MINERAL LIBERATION EVOLUTION AFTER DIFFERENT DEGREES OF COMMINUTION ON ITABIRITES*

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Abstract

This paper presents a mineral liberation study on two iron ore samples from the Quadrilátero Ferrífero, in Brazil: compact itabirite of low-grade metamorphism (CI) and friable itabirite of high-grade metamorphism (FI). The quartz liberation spectrum for both samples have been obtained through QEMSCAN analyses, carried out for several comminution degrees, to compare the liberation characteristics and to evaluate the evolution of the quartz's liberation degree after fragmentation. The results have shown significant differences in liberation spectrum between the itabirite samples studied. The liberation process of FI was easier than of CI, due its high porosity and larger crystal size compared with CI, which has shown more complex mineral assemblages with low porosity and smaller crystals. The quartz liberation spectrum has shown that grinding at P_{95} of 0.210 mm for the FI sample, and of 0.045 mm for CI sample, should result in satisfactory liberation for subsequent concentration step.
Keywords: Itabirite; Iron ore; Mineral liberation spectrum; QEMSCAN.

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1 INTRODUCTION

1.1 Geological Aspects

Brazil has huge iron ore reserves, such as the Quadrilátero Ferrífero`s deposits (Minas Gerais State), where, in addition to the high-grade ores, occurs the quartz itabirites from the Caue Formation, Minas Supergroup, which are banded iron formations formed by the metamorphism of a "laminated rock with chert and iron oxides" as described by Dorr [1]. These rocks are composed mainly of quartz and iron minerals, with some other trace phases. In some mines, the high-grade and friable itabirite are currently exploited. Characterisation studies are being developed to allow the exploitation of compact and poor itabirites. Due to the lower Fe content and the mineralogical associations, the process flowchart for poor itabirite ores is constituted by grinding of the whole run of mine, with subsequent concentration by inverse flotation of quartz to produce pellet feed fines. In this context, mineral liberation studies are important to define the size at which the ore must be comminuted to liberate the mineral quartz and to ensure good performance of the concentration without, however, overgrinding the ore.

As well known, the texture and the arrangement of mineral grains in the rock greatly influence the manner of particle fragmentation and therefore the mineral liberation. Metamorphism and strain processes beyond the supergene processes were the main factors that changed the texture of iron ore from Quadrilátero Ferrífero. The region was subjected to at least two major events of strain and metamorphism which have altered the structural and textural characteristics of the rocks. However, the intensity of metamorphism and deformation was not uniform across the area, varying with the differences in tectonic processes and according to the rocks' rheological behaviour (Rosière et al. [2]; Chemale Jr. et al. [3] and Pires [4]).

The Quadrilátero Ferrífero tectonic events decreased from east to west, and divided the region into two main areas: high strain and low strain (Rosière et al., [2]). Pires [4] proposed the subdivision of the region in four metamorphic zones: Grunerite Zone (GZ), Cummingtonite Zone (CZ), Actinolite Zone (AZ), and Tremolite and Anthophyllite Zone (TAZ). The metamorphic grade increases from the first to last. Figure 1 shows a schematic map of Quadrilátero Ferrífero divided into the areas mentioned above. As concluded by Rosière and Chemale Jr. [5], the deformation gradient follows the metamorphic zoning.
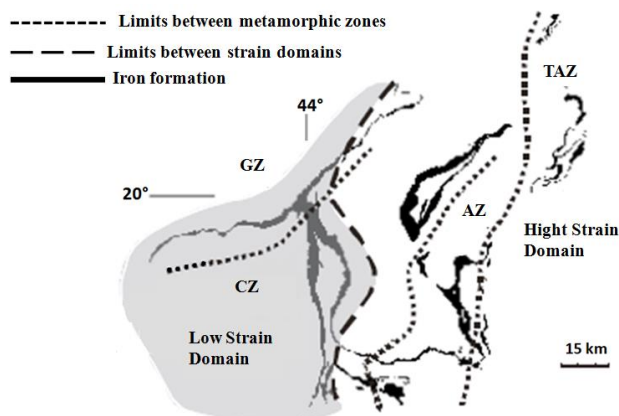

Figure 1. Metamorphic zones and strain domains in the Quadrilátero Ferrífero region. (Rosiére and Chemale Junior, [5]).

