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ORIGINAL PAPER

Iron recovery from the waste generated during the cutting of granite

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Abstract Metallic iron is present in the waste left when granite blocks are cut. Thus, the purpose of this study was to characterize this waste using chemical and particle size analyses. To achieve this, X-ray diffraction and scanning electron microscopy coupled with electron back-scattered diffraction were used. To find the method with the best metallic iron recovery from the waste of ornamental rock, three distinct methods were examined: magnetic separation, table concentration and cyclone processing. The first method involved three steps: (1) use of a wet high-intensity magnetic separator, where only the equipment's remaining magnetic field was present; (2) the material from the first step was then submitted to separation again, this time using a magnet for rare earth particles; and (3) this material after two separation processes was finally submitted to ferromagnetic separation. The second method used a concentration table set at various inclinations, oscillation frequencies and wash flow rates. Meanwhile, for the third method, the cyclone tests, only the water pressure was varied. After each test, a chemical analysis was performed to determine the metallic iron present in each sample. The tests revealed that magnetic separation presents the best results. Using this technique, a ferrous concentrate with 93 % metallic iron content and a granite concentrate with only 0.6 % metallic iron were obtained. On the other hand, in the table concentrator tests, the ferrous concentrate only had a metallic concentration of 13.6 %. In separation by the cyclone processing, the product barely contained metallic iron (7.2 % maximum).

Keywords Granite waste · Solid waste · Recycling · Waste management

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Introduction

The industrial activities involving ornamental rocks involve the extraction, cutting and polishing of rocks such as granite, marble, slate, gneiss and quartzite (Souza et al. 2010).

However, these activities generate solid wastes that are becoming a serious problem for the industry and the environment (Acchar et al. 2006; Nouri et al 2012).

Ornamental rock extraction usually involves three steps. The first is to extract the blocks, which come in various sizes, from the quarry. Then, the blocks go through primary processing where a slab-cutting machine transforms them into slabs of a predetermined thickness. In the final step, a polishing machine gives the finishing to the slab, which is now a final product.

During the cutting step, it is estimated that approximately 20–25 % of the total granite block is lost (Saboya et al. 2007; Vijayalakshmi et al. 2013). In Brazil, in 2007, approximately 1.8 million tons of wastes was generated by this sector (Silva et al. 2011). In this step, the waste is composed of water, lime, steel shot and rock particles. After drying, this residue becomes dust, and it is classified as hazardous waste because of the amount of soluble iron. Generally, it is deposited in outdoor piles or in rivers, without any type of treatment (Silva et al. 2011; Binicia et al. 2008). The presence of iron in the granite waste is a consequence of the cutting process, where steel shots, a source of iron, are frequently added during this stage.

According to Seifelnassr et al. (2013), the most commonly used beneficiation methods for iron ores are the gravity and magnetic separation techniques.

Magnetic separation is an important beneficiation process applied in a lot of types of ore, such as manganese, nickel and iron (Dwari et al. 2013). This is a method to separate and capture fine magnetic particles by the magnetic force acting on the particles in a gradient magnetic field (Satoshi 2002). Gravity concentration has been accepted as a low-cost and an environmental-friendly process for the separation of minerals (Richards et al. 2000). The gravimetric separation is the result of relative movement between two or more minerals in response to gravity and one or more other forces (Burt 1999). However, particle gravity separation is difficult for particle sizes below 0.100 mm (Galvin et al. 2010; Traore et al. 1995).

Thus, to perform this research, the ornamental rock dust came from state of Espírito Santo, Brazil, between years 2008 and 2010. The purpose of this paper was to carry out the characterization of the ornamental rock dust and iron recovery from that dust using three different methods: magnetic separation, concentration table and cycloning.

Materials and methods

The granite waste used in this study came from the slab-cutting process, where steel shots were used. The sample was taken from a typical cutting company. The sample weighed around 300 kg and was stored in barrels to avoid loss or contamination. Before beginning its metallic iron recovery, the sample was characterized.

Waste characterization

To perform the characterization, a portion of the sample was chosen and dried on a heated plate. With drying, the sludge turned into powder, which, in turn, was submitted to the conical pile quartering method. This involved the quartering of a conical pile of powder until homogeneous portions of 20 g were obtained.

Characterization then began and involved the quantification of the metallic iron content percentage, together with the other minerals present, such as SiO_2, Al_2O_3, CaO, K_2O, Na_2O, MgO and Fe_2O_3. Besides this, the material was also subjected to X-ray diffraction, particle size distribution analysis and scanning electron microscopy.

