results of microtumbler was identified.

1. Introduction

The total world resources of iron ore (indicated + inferred) are 170 billion tonnes. Approximately 12% of these resources, with an average Fe grade of 50.3%, are located in Brazil and are distributed among the following states: Minas Gerais — 70% (46.9% Fe), Mato Grosso do Sul — 15.3% (55.4% Fe), Pará — 13.1% (64.8% Fe) and others — 1.6% (Jesus, 2013).

More than 90% of the total world's iron ore production is used in the steel industry (blast furnace and direct reduction) (Wolff, 2009). Iron ore with a Fe grade less than 64.0%, such as the actual ores of Minas Gerais and Mato Grosso do Sul, must be concentrated to achieve satisfactory specifications. The choice of beneficiation route for a specific iron ore depends on its mineralogical composition, Fe and SiO_2 grades and size distribution (Araujo et al., 2003; Al-Wakeel and El-Rahman, 2006).

Most of the iron ores from Iron Quadrangle, Brazil are related to Proterozoic banded iron formations (BIFs) of the Cauê Formation (Carlos et al., 2014). The mineralogy of these ores is basically constituted by quartz and iron oxide minerals. In high-grade iron ores, hematite is the main iron mineral. The iron ore deposits of Iron Quadrangle were subjected at least to two metamorphism and deformation events. The metamorphic and deformation intensity increase from west to east and from south to north in the region. These processes beyond the supergene processes were the main factors that changed the texture of the iron ore of this region. In general, the mineral crystal size increases with metamorphism degree (Rosière, 1996; Mendes and Lagoeiro, 2012).

In Brazil, iron ore with a high grade of goethite (FeOOH) (LOI > 3.5%), independent of its Fe grade, is considered marginal because goethite causes excessive mud generation in the wet processing step, which is very harmful in flotation (slime coating phenomena) and filtration (blinding) operations. In agglomeration processes (pelletising and sintering), high goethite contents affect both the process control and the final quality of agglomerate (pellet or sinter) (Magalhães et al., 2007; Loo et al., 1994; Yang et al., 2000; Okazaki et al., 2003; Leonel, 2011).

* Corresponding author at: Departamento de Engenharia de Minas, Universidade, Federal de Ouro Preto, Campus Universitário, Morro do Cruzeiro, S/N, CEP: 35400-000, Ouro Preto, MG, Brazil.
E-mail address: rosa@demin.ufop.br (R.M.F. Lima).

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Loo (2005) reviewed several studies conducted with mix sinter of iron ore containing a high proportion of goethitic (pisolitic) ore; the first of these studies was conducted in Australia in 1990. Although the high porosity and low density of goethitic ore increase its melt volume during sintering, which causes deterioration in granulation and low productivity, among other problems. Operation control (e.g. the addition of more water during granulation, the use of a faster flame front speed than that used in a sinter bed with a denser ore, maintenance of the green bed permeability during the sintering operation and other factors) can provide good performance for a sinter mix with a high proportion of goethite ores.

Detailed technological characterisations (i.e. physical, mineralogical and chemical characterisations) and specific technological experiments such as bench calcination and sintering of Brazilian iron ores with high goethite contents must be conducted to predict the behaviour of such ores in industrial processes. Such predictions would facilitate modifications to the actual beneficiation routes, which could support the rational use of these ores. This information will be very important for increasing the lifetimes of the mines located in the Iron Ore Quadrangle region.

This work presents the results of bench calcination and sintering studies performed with a natural iron ore sample with high goethite content from the Iron Quadrangle, Brazil; the samples contained different proportions of nucleating, intermediate and adherent particles.

2. Experimental

2.1. Materials

The raw iron ore sample (52 kg) preparation was constituted by comminution by jaw and roll crushers to −3.36-mm size. This size was determined based on work carried out by Umadevi et al. (2011), who studied the influence of iron ore fines (mean particle size from 1.22 to 3.95 mm) on mineralogy, productivity, physical and metallurgical properties of the sinter. The comminuted ore, after being homogenised, was split into sub-samples for the physical, chemical and mineralogical characterisations. For calcination and sintering tests, the sample was size classified by wet sieving in the following fraction sizes: −3.36 + 1.182 mm (nucleating particles), −1.186 + 0.209 mm (intermediate particles) and −0.209 mm (adherent particles), which were mixed in different proportions (Table 1).