According to Rosière [6], the increase in the degree of metamorphism contributed to a general increase of the mineral crystals' size. James, apud Dorr [1], stated that the size of quartz and hematite grains varies systematically with the degree of metamorphism. James and Dorr, apud Klein [7], defined the metamorphic grade of the iron formations through the average dimensions of quartz crystals. Therefore, one expects some variation in the crystal size between orebodies from the different zones.

Supergene processes also have changed the itabirite's texture. The friable ore has suffered a natural softening due to the leaching of silica from the jaspilites (or itabirites) that occurs in the aerated zone above the water table, where the percolation of meteoric water is free. These would be conditions in which most of the Fe stays immobile and the silica would be leached, producing a porous and friable rock (Dorr, [1]; Biondi, [8]).

1.2 Mineral Liberation

There is an intuitive correlation between fragmentation and mineral liberation, i.e., the finer the fragmentation, the more liberated will be the mineral phases. However, there are two reasons why one should limit the fragmentation to degree of liberation strictly necessary: first, the comminution operations generally represent a high-cost step of mineral processing; second, because the thinner the mineral particles, the worse separation results, overall. The problem that arises in each case is to define the extent to which lead fragmentation obtains sufficient mineral liberation, and this is determined by the recovery and the quality of the concentrate.

According to Gaudin [9], one generally divides the ore dressing in two major steps— liberation and separation—and the second stage would be impractical if the first is not performed successfully. According to that author, this statement is partially true, because it is possible to obtain some degree of separation of locked particles of various types. Following the same concept, Miller *et al.* [10], and Gottlieb *et al.* [11], stated that for practical applications in mineral processing, a particle can be considered free if its composition by volume is greater than 90% of the mineral phase of interest.

Beyond the degree of liberation, it is also important to know the relative amounts of locked particles with different proportions of the mineral of interest and of gangue— which can be obtained in the liberation spectrum—which consists of a mineralogical composition distribution of particles in a population. This enables the quantification of the amount of free particles of the mineral of interest and the gangue mineral, as well as the relative amounts of locked particles (Neumann et al., [12]; Lin and Miller, [13]). The liberation spectrum may be obtained by various methods, including the QEMSCAN® (FEI Company), an automatic system for quantitative mineralogical analysis, which comprises SEM (Scanning Electron Microscope) and EDS (Energy Dispersive X-ray spectrometers) technology, plus software for automatic image analysis and data processing (Liu et al., [14]; Jaime et al., [15]; Spencer and Sutherland, [16]).

2 MATERIALS AND METHODS

Two itabirite samples of different degrees of metamorphism and supergene alteration have been collected—a compact itabirite (CI) and a friable itabirite (FI)—from the western and eastern regions of Quadrilátero Ferrífero/MG respectively, in mines

currently operated by Vale S/A. Both samples are composed mainly of iron oxides and quartz, with about 50 wt.% Fe (Table 1).

Table 1. Chemical analysis

Sample	Chemical Analysis (%)								
	Fe	SiO_2	P	Al_2O_3	Mn	TiO_2	CaO	MgO	PPC
IC	49.57	27.02	0.073	0.60	0.075	0.025	0.112	0.043	1.07
IF	47.37	28.86	0.071	0.43	0.086	0.021	0.005	0.001	2.43

Each sample, approximately 100 kg, has been crushed in laboratory by a roll crusher in 1.0 mm. After, the crushed ores were splitted through a rotary splitter on representative subsamples and then subjected to laboratory ball grinding, with P_{95} in several sizes (0.210 mm, 0.150 mm, 0.105 mm, 0.075 mm and 0.045 mm). These tests have been carried out with pulp at 65 wt.% solids, in a laboratory mill with 203.2 mm diameter and 304.8 mm length, without liners, rotating at 7.54 rad/s. Table 2 shows the distribution of the ball load in the mill.

