Original Article

High grade quartz products from magnetic separation and flotation of iron ore tailings

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Abstract

More than 80 Mty of iron ore tailings have been generated in the Iron Ore Quadrangle last decades. Reverse flotation of quartz and magnetic separation are the main concentration processes on the beneficiation plants in this region, with SiO_2 content on the deslimed tailings close to 90%, with potential to be destinated as sand for the civil construction industry. This paper investigated the possibility of obtaining high grade quartz (>99% SiO_2) products from a typical iron ore deslimed tailing to be destinated to different markets, as alloy, artificial stone, glass, foundry, semiconductor, silicates and solar panel. A typical iron ore deslimed tailing sample was characterized by chemical, size distribution and mineralogical analyses using optical microscope and QUESCAM, showing the quartz as the main mineral and Fe-oxides the second more abundant mineral in the iron ore tailing, occurring almost 83% as free particles and 17% associated with quartz. Other minerals as phosphates and silicates were observed in associations with quartz. The addition of a high-intensity vertical magnetic separation stage to remove the Fe-oxides can increase the SiO_2 grade to 97.46%. A final concentration stage by flotation using specific collectors was evaluated in this paper aiming at the production of high grade SiO_2 products. Cationic collectors for quartz and alternative anionic collectors of Fe-oxides showed great potential to achieve premium quartz with 99.20 to 99.46%% SiO_2, 0.79 to 0.96% Fe_2O_3 and less than 0.260% of other impurities with possibility to be destinated for glass, silicates, foundry and artificial stone industries. The presence of small associated and included impurities particles, mainly Fe-oxides, prevents obtaining higher purity quartz (> 99.46% SiO_2). Leaching with different acids can be an alternative to remove these impurities and obtain high-purity quartz from iron ore deslimed tailing. This paper contributes to the circular economy through better understanding the iron ore tailings from the Iron Quadrangle by deep characterization methods, showing the potential to obtain premium quartz products.

Keywords: Iron ore tailings; High grade quartz products; Magnetic separation; Flotation.

1 Introduction

Silicon (Si) is the 2nd most abundant chemical element in the Earth's crust, present mainly in the form of quartz: mineral of the hexagonal system with a density of $2.65 g/cm^3$. Transparent, colourless, high hardness, good polishing and easily distinguishable edges. Crystals are usually compact (little porous), with irregular contacts and edges ranging from slightly rough (smooth) to very rough and with cavities. It does not show cleavage, but it does have a conchoidal fracture. Granular appearance with equidimensional and inequidimensional crystals of very variable granulation. Quartz is the most important silica (SiO_2) polymorph in nature, and is widely found in all rocks (magmatic, metamorphic and sedimentary) and hydrothermal veins. Various impurities are introduced inside the quartz during the crystal grouth process, some of these impurities occur as small inclusions [1]. Considering the geology of the Iron Quadrangle (IQ) of Minas Gerais, different minerals further than Fe-oxides can be associated with the quartz as shows Figure 1 [2,3].

