

Dolomitic BIF beneficiation of Conceição – MG mine

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1. Introduction

The high demand for iron ore products and the exhaustion of reserves with a high iron grade in the “Quadrilátero Ferrífero” of Minas Gerais in Brazil have led to the development of beneficiation of low grade “banded iron formations” (BIF) with the removal of overburden materials. They are composed of minerals without economic value, which are deposited in piles or replaced after mining. The overburden material represents costs in the operation of a mine, as well as issues related to safety and the environment (Aragão and Oliveira Filho, 2011). When there is a process route for its concentration, this material can be considered as ore. The dolomitic BIF is the main overburden material of iron ore exploitation at the Conceição mine (Itabira, Minas Gerais,

2. Materials and methods

The sample of dolomitic BIF was obtained in blocks at the Conceição mine (Figure 1). The sample was analyzed by X-ray diffraction (XRD), X-ray fluorescence (FRX), scanning electron microscopy (SEM), conversion electron Mössbauer spectroscopy (CEMS), loss on ignition, thermogravimetry and density by helium pycnometry. The liberation size was deter-

Abstract

In the Conceição – MG mine, there are significant reserves of dolomitic itabirite, which is considered overburden material. This study aimed to develop a flotation route for the concentration of this dolomitic BIF according to the requirements of the steel industry. The characterization of samples, comminution, liberation size, microflotation of the pure dolomite and hematite minerals were performed to establish the conditions for selective separation in bench flotation tests. The microflotation tests showed that the soybean oil at pH 6 preferably floats the dolomite in relation to the hematite. The corn starch depressant at a pH higher than 10 preferably depresses the hematite relative to the dolomite. The bench flotation tests revealed interactions between the factors pH, percentage of solids in the pulp, soybean oil and starch dosage. The optimal results for bench flotation tests were achieved under the following conditions: pH at 10.5, percentage of solids of 27wt%, dosages of soybean oil and corn starch of 400g/ton and 351g/ton, respectively.

keywords: dolomitic BIF, iron concentration, flotation.

Brazil). It has a similar texture of the siliceous BIF, with alternating bands of carbonate and iron, composed of dolomite, hematite and smaller quantities of quartz, and may also contain varying amounts of calcite, talc, chlorite and amphiboles (Rosière *et al.*, 2008).

The concentration by flotation is the most used process in mining, both in quantity of processed ores and in diversity of applications (Peres *et al.*, 2007). It is based on the principle of selectively inducing hydrophobicity to the surface of the mineral to be floated by chemical agents, separating it from other hydrophilic minerals. Saponified vegetable oils are surfactants widely used in flotation as a collector. They are composed of mixtures of carboxylic acids (RCOOH) of

long hydrocarbon chain, which presents a hydrophobic character, and the carboxyl polar group, which is hydrophilic (Leja, 1982; Pearce, 2005). Soaps or salts of these oils, obtained by saponification, are used in the anionic flotation of slightly soluble and oxide minerals (Kulkarni and Somasundaran, 1980; Guimarães *et al.*, 2005). Starches of different origins are used as depressants in several flotation systems (Leja, 1982; Hanna and Somasundaran, 1976). Nunes and Peres (2011) point them out as being excellent carbonate depressants. In Brazil, starches are widely used in flotation of siliceous BIF as a depressant of iron minerals (Monte and Peres, 2004).

The objective of this study was to obtain a concentrate of iron ore by means of flotation from the dolomitic BIF.

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out long enough for 5% of the material to be retained in the 74µm sieve. The ground material underwent wet sieving, using the 37µm sieve. The +37µm fraction was dry classified in an electromagnetic stirrer using the 100, 74, 53, 44 and 37µm sieve series. The -37µm fraction was classified in a cyclosizer at the sizes 24.7, 18.4, 12.6, 9.0 and 7.2µm.

The dolomite and hematite for the microflotation tests were obtained from the ore itself (Figure 1). The ore blocks were comminuted in a jaw crusher, followed by manual pick up of fractions rich in dolomite and hematite. Each of these fractions were ground in a disc mill and classified in the 149µm +74µm range. The purification of these fractions was done in a dry magnetic separator of high intensity field. The microflotation tests were performed on a modified Hallimond tube using distilled water, 1.0 g of mineral, 4 minutes of conditioning for soybean oil

and 2 minutes for corn starch, 1 minute of flotation and nitrogen flushing of 70 cm³/min. The concentrations of soybean oil were 2, 4, 8, 16 and 32 mg/L, the concentrations of corn starch were 1.25, 2.5, 10, 20 and 40 mg/L, and the pH were 4, 6, 8, 10 and 12.