Table 2. Ball load for the laboratory grinding tests

Diameter (mm)		Mass (g)	
Value	Acceptable Deviation	Value	Acceptable Deviation
38,1	± 3	11007	± 50
25,4	± 2	3430	± 20
19,1	± 2	1527	± 10
12,7	± 2	913	± 5

The milling time has been controlled by a programmable timer. Several tests have been performed at different times until P_{95} was achieved in the control size of the test. Such verification has been conducted by wet sieving with a subsample of the ground product obtained in a pulp splitter. After grinding, the samples were divided into pulp splitter, sieved into narrow size fractions, and after, they were subdivided into micro rotary splitter for mineralogical analysis.

Mineralogical characterisation has been performed through QEMSCAN® for all comminution products, with the main objective to assess the evolution of quartz liberation spectrum with different degrees of fragmentation. The mean size of mineral grains (ferrous and gangue) has been measured in equivalent circular diameter by QEMSCAN, from the sample crushed in 1.0 mm.

QEMSCAN® model Quanta 650 (FEI Company), with tungsten source, has been used. Polished sections of 30 mm diameter have been prepared with the addition of ultrapure graphite in the same particle size range of ore particles for deagglomeration. The PMA analysis method (mineral particle analysis) with two-dimensional measurements has been applied, by individual particle, to obtain the quartz liberation spectrum.

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3 RESULTS AND DISCUTION

3.1 Macroscopic Description

Figure 2 depicts photographs of macroscopic aspects of compact itabirite (CI). As can be observed, there is a considerable quantity of blocks (Figure 2A). It is possible to see the typical occurrence of alternating laminations of ferrous minerals and quartz, both very fine-grained, while in most cases it is impossible to differentiate the crystals with the naked eye (Figures 2B, 2C and 2D). There is dispersion of ferrous minerals in laminations composed predominantly of quartz and quartz crystals scattered in predominantly ferruginous layers, i.e., the laminations are not mineralogically homogeneous. The rock is very resistant, and apparently it is hard to comminute it.
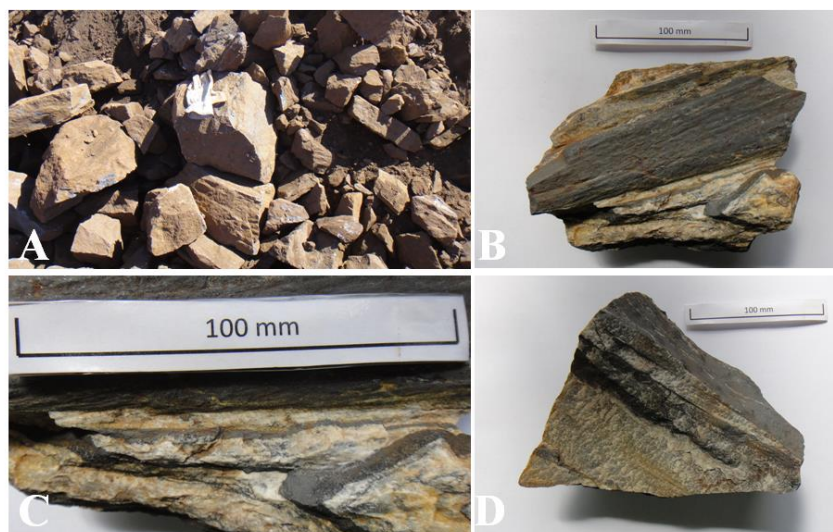
Figure 2. General aspects of the compact itabirite (A) and some fragments of the sample (B, C, D).