Premium quartz (98% < SiO_2 < 99.9%) and high-purity quartz (SiO_2 > 99.95%) can be destinated for different markets according to the Figure 2. Considering the high-purity quartz (HPQ) products, the total impurities should be maximum 50 ppm, with Al < 30 ppm, Ti < 10 ppm, K < 8 ppm, Na < 8 ppm, Li < 5 ppm, Ca < 5 ppm, Fe < 3 ppm, P < 2 ppm and B < 1 ppm [1,4]. Size distribution of premium and high-purity quartz is also controlled, normally above 45 or 100 μm [1,4].
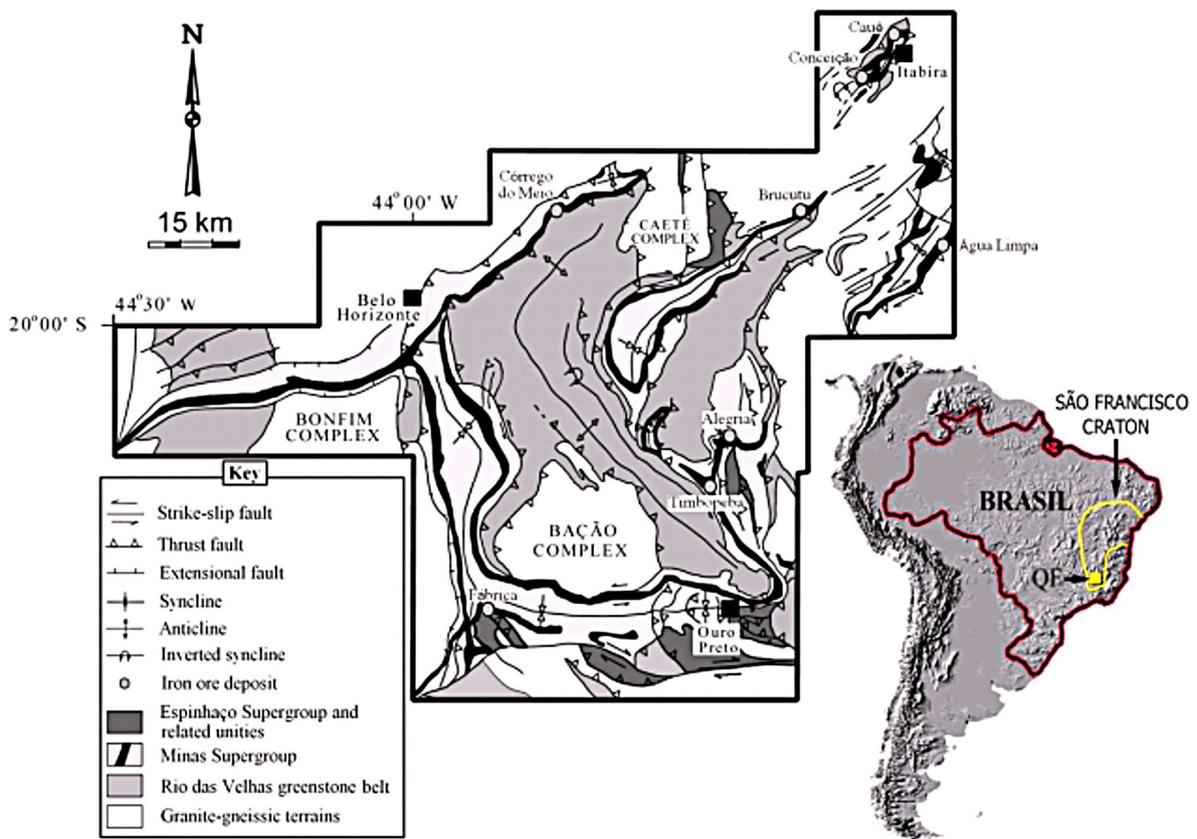
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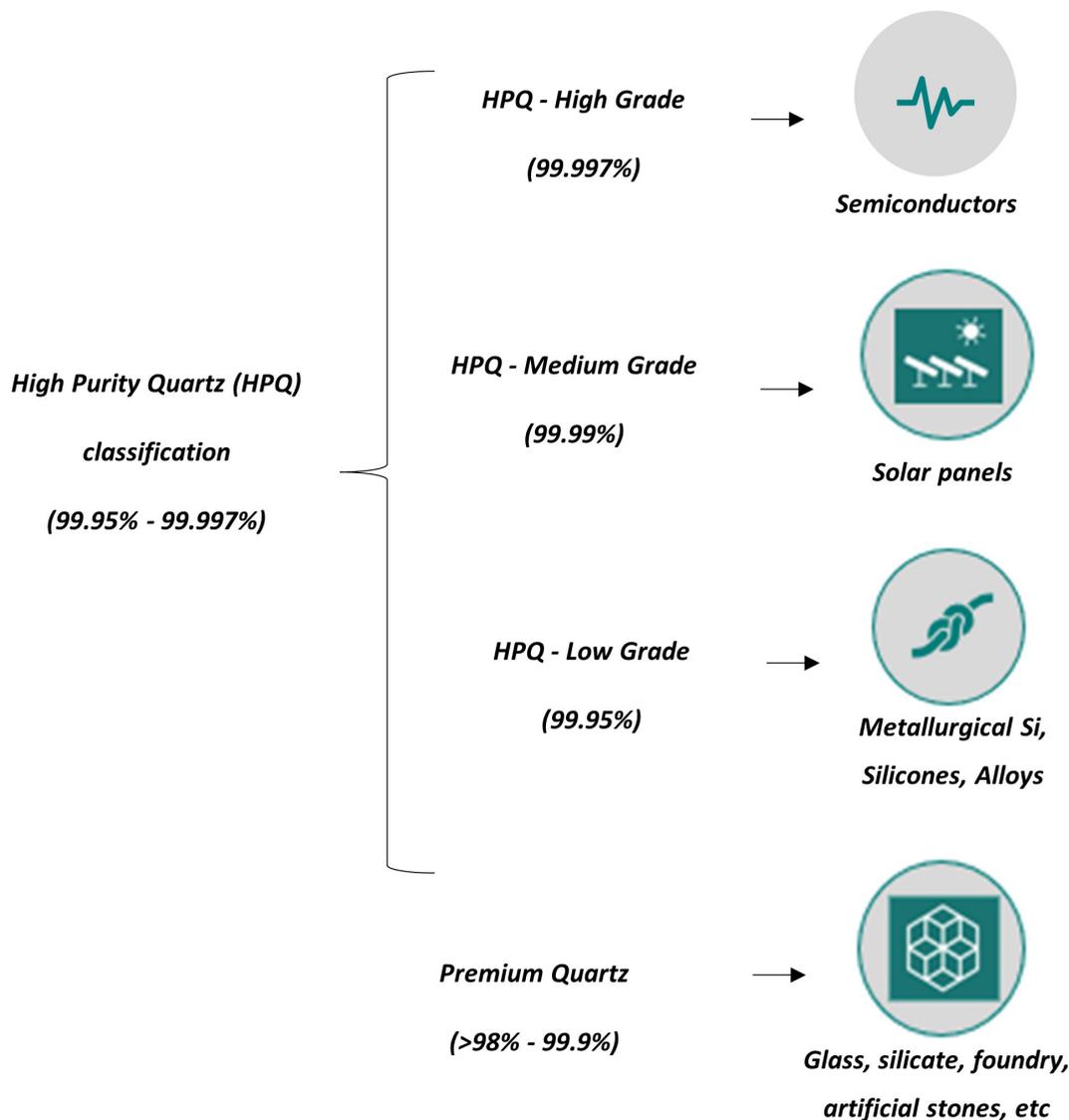
Figure 1. Iron Quadrangle map and types of ores according to mineral criteria [2,3].