The reverse anionic flotation tests were conducted in a bench flotation cell using 1kg of ore, conditioning time of 4 minutes for soybean oil and 2 minutes for corn starch, 6 minutes of flotation, 1200 rpm of the rotor. A 24 factorial design (Table 1) was performed to evaluate the

effects of the factors pH, concentration of solids in the pulp (CS), dosage of soybean oil (SO) and dosage of corn starch (ST) on grade of iron (Fe), CO₃²⁻ (carbonate), and SiO₂ (silica) in the concentrate and in the tailings. The results were analyzed with the aid of Minitab 17 software.

The soybean oil was supplied by Pirapora and saponified with sodium hydroxide at 70°C. The corn starch (corn meal, Flotamil 75), supplied by Caramuru Alimentos SA, was gelatinized with sodium hydroxide. Sodium hydroxide and hydrochloric acid were used for pH adjustments.



Figure 1
Sample of dolomitic BIF from Conceição mine.

Factor	Low level	Coded unit	High level	Coded unit
pH	6.0	-1	10.5	+1
CS, wt%	27	-1	44	+1
SO, g/ton	200	-1	400	+1
ST, g/ton	250	-1	500	+1
Test	pH	CS	SO	ST
T1	+1	+1	+1	-1
T2	+1	-1	+1	-1
T3	+1	-1	-1	-1
T4	-1	-1	+1	-1
T5	-1	+1	-1	+1
T6	-1	+1	+1	-1
T7	+1	+1	+1	+1
T8	+1	-1	-1	+1
T9	+1	+1	-1	+1
T10	-1	-1	+1	+1
T11	-1	+1	+1	+1
T12	+1	+1	-1	-1
T13	+1	-1	+1	+1
T14	-1	-1	-1	-1
T15	-1	-1	-1	+1
T16	-1	+1	-1	+1

Table 1
24 factorial design for the flotation tests.

3. Results and discussion

3.1 Characterization

The mineralogical analysis by X-ray diffraction showed that the sam-

ple consists of dolomite (majoritarian) and hematite (intermediate) and small

amounts of chlorite and talc. In addition to the hematite, in the Fe³⁺ oxidation

state, analysis by conversion electron Mössbauer spectroscopy, identified another iron phase, ferrihydrite, with the hematite being the predominant phase

(98.00%).

The results of the X-ray fluorescence are shown in Table 2. The thermogravimetry showed that at 691°C the

sample lost 11wt% due to the release of CO₂ from the carbonated phase, which is the explanation for the high value of the loss on ignition (LOI).

Table 2
Grade in the sample of dolomitic BIF (%)

Fe	Mg	Ca	SiO ₂	Al ₂ O ₃	P ₂ O ₅	MnO ₂	CO ₃ ²⁻	LOI
38.94	7.32	5.93	1.96	0.53	0.58	0.50	27.14	13.00

In the determination of the liberation size of the minerals, the hematite/gangue ratio was considered,

where the gangue constitutes all the other minerals of the sample. The liberation degree of 88.56% is when the

particles are practically all liberated (Figure 2a).