Figure 3 shows the friable itabirite (FI). Also found in this sample were ferrous minerals and quartz, predominating in alternating layers and laminations. It is possible to see goethite or limonite lenses, which in some cases is dispersed on the other mineral crystals. The crystals, in general, are noticeably larger, being distinguishable with the naked eye. This fact is evidence of the trend of occurrence of larger crystals occurring in the eastern region of the Quadrilátero Ferrífero, which has been submitted to most intense metamorphism (Rosiere and Chemele Jr., 2000). In the FI sample, there is a wide variation in the particle size range (Figure 3A). The ore is fragile, it is possible to detach layers by hand (Figure 3B), and when detaching these layers, a considerable amount of quartz crystals is easily detached from the rock (Figure 3D).
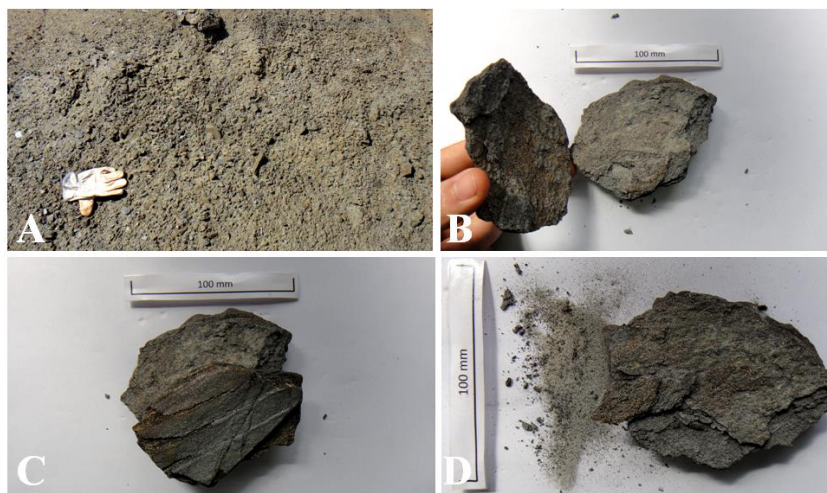
Figure 3. General aspects of the friable itabirite (A) and some fragments of the sample (B, C, D).

3.2 Size Distribution of the Comminuted Samples

The particle size distributions of the comminuted samples are grouped in Figures 4 and 5, for CI and FI respectively. As can be observed, all specifications in grain size control proposed for the study have been met: P95 of 0.210 mm, 0.150 mm, 0.105 mm, 0.075 mm and 0.045 mm.
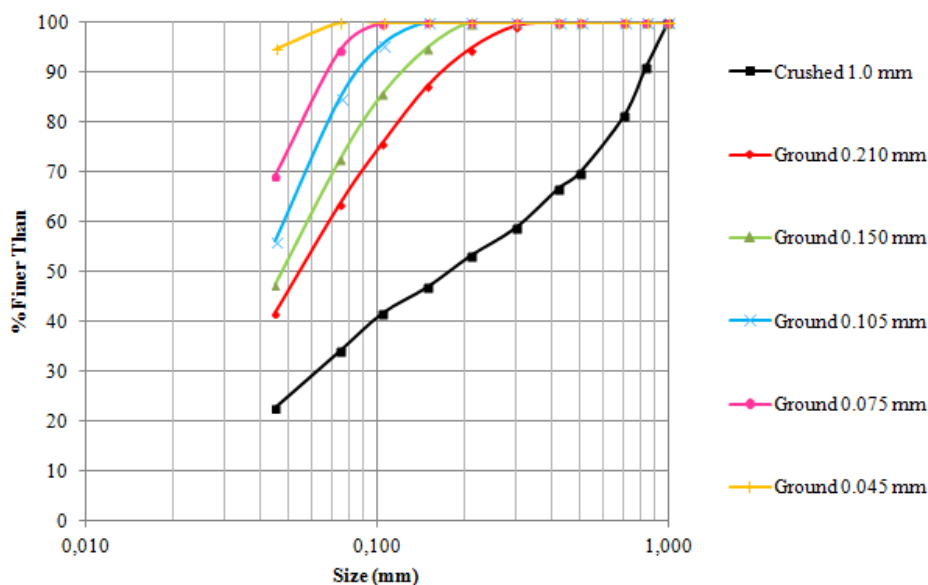