Types of BIF	Main components	Accessory components
Common itabirite	Hematite, martite, magnetite, quartz	Chlorite, sericite, Fe-dolomite, kaolinite, kyanite, Mn oxides, sulfides, apatite, pyrophyllite
Dolomitic itabirite	Martite, hematite, magnetite, Fe-dolomite	Calcite, grunerite-cummingtonite, chlorite, stilpnomelane, biotite, tremolite, actinolite, quartz (chert), sulfides, pyrophyllite
Amphibolitic itabirite	Martite, hematite, magnetite, maghemite, grunerite–cummingtonite, tremolite, actinolite, quartz	Carbonates, talc, Fe-dolomite, aegirine, biotite, riebeckite, kupferite, sulfides
Hematitic phyllite	Hematite, sericite	Quartz, chlorite

Li et al. [7] investigated the separation and purification of high-purity quartz from an iron ore tailing sample with 81.39% SiO_2 and 15.21% Fe_2O_3. The use of a modified magnetic separador [8,9] with 5 Tesla of magnetic field, 4% to 8% of solids concentration and slurry flow velocity from 0.12 to 0.23 m/s, enabled obtaining low-grade quartz product with 98.56% SiO_2 with 60.59% of mass recovery and 39.41% of no-magnetic with 46.78% of Fe. High grade quartz with 99.92% SiO_2 was obtained from the low-grade quartz of the magnetic separator after acid leaching using HNO3, HCL and H2SO4, molarity ratio of 1:4:1, solid-liquid radio of 1:4, temperature of 80ºC and stirring time of 10 h.

HPQ - High Grade

(99.997%)

Semiconductors

High Purity Quartz (HPQ)

classification

(99.95% - 99.997%)

HPQ - Medium Grade

(99.99%)

Solar panels

HPQ - Low Grade

(99.95%)

Metallurgical Si,

Silicones, Alloys

Premium Quartz

(>98% - 99.9%)

Glass, silicate, foundry,

artificial stones, etc

Figure 2. The High Purity and Premium Quartz classifications and markets, modified from [5,6].

Table 1 summarizes the results of this study using a green S-HGMS coupling F-freee MAL technology.

Flotation is the main concentration process applied for all minerals. In the iron ore industry, the reverse flotation of quartz is performed in circuits consisting of mechanical cells, columns, or a combination of both types of machines. The feed consists of particles in the size range between 10 and 300 μm. The slimes (fraction < 10 μm) are removed in hydrocyclones [10]. This wide range of particles size impairs the process selectivity due to possible differences in behavior concerning properties such as hydrophobicity, specific surface area, weight, etc. Lima et al. [11] showed the entrainment effect as one responsible for the presence of Fe oxides on the froth. Direct flotation of these oxides by the electrostatic interaction with the etheramine also contribute to decrease the SiO_2 grades of the tailings. Figure 3 represents the entrainment effect on the flotation. Nykänen et al. [12]

demonstrated the presence of iron-bearing minerals on the froth can be explained by hydrodynamic drag (entrainment) and true flotation (adsorption of etheramine with Fe minerals).

Fu et al. [13] evaluated dodecylamine adsorption characteristics of hematite and quartz surface active sites to forecast the flotation behavior of minerals. Results of surface complexation model indicate that the adsorption ability of DDA on the quartz surface is stronger than that of hematite.

Direct flotation of Fe-bearing minerals can be done using alkyl sulfonate (RSO3-) and alkyl sulfate (RSO3-) colletctors at pH´s below the mineral´s isoeletric point [14]. The interaction between these collectors and the Fe-bearing minerals is controlled by electrostatic phenomena, so the flotation´s pH is very important [14]. Although the direct flotation of iron oxides/hydroxides has not been implemented in Brazil, it has been studied since the 60's with fatty acids, the most common anionic collector. As Figure 4 illustrates,

Table 1. Conditions of tests for the purification of high-purity quartz from an iron ore tailing [modified from [7]].

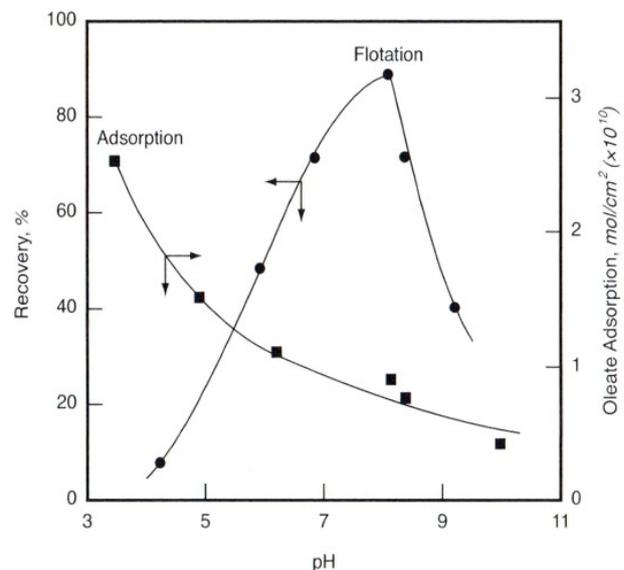| Test	SiO₂ grade in quartz concentrate (%)	Iron oxide content in iron-rich substance (%)	SiO₂ recovery (%)	Feed conditions	SiO₂ purity in quartz after leaching (%)	Leaching conditions
1	98.56 ± 0.13	46.78 ± 0.14	60.59 ± 0.13	Average data: solid concentration, 4%; slurry flow velocity, 0.12 m/s; mass weight, 33.91 kg/h.	99.92 ± 0.01	HNO3, HCl, and H2SO4 molarity ratio of 1:4:1, solid–liquid ratio of 1:4 (g: mL), leaching temperature of 80°C, and stirring
2	96.48 ± 0.87	38.42 ± 0.65	44.21 ± 0.09	Average data: solid concentration, 6%; slurry flow velocity, 0.12 m/s; mass weight, 50.87 kg/h.	99.40 ± 0.03	HNO3, HCl, and H2SO4 molarity ratio of 1:4.5:1.5, solid–liquid ratio of 1:4 (g: mL), leaching temperature of 80°C, and stirring time of 10 h.
3	94.34 ± 0.65	37.45 ± 0.39	43.41 ± 0.08	Average data: solid concentration, 4%; slurry flow velocity, 0.23 m/s; mass weight, 65.99 kg/h.	98.05 ± 0.04	HNO3, HCl, and H2SO4 molarity ratio of 1:4.5:1.5, solid–liquid ratio of 1:4 (g: mL), leaching temperature of 80°C, and stirring time of 15 h.
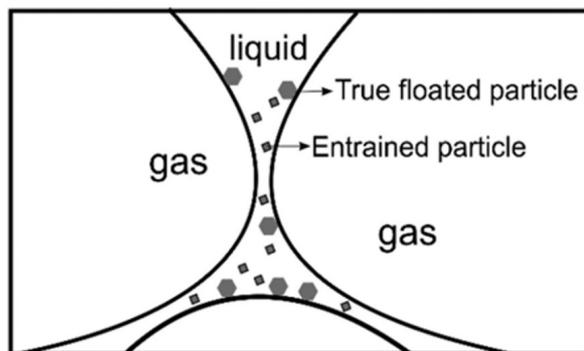
Figure 3. Illustration of the entrainment phenomenon [12].