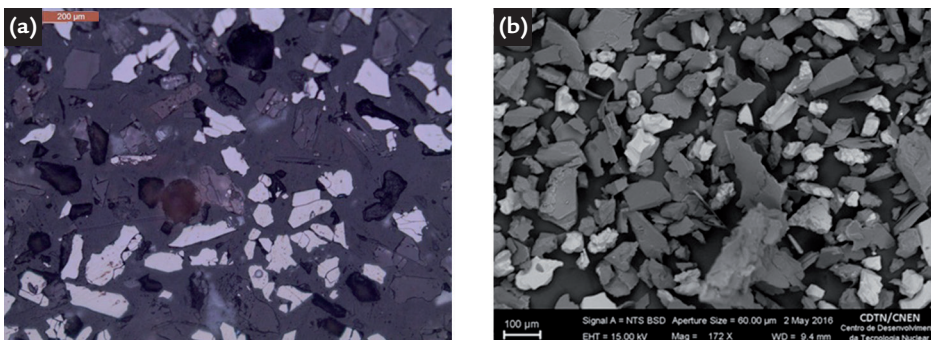
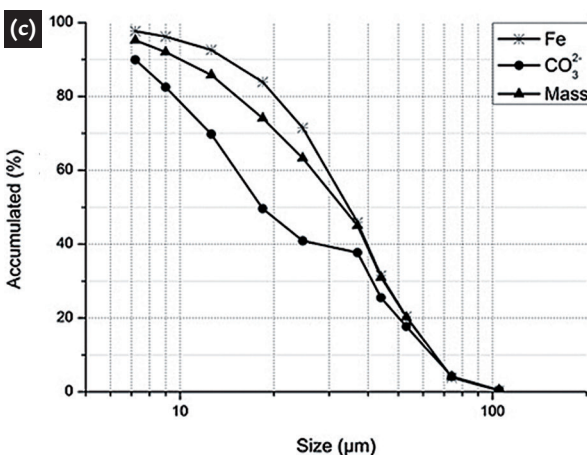


Figure 2
(a) Optical microscopic image of the fraction -74 + 53 μm, showing free particles of hematite (light particles); (b) BSD/MEV image of the dolomitic BIF: milling time 12.5 min, P₉₅ at 74 μm, showing free particles of hematite (light particles); (c) mass, Fe and CO₃²⁻ distribution as a function of particle size.



According to the liberation size, the results of the grinding tests showed that for 12.5 min, P₉₅ is 74 μm. Figure 2b shows the backscattered electron image (BSD) generated in scanning electron microscopy of the ground sample for 12.5

min. In this size fraction, the particles are practically all liberated, being possible to observe the existence of individualized fractions of the hematite (white fractions). The size analysis (Figure 2c) showed that the CO₃²⁻ distribution is concentrated in

the finer fractions and the Fe fraction in the coarser fractions, with 45.9% Fe being distributed in the fractions >37 μm and 37.7% in CO₃²⁻ in the fractions <37 μm. The density of the sample by helium pycnometry was 4.13g/cm³.

3.2 Microflotation

Figure 3 shows the results of the microflotation tests. The flotability of hematite as a function of pH and soybean oil concentration (Figure 3a) is more complex. It decreases with the increasing of the pH, showing a maximum around 10 mg/L, and increases steadily with the increasing of soybean oil concentrations higher than 15 mg/L. The dolomite flotability (Figure 3b) is low for high pH and low concentrations of soybean oil and increases with the increasing of the soybean oil concentra-

tion. It shows a maximum around pH 6 for low concentrations of soybean oil. The hematite flotability decreases very fast with the increasing the starch concentration for an addition of 32 mg/L of soybean (Figure 3c); the dolomite flotability decreases with the increasing of the starch concentration for an addition of 32 mg/L of soybean oil. According to Chen and Tao (2004), for dolomite the Mg²⁺ and Ca²⁺ species predominate at pH 6, which favor the adsorption on the collector and

lead to the high flotability of this mineral.

The flotability of both minerals decreased under the alkaline conditions (Figures 3a and 3b), especially for hematite. This behavior was also observed by Lopes and Lima (2009) and Kulkarni and Somasundaran (1980), who obtained low hematite flotabilities for pH > 10 using a fatty acid as the collector. Figures 3c and 3d show that the corn starch preferentially depresses the hematite, as also reported by Brandão (2005).

