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MINERALOGICAL AND TECHNOLOGICAL CHARACTERIZATION OF PHOSPHATE TAILING AS A PRELIMINARY STEP FOR REPROCESSING STUDIES

CARACTERIZAÇÃO MINERALÓGICA E TECNOLÓGICA DE UM REJEITO FOSFÁTICO COMO ETAPA PRELIMINAR PARA ESTUDOS DE REPROCESSAMENTO

CARACTERIZACIÓN MINERALÓGICA Y TECNOLÓGICA DE UN RECHAZO FOSFÁTICO COMOETAPA PRELIMINAR PARA ESTUDIOS DE REPROCESAMIENTO

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Abstract

This study investigates the mineralogical and technological characterization of a phosphate mining tailing, with the objective of providing technical support for the assessment of its potential in future reprocessing studies. The increasing generation of mining tailings and the need for higher phosphorus recovery make the search for alternative approaches to reuse these materials essential. The analyzed tailing, originating from desliming stages of the beneficiation process, exhibits predominantly ultrafine particle size distribution, with approximately 50% of the particles smaller than 0.012 mm. Chemical analysis indicated an average P_2O_5 grade of 7%, with the highest concentrations occurring in the particle-size range between 0.011 and 0.044 mm. The tailing mineralogy is predominantly composed of apatite, goethite, micas, and clay minerals, which hinder conventional concentration processes. Pure apatite collected for complementary studies exhibited electrokinetic behavior compatible with the use of anionic collectors, such as fatty acids, at pH values above 8.5, providing relevant information on the surface properties of the mineral. The results obtained provide technical indications that contribute to the continuation of future studies aimed at evaluating recovery routes adapted to the mineralogical and granulometric characteristics of the tailing.

Keywords: Mining Tailings; Mineral Characterization; Phosphate Ore.

Resumo

O estudo investiga a caracterização mineralógica e tecnológica de um rejeito fosfático de mineração, com o objetivo de fornecer subsídios técnicos para a avaliação de seu potencial em estudos futuros de reprocessamento. A crescente produção de rejeitos na mineração e a necessidade de maior recuperação de fósforo tornam essencial a busca por alternativas de reaproveitamento desses materiais. O rejeito analisado, proveniente das etapas de deslamagem do beneficiamento, apresenta granulometria predominantemente ultrafina, com cerca de 50% das partículas abaixo de 0,012 mm. A composição química apontou teor médio de 7% de P_2O_5, com as maiores concentrações ocorrendo nas faixas de tamanho entre 0,011 e 0,044 mm. A mineralogia do rejeito é composta predominantemente por apatita, goethita, micas e argilominerais, que dificultam processos convencionais de concentração. A apatita pura coletada para estudos complementares apresentou comportamento eletrocinético compatível com o uso de coletores aniônicos, como ácidos graxos, em pH acima de 8,5, fornecendo informações relevantes sobre as propriedades superficiais do mineral. Os resultados apresentados fornecem indicativos técnicos que contribuem para o seguimento de estudos futuros voltados à avaliação de rotas de recuperação do rejeito, adaptadas às suas particularidades mineralógicas e granulométricas.

Palavras-chave: Rejeitos de Mineração; Caracterização Mineral; Minério Fosfático.

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Resumen

Este estudio investiga la caracterización mineralógica y tecnológica de un residuo fosfático de minería, con el objetivo de proporcionar fundamentos técnicos para la evaluación de su potencial en estudios futuros de reprocesamiento. El aumento en la generación de residuos mineros y la necesidad de una mayor recuperación de fósforo hacen esencial la búsqueda de alternativas para el reaprovechamiento de estos materiales. El residuo analizado, procedente de las etapas de deslamado del proceso de beneficio, presenta una granulometría predominantemente ultrafina, con aproximadamente el 50% de las partículas por debajo de 0,012 mm. El análisis químico indicó un contenido medio de 7% de P_2O_5, con las mayores concentraciones en el intervalo de tamaño entre 0,011 y 0,044 mm. La mineralogía del residuo está compuesta predominantemente por apatita, goethita, micas y minerales arcillosos, que dificultan los procesos convencionales de concentración. La apatita pura recolectada para estudios complementarios mostró un comportamiento electrocinético compatible con el uso de colectores aniónicos, como los ácidos grasos, en valores de pH superiores a 8,5, proporcionando información relevante sobre las propiedades superficiales del mineral. Los resultados obtenidos aportan indicativos técnicos que contribuyen a la continuidad de estudios futuros orientados a la evaluación de rutas de recuperación adaptadas a las particularidades mineralógicas y granulométricas del residuo.