Figure 4. Hematite recovery after oleate adsorption as a function of pH [15].

the maximum response to flotation with fatty acids occurs at a pH level close to the mineral's IEP [15], in that case, pH = 8.

Quast [16] carried out the measurement of adsorption of oleate onto hematite at pH 4 and as a function of pH at constant oleate addition using differential scanning calorimetry, where oleate adsorption on hematite as a function of pH showed maximum at pH 4 and 9, suggesting co-adsorption of molecular and ionic oleate species at pH 4. Hematite flotation with 10^{-4} molar oleate addition was high for pH values above 4.

Fu et al [17] evaluated the adsorption mechanism of sodium oleate at hematite/quartz-water interfaces, showing there are more positive $FeOH_2^+$ site on the hematite surface, and it has strong adsorption capacity with anionic surfactant.

Magnetic concentration is an older process using the difference of magnetic susceptibility to separate Fe-bearing minerals from gangue minerals as quartz and other silicates and aluminum minerals. According to Dobbins et al., 2009 [18] high intensity magnetic separators (WHIMS) can be used to separate paramagnetic minerals such as hematite, martite and goethite from silicates as quartz, using magnetic

field from 0.7 to 1.2 Tesla. Horizontal WHIMS are the most common machine used in Brasilian´s iron ore beneficiation plants. The vertical WHIMS was developed last two decades combining high magnetic field with pulp pulsation to increase the recovery of coarse and fine particles. Figure 5 shows the differences between the conventional (horizontal) WHIMS and the vertical WHIMS.

More than 80 Mty of iron ore tailings have been generated in the Iron Ore Quadrangle last decades [19], from which more than 85% are generated after removal of slimes and concentration processes with quartz and Fe-oxides as the main minerals. The presence of the Fe-oxides after desliming and concentration processes can be explained by

the entrainment effect and the adsorption of etheramine with Fe minerals [11] and [12]. Premium or high-grade quartz products can be produced from these deslimed tailings after the removal of minerals especially Fe-bearing minerals. The deep understanding and characterization of tailings can determine the alternatives of beneficiation process to remove the iron oxides and other minerals. This paper describes alternatives of concentration of a typical tailing by high intensity magnetic separation followed by alternatives of flotation of quartz and flotation of Fe-oxides, using deep characterization techniques prior the concentration alternatives to evaluate the mineralogical composition and associations with quartz.

2 Materials and methods

Two size tailings are typically generated by the beneficiation plants in the Iron Ore Quadrangle, after grinding, desliming and concentration processes (reverse flotation of quartz and magnetic separation) to obtain high grade pellet feed for the steel industry. A typical iron ore deslimed tailing from the Iron Quadrangle region was sampled for this work, where different characterization methods followed by high intensity magnetic concentration and two flotation routes were evaluated aiming to achieve the quartz products. The slime tailing was not considered in this study. Figure 6 ilustrates the sample collected and the steps of this study.
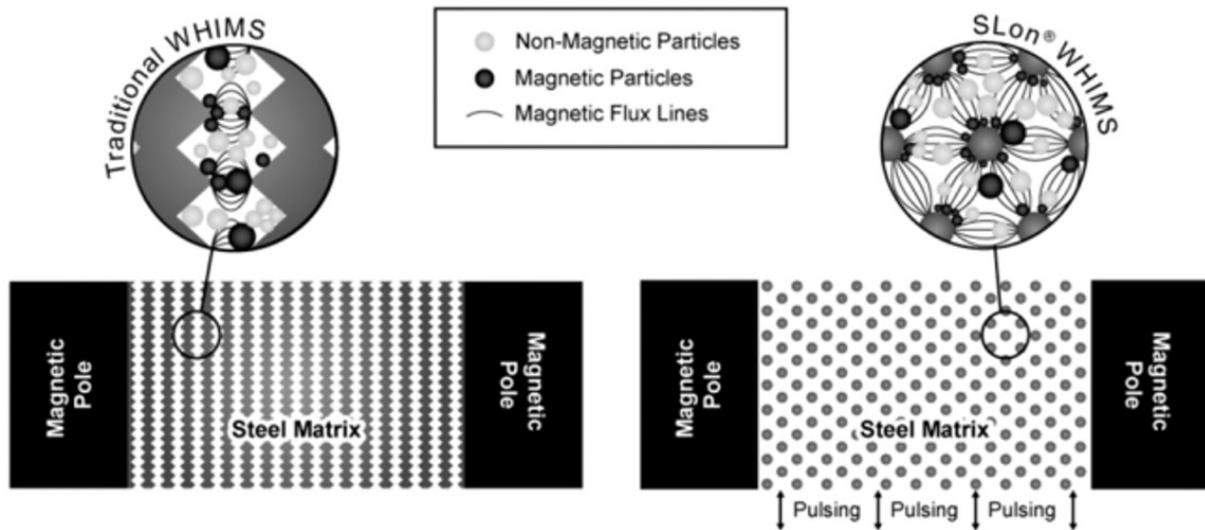