Figure 4. Size distribution for the different degrees of comminution – compact itabirite.
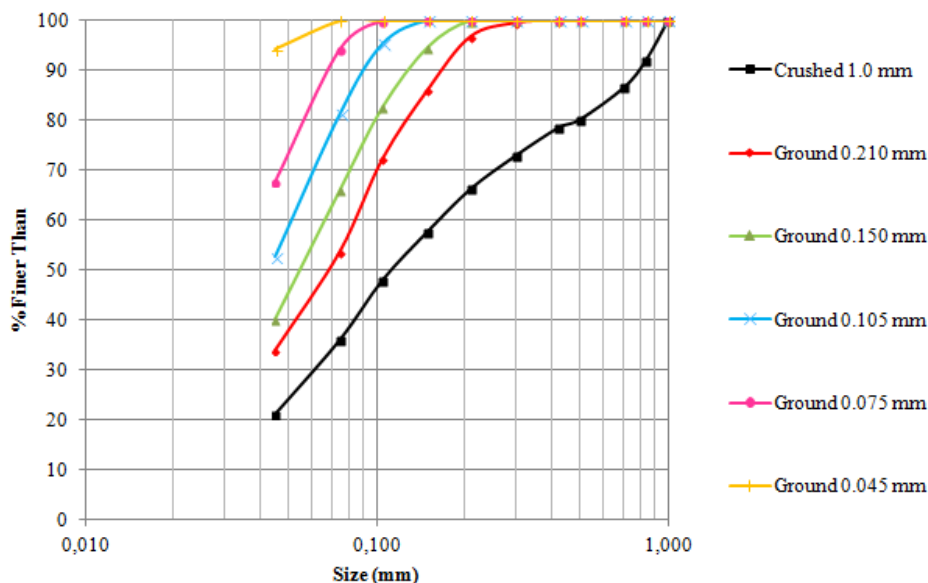
Figure 5. Size distribution for the different degrees of comminution – friable itabirite.

3.3 Crystal Size

The global average size of the crystals of ferrous minerals and quartz are presented in Table 3. As can be observed, even with some variations in certain size fractions, it was found that, in general, the crystals are larger in the FI sample compared to the CI sample, for both ferrous and gangue minerals, as already observed in the macroscopic evaluation. This difference is attributed to the FI sample from the east region of Quadrilátero Ferrífero, which was submitted to higher intensity of metamorphism; these data corroborate the statements of Rosiere [6] and James (1955) on the correlation between metamorphic grade and crystal minerals size. Of course, it is noteworthy that the comminution at 1.0 mm may have reduced the differences in crystal size between the two samples.

Table 2. Crystal size for the samples crushed at 1.0 mm

Sample	Mineral	Size µm							
		-1000+500 µm	-500+210 µm	-210+150 µm	-150+105 µm	-105+75 µm	-75+45 µm	-45 µm	Average
IC	Quartz	79,73	55,61	53,18	42,60	47,70	38,00	19,09	51,11
IF		156,49	117,66	110,88	82,95	68,09	46,19	16,68	88,63
IC	Fe Oxides	123,00	97,02	87,22	75,30	62,88	46,46	22,09	77,68
IF		188,16	124,99	102,72	79,65	60,19	45,64	17,80	94,45