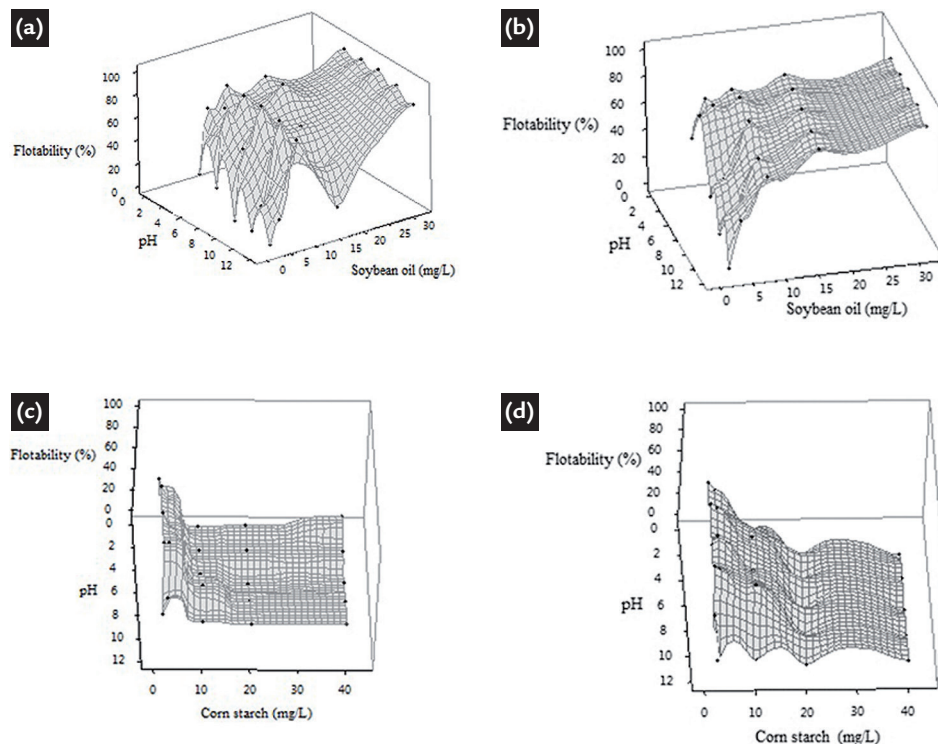


Figure 3
Surface plots for the microflotability. The dots are the experimental points and the surfaces were interpolated with the software Minitab 17 by the distance method. Flotability of hematite (a) and of dolomite (b) as a function pH and soybean oil concentration; Flotability of hematite (c) and dolomite (d) as a function of pH and starch with the addition of 32 mg/L of soybean oil.

3.3 Bench flotation tests

Table 3 shows the results of the grade of Fe, CO₃²⁻ and SiO₂ in the concentrate and in the tailings. Tables 4 and 5 show the results of the calculations of the main effects and the interactions carried out with Minitab 17 for the concentrate and the tailings by means of a stepwise analysis with input alpha of 0.10. Note the complex-

ity of the system due to the interactions between the factors, suggesting the existence of physical and chemical interactions between the reagents. Only the effects of factors and interactions with p≤0.100 are considered statistically significant.

The optimal conditions were calculated with the Response Opti-

mizer of Minitab 17 to obtain in the concentrate the maximum grade of Fe and the minimum grade of CO₃²⁻ and SiO₂, as well as in the tailings the minimum grade of Fe and maximum grades of CO₃²⁻ and SiO₂. The optimal settings, the expected grades and the results obtained in confirmatory tests are shown in Tables 6 and 7.

Test	Concentrate			Tailings		
	Fe	CO ₃ ²⁻	SiO ₂	Fe	CO ₃ ²⁻	SiO ₂
T1	61.18	8.17	1.10	15.69	47.00	4.09
T2	55.10	12.45	1.96	8.67	53.30	5.53
T3	50.00	16.59	2.48	16.20	47.00	4.35
T4	54.26	12.40	3.05	32.17	43.60	4.00
T5	48.13	18.82	2.08	9.89	52.30	3.01
T6	54.09	12.47	1.83	23.19	52.20	1.51
T7	49.00	18.14	1.95	27.00	44.00	2.39
T8	48.74	17.45	0.81	13.10	50.30	2.45
T9	57.74	10.08	0.83	11.43	51.10	3.77
T10	58.74	8.62	2.17	28.78	37.00	1.71
T11	52.42	14.14	1.62	22.20	43.20	1,67
T12	56.03	11.12	1.01	14.41	48.80	3.15
T13	62.80	5.69	0.86	12.94	50.40	2.81
T14	56.25	10.86	2.17	23.29	42.10	1.64
T15	51.00	14.98	2.20	23.22	41.80	1.66
T16	42.71	21.67	2.12	18.16	44.80	3.11

Table 3
Grades of Fe, CO₃²⁻ and SiO₂ (%) in the concentrate and in the tailings obtained in the bench flotation tests (the settings of each test is shown in Table 1).