The higher proportion of nucleating particles (70%) compared to adherent particles (30%) in SF1 aimed to intensify the granulation of mixture and, consequently, it could directly affect the permeability of bed sintering. The substitution of 15 wt.% of nucleating particles and adherent particles by intermediate particles, respectively in SF2 and SF3, was based on practise of industrial plant of sintering, which uses the maximum of 15 wt.% intermediary particles. This is done because intermediate particles do not have defined behaviour like nucleating or adherent particles. The higher proportion of adherent particles (70%) compared to particles nucleating (30%) in SF4 aimed to assess the main mechanisms of granulation using fine particles in mixture of sintering, which provide the increase in bed of sintering (Silva, 2014).

Table 1
Constitutions of the natural sinter feed samples.

Natural sinter feed sample	Proportions of particles (wt.%)		
	Nucleating (−3.36 + 1.182 mm)	Intermediate (−1.182 + 0.209 mm)	Adherent (−0.209 mm)
1	70	…	30
2	55	15	30
3	30	15	55
4	30	…	70

Table 2
Chemical composition and ignition loss (LOI) of the samples studied: natural sinter feed (SF), calcined sinter feed (C) and sinter (S).

Chemical composition (wt.%)	Sample				
	Type	Condition			
		1	2	3	4
$Fe_{(T)}$	SF	64.10	64.20	64.40	64.60
	C	67.58	67.60	67.65	67.72
	S	58.70	58.55	59.96	59.22
FeO	SF	0.20	0.20	0.20	0.20
	S	0.93	0.74	0.38	0.42
SiO_2	SF	1.34	1.39	1.36	1.29
	C	1.26	1.36	1.36	1.19
	S	2.02	1.40	1.41	1.26
Al_2O_3	SF	0.72	0.73	0.76	0.77
	C	0.84	0.83	0.94	0.92
	S	0.89	0.74	0.88	0.82
CaO	SF	0.04	0.04	0.03	0.03
	S	12.42	12.51	11.37	12.50
MgO	SF	0.03	0.04	0.04	0.04
	C	0.06	0.06	0.07	0.06
	S	0.15	0.18	0.15	0.15
TiO_2	SF	0.05	0.05	0.06	0.06
	C	0.07	0.07	0.08	0.08
	S	0.06	0.05	0.07	0.06
P	SF	0.17	0.17	0.16	0.16
	C	0.17	0.17	0.17	0.15
	S	0.17	0.18	0.16	0.15
Mn	SF	0.06	0.06	0.06	0.06
	C	0.07	0.06	0.07	0.06
	S	0.06	0.06	0.06	0.06
LOI	SF	6.11	6.07	5.67	5.46

2.2. Methods

2.2.1. Calcination and sintering tests

For calcination tests, approximately 20 g of each sinter feed sample (SF1, SF2, SF3 and SF4) was placed in an alumina crucible that was previously weighed. The crucible with the sinter feed sample was again weighed and introduced into a furnace (FortLab, model ML 1300).

The run conditions of the calcination tests were as follows: natural atmosphere (air), ramp of 10 °C/min from 25 to 700 °C and an isotherm of 35 min at 700 °C, as previously used by Silva (2014); the tests were conducted on a TA Instruments model TGA Q50 thermogravimetric analyser. After the calcination process, the crucible with the calcined sample was removed from the furnace and cooled in a normal laboratory environment until it reached room temperature (~25 °C). The crucible was then reweighed to determinate the sample weight loss. This procedure was performed three times for each sinter feed sample to produce samples of sufficient mass for the characterisation (physical, chemical and mineralogical) and sintering tests.

For the sintering tests, the calcined sinter feed samples (C1, C2, C3 and C4) were mixed with lime (CaO) and activated charcoal in the following proportions: 80 wt.% calcination product, 19.35 wt.% CaO and 0.65 wt.% charcoal. Because of the intrinsic characteristics of this iron ore sample, water was carefully added to the mixture during the granulation step to avoid deformation of the sintering bed (Sakamoto et al., 1993; Sakamoto et al., 1997). The control was done by visual inspection. Then, the granulated mixture (approximately 100 g) was placed over a refractory brick and sintered in the same furnace used in the calcination tests. After the sintering process, the samples were cooled in the furnace under natural atmosphere until they reached environmental temperature (~25 °C).

The run conditions for the sintering tests were as follows: i) natural atmosphere (air), ramp of 5 °C/min from 25 to 300 °C, isotherm of 10 min at 300 °C; ii) ramp of 5 °C/min to 700 °C, isotherm of 10 min at 700 °C; iii) ramp of 10 °C/min to 1000 °C, isotherm of 10 min at 1000 °C; iv) ramp of 10 °C/min to 1200 °C, isotherm of 10 min at