TECHNOLOGICAL CHARACTERIZATION OF PHOSPHATIC TAILINGS BY MEANS OF MINERAL LIBERATION ANALIZER-MLA[1]

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Abstract

This paper shows the results of investigation of feed characteristics of residues from the Cajati Mine and how these characteristics would affect the selective flotation of dolomite from calcite. The study was conducted using both traditional analytical techniques and Secondary Electron Microscopy (SEM) combined with Mineral Liberation Analyzer (MLA) technique. The results showed that the sample was composed by calcite, dolomite and in minor proportion, apatite, quartz, diopside, plhogopite, forsterite, pyrrotite, pyrite and magnetite. Both calcite and dolomite had 96 % of free area surface, approximately, and the higher liberation of calcite and dolomite particles has suggested that a higher recovery can be obtained by flotation.

Key words: Mineral liberation analyzer; Characterization; Technology of residues; Phosphate ore.

CARACTERIZAÇÃO TECNOLÓGICA DE REJEITOS DE ROCHA FOSFÁTICA POR *MINERAL LIBERATION ANALAIZER*-MLA

Resumo

Este trabalho mostra os resultados do estudo das características mineralógicas do rejeito da mina Cajatí e como essas características poderiam afetar a separação seletiva, por flotação, da dolomita da calcita. Este estudo foi realizado empregando os métodos tradicionais para caracterização de minérios, bem como utilizando a microscopia eletrônica de varredura combinado com a técnica *Mineral liberation analizer* (MLA). Os resultados mostraram que a amostra está composta por calcita, dolomita e em menor proporção, apatita, quartzo, diopsidio, flogopita, forsterita, pirrotita, pirita e magnetita. As partículas de calcita e dolomita possuem 96% de área livre de superfície, aproximadamente. Os resultados sugerem que pode ser obtida uma alta recuperação dos minerais de interesse por flotação.

Palavras-chave: *Mineral liberation analizer*; Caracterização; Tecnologia de resíduos, Minério fosfático.

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1 INTRODUCTION

The Fosfertil Industry has a phosphate mine located at Cajati-SP District. The mineral benefit consists in crushing, milling, magnetic separation and flotation steps.(1) The tailing of this plant has a high grade of carbonatic minerals, mainly, calcite ($Ca(CO_3)_2$) and dolomite ($CaMg(CO_3)_2$).

The calcite and dolomite minerals are widely use in the construction, pharmaceutical and agriculture industry. Dolomite and calcite are sat-type minerals that show similar flotation behaviors due to the same characteristic. According to Amankonah and Somasundaram(2) the separation of minerals contained in phosphate rocks by flotation has been a major problem in the phosphate industry.

Mineral processing operations demand detailed characterization of the ore and plant feed. Textural parameters, such as mineral liberation size and mineral association, combine with modal mineralogy data to strongly influence mineral processing conditions and recovery. Liberation is an essential factor which strongly affects recovery and product grade. If there is inadequate liberation the separation efficiency will be limited.(3)

The innovation on the mineral characterization by mineral liberation analysis was the focus of this study. The mineral liberation analyzer (MLA) consists in a unique method of combining Back-scattered electrons (BSE) image analysis and X-ray mineral identification to provide automated quantitative mineral liberation characterization.(4,5) This technique explores, widely, the characteristics of samples such as mineral association, liberation, chemical and mineralogical composition.

The present work aim at characterized the sample of Cajatí tailing using both traditional analytical techniques and Secondary Electron Microscopy (SEM) combined with Mineral Liberation Analyzer (MLA) technique. The results of mineralogical, chemical and liberation analyses are able to elucidate the particle behavior in the future flotation tests.

2 MATERIALS AND METHODS

A sample of phosphate tailing, product of apatite flotation step, was studied. This representative sample was collected from Cajatí mine located at Sao Paulo State. The particle size distribution was made in wet sieving. After that, an amount of 4 g of each fraction was separated and embed in epoxy resin. The section was polished with water and diamond powder (125 to 1 µm) using the automatic polishing machine *Struers Tegra.* The polished face was covered afterwards with a surface of conductor carbon for scanning in SEM.