Figure 5. Matrix comparison between conventional WHIMS and vertical WHIMS [18].
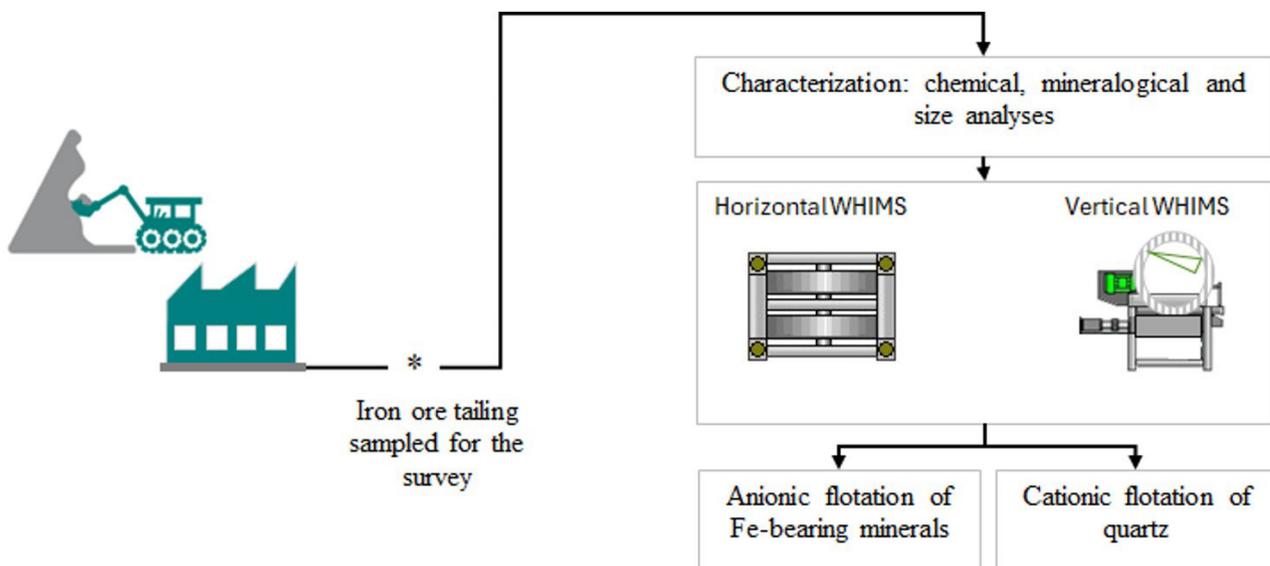

Figure 6. Typical flotation circuit where the sample was collected with the characterization and tests to achieve quartz products with different qualities. (*) Iron ore tailing sampled for the survey.

Chemical composition (Fe, SiO_2, Al_2O_3, P, Mn, CaO, MgO, and LOI) was analysed by X-ray fluorescence (XRF using the Thermo Fisher Scientific Niton XL3t).

The mineralogical composition was determined using an X-ray diffraction technique and the automated scanning electron microscope (QEMSCAN® system) with a tungsten source from the FEI company (Hillsboro, OR, USA). The iDiscover software (everly Hills, CA, USA) was used for the measurements and the data processing. The PMA (Particle Mineral Analysis) analysis model was used. The QEMSCAN® was operated using a voltage acceleration of 15 kV and a current of approximately 5 nA. For the X-ray diffraction, the samples were pressed and analyzed in a PANalytical X-ray diffractometer (Empyrean model) while using a Co tube. The crystalline phases were identified and interpreted by the High Score Plus software (Malvern Panalytical, Worchestershire, UK) and the mineralogical composition was quantified by the Rietveld method. An optical microscope (Leica DM 2500P) was used to evaluate the morphology of the minerals. X-ray microtomography was used to evaluate the 2D and 3D images of different phase densities.

The size distribution was wet determined using square 500 × 500 millimetres (mm) sieves on the sizes: 1.0mm, 0.500mm, 0.300mm, 0.210mm, 0.150mm, 0.106mm, 0.074mm, 0.044mm and 0.037mm. Laser sizer instrument (Malvern® Mastersizer 2000, Malvern, Malvern, UK) was combined to determine particles smaller than 0.037mm.

The sample was first submitted to magnetic concentration pilot tests using the horizontal and the vertical WHIMS, aiming to compare the performance of these machines to achieve the highest % SiO_2.

A Minimag GS-50 was used for the horizontal WHIMS tests and a 500mm of ring diameter was used for the vertical WHIMS tests, both with 1,0 Tesla, matriz of 1.5mm and 35% of solids on the pulp.

Bench scale flotation tests in rougher-cleaner stages were conducted in a 2.4 Liters Denver cell machine with 1000 rpm and 40% of solids on the pulp. Two different flotation strategies were studied using the no-magnetic from the magnetic concentration tests to yield a quartz concentrate with minimum of 99% SiO_2: a) Anionic flotation of iron-bearing minerals; b) Cationic flotation of quartz.