3.4 Quartz Liberation Spectrum

The quartz liberation spectrum for CI and FI samples obtained through QEMSCAN® are shown in Figures 6 and 7, including all of the comminuted samples.
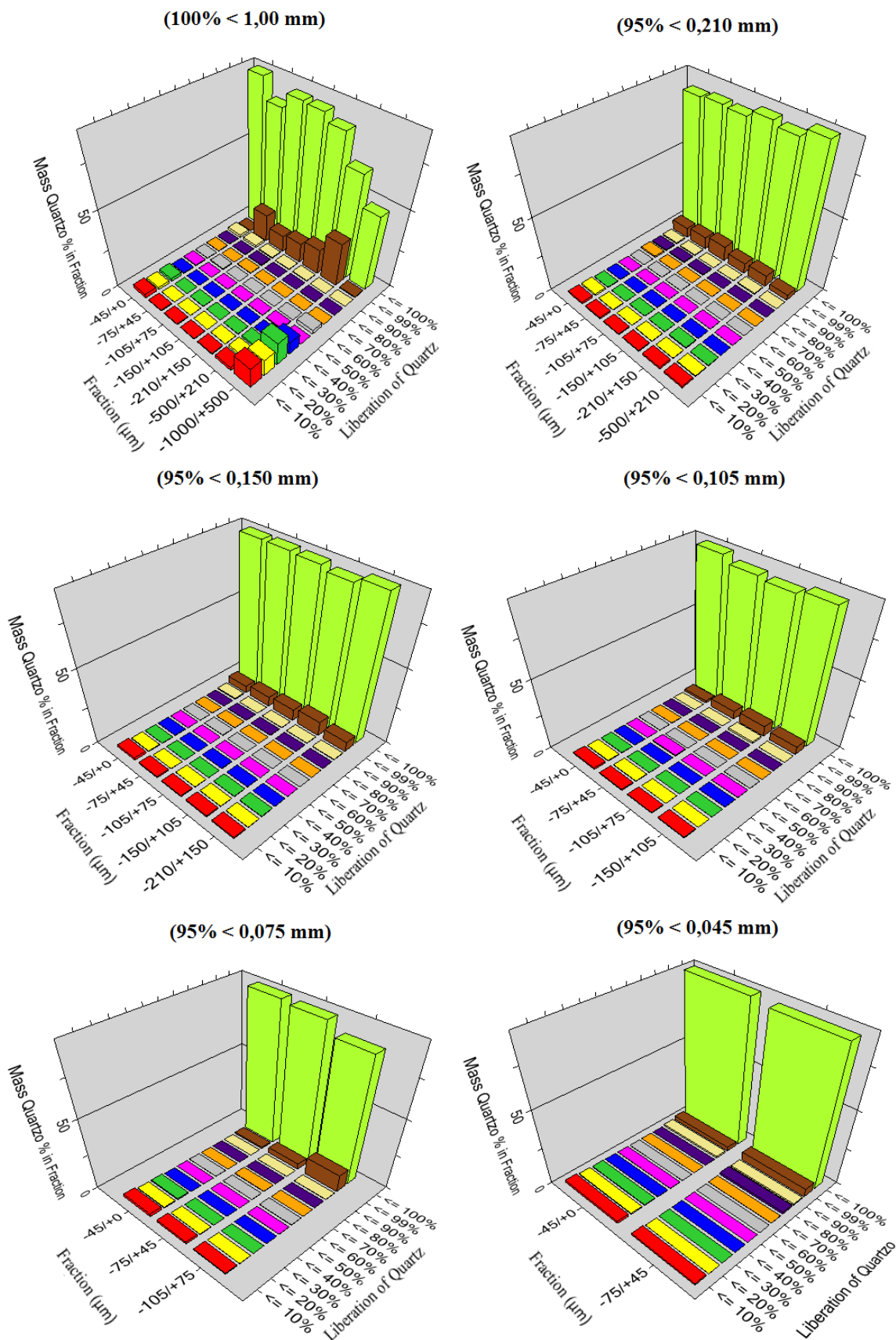
Figure 6. Quartz liberation spectrums for the friable itabirite sample.