Palabras clave: Residuos Mineros; Caracterización Mineral; Mineral de Fosfato.

1. Introduction

The generation of mining tailings constitutes one of the main challenges currently faced by the mining sector. These materials, considered a significant environmental liability, are commonly disposed of in tailings dams, which may cause substantial social and environmental impacts. Among the associated risks are the contamination of soils and bodies of water by heavy metals, as reported in the Gironde Estuary in France, as well as large-scale accidents such as the failures of the Fundão and Córrego do Feijão dams in Minas Gerais, Brazil, which occurred in 2015 and 2019, respectively, representing the largest mining incidents in the country's history. (Coimbra, Alcântara and Souza Filho, 2020; Parente et al., 2020; Paulelli et al., 2022; Schäffer, Coynel and Blanc, 2022; Vergilio et al., 2020).

In addition, the progressive depletion of high-grade ore deposits has led the

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mining industry to exploit increasingly complex ores with lower concentrations of the target elements. Combined with the growing demand for mineral resources, this condition intensifies the generation of tailings, making essential the development of alternatives for the recovery and utilization of currently discarded materials (Maus et al., 2020).

In this context, phosphate ore deposits stand out due to their mineralogical diversity and low phosphorus recovery rates, phosphorus being the main element of interest in this type of mining activity and predominantly used in fertilizer production. Consequently, tailings accumulated from phosphate ore beneficiation contain significant amounts of phosphorus, expressed as oxides (P_2O_5), as well as other elements of relevant economic value in some mines, such as iron, titanium, and rare earth elements, indicating their potential for reuse (Banihashemi et al., 2019; Guan et al., 2022; Liang et al., 2018).

A representative example of phosphate mining in Brazil is found in the western region of Minas Gerais, which hosts several mines and beneficiation plants located in municipalities such as Tapira, Patrocínio, and Araxá. Phosphate mining activities in this region have been carried out since the 1970s and include the largest phosphate mining operation in Brazil. The storage of tailings from these operations occurs in dams, one of which is the second largest tailings dam in Latin America, with approximately 170 million m^3 of stored tailings (MOSAIC, 2022).

Given this scenario, considering the volume of tailings generated from phosphate ore beneficiation in this region, the continuous demand for phosphate fertilizers, and the economic context that drives the maximization of phosphorus utilization in mining activities, this article aims to characterize a phosphate tailings sample originating from the mineral beneficiation stages of a production unit in the region. Based on this characterization, the study seeks to identify the main limiting factors and technical indicators associated with phosphorus recovery from the tailings, providing a technical foundation for future studies aimed at the potential reprocessing of this material.

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2. Literature Rewiew

The phosphate ore from the region under study is associated with an alkaline–carbonatitic complex covering approximately 35 km² and is processed in a beneficiation plant with a feed capacity of up to 2000 t/h (Barros, 1997; Ferreira et al., 2023). Its mineralogy is characterized by the predominance of apatite as the main phosphorus-bearing mineral, typically associated with silicates, carbonates, and magnetite, which act as contaminants and significantly affect processing efficiency and P_2O_5 concentration.

The primary technique used for phosphate ore recovery is flotation, a process in which the ore, after comminution and particle size adjustment, is subjected to interaction with chemical reagents that promote the hydrophobization of the mineral of interest, called collectors. Air is then injected into the mineral pulp, enabling the hydrophobic particles to attach to air bubbles and be transported to the pulp surface, forming a froth enriched in apatite concentrate (Ferreira et al., 2023).

The non-concentrated product consists of flotation tailings, generally composed of a pulp rich in contaminant minerals, fine particle size, and structurally altered apatite. In addition, mineral processing includes stages such as magnetic separation and desliming, which generate additional tailings streams. As a result, approximately 30–40% of the total phosphorus oxide (P_2O_5) extracted from phosphate mines is discarded in tailings dams (Teague and Lollback, 2012; Seifelnassr and Ahmed, 1998).

