

Simultaneous use of direct and reverse flotation in the production of iron ore concentrate plant

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Abstract

The exploitation of low-grade iron ore deposits represents the current scenario for this business. Thus arise several types of research that aim at improvements and innovations as solutions to such situation. Therefore, herein, suggested is an alternative route for iron ore flotation that targets a higher mass recovery with concentrate within the market specification. First, the sample was deslimed at 38 μm and with the underflow a cut was performed in the 74 μm cyclone in order to separate the coarse particles from the fine. With the coarse particles, i.e. with the underflow, there a reverse flotation was carried on obtaining a concentrate with 67.7% of Fe and 0.9% of SiO_2 . In the fine particle overflow, direct flotation was done, generating a low-quality concentrate with 40.9% of Fe and 39.7% of SiO_2 . The mass recovery in direct flotation was 88% and in the reverse flotation was 61.1%. The concentrate generated from two flotations had a mass recovery of 67.4% with a content of Fe of 53.4% and 21.6% SiO_2 .

Keywords: iron ore, flotation circuits, reverse flotation.

1. Introduction

The processes for iron ore concentration that stand out are the gravitational methods, magnetic separation, and flotation. And among these, flotation has allowed the concentration of low-grade ore fines. Flotation is a procedure that began to be applied in the mineral industry in the early twentieth century. With a few innovations, the use of flotation was intensified and today it is the predominant technique in the area of mineral concentration (Sampaio and Baltar, 2007).

Iron ore flotation shows good performance for the concentration of oxidized ores with low iron content, allowing the reduction of silica found in magnetic concentrate and therefore it is the most suitable process for the production of concentrates intended for metallurgical processes that use a direct reduction process (Iwasaki, 1983).

According to Araujo and Peres

(2006), the flotation routes that can be used for iron ore are:

- Cationic reverse flotation of quartz;
- Anionic flotation of iron oxides;
- Anionic reverse flotation of activated quartz.

The cationic reverse flotation of quartz is the most commonly used route: quartz is floated with ether amines partially neutralized with acetic acid, and the iron ore is depressed by unmodified starches (Yang, 1988).

The cationic reverse flotation of quartz is preferred for iron ores, due to the high selectivity achieved and the fact that the iron-bearing minerals are in greater portion than the gangue, minimizing collector consumption (Costa, 2009).

Another relevant fact on reverse flotation is the high density of minerals of interest compared to the gangue. It is important to note that for better

flotation with foam, the mineral ores should be finely ground (Chaves and Leal Filho, 2004).

The direct flotation of iron oxides consequently has potential application for low-grade ores and recovery materials in dams (Araujo and Peres, 2006).

The anionic direct flotation of iron oxides is an attractive route for low-grade ores and tailings deposited in the tailings dams. The anionic reverse flotation of activated quartz was used during the time that the amines were not available.

A large portion of the Brazilian iron ore deposits was highly weathered, which generates significant amounts of fine mineral particles. In addition to the fines, friable fine dust, generated during mining operations, as well as in the process of comminution, was present in large amounts in the low-grade iron ores (Mapa, 2006).

Usually with desliming before the reverse flotation and in certain types of iron ore, the attrition increases the mass recovery and reduces the levels of both iron as contaminants, with a gain of Gaudin's selectivity. The attrition improves performance and reduces consumption of the collector (Krishnan and Iwasaki, 1984).

The fine particles removed in the process of desliming usually are rich in silica and alumina, and other undesirable

elements like phosphorus because of the concentration of friable minerals, such as goethite and kaolinite, is greater in the fines. So the desliming process increases the total iron content of ore to be floated (Queiroz, 2006; Donskoi *et al.*, 2008; Totou, 2010).

Herein presented are the results of the study of an alternative route to flotation processes conventionally used for iron ore, without the inclusion of unit operations, in order to increase the mass

recovery and ensure the quality of the concentrates generated.

The proposed route is that after the desliming operation adopted in traditional reverse flotation circuit, a cut is made in the cyclone for separating the fine particles from coarse particles. The coarse particles are floated by the reverse flotation method with the use of conventional reagents. The fine particles are floated directly with the use of reagents studied in partnership with the Clariant Company.

2. Materials and methods

Initial characterization

The study was conducted with an iron ore sample from the Quadrilátero Ferrífero, Minas Gerais, Brazil. After milling until 95% of the material passes in 150 μm , wet sieving and a chemical analysis of each fraction were carried out.