(100% < 1,00 mm)

(95% < 0,210 mm)

(95% < 0,150 mm)

(95% < 0,105 mm)
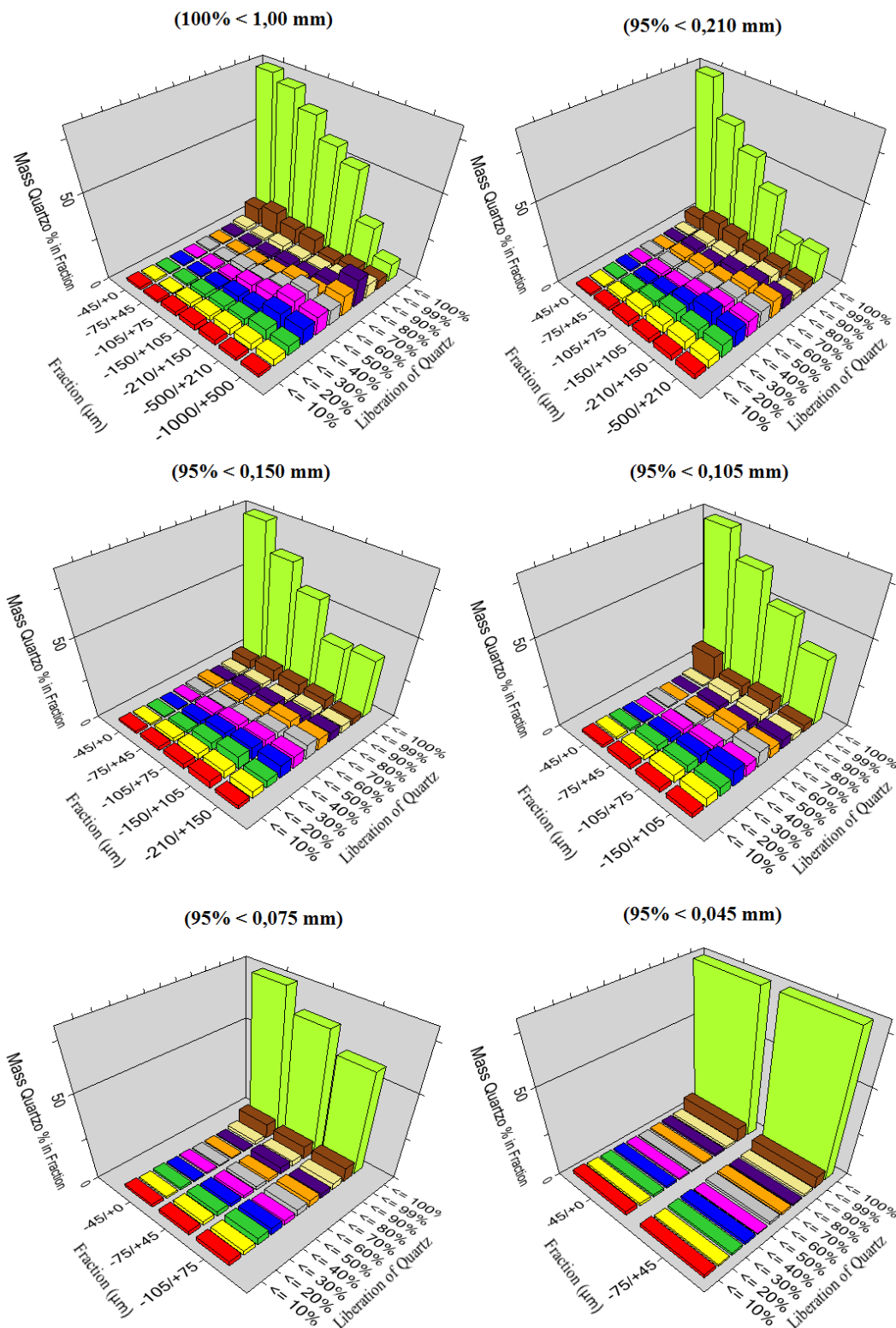
(95% < 0,075 mm)

(95% < 0,045 mm)

Figure 7. Quartz liberation spectrums for the compact itabirite sample.