Chemical analysis

The metallic iron content was measured by volumetric analysis, and the SiO_2, Al_2O_3, CaO, K_2O, Na_2O, MgO and Fe_2O_3 concentrations were determined by X-ray spectrometry.

X-ray diffraction

The objective of using X-ray diffractions was to determine the principal phases present in the granite waste. For this purpose, a Philips piece-of-equipment, model MPD 1880, with a copper-alpha radiation of Kα (λ = 1.5418A), having 40 kV and 40 mA of power, was employed.

Scanning electron microscopy

For the scanning electron microscopy, a Philips Model XL-30 device equipped with an electron back-scattered diffraction detector was used.

The analyzed samples were in powder form and as such were mounted on double-faced tape. As the granite waste does not conduct electrons, it was necessary to coat the samples with conduction material; in this case, gold was used.

Particle size distribution

The particle size distribution analysis used two methods. In the first method, sieves having the openings (mm) 0.217, 0.150, 0.105, 0.075, 0.053 and 0.044 (65, 100, 150, 200, 270 and 325 mesh) were used. Sample mass weight was 2,205 g. Volumetric chemical analysis was performed to quantify the metallic iron content of each obtained fraction.

In the second method, a Malvern Mastersizer 2,000 equipment was employed, since it uses laser diffraction to define the material's particle size distribution. The device contains a system of infrared light detectors that identify frontal, lateral and rear scattering. The source of the infrared light was a helium neon laser whose wave length defines the size range of the particle being measured.

Sample preparation

Before beginning the metallic iron recovery tests using magnetic separation, table concentration and cyclone processing, it was necessary to prepare the samples. Sample quartering was necessary in the first two methods, but in the cycling process, this was not necessary, since the whole remaining sample was used.

Initially, sample quartering was performed to obtain a homogeneous sample. Then, the humidity of each sample was determined with an infrared measurer. Finally, the samples were distributed among the different processes.

Samples were not dried in the recovery tests. All tests were performed using the raw material. Consequently, a pulp containing 70 % of solids was used.

Concentration tests

In this study, three types of equipment were used, one for each type of the previously mentioned metallic iron recovery methods.

Magnetic separation

The magnetic separation test was divided into three stages. In the first stage, the test was performed on the pulp with a wet high-intensity magnetic separator that used a ferromagnetic matrix of the Jones type. In this stage, only the equipment's remaining magnetic field was used.

To obtain this remaining magnetic field, the device was left functioning for 10 min and turned off. At this point, only a low-intensity magnetic field remained and was used for the sample's magnetic separation.

The magnetic fraction obtained in the first magnetic separation was submitted to the second stage, while the non-magnetic fraction was dried on a heated plate and quartered for volumetric chemical analysis to determine the metallic iron content. This non-magnetic material was denominated as *granite concentrate*.

In the second stage, the magnetic material from the first stage was submitted to another separation processed with a rare earth magnet.

Finally, the material obtained from the second stage was submitted to separation with a standard iron magnet and was denominated as *ferrous concentrate*.

The ferrous concentrate obtained was dried on a heated plate, weighed and quartered, so that a chemical analysis with the volumetric method could be performed to determine the metallic iron content.

Table 1 Concentrator table parameters

Test	Table inclination	Oscillation frequency	Water flow (L/min)
1	7.7°	30	4
2	7.7°	50	
3	5.3°	30	
4	7.7°	30	8
5	7.7°	20	

Concentration table

In another test, a portion of the pulp was transferred to the concentrator table's feeder to initiate the homogenization process that took 5 min. Homogenizing was performed by a device coupled to the concentrator table that had a motor with a propeller mounted to the shaft end. The objective of installing the feeder on the table was to store the waste and feed the table at a constant flow. The homogenizer had the function of not letting the waste precipitate to the bottom of the feeder. The concentrator table parameters can be seen in Table 1.

At the end of each test, the product was dried on a heated plate, weighed and then quartered according to the pile method to obtain homogeneous portions for determining the metallic iron content.

Cycloning

To perform the cyclone processing, a device was used, whose dimensions are demonstrated in Fig. 1.