All prepared polished sections were analyzed at the equipment MEV FEI Quanta 400, initially suffering a detection of backscattered electrons in which the particles and their average atom number were obtained. Thereafter, a energy dispersive spectrometry (EDS) analysis provided the punctual mineral composition of each particle. The images generate were analyzed with the use of the software MLA *Suite* 2008.

Moreover, the representative sample was milled under 105 µm in agate planetary mill for determining the mineralogical and chemical composition of head sample. The X-Ray Diffraction (XRD), performed by the powder method, was made with 5g of sample in order to identify the mineral phases. They were conducted by the equipment *Bruker-D4 Endeavor* at conditions of radiation of Co Kα (40kV/40mA). The

qualitative interpretation of the spectrum were made by comparison with patterns at the database PDF02 (ICDD, 2006) using the software *Bruker DiffracPlus.*
The chemical determination of the sample was carried out by several analytical methods. The phosphorus and silicon concentrations were determined by calorimetry and gravimetry, respectively. The atomic absorption technique (AAS) through flame with C_2H_2, N_2O and air were used to obtain the concentration of calcium and magnesium. The previous technique, without the use of N_2O, was employed to obtain iron and potassium concentrations. Combustion was used for determination of the total carbon concentration.

3 RESULTS & DISCUSSION

3.1 Particle Size Analyze

The particle size distribution of the sample is shown in Table 1. The P_{80} of the sample is 325 µm. For flotation process, the sample is composed by coarse particles (> 210 µm) and most of the particles are at the intermediate size, which is a favorable fact because there are only few mass of extra fine material that can interfere in a future beneficiation.

Table 1. Paticle size distribution of Cajati tailing

Fraction (µm)	Overweight (g)	Overweight (%)	Cumulative overweight (%)	Cumulative passing (%)
+600	7.2	1.5	1.5	98.5
+600 -425	26.2	5.3	6.8	93.2
+425 -300	78.7	16.0	22.8	77.2
+300 -212	85.1	17.3	40.1	59.9
+212 -150	74.4	15.1	55.2	44.8
+212 -106	70.9	14.4	69.6	30.4
+106 -75	45.9	9.3	79.0	21.0
+75 -53	26.9	5.5	84.4	15.6
+53 -38	34.6	7.0	91.5	8.5
-38	41.9	8.5	100.0	0.0

3.2 Mineralogical Composition

The mineralogical composition obtained by the XRD technique is shown in Figure 1. The minerals species identify were: calcite ($Ca(CO_3)_2$), dolomite ($CaMg(CO_3)_2$), phlogopite ($KMg_3AlSi_3O_{10}F(OH)$) and quartz (SiO_2). The high intensity peaks of calcite and dolomite suggests a higher concentration of these minerals in the sample. The peaks of calcite were dislocated; it is mean that, the calcite found in the sample is rich in magnesium.