For the mineral liberation study, the Gaudin method was used, where at least five hundred particles were counted and their mineral of interest

composition estimated.

All chemical analysis performed in this study were made through atomic emission spectroscopy with a plasma source.

The mineralogical characterization was conducted by an optical microscope (Zeiss—Axioplan model), whereupon the results were refined in a Scanning Electron Microscope (SEM) and QS (FEI Quanta

650 SEM operating at 25 kV and 10 nA equipped with two EDS Bruker SDD 5030).

The analyses by x-ray diffraction (XRD), using the powder method, were performed with a PANalytical device and the qualitative interpretation of the spectra was performed by comparison with PDF02 standards contained in the database (ICDD 2006) Plus software in HighScore.

Ore preparation

The formation of mass for the tests included initial screening stages, crushing the retained material in a conical Crusher (opening equal to 4.0 mm) and then in a roller crusher (opening of 0.6 mm). About 60 kg were formed, homogenized and divided into fractions of 2 kg destined to:

- initial wet sieve analysis in the following mesh sieves: 212; 150, 106, 74, 44 and 38 μm in order to estimate the initial time of grinding.

- wet grinding in a mill jar of 0.20 x 0.30 m, using 20 steel bars 1" and 60% solid concentration.

The milled product was homogenized and divided in a rotating divisor of

pulps for the following analyses:

- 01 fraction of approximately 50 g for the determination of the actual density by gas pycnometer (1200 Quatachrome);

- 01 fraction of approximately 250 g for mineralogical analysis;

- 01 fraction with the rest of the material for chemical analysis for the range of sizes.

Particle size analysis was done by wet sieving (sieve meshes were equal to 150; 106; 74; 44 and 38 μm). Pulverization and sending the fractions retained by each sieve to chemical analysis.

The sample (after grinding, $P_{95} < 150 \mu\text{m}$) was deslimed in a

cyclone with cut of 38 μm and the overflow and underflow were chemically analyzed. The overflow was filed. And part of underflow obtained was subjected to a reverse flotation method on the terms traditionally used in iron ore projects in the CDM: Denver cell D12; 50% solids; 2.5 L tank; 1200 rpm rotation; depressant: corn starch in 550 g/t of dosage; NaOH for gelatinization; Collector: Flotigan amine 3C Clariant (EDA) with dosage of 400 g/t of SiO_2 ; pH 10.5 (adjusted with NaOH). The conditioning time was 3 minutes for the starch and 1 minute for the amine.

Reverse and direct flotation tests

The remainder of the underflow was forwarded to a cyclone device again (classification cut of 74 μm), and with the new underflow obtained a reverse flotation was performed, under the same conditions described above. A direct flotation was made with the overflow from the same classification under the following conditions: Denver cell D12; 50% solids; 2.5 L tank; 1200 rpm rotation; depressants: Genamin (FQ7) of Clariant with dosage of 600 g/t; collector: 5806 Floti-

gam-25 (fatty acid) of Clariant with dosage of 600 g/t; pH 7 (adjusted with HCl). Conditioning times were of 5 minutes for the FQ7 and 2 minutes for the fatty acid. Another set of tests in the same dosages involved the collector: Flotigan EDA of Clariant and the depressor: cornstarch of Unilever S.A. Finally, a total of 18 independent tests were run. Chemical analyses were performed on all products generated in each step described above. The results obtained in the traditional test were

compared with the results obtained in the alternative circuit in terms of mass recovery and concentrate levels to assess the proposed route.

The tests were designated as follows (Figure 1):

Test 1: reverse flotation products made with the underflow of desliming;

Test 2: reverse flotation products made with the underflow of classification;

Test 3: direct flotation products made with the overflow of classification.

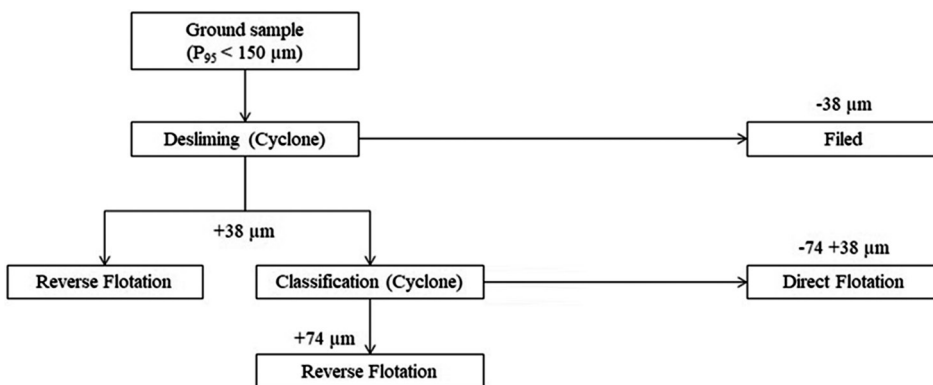


Figure 1 Testing flowchart.