A sample of the pulp was transferred to the feed box of the cyclone, where it was homogenized by two mechanisms. The first was the flux from the return of the

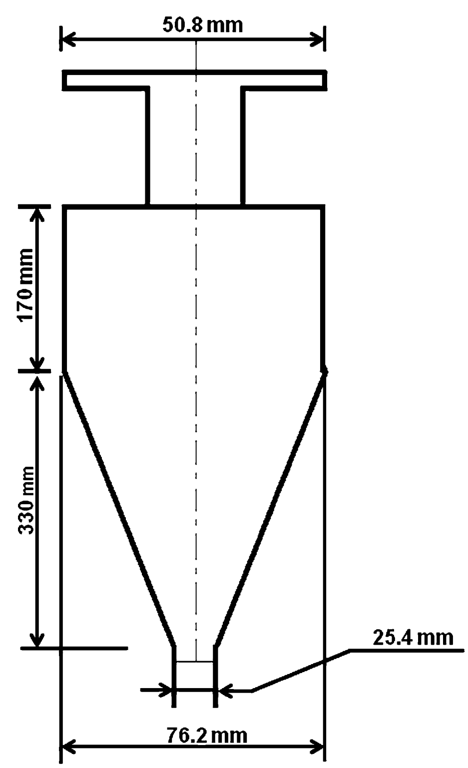

Fig. 1 Dimension of the cyclone used in the test

underflow and overflow, and the second was the return flux caused by the cyclone's feeder pump.

At the cyclone's underflow and overflow outlets, hoses were adapted so that the flux from each one returned to the feed box, creating a continuous system.

The homogenizing time in the feed box was 10 min, after which the cyclone processing began.

At the end of each test, the pulp was homogenized for 5 min before it was newly removed under a variety of parameters. In the tests, the only variation was the feed pressure. To measure this, a pressure gauge was installed at the entrance of the equipment. Table 2 presents the values of the parameters used in the cyclone processing tests.

At the end, the samples obtained from the under- and overflow were sent to the same procedures as the previous test.

Results and discussion

Characterization of granite waste

Chemical analysis

The results obtained by the chemical analysis of the granite waste sample are presented in Table 3.

From Table 3, it can be observed that the main component of the granite waste is SiO_2 with 65.9 wt%. Noticeable also is the presence of other elements of lesser proportion in relation to SiO_2, for example, Al_2O_3, CaO and Na_2O. According to Menezes et al. (2005), the granite waste is mostly composed of SiO_2, Al_2O_3, Fe_2O_3 and CaO. The sodium and potassium encountered in the residue's composition could have originated from the feldspar found in granite (El-Taher 2010). The metallic iron present is from the steel shot used in the cutting process (Torres et al. 2009). According to Moreira et al. (2008), the high level of SiO_2 and Al_2O_3 content of approximately 79.3 % is typical of metamorphological rocks.

A majority of the granite waste constituents are expressed in the form of oxides, such as (SiO_2) and alumina (Al_2O_3), followed by lime (CaO) and the alkaline oxides (Na_2O and K_2O) (Segadães et al. 2005; Menezes et al. 2005).

Table 2 Cyclone processing parameters

Test	Vortex diameter (mm)	Apex diameter (mm)	Pressure (kPa)
1	50.8	25.4	24.5
2			14.7
3			9.8
4			4.9
5			0

Table 3 Chemical analysis of the dried granite waste

Elements	Mass (%)
SiO_2	65.9
Al_2O_3	13.4
Fe_2O_3	1.4
MgO	1.0
CaO	4.2
Na_2O	2.6
K_2O	4.4
Fe	4.8
Loss on ignition	2.3

X-ray diffraction

Figure 2 presents X-ray spectrum of the studied granite waste sample.

In Fig. 2, the peaks for quartz (SiO_2), albite ($Na(AlSi_3O_8)$), orthoclase ($K(AlSi_3O_8)$) and muscovite ($(K,Na)(Al,Mg,Fe)_2(Si_{3,1}Al_{0,9})O_{10}(OH)_2$) can be observed.

The albite and orthoclase belong to the feldspar mineral family (El-Taher 2010; Ângulo et al. 2009). Muscovite is a mineral derived from micas (Hojamberdiev et al. 2011; Bennadji et al. 2008).

Scanning electron microscopy

Figure 3 presents the image obtained by scanning electron microscopy of the granite waste. Observed were two different points represented by A and B, respectively. Point A is metallic iron, which comes from the steel shot. It was added in the preparation of the abrasive pulp at the cutting-stage slab. Point B is predominantly composed of Si. However, other elements were found in point B, such as K, Al, Mg, O and Na. The K, Al, Mg and Na peaks are due to the albite, orthoclase and muscovite.

Particle size distribution

Table 4 presents the particle size distribution results for the granite waste. It also presents the metallic iron content present in each fraction.

Notice that in Table 4, a greater part of the particles of the granite waste (82.6 %) is <0.044 mm in size and only 15.1 % of the metallic iron present in the granite waste is above 0.105 mm. Similar results were obtained in Vieira et al. (2004). The authors said that only 14 % of the particles retained were of the 0.044 mm size.