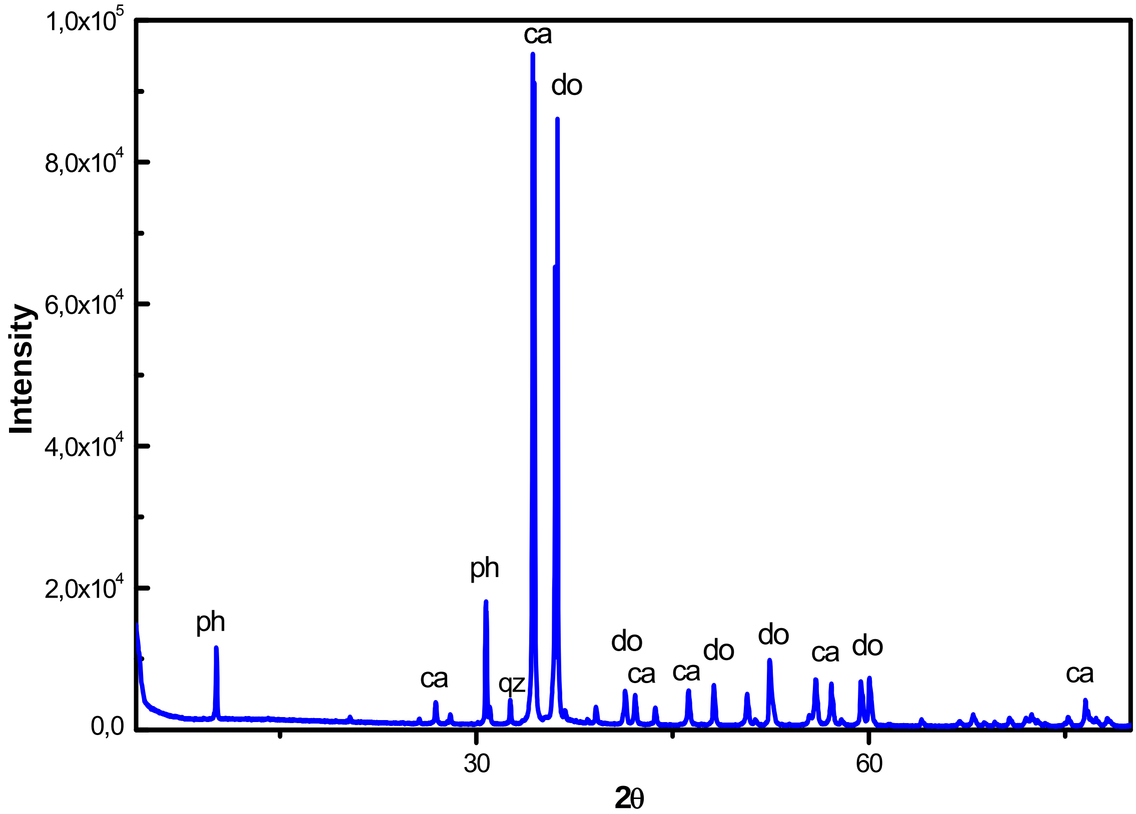

Figure 1. XRD spectrum of cajati tailing sample. Magnesian calcite (ca), dolomite (do), phlogopite (ph), quartz (qz).

Figure 2 shows the mineralogical composition generated by MLA from the combined of the fractions (-600µm +38µm). This technique permitted to indentify more phases minerals than the DRX technique. The MLA results showed that the sample is composed mainly of calcite and dolomite, and a relative low abundance of apatite ($Ca_5(PO_4)_3(OH,F,Cl)$), diopside ($CaMg(Si_2O_6)$), phlogopite, forsterite ($Mg_2(SiO_4)$), pyrrhotite ($Fe_{1-x}S$), magnetite (Fe_3O_4), pyrite (FeS_2), chlorite ($(Mg,Al,Fe)_{12}(Si,Al)_8O_{20}(OH)_{16}$), quartz and ilmenite ($FeTiO_3$). In traces were found fayalite ($Fe_2(SiO_4)$), barite ($Ba(SO_4)$), galena (PbS), phentlandite ($Fe_9Ni_9S_{16}$), plagioclase ($(Na,Ca)(Si,Al)_3O_8$), fluorite (CaF_2), rutile (TiO_2), chalcopyrite ($CuFeS_2$), arsenopyrite (FeAsS), scheelite ($Ca(WO_4)$), sphalerite (ZnS) e titanite ($CaTiSiO_5$).

The results obtained by MLA corroborate with those achieved by XRD technique and the literature.(6) The use of electronic microscopy together with MLA got a better limit of detection since it was observed a higher sensibility in the identification of species. Only by this method it was possible to detect low concentrations of materials with environmental implications such as arsenopyrite. In addition, it was possible indentify chlorite, mineral that was able to interfere in a flotation processes.

Apatite is a mineral which occurs in abundance in phosphate rocks, but since the sample is a tailing, the concentration expected of apatite should be low. However, 1% of apatite in a tailing is still a important concentration, which can denote imperfections at the beneficiation process.