The following anionic collectors produced by Clariant were evaluated on the flotation of Be-bearing minerals at pH values of 5 and 8: Flotinor 5806 (mixture of fatty acids and sarcosine), Flotinor 7580 (mixture of alkyl sulfonates), Flotinor 16261 (alkylsulfosuccinamate), Flotinor 17744 (wetting agent mixture with sulfosuccinate). Prior to the reverse flotation, the pulp pH was adjusted with HCl (10% in weight) and conditioned with the anionic collectors for 5 minutes.

The following cationic collectors for the flotation of quartz were evaluated at pH = 10: Flotigam 7100 (alkyl ether-amine), Flotigam 7848 (alkyl ether-amine), Flotigam 18957 (mixture of quaternized ammonium salts). The pulp was conditioned with 300 g/t of corn starch 5:1 (2% in weight) for 5 minutes to ensure Fe-bearing minerals and non-liberated quartz-hematite particles were depressed at pH = 10 (adjusted with NaOH 10% in weight). Thereafter, the cationic collector was added to interact only with the quartz particles for 1 minute.

The products of magnetic and flotation tests were conducted by X-Ray fluorescence (XRF using the Thermo Fisher Scientific Niton XL3t) and by ICP-OES to detect traces of iron in the high purity quartz concentrates.

3 Results and discussions

Table 2 shows the grades by sizes of the sample used for the tests, with lower grade of SiO_2 on the coarse sizes especially +300 μm and +210 μm, what represent less than 5% of the total feed as show on the size distribution on the Figure 7.

The mineralogical analysis of the optical microscope is shown on the Table 3, where quartz is the main mineral

Table 2. Grades by sizes of the iron ore tailing feed

Size (μm)	Chemical (%)				
	SiO_2	Al_2O_3	Fe_2O_3	P_2O_5	LOI
+1000	40.00	0.84	58.00	0.180	3.07
+500	59.60	0.63	40.40	0.120	2.09
+300	83.50	0.31	16.20	0.050	0.79
+210	75.12	0.01	23.88	0.010	0.18
+150	87.39	0.01	9.61	0.010	0.15
+106	92.53	0.01	7.47	0.010	0.06
+74	94.70	0.37	6.05	0.010	0.10
+44	88.60	0.19	11.40	0.010	0.26
+37	84.50	0.27	16.20	0.010	0.16
-37	84.50	0.71	15.50	0.030	0.74
Global	**86.25**	**0.17**	**13.14**	**0.020**	**0.32**

and hematite is the main Fe-oxide mineral. The results of AMICS with mineral liberation in different sizes is shown on the Figure 8, where quartz have maximum liberation at -210+106 µm and Fe-oxides maximum 90.9% liberation at the finest sizes. In global terms, quartz particles are 97.7% of liberation while Fe-oxides 83.3% of liberation.

Figures 9A and 9B from the MEV / QUESCAM show different associations and micro inclusions. Fe-oxides are the main mineral associated with quartz. Other associations with quartz include apatite and other silicate minerals. These associations and micro inclusions can be a challenge to obtain high-purity quartz products using traditional beneficiation methods without fine grinding, considering also the size limits of quartz products for the markets [1] and [6].

The presence of Fe-oxides inclusions on quartz particles can be observed also with the X-ray microtomography analysis as show Figure 10, where the yellow parts represent quartz (lower density) the blue parts are Fe-oxides (higher density).