Considering the significant amount of valuable elements lost in conventional mineral beneficiation processes, studies addressing the treatment and utilization of mining tailings have become increasingly common, particularly with respect to the recovery of P_2O_5 from phosphate residues. Table 1 summarizes selected studies in which different processing techniques were applied to produce new concentrates from phosphate tailings.

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Table 1 – Phosphate tailings reprocessing studies

Method	Reagent	Particle size (mm)	Initial P_2O_5 (%)	Concentrate P_2O_5 (%)	Recovery (%)	Reference
Leaching	Hydrochloric acid	−0.074	10.0	34.18	96.3	Yu and Du (2023)
Conventional flotation	Sodium oleate	−0.125 +0.045	21.6	28.4	>73	Alsafasfeh and Alagha (2017)
Conventional flotation	Synthetic collector and rice oil	−0.290	9.5	29.4	46.2	Oliveira et al. (2011)
Conventional flotation	Alkyl hydroxamic acid	−0.100 +0.038	19.5	30.9	81.6	Nettour et al. (2019)
Jameson cell flotation	Tall oil	100% −0.150 to 80% −0.020	≈15.6	>32.0	>80	Teague and Lollback (2012)
Conventional flotation	Amine	Tailings, slimes, and concentrate blend	–	32.6	94.2	Tamm et al. (2021)

Source: Elaborated by the author

Given the high mineralogical, granulometric, and compositional variability of phosphate deposits worldwide, it becomes impractical to establish direct and universal comparisons among different studies on phosphate tailings reprocessing. Differences related to the type of apatite, associated gangue minerals, degree of mineral liberation, and tailings generation conditions impose clear limitations on the comparability of the systems analyzed in the literature. Nevertheless, the studies reviewed provide relevant and complementary information, particularly regarding trends in apatite behavior, technological routes employed, and recurring challenges associated with the treatment of fine and

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ultrafine materials, thus playing a fundamental role as a conceptual and methodological basis to support the decisions and interpretations adopted in the present work.

Oliveira et al. (2011), for instance, carried out the characterization of a phosphate tailing originating from the same region as the material investigated in this study, using particle size analysis and X-ray diffraction as a preliminary step prior to flotation tests. The authors employed an anionic collector based on fatty acids derived from rice oil, blended with a synthetic collector, to evaluate its performance in apatite concentration.

Although the tailing analyzed by Oliveira et al. (2011) exhibited a significantly coarser particle size distribution and a higher initial P_2O_5 grade (9.5%) compared to the material evaluated in the present study, the results evidenced the efficiency of the proposed flotation route, achieving P_2O_5 grades of up to 29% and metallurgical recoveries on the order of 49%. Selectivity indices were directly related to increased dosages of the synthetic collector, known for its higher selectivity toward ores from the region. These results reinforce the recovery potential of phosphate tailings in the region, even under granulometric conditions different from those addressed in this work.

Similarly, Alsafasfeh and Alagha (2017) conducted a study focused on the mineralogical and technological characterization of industrial phosphate tailings, aiming at apatite recovery through direct flotation. The authors performed a comprehensive characterization of the tailing using techniques such as X-ray diffraction, X-ray fluorescence, scanning electron microscopy, and mineral liberation analysis to better understand the mineralogy, particle morphology, and degree of liberation of the target mineral prior to flotation tests. Flotation was carried out using sodium oleate as an anionic collector, combined with sodium silicate as a dispersant. Although the analyzed tailing presented a significantly higher initial P_2O_5 grade than that evaluated in the present work (21.6%) and a less extreme ultrafine particle size distribution, the results demonstrated the feasibility of the proposed route, yielding concentrates containing approximately 28% P_2O_5 and recoveries above 70%.

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Finally, Yu and Du (2023) evaluated phosphorus recovery from phosphate tailings through leaching. Using samples with particle sizes smaller than 0.074 mm, similar to those of the tailing investigated in this study, the authors obtained a leach liquor with metallurgical recoveries exceeding 90% and a P_2O_5 grade of 34% through hydrochloric acid leaching, already demonstrating commercial feasibility. Thus, despite the higher operating costs and environmental and safety concerns associated with acid use, leaching of ultrafine phosphate tailings appears as a technically promising alternative when conventional beneficiation methods are insufficient.