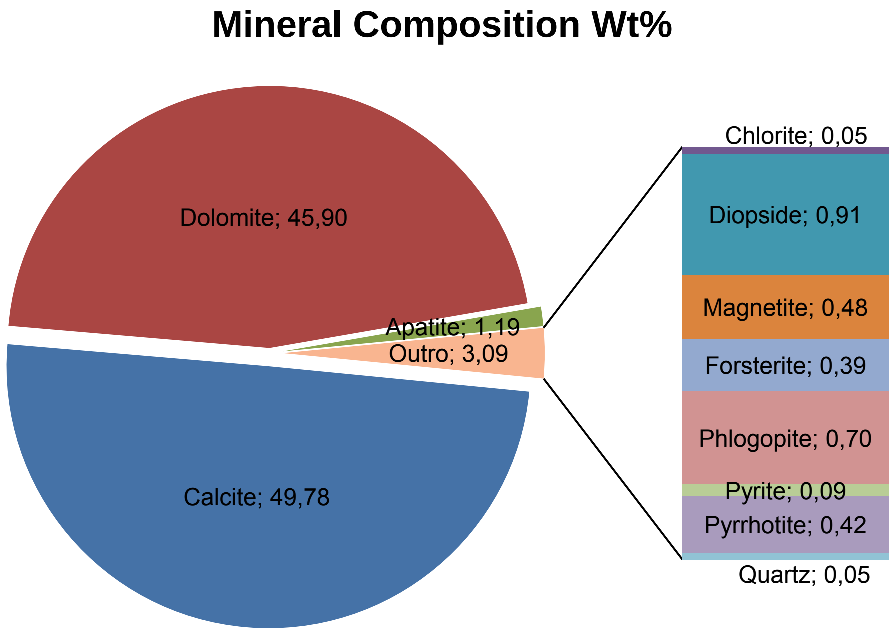

Figure 2. Combined mineralogical composition (fractions -600 +38µm) of Cajati sample by MLA analyze.

3.3 Chemical Composition

The chemical composition obtained by previous described analytical chemical methods is included in Figure 3. The chemical composition obtained by, both MLA and traditional technique were similar. However, extra care should be taken because MLA only analyze the average concentration of combine fractions (-600 +38µm).

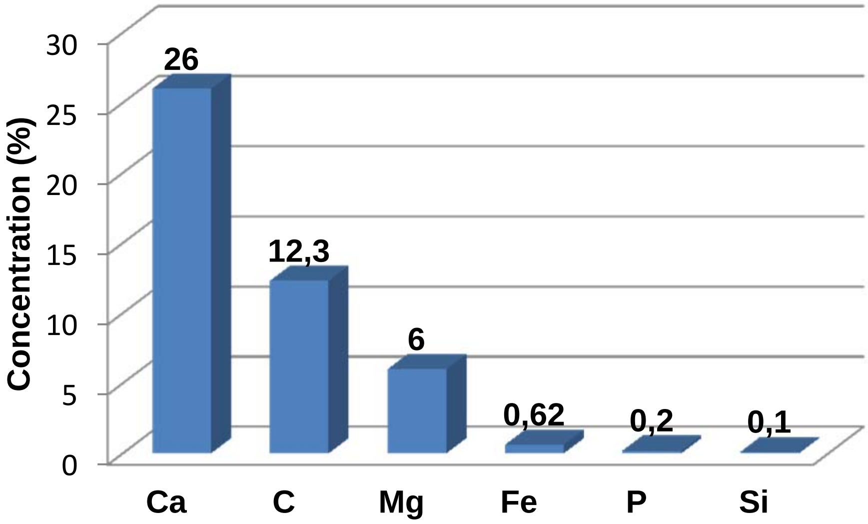

Figure 3. Chemical composition of sample obtained by analytical techniques

Figure 4 shows the chemical composition given by MLA. The analysis revealed a high content of calcium, magnesium and carbon. The amount of calcium is elevated due to this element compose both calcite and dolomite. The magnesium concentration is high because it is present in dolomite and magnesium calcite structure too. The presence of carbonate minerals confirmed the quantity of carbon in the sample. A low abundance of phosphorus is due to low quantity of apatite particles. Silicon comes from low concentrated silicates and iron concentration is attributed to magnetite, pyrite and pyrrhotite.