Table 3. Mineralogical analysis of the iron ore tailing sample by optical microscope
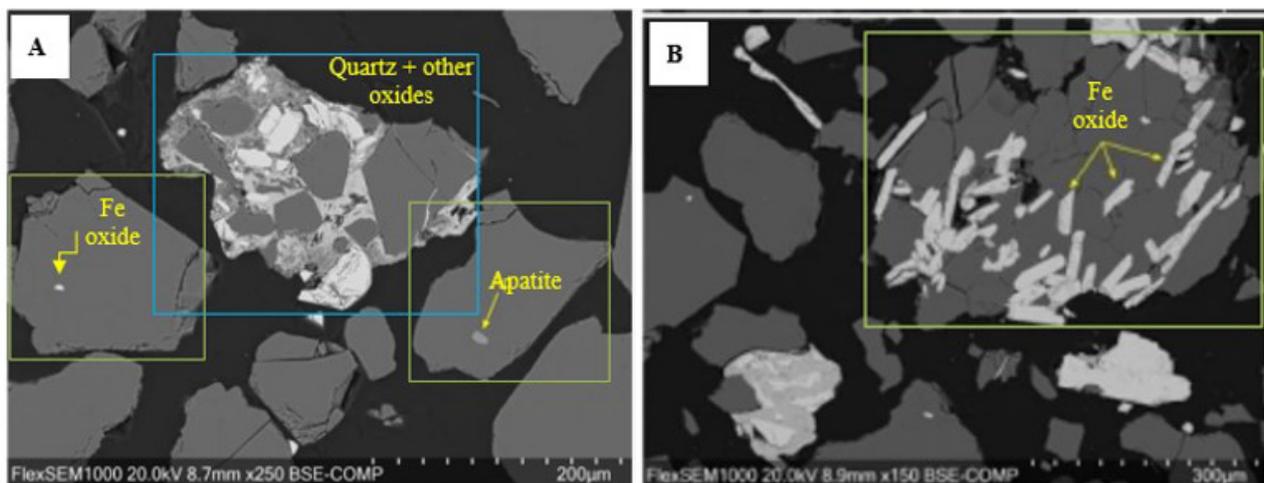
Mineralogical analysis (%)				
Quartz	Hematites	Martite	Goethite	Others
87.53	10.59	1.31	0.53	0.04
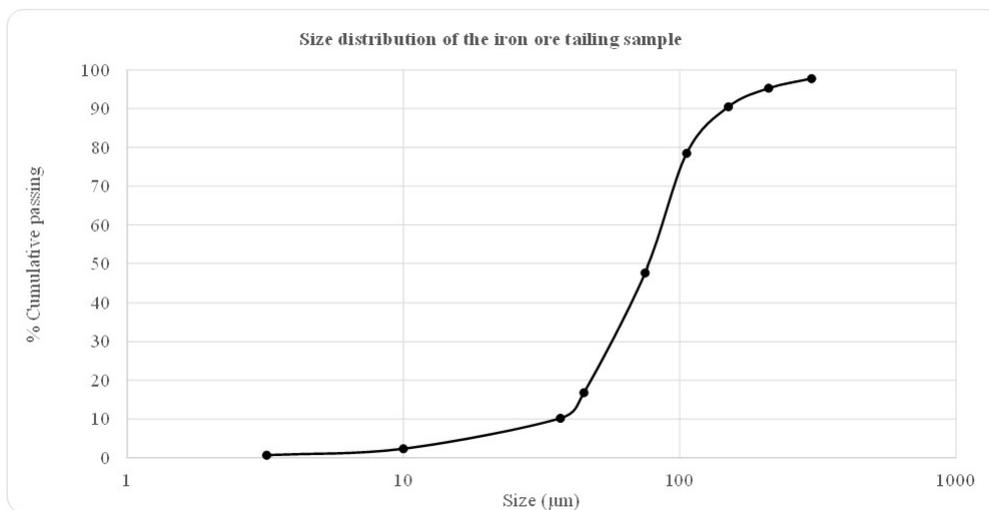
Figure 7. Size distribution of the iron ore tailing sample.
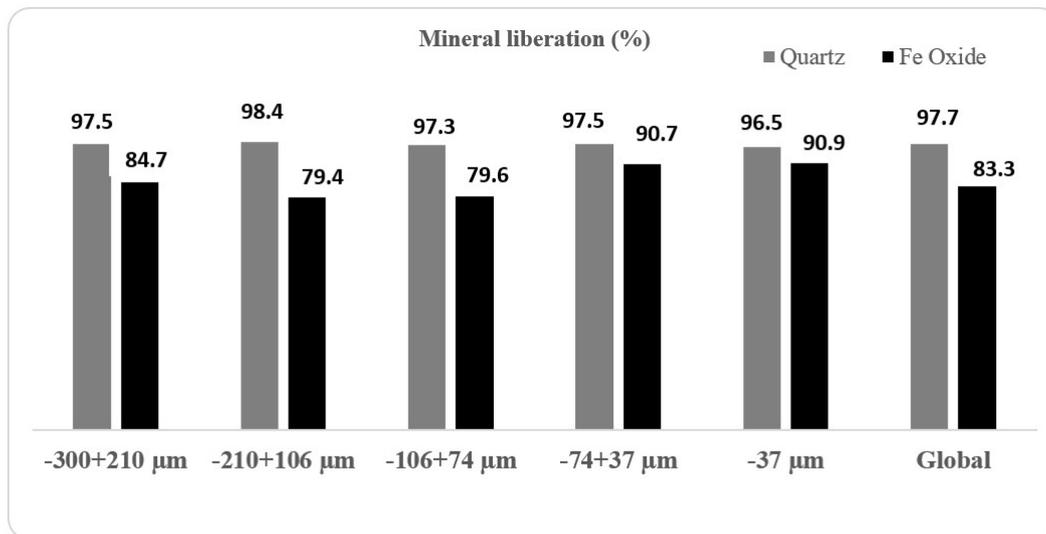
Figure 8. Liberation degree of quartz and Fe oxide for different sizes.

Figure 9. A- Micro inclusions of Fe-oxide and apatite with quartz. B: Fe-oxide association with quartz.

The results of magnetic separation are shown on the Table 4, indicating the possibility of achieving 81.49% of quartz with 97.46% SiO_2 with the vertical WHIMS. Furthermore, the vertical WHIMS enabled Fe product with better quality. Considering the results of Figure 8 where 97.7% of quartz occur as free particles, the results of Table 4 suggest that all free-quartz particles were recovered by the vertical WHIMS. The combined effect of magnetic field and pulsation can explain the better results of the vertical WHIMS with more removal of Fe-oxides from the quartz.

Bench scale flotation tests were performed with the no-magnetic (quartz) obtained with the vertical WHIMS via cationic flotation with different collectors and dosages aiming the recovery of particles composed mainly of quartz with few inclusions of Fe-oxides and other minerals. The traditional collector Flotigam 7100 (alkyl ether-amine) had a very low performance, so its results were not considered in this paper. The collector Flotigam 18957 (400g/t) at pH 10,0 enabled achieving 94% of quartz with 99.45% SiO_2 and 0.79% Fe_2O_3, classified as premium quartz as the results of Table 5. These results indicate the attempting of the cationic flotation of quartz particles with few inclusions of Fe-oxides can be an option to obtain premium quartz with high recovery, the challenge is the high froth rate and high dosage of collector.

The other strategy to increase the %SiO_2 after magnetic separation was via anionic flotation of Fe-oxides, considering this mineral is the 2º most abundant occurring as free particles or in associations and inclusion with quartz particles. According to the results of Table 6, 180 g/t of Flotinor 7580 at pH 5 was the best condition to achieve 99.20% SiO_2 and 0.98% Fe_2O_3 with 81% recovery.