From the liberation spectrum of the FI sample crushed to 1.0 mm (Figure 6), it was found that in the fraction -1.0 + 0.5 mm, approximately 48% of quartz is free, and the remainder is distributed mainly in particles of less than 40% of quartz in the composition. For sizes less than 0.5 mm, the percentage of free quartz rises further, and it is no longer observed large percentage of particles with composition of quartz

less than 40 wt.%, but above 90 wt.%. In the fraction size -0.21 + 0.15 mm, about 80% of quartz is free, and in the lower size fractions the degree of liberation exceeds 85%, except for the fraction -0.075 + 0.045 mm, in which the percentage of free quartz kicks to 78%, probably due to some measurement deviation. The degree of liberation reaches 91% in the fraction -0.045 mm.

For the FI sample, there has been a considerable increase in the liberation of quartz with grinding in 0.210 mm, for all size classes (Figure 6). The liberation spectrum has shifted almost entirely to the classes upper to 99% quartz in the particle, and thus occurred after grinding in the other control sizes, being obtained very similar distributions. These data have shown that is easy to liberate the crystals in the friable itabirite, and this fact may be associated with the textural characteristics of the ore, as the significant occurrence of connected pores forming zones of weakness between the crystals, as well as the crystal size, which is larger in this case.

The liberation spectrum of the 1.0 mm crushed product of the CI sample (Figure 7) is more complex when compared to the FI sample (Figure 6). The liberation degree of quartz in the CI sample is lower than that of the FI sample in all size fractions. In thicker fractions, -1.0 + 0.5 mm, there is a broad spectrum, with particles distributed across all classes of composition, with the highest concentration between 80 and 40% classes. The liberation degree of this fraction is only 11%. For the finer size fractions, the degree of liberation has increased gradually, peaking at 80% in the fraction - 0.045 mm.

Despite the presence of a large proportion of free quartz particles from the first degree of comminution (1.0 mm), the CI sample spectrum remained more distributed until grinding in 0.075 mm. The spectrum relating to 0.045 mm ground product presents the vast majority of the particles into the 99% and 100% classes. These data have shown that is relatively difficult to liberate mineral phases in the compact itabirite, reflecting the textural characteristics of the ore, whose particles are more resistant, along with less porosity, and smaller and overlapping crystals.

For both studied samples, it was observed that a considerable proportion of free quartz particles were already in the early stages of comminution. This fact highlights the importance of applying pre-concentration processes in the processing of these ores. The free particles of the gangue mineral must be removed as soon as possible to minimise energy consumption in fragmentation. Every effort to reduce size for liberation must be applied to locked particles.

Based on the results presented in Figures 6 and 7 and considering that particles in which quartz proportion is more than 90% are free [10,11], grinding in 0.210 mm and 0.045 mm, for FI and for CI respectively, should result in a satisfactory degree of liberation for subsequent concentration through inverse flotation.

4 CONCLUSION

Based on liberation studies carried out with the iron ore samples, friable and compact itabirite, it was concluded that there are significant differences between the two ore samples in mineral liberation characteristics. The easer liberation of quartz on FI compared with CI can be due to its larger crystal size, which implies in the trend to satisfactory degree of liberation by comminution in a coarser size. The CI sample has more complex texture, with low porosity and no occurrence of zones of weakness between the crystals, which are smaller, thus making it necessary to apply grinding into finer sizes for satisfactory liberation. The liberation spectrum of the comminuted samples have shown that grinding in 0.210 mm for FI sample, and 0.045 mm for CI

sample, should result in a satisfactory liberation for subsequent concentration, assuming that particles with more than 90% quartz can be considered free.

Acknowledgements

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