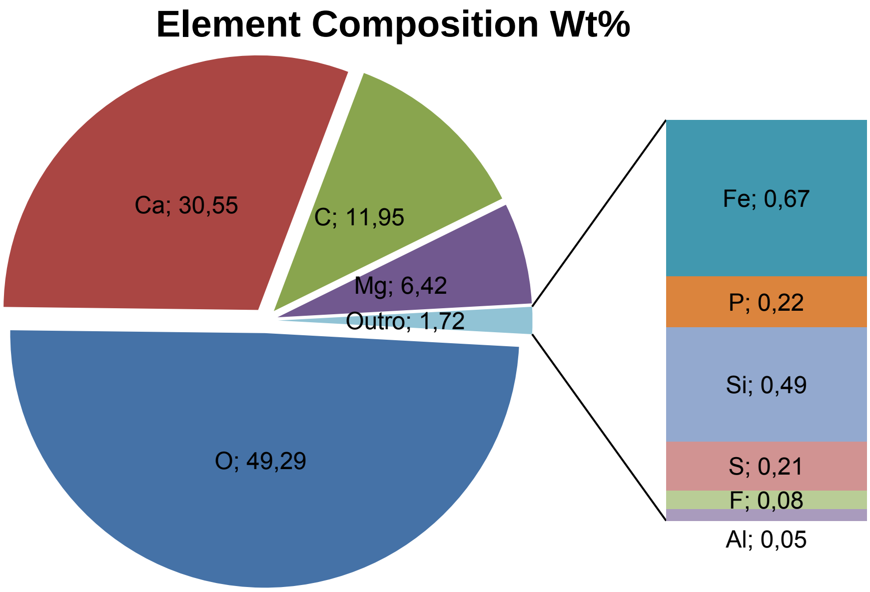

Figure 4. Combined Chemical composition (fractions -600 +38µm) of Cajati sample by MLA analyze.

3.4 Mineral liberation analyser

Figure 5 shows one of the images taken by BSE/SEM. In Figure 6 were identified all different mineral phases of particles and define their boundaries. Each particle was given a color according to their chemical composition. X-ray mapping imposes a grid over an entire particle image, or specific grains thereof, and collects X-ray data at each grid point to determine the mineral identity(4) (Figure 7).