3. Results

Initial characterization

In Table 1, the results of chemical analysis by range of sizes and global sample are presented. It is observed that the initial content of Fe in the sample

was 44% and 34.9% of SiO₂. Although the fraction 38 μm has high iron content, the levels of deleterious elements are very high compared with the other

fractions. For that reason, desliming was adopted that aimed at a separation of 38 μm in order to reduce the levels of undesirable elements.

Sample granulometry (μm)	(%)									(ppm) P
	Al ₂ O ₃	CaO	MgO	Mn	SiO ₂	TiO ₂	FeO	Fe	PF	
Global	0.485	0.041	0.082	0.192	34.9	0.029	1.68	44.0	1.43	374.34
150	0.257	0.042	0.084	0.268	39.3	0.023	2.87	41.5	1.44	367.86
106	0.183	< 0.031	0.035	0.182	46.7	0.016	2.30	36.1	1.14	296.86
74	0.186	< 0.031	0.034	0.141	46.0	0.016	2.48	36.8	0.99	275.67
44	0.205	< 0.031	0.043	0.135	38.3	0.022	2.27	42.2	1.02	282.08
38	0.239	< 0.031	0.063	0.135	28.8	0.031	1.96	48.5	1.00	298.47
< 38	1.091	0.059	0.148	0.290	21.8	0.049	1.42	51.5	2.18	585.98

Table 1 Chemical analysis by size range of the sample that was grinding.

The mineralogical composition is presented in Table 2. The main source of iron is hematite; quartz

represents more than 90% content of silica. The kaolinite is responsible for more than 30% content of Al₂O₃, and

almost 70% of Al₂O₃ was associated with goethite.

Minerals	Structural formula	Proportion (%)
Hematite	Fe ₂ O ₃	49
Goethite and Limonite	FeO·OH	7
Magnetite	Fe ₂ ³⁺ , Fe ²⁺ O ₄	7
Mn oxo-hydroxides	MnOx	< 1
Quartz	SiO ₂	37
Kaolinite	Al ₂ Si ₂ O ₅ (OH) ₄	Dash
Talc	Mg ₃ Si ₄ O ₁₀ (OH) ₂	Rare
Amphibole	(Mg,Ca, Na, K) ₂₋₃ (Mg,Fe,Al,Ti,Mn,Cr,Li,Zn) ₅ (Si,Al) ₈ (OH) ₂	Rare

Table 2 Mineralogical composition of initial sample.

By washing, 6.5% of the sample was discarded as slimes. And the remaining 93.5% proceeded to the next step and whose iron content was 44.3% and silica equal to 34.7%. The mineral-

ogical characterization by X-ray diffraction showed that the overflow sample was rich in talc and kaolinite, while in the sample from the underflow, these minerals were not detected.

In general, the liberation degree (LD) of oxides/hydroxides and gangue (LD 95%-100%) of the sinter feed and pellet feed samples was above 90% (Figure 2).

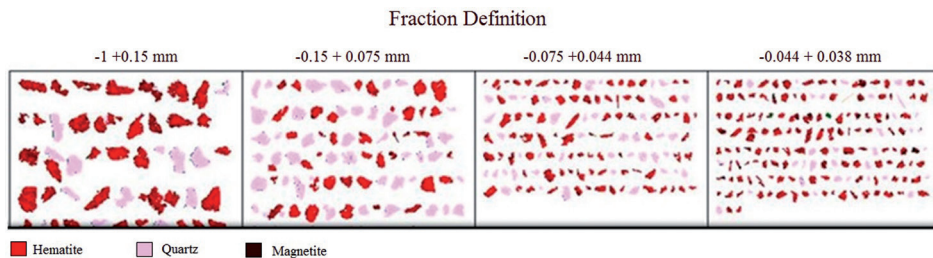


Figure 2 Liberation degree (LD) of oxides/hydroxides and gangue.