Table 4 also shows the metallic iron is uniformly distributed in all the particle size levels, except in the smallest

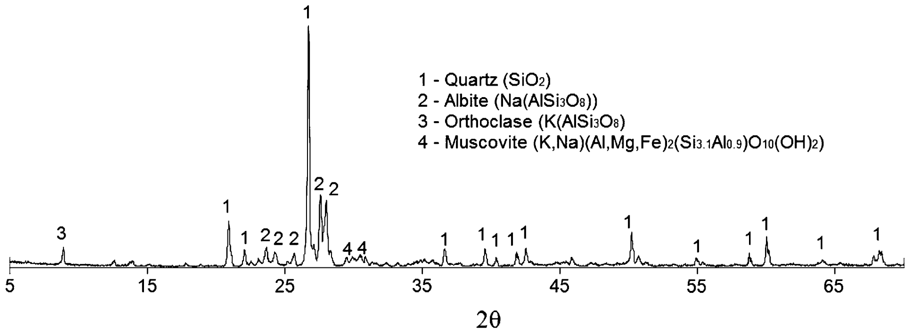

Fig. 2 X-ray spectrum of the granite waste

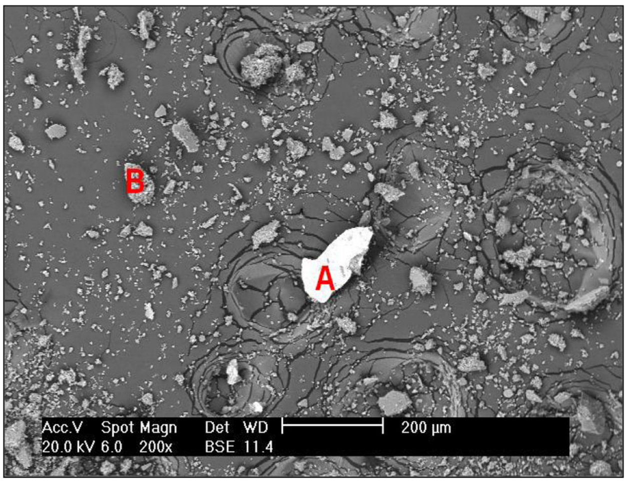

Fig. 3 Image of the back-scattered electrons in the granite waste

opening where the observed content is 2 %. In spite of the low quantity of iron in the finest portion of the residue, it is the fraction with the greatest iron quantity in weight, around 35.69 %. As such, it is impossible to concentrate the iron for particle size classification.

Figure 4 presents the particle size distribution performed with the Mastersizer 2,000 equipment.

The mean particle size of the waste is situated between 0.50 and 563.67 µm, and 4 % is above 100 µm. These values are in accordance with those obtained by the particle size distribution performed with sieves.

Metallic Fe recovery

Magnetic separation

To perform the magnetic separation tests, a wet high-intensity magnetic separator was initially used, in which only the remaining magnetic field was used.

The concentrate granite obtained represents 92 % of the initial sample. This granite concentration presented a 0.6 % metallic iron content.

After the first magnetic separation stage, the magnetic material obtained (which corresponds to 8.2 % of initial mass of the granite waste) was ready for the second stage that used a rare earth magnet.

To finalize the magnetic separation test, this material was then submitted to another separation process, this time with a Fe magnet. In this step, the concentrated iron obtained represents 4.5 % of the initial sample. This iron concentration had a metallic iron content of 93 %.

Considering that the magnetic fraction contains a metallic iron concentration of 93 % and the mass of magnetic fraction is 4.6 % of the initial granite waste mass, an iron recovery of 88 % was observed. On the other hand, a granite concentrate was produced, which contains only 0.6 % of metallic iron.

Concentration table

Tests were performed at the concentration table, which was adjusted to attend parameter variations. That is, its inclination angle and water flow were varied. The vibration amplitude remained constant. Table 5 demonstrates the values obtained at this concentration table.

In magnetic separation, it was possible to obtain an iron concentration of 93 %, while the concentration table produced an iron concentrate that had only 13.7 % of metallic Fe. This value was obtained when the inclination was 7.7°, the oscillation frequency was 30 per minute, and the water flow was 4 L/min.

When the oscillation frequency increased from 30 to 50 oscillations per minute, the metallic iron content in the iron concentration diminished from 13.7 to 11.4 %. The same fact occurred when the table's inclination angle was decreased. Increasing the water flow also hindered metallic iron retrieval from the granite waste.