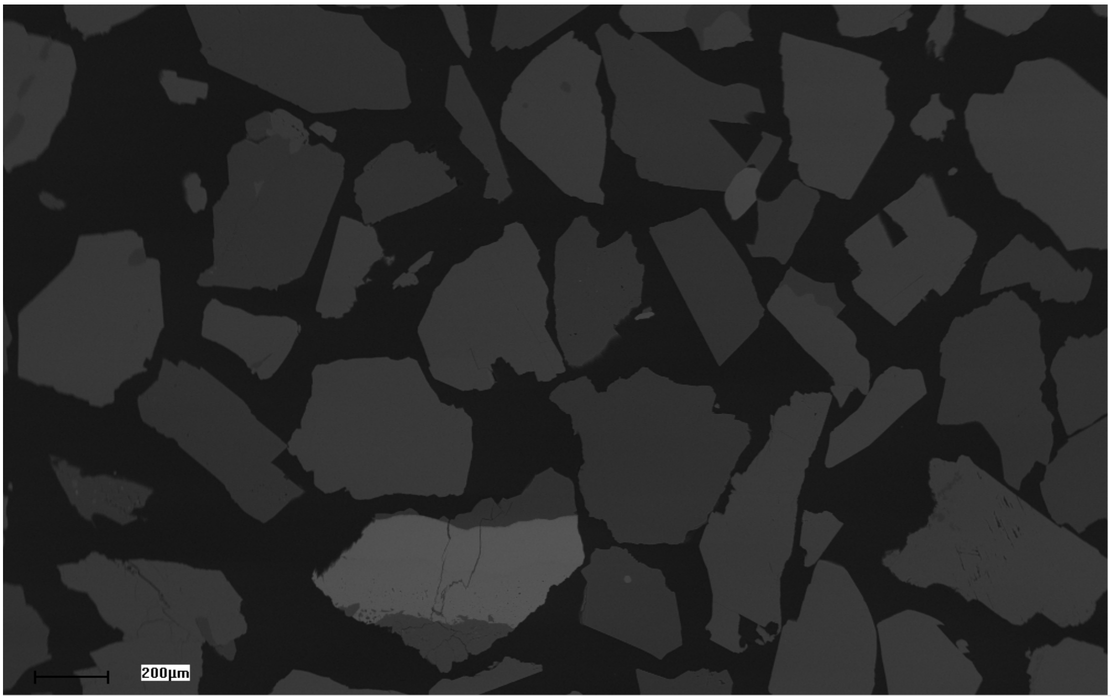

Figure 5. Original BSE image. Fraction -425 +300µm.

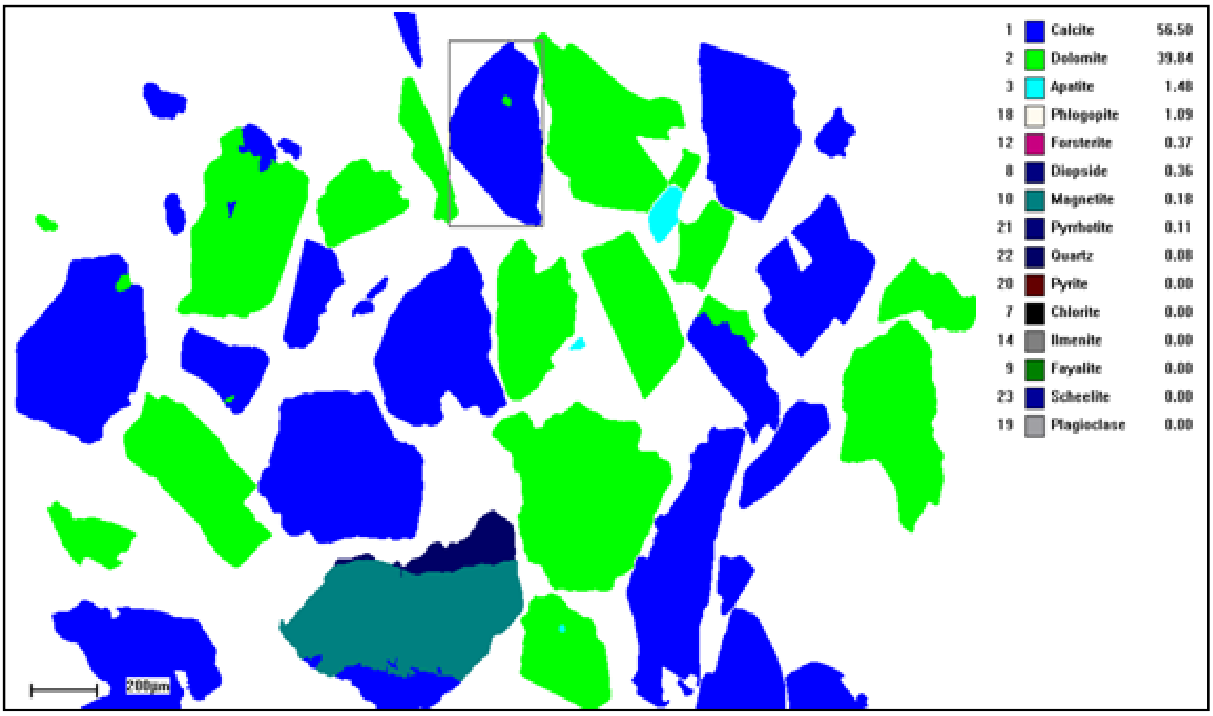

Figure 6. Segment particle image. Fraction -420 +300 µm.