Factor	Fe			CO ₃ ²⁻			SiO ₂			
	Value	SE	p	Valor	SE	p	Value	SE	p	
Constant	53.64	0.86	0.000	13.35	0.64	0.000	1.77	0.11	0.000	
pH	2.87	1.71	0.169	-1.78	1.28	0.233	-0.78	0.22	0.011	
CS	-1.95		0.319	1.95		0.201	-0.40		0.117	
SO	4.62		0.054	-3.69		0.044	0.11		0.644	
ST	-1.49		0.434	0.99		0.481	-0.39		0.121	
pH*CS	3.78		0.092	-3.11		0.071	0.09		0.691	
pH*SO	-0.73		0.691	0.99		0.480	0.08		0.724	
CS*SO	-1.60		0.420	1.49		0.305	0.01		0.965	
CS*ST	-2.90		0.165	2.38		0.135	0.52		0.054	
SO*ST	1.07		0.566	-0.71		0.606	*		*	
pH*CS*SO	-4.08		0.076	3.76		0.042	0.41		0.22	0.106
CS*SO*ST	-3.60		0.103	3.17		0.067	*		*	*

Table 4
Calculation of the main factors effects and interactions for the concentrate by means of stepwise analysis ($\alpha=0.100$).

* Not considered in the stepwise analysis

Factor	CO ₃ ²⁻			SiO ₂			Fe		
	Value	SE	p	Value	SE	p	Value	SE	p
Constant	46.50	0.56	0.000	2.93	0.16	0.000	18.77	1.06	0.000
pH	3.75	1.12	0.010	1.28	0.31	0.003	-7.68	2.12	0.011
CS	1.63		0.185	-0.18		0.575	-2.05		0.371
SO	-0.30		0.796	0.07		0.824	5.11		0.052
ST	-2.35		0.069	-0.96		0.015	1.67		0.460
pH*CS	-5.38		0.001	*	*	*	6.46		0.022
pH*SO	*	*	*	*	*	*	-2.83		0.229
pH*ST	3.50	1.12	0.014	*	*	*	0.71		0.747
CS*SO	*	*	*	-0.92	0.31	0.018	*	*	*
CS*ST	*	*	*	0.76		0.040	*	*	*
SO*ST	-3.05	1.12	0.026	-0.67		0.062	1.13	2.12	0.612
pH*CS*ST	*	*	*	*	*	*	4.28		0.090

Table 5
Calculation of the main factors effects and interactions for the tailings by means of stepwise analysis ($\alpha=0.100$).

* Not considered in the stepwise analysis

Table 6
Result for the response optimizer calculated with Minitab 17 to achieve in the concentrate: the maximum grade of Fe and the minimum the grades of CO₃²⁻ and SiO₂; in the tailings: the minimum grade of Fe and the maximum grades of CO₃²⁻ and SiO₂.

*D:0.7765	Level	pH	CS	SO	ST
	High	1.0	1.0	1.0	1.0
Optimal	[1.0] (10.5)	[-1.0] (27 wt%)	[1.0] (400 g/ton)	[-0.1933] (351 g/ton)	
Low	-1.0	-1.0	-1.0	-1.0	

*D: desirability function

Test	Concentrate grade (%)			Tailings grade (%)		
	Fe	CO ₃ ²⁻	SiO ₂	Fe	CO ₃ ²⁻	SiO ₂
T17	57.27	8.40	1.93	14.41	53.33	5.44
T18	57.30	8.28	1.30	17.98	47.28	5.32
Mean	57.29	8.34	1.62	16.20	50.31	5.38
SE	0.02	0.08	0.45	2.52	4.28	0.08
Expected	58.34	9.58	1.50	13.12	52.09	4.38

Table 7
Confirmatory tests for the optimized settings of the factors

Figure 4 shows the mass and metallurgical balance of the flotation test performed under optimized conditions (T18 of Table 7) and with roughing, cleaning and scavenging stages. These stages were

carried out to reduce the CO₃²⁻ and Fe grades in the concentrate and in the tailings, respectively. In the cleaning stage, 50g/ton of soybean oil was added to float carbonates to clean the final concentrate.