The occurrence of these minerals is shown in Figure 3, representing the electronic image obtained by SEM. In

textural terms, the samples were characterized by the presence of granular quartz crystals with granulation "average" and

"thin" having minor variations related to amphibole and goethite, according to Figure 3.

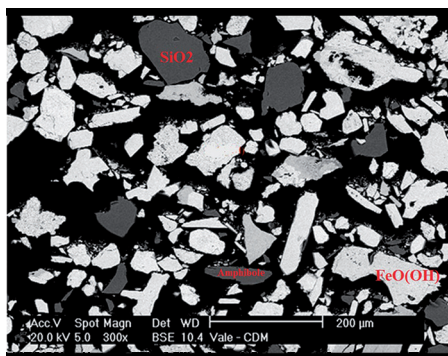


Figure 3 The electronic image of iron ore sample - Quanta 650 SEM.

Flotation tests

Table 3 presents the data regarding the mass recovery, along with the levels of concentrate and tailings generated in the test 1, test 2 and test 3 shown in the order of largest recovery and iron content.

Chemical analysis of the concentrated revealed a 67.70% iron content and 0.68% silica, the mass recovery of concentrate was 54.4% and the metallurgical

recovery was 85.5% (Test 1).

The next step was the classification (cut of 74 µm) in which there were obtained portions of 55.76% of mass for the underflow, with Fe content of 48.13% and 29.7% of silica. With 44.24% of the total, the overflow presented levels of Fe and 37.1% and 44.0% of SiO₂. The overflow contents are consistent for applications

using the direct flotation method.

Table 3 also presents the data for the mass recovery and the contents of concentrate and tailings generated in test 2. The concentrate obtained from the reverse flotation of underflow after classification had in its composition about 67.71% of Fe and 0.88% of silica. Mass recovery was 61.1% and metallurgical recovery was 85.4%.

Table 3 Flotation test results.

	Product	(%)										(ppm)	
		Mass recovery	Al ₂ O ₃	CaO	MgO	Mn	SiO ₂	TiO ₂	FeO	Fe	LI	P	
Test 1	Concentrated	54.4	0.203	0.037	0.035	0.256	0.68	0.036	3.05	67.70	1.72	458.55	
	Tailings	45.6	0.242	0.034	0.092	0.050	76.42	0.019	0.70	15.16	0.45	232.12	
Test 2	Concentrated	61.1	0.203	0.037	0.035	0.256	0.88	0.036	3.05	67.71	1.72	394.15	
	Tailings	38.9	0.242	0.034	0.092	0.050	76.42	0.019	0.70	15.16	0.45	230.01	
Test 3	Concentrated	88.04	0.203	0.037	0.035	0.256	39.67	0.036	3.05	40.93	1.72	368.77	
	Tailings	11.96	0.242	0.034	0.092	0.050	76.42	0.019	0.70	15.16	0.45	< 230	

LI: Loss on Ignition

Mass recovery data and contents of the concentrate and tailings generated in test 3 are presented in Table 3

too. The iron content of the concentrate was 40.93% and 39.67% silica. The mass recovery of concentrate was

88.04% and metallurgical recovery was 89.0%.

4. Discussion

The results obtained in the traditional circuit met the standards of market specification, with exception to the

phosphorous content which was higher. Already concentrated results generated in reverse flotation of the coarse fractions

and of direct flotation of the fine fraction showed themselves outside of the specifications.

The Figure 4 shows the mass balance and contents obtained in the traditional circuit of flotation and the suggested circuit. Note that the mass recovery in the proposed circuit was 67.41%, much higher than the standard circuit which was 50.2%. However, the levels of Fe and SiO₂, unlike the traditional circuit, were below the market's specifications. The value of Fe in the proposed route was 53.4% and SiO₂ was 21.6%, while the traditional route were 67.7% levels of Fe and 0.7% of SiO₂. The P values remained high in both circuits.

Analyzing separately the proposed route data, the results obtained in reverse flotation after classification ("cycloning") were very satisfactory, both in terms of mass recovery, which was 67.71%, and in terms of levels of Fe and SiO₂, 67.76% and 0.88%, respectively.

However, the results obtained in direct flotation were not within the expected. Despite the high mass recovery, of 88.0%, concentrate levels were way outside of specifications, the Fe content was 40.9% and 39.7% of SiO₂. Possibly, there occurred too much drag by the quartz concentrated foam obtained by direct flotation, increasing the SiO₂ content of this. In this way it is concluded that the depressant used (quaternary ammonium salt—Genamin PQ7) was not efficient, not allowing good results for direct flotation.