Table 7 shows the comparative results between cationic flotation of quartz and anionic flotation of Fe-oxides, where the first option enabled quartz with slightly higher % SiO_2 and lower Fe content. The use of starch can explain the best performance of the cationic collector, contributing for
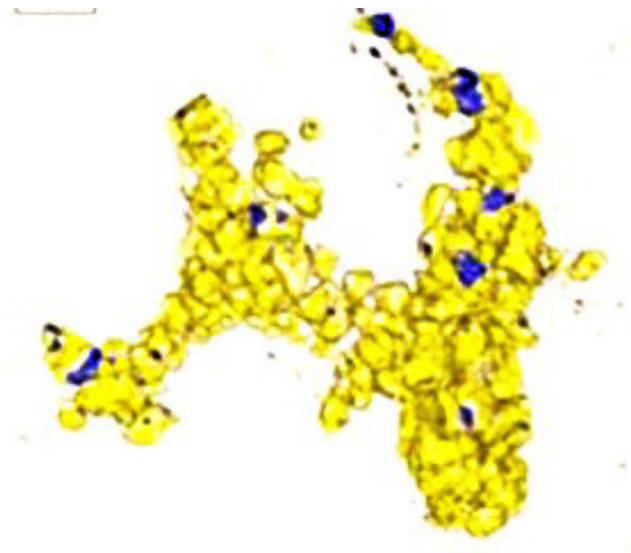
Figure 10. X-ray microtomography picture, where quartz is represented by the yellow parts and Fe-oxides the blue parts.

the depression of associated particles with greater amount of Fe-oxides. The total impurities, except %Fe_2O_3, on the quartz products obtained in both flotation options was smaller than 0.260% or 2600 ppm with possibility to be destinated for glass, silicates, foundry and artificial stone industries. Economic analyses shall be done for better evaluation between the two flotation options, talking into account the chemicals prices and dosages and the effect of the amount of froth and residence time on the flotation´s machines scale-up.

The use of acid leaching, according to prior studies [7-9] after magnetic separation and flotation shall be done aiming the removal of associated and included impurities particles, especially Fe-oxides, on the quartz particles to achieve higher grade SiO_2 products and trying to obtain high-purity quartz from iron ore tailings.

Table 4. Results of magnetic separation

WHIMS	ID	Mass (%)	SiO$_2$ (%)	Fe$_2$O$_3$ (%)
Horizontal	Magnetic	21.28	47.49	50.76
	No-Magnetic	**78.72**	**96.73**	**2.97**
Vertical	Magnetic	18.51	35.09	62.91
	No-Magnetic	**81.49**	**97.46**	**1.84**

Table 5. Results of cationic flotation of quartz with different collectors and pH 10,0

Colector	Recovery of quartz (%)	% SiO$_2$	% Fe$_2$O$_3$
Flotigam 18957 (300 g/t)	87.0	98.84	1.14
Flotigam 18957 (400 g/t)	**94.0**	**99.45**	**0.79**
Flotigam 7848 (120 g/t)	82.0	99.06	0.92

Table 6. Results of anionic flotation of Fe-oxides with different collectors, pH 5 or 8

Colector	Recovery of quartz (%)	% SiO$_2$	% Fe$_2$O$_3$	Froth (%)
Flotinor 7580, 180 g/t, pH 5	**81**	**99.20**	**0.98**	19
Flotinor 1626 1, 180 g/t, pH 5	80	98.91	1.19	20
Flotigam 5806, 180 g/t, pH 5	62	98.83	1.27	38
Flotinor 17744, 180 g/t, pH 8	84	98.81	1.29	16
Flotinor 7580, 180 g/t, pH 8	71	98.77	1.33	29
Flotinor 17744, 180 g/t, pH 5	70	98.76	1.34	30
Flotinor 1626 1, 180 g/t, pH 8	97	98.64	1.46	3

Table 7. Comparative results of cationic flotation of quartz and anionic flotation of Fe-oxides

Parameter	Cationic flotation of quartz	Anionic flotation of Fe-oxides
Recovery of quartz (%)	94.0	81.0
Froth (%)	94.0	19.0
% SiO$_2$	99.46	99.20
% Fe$_2$O$_3$	0.79	0.96
% Al$_2$O$_3$	0.200	0.180
% P$_2$O$_5$	0.010	0.010
% CaO	0.024	0.020
% MgO	0.015	0.012
% Mn	0.001	0.001
% LOI	0.007	0.005

4 Conclusions

Different methods were used on the characterization of typical iron deslimed tailing, indicating different minerals associated and included with quartz particles as Fe-oxides, phosphates and other silicates.

Magnetic concentration using vertical WHIMS enabled obtaining quartz with 97.46% of SiO$_2$, after the removal of the total free Fe-oxides, further obtaining Fe product with 62.91% Fe$_2$O$_3$.

Premium quartz with 99.20 to 99.46%% SiO$_2$, 0.79 to 0.96% of Fe$_2$O$_3$ and less than 0.260% of other impurities was obtained thought the combination of the vertical WHIMS and cationic flotation of quartz or anionic flotation of Fe-oxides.

This quartz quality can be destinated for for glass, silicate, foundry and artificial stone industries.

The presence of small associated and included impurities particles, mainly Fe-oxides, prevents obtaining higher purity quartz (> 99.46% SiO$_2$). Leaching with different acids can be an alternative to remove these impurities and obtain high-purity quartz from iron ore deslimed tailing.

This paper contributed to the circular economy through better understanding the iron ore tailings from the Iron Quadrangle by deep characterization methods, showing the potential to obtain premium quartz products by the combination of wet high intensity magnetitic separator with cationic flotation of quartz or anionic flotation of Fe-oxides.

Acknowledgements

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