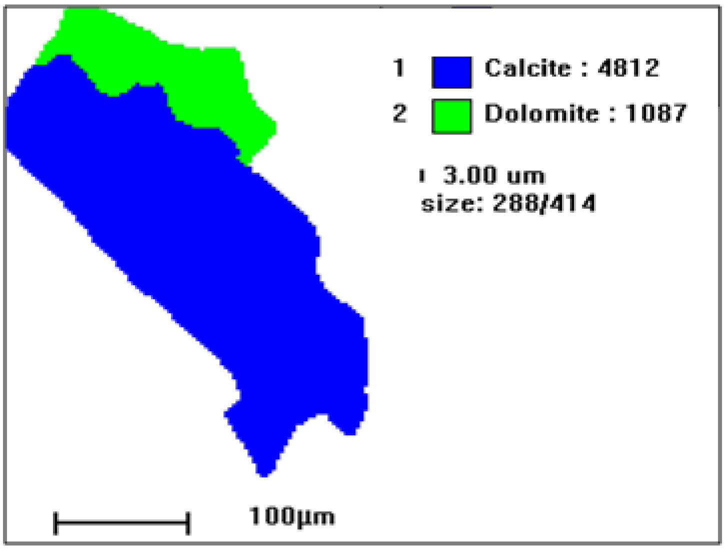

Figure 7. Illustration of X-ray mapping of calcite associated with dolomite. Fraction -420 +300 µm.

Figure 8 shows the cumulative mass recovery vs. liberation class for calcite, dolomite and apatite. The results showed that a high liberation of calcite and dolomite particles. That means, a good mass recovery. However, the apatite showed a minor liberation, to get a purity of 100%, the recovery of mass reach 75%. These figures confirm too, previous chemical and mineralogical results that some of the phosphate loss in the tailings product is due to liberation limitations.