In the tests performed with the table concentrator, it was only possible to retrieve 4 % metallic iron from the granite concentrate.

According to Manser et al. (1991), increasing water flow affects the material, principally that of lesser density. The

Table 4 Particle size distribution of the granite waste

Sieves (mm)	+0.217	−0.217 +0.150	−0.150 +0.105	−0.105 +0.075	−0.075 +0.053	−0.053 +0.044	−0.044
Retained residual mass (g)	13.08	18.49	50.84	75.30	134.40	92.22	1,820.90
% retained residue	0.6	0.8	2.3	3.4	6.1	4.2	82.6
% metallic Fe in each fraction	12.1	14.7	22.9	22.9	17.0	14.4	2
Metallic Fe mass (g) in each fraction	1.58	2.72	11.63	17.22	22.83	13.26	35.69
Accumulated % retained metallic Fe	1.5	4.1	15.1	31.6	53.3	66.0	100

Fig. 4 Particle size distribution of the granite waste

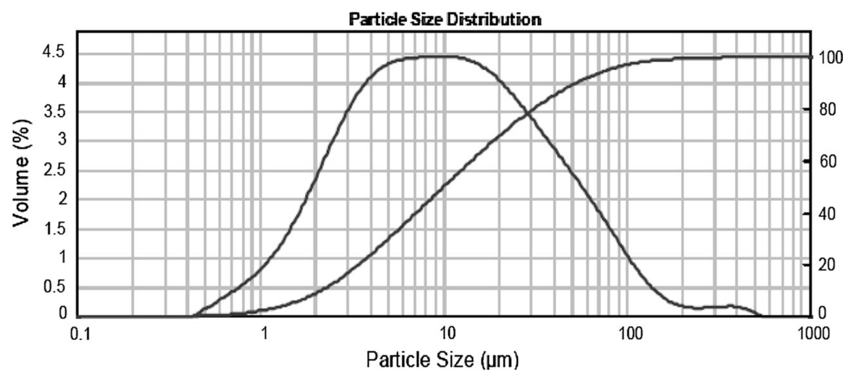

Table 5 Concentration table results

Test	Ferrous concentrate			Granite concentrate		
	Mass (g)	% (mass)	% metallic Fe	Mass (g)	% (mass)	% metallic Fe
1	216.67	8.58	13.7	2.310	91.42	4
2	76.11	7.23	11.4	976.5	92.77	4.4
3	206.03	22.38	6.69	714.5	77.62	4.3
4	224.49	17.39	5.9	1,066.64	82.61	4
5	68.43	6.37	7.6	1,006.07	93.63	4.7

Table 6 Cyclone processing results

Feed pressure (kPa)	Underflow			Overflow		
	Mass (g)	% (mass)	% metallic Fe	Mass (g)	% (mass)	% metallic Fe
24.5	1,784.6	34.55	5.0	3,381.2	65.45	4.7
14.7	1,630.1	32.88	6.5	3,327.4	67.12	4.0
9.8	1,687.7	33.71	6.0	3,318.1	66.29	4.2
4.9	1,746.9	33.43	7.3	3,479.3	66.57	3.5
0	1,689.8	38.21	6.2	2,732.7	61.79	3.9

author also states that inclination angle change is not normally necessary, unless the composition of the material being fed has been altered considerably.

Particle size also can affect the analysis once the difficulty in the gravity separation increases with decreasing the granularity of particle (Sébastien et al. 2012; Maharaj et al. 2012).

Cyclone processing

The tests with the cyclone processing were performed in order to recover the metallic iron present in the granite waste. However, the results were not satisfactory, since the cyclone only produced a product having a maximum of 7.3 % metallic iron. Table 6 shows the results obtained in the cyclone processing.

Comparing the metallic iron recovery results from the magnetic separation (93 %), concentration table (13.7 %) and cyclone processing (7.3 %), it is evident that the latter was the worst of the three recovery methods studied.

Conclusion

The granite waste presented the following constituents: SiO_2 (65.9 %), Al_2O_3 (13.4 %) and metallic iron (4.8 %). The particle size distribution of granite waste showed that 82.6 % of the particles are smaller than 0.044 mm. In regard to metallic iron recovery, the magnetic separation method presented the most efficient results, since it produced a mass of 4.5 % of the initial mass, of which 93 % was metallic iron. The granite concentrate obtained presented 95.5 % of the initial mass of the sample, with a 0.6 % metallic iron content. The tests performed at the concentration table produced an iron concentrate of 8.5 % of total initial mass, containing approximately 13.5 % of metallic iron. The metallic iron recovered by cyclone processing proved to yield the worst results.

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