The results reported in the literature highlight the potential for apatite recovery from phosphate tailings and reinforce the importance of detailed characterization as a preliminary step in selecting beneficiation routes, even when mineralogical and granulometric conditions are not directly comparable to those of the tailing studied in this article.

In this context, the development of the present work is fully justified, as each mineral system exhibits specific behaviors associated with its physicochemical characteristics, for which optimal operating ranges exist at each stage of concentration. Therefore, detailed ore characterization constitutes an essential element not only for understanding the mechanisms involved but also for optimizing process flowsheets and defining the most promising beneficiation routes (Ahmed and Jameson, 1989; Nazari et al., 2019; Reis et al., 2019).

3. Metodology

The experiments described in this study were carried out at the Characterization and Flotation Laboratories of the Federal University of Ouro Preto (UFOP) and were structured into two main stages. The first stage comprised the technological and mineralogical characterization of the tailings generated from the beneficiation of phosphate ore. The second stage focused on the development of fundamental studies using pure apatite samples from the same region as the tailings, with the aim of deepening the understanding of their physicochemical

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properties and behavior in mineral beneficiation processes.

3.1 Phosphate tailing characterization

A sample of approximately 15 kg of tailings originating from the desliming stages of the phosphate ore beneficiation plant under study was used. The sample, collected in slurry form, was dried in an oven at 80 °C and subsequently homogenized and quartered using a Jones riffle splitter. The resulting aliquots were then directed to the characterization tests.

Initially, the particle size distribution of the tailings sample was determined by dry sieving, using screens with openings of 0.106, 0.075, 0.053, 0.044, and 0.038 mm. The finest fraction, passing through the last sieve, had its particle size distribution determined using a CILAS 1046 laser granulometer.

In addition, the fine fraction was subjected to classification tests using a Cyclosizer, generating samples corresponding to specific size ranges. This procedure enabled detailed chemical analyses of each size fraction, which is a relevant aspect for the present study. The size ranges adopted for classification were 0.033, 0.023, 0.015, and 0.011 mm.

Consequently, both the bulk tailings sample and the samples obtained after Cyclosizer classification had their chemical composition determined by X-ray fluorescence spectroscopy (XRF), using an Axios Fast spectrometer from Malvern Panalytical. This procedure allowed for the granulochemical characterization of the sample, identifying particle size ranges with higher P_2O_5 contents and, therefore, greater potential for beneficiation.

Finally, the mineralogical composition of the analyzed tailings was determined by X-ray diffraction (XRD) using the total powder method. The analyses were performed using a diffractometer located at the facilities of the company supplying the tailings. The diffractometer operating conditions included an analysis time of 14 minutes, a scanning range from 5° to 90°, an operating voltage of 45 kV, and a current of 40 mA. The diffraction data were processed using Datacollector and HighScore Plus software.

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3.2 Apatite pure sample characterization

In order to better understand the characteristics of the main mineral of interest present in the phosphate tailings under investigation, samples of pure apatite were collected. This approach allows for the evaluation of the conditions under which the mineral occurs in the mine and, based on this understanding, the development of potential routes for its recovery from the tailings.

The true density of the pure apatite was determined by helium gas pycnometry using a Quantachrome Instruments ULTRAPYC 1200e pycnometer. The test consisted of placing a finely pulverized sample of the material into the sample chamber, from which air was initially evacuated, followed by filling the chamber with helium gas. This procedure allowed the determination of the total volume of gas displaced by the sample. Using the known gas density and the measured volume, the true density of the material was calculated. All measurements were performed in triplicate, and the average value was adopted as the representative result.

The specific surface area of the mineral used in the experimental tests was determined by gas adsorption techniques. For this purpose, the multipoint Brunauer–Emmett–Teller (BET) method was applied, which is based on the physical adsorption of nitrogen gas onto the particle surface, enabling accurate determination of the specific surface area. The analyses were carried out in triplicate using a Quantachrome NOVA 1200e porosimeter, available at the Federal University of Ouro Preto.

Finally, zeta potential measurements of the apatite were performed in order to evaluate its electrokinetic behavior at different pH values. The results provide important information regarding both suspension stability and the electrical properties of the mineral surface, which are essential for selecting reagents to be used in future flotation tests should the technical potential of the method be observed. The measurements were conducted using a Zeta Meter 4.0, also located at the Federal University of Ouro Preto.