According to Baltar (2010) any salt can be used as a depressant and its efficiency is in the competition that is created between ionic entities from the collector and depressor, tending to a decrease of ions and collectors, so there is adsorption on mineral particles. In this

way, the time of contact or residence time of flotation is of fundamental importance in this regard involving the interaction between collector and depressant for mineral depression.

Still, there must be taken into consideration the complete dissociation of quaternary ammonium salts in almost the entire pH range, with their accelerated consumption and unavailability in the solution.

When compared to studies done by (Nascimento, 2010), the results of the proposed circuit obtained a concentrate of best quality, but was also outside the market specification. But (Lopes and Lima, 2009) managed to obtain the highest content of Fe and the lowest content of SiO₂ in the concentrates produced. The data for direct comparison can be appreciated in Table 4.

Table 4
Comparison of the results of the proposed circuit and of direct flotation studies.

Identification	Collector (g/t)	Depressor (g/t)	%Fe	%SiO ₂
Proposed circuit	Fatty acid = 600	Quaternary ammonium chloride and acrylamide = 600	53.40	21.60
Nascimento (2010) ¹	Sodium oleate = 2400	Sodium fluorosilicates = 1200	46.71	28.85
Lopes and Lima (2009)	Commercial hydroxamate (AERO 6493) = 1200	Sodium silicate = 1500	61.50	9.80

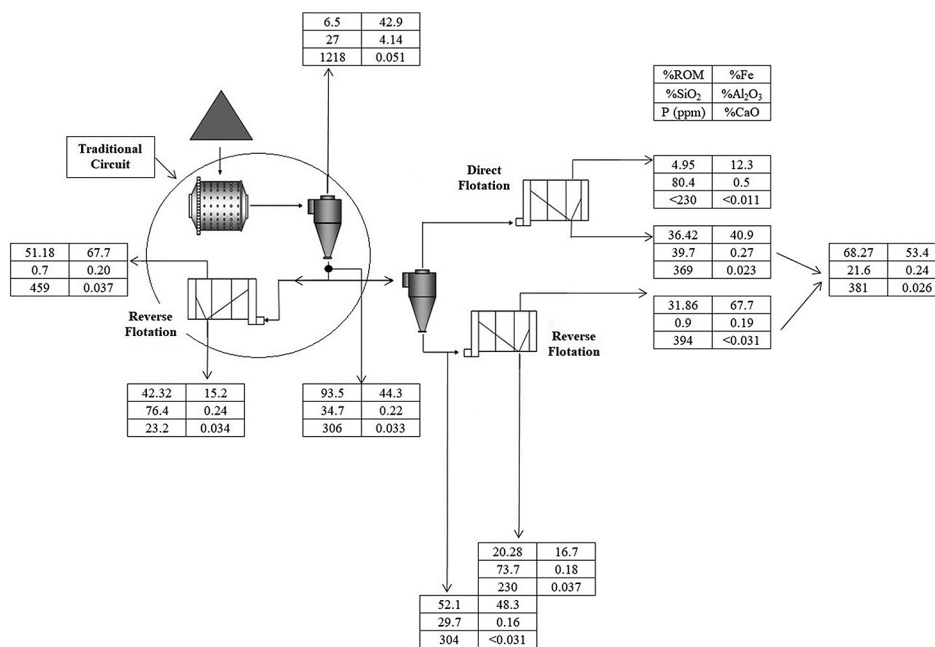


Figure 4
Flowchart with mass balance and contents of standard and alternative industrial circuit proposed.

5. Conclusions

Bench-scale tests carried out in this study reached the goal of elevating the mass recovery after the alteration proposed.

The step of coarse and fine particle separation using a cyclone proved to be promising, since the results of reverse flotation in large particles have achieved superior results.

In terms of mass recovery of concentrate, compared to the traditional circuit, there were no decreases.

However, in terms of the fine particles, a large volume of gangue minerals that were to foam due to drag from the flotation, the concentrate directly generated a concentrated with high levels of

SiO₂ (39.7%). The depressor chosen was not effective, requiring its replacement for the maintenance of the proposed procedures, in order to re-evaluate its functionality and this demonstrates the need for more research and experimental work in terms of direct flotation reagents for iron ore.

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