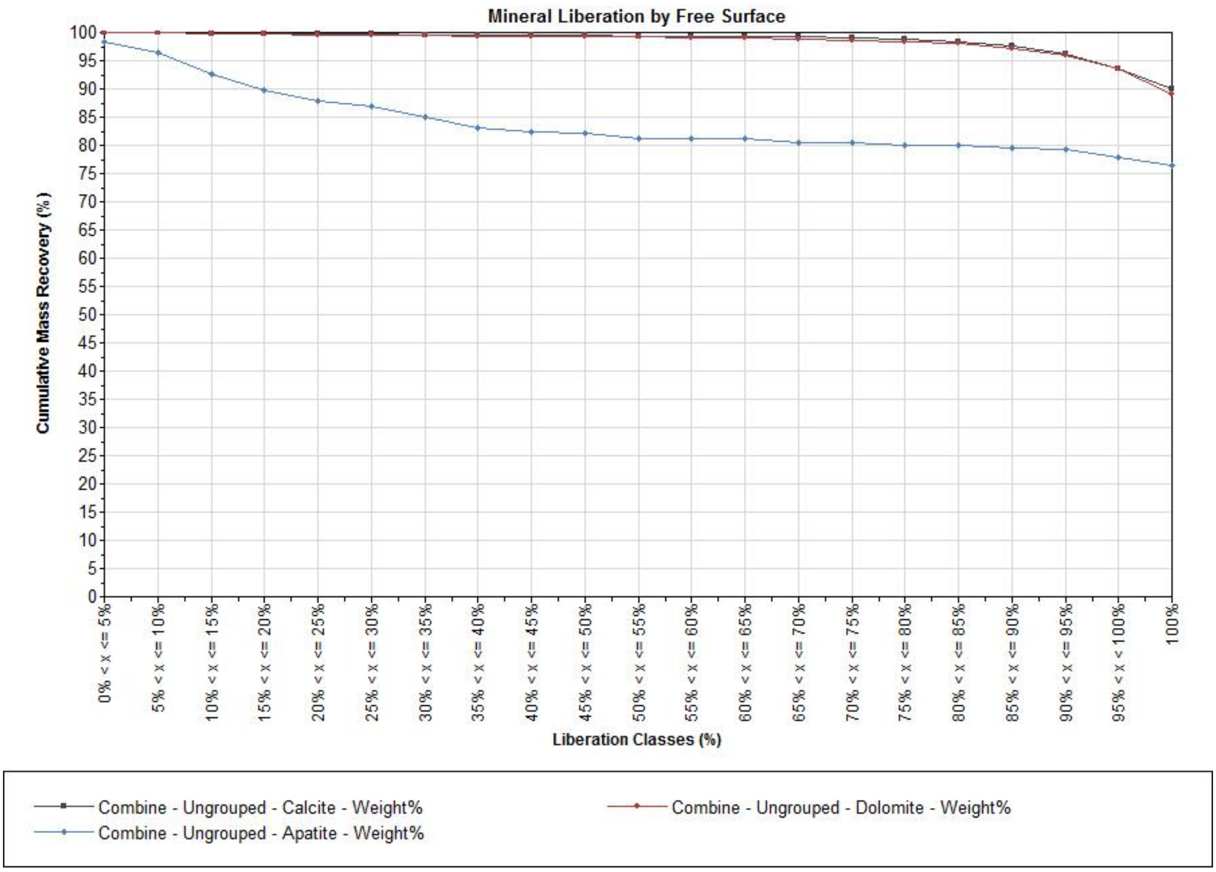

Figure 8. Combine fraction (-600 +38µm) mineral liberation by free surface for calcite, dolomite and apatite.

Table 2 shows the mineralogical association of calcite, dolomite and apatite obtained by MLA analyze.

Table 2. Mineral association of calcite, dolomite and apatite. Data generated by MLA. (combined fractions (-600 +38µm))

Mineral	**Calcite (%)**	**Dolomite (%)**	**Apatite (%)**
Calcite	0	3.86	9.08
Dolomite	3.54	0	20.22
Apatite	0.31	0.68	0
Chlorite	0.01	0.01	0
Diopside	0.02	0	0.04
Magnetite	0.05	0.06	0.51
Forsterite	0.03	0.02	1.32
Phlogopite	0.04	0.11	3.07
Pyrite	0.03	0.01	0.02
Pyrrhotite	0.01	0.02	0.04
Quartz	0	0	0.08
Scheelite	0	0.01	0
Free Surface	95.95	95.22	65.62

Calcite free surface in the tailing sample was high, about 96% of the particles are liberated. The remaining, 4% are, mainly, associated with the dolomite (3.5%) and the apatite (0.3%).

In the same case, most of the dolomite particles were unlocked (95%). The remaining 4.8% were interlocking with the calcite (3.9%) and the apatite (0.7%).

Apatite was mainly associated with dolomite (20%), calcite (9%), phlogopite (3%), forsterite (1.3%) and magnetite (0.5%). Most of the apatite particles are liberated, but his free surface is 65%, lower than the calcite and dolomite.

4 CONCLUSION

The Cajati tailing sample is composed by magnesian calcite, dolomite, and in minor proportion, apatite, diopside, phlogopite, forsterite, magnetite, pyrite, chlorite, quartz, ilmenite and scheelite. In concentration lower than 0,01% w/w were detected fayalite, barite, feldspar, galene, phentlandite, plagioclase, fluorite, rutile, chalcopyrite, arsenopyrite, esphalerite and titanite.

Although the arsenopyrite is the main mineral that has environmental implications, it is in lower concentration (> 0.01%). Furthermore, it was possible to observe the presence of materials which may adversely affect the flotation process, such as chlorite and micas.

The liberation results showed that a high free surface area from calcite and dolomite (95%), suggesting high recoveries in the selective separation by froth flotation. The calcite and dolomite particles, no liberated, are associated between them. However, One of the reasons for the phosphate loss in the tail is the lack of liberation of coarse interlocked particles.

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