In the scavenging stage, the addition of reagents was not necessary, since the material of this stage comes from the flotation roughing. At all stages, the pH was adjusted to 10.5. The final (cleaner)

concentrate had grades of Fe, CO₃²⁻ and SiO₂ of 62.94, 4.13 and 0.82%, respectively. It should be noted that the tailings

of the cleaner as well as the scavenger concentrate still contain high iron grade, close to that of the feed, so they can be

returned to the roughing stage. Tables 8 shows the results of the complete chemical analysis of the final concentrate.

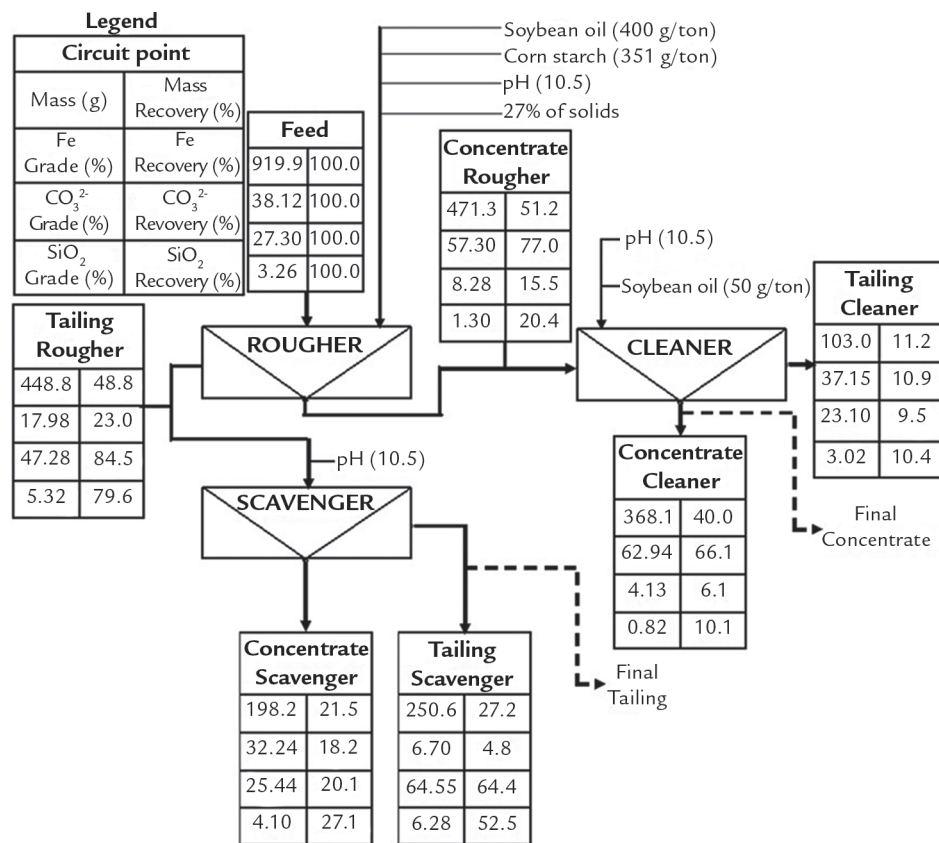


Figure 4 Mass and metallurgical balance of the flotation test performed under optimized conditions (T18 of Table 7) and with rougher, cleaner and scavenger stages.

Cleaner stage	Final concentrate grade (%)								
	Fe	CO ₃ ²⁻	SiO ₂	Ca	Mg	Al ₂ O ₃	MnO ₂	P ₂ O ₅	LOI
	62.94	4.13	0.82	0.75	1.82	0.51	0.14	0.24	3.02

Table 8 Complete chemical analysis of the final concentrate.

4. Conclusion

The dolomitic BIF of the Conceição mine is composed of dolomite and hematite, being the dolomite the majoritarian mineral. At -74µm the dolomite and hematite are practically liberated. Microflotation tests showed that at alkaline conditions, the corn starch preferably

depresses the hematite and the soybean oil preferably floats the dolomite. The results of the studies of characterization, liberation size, milling and microflotation allowed the concentration of this ore by flotation. According to the optimization of bench flotation tests,

good performance is achieved under the following conditions: pH 10.5, 27wt% of solids, 351 g/ton of corn starch and 400 g/ton of soybean oil. The cleaner stage improved the levels of Fe, CO₃²⁻ and SiO₂ in the concentrate to 62.94, 4.1 and 0.82%, respectively.

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