Prior to the analyses, the samples were classified to obtain a particle size

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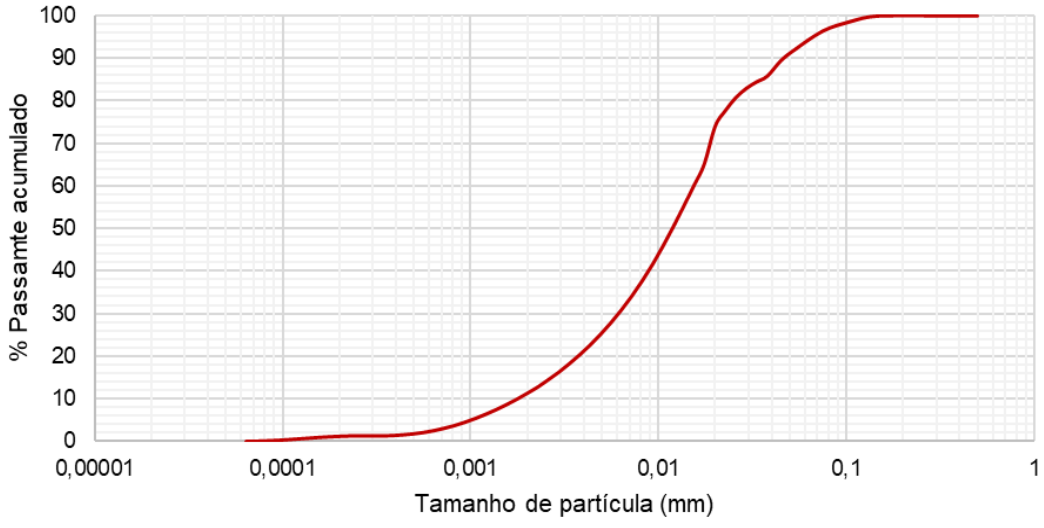
fraction smaller than 0.010 mm, suitable for electrokinetic measurements. The selected fraction was subsequently dispersed in an aqueous solution containing an indifferent electrolyte (KCl at a concentration of 10^{-3} M) to ensure constant ionic strength throughout the tests. Ten measurements were performed for each pH value, with the average of the results adopted as the final value, accepting a maximum deviation of 5%.

4. Results

4.1 Phosphate tailing characterization

The particle size analysis of the phosphate tailings, shown in Figure 1, indicates a marked predominance of fine and ultrafine particles, with approximately 50% of the mass below 0.012 mm and about 80% finer than 0.025 mm. As this material originates from successive classification stages following grinding, the tailings can be characterized as a stream with low amenability to recovery by conventional mineral processing routes.

Figure 1 – Tailings sample size distribution



Source: Elaborated by the author

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The chemical composition of the phosphate tailings under study is presented in Table 2. The average P_2O_5 content of the sample was approximately 7%, with iron, magnesium, calcium, and silicon identified as the main contaminant elements. It is observed that the highest P_2O_5 contents are concentrated in the particle size range between 0.011 and 0.044 mm, indicating a higher proportion of apatite within this size fraction.

Table 2 – Grade distribution by particle size

Size	**Grades**							
Interval (mm)	**P_2O_5**	**Fe_2O_3**	**MgO**	**CaO**	**Al_2O_3**	**TiO_2**	**SiO_2**	**Mass (%)**
+0.106	3.9	13.6	13.9	8.3	8.1	3.6	35.7	1.3
-0.106 +0.075	6.2	13.8	11.5	12.6	6.3	4.5	33.2	2.3
-0.075 +0.053	5.7	14.7	11.8	11.9	6.6	4.5	34.0	4.4
-0.053 +0.044	6.8	23.8	5.1	12.8	4.9	8.8	24.6	2.5
-0.044 +0.038	7.8	16.5	10.9	15.3	6.2	5.9	34.2	3.7
-0.038 +0.033	8.0	20.9	4.6	15.8	4.3	9.8	26.0	1.3
-0.033 +0.023	8.6	19.2	4.4	17.4	3.2	10.4	29.5	6.7
-0.023 +0.015	8.4	18.1	5.8	17.1	3.2	10.4	31.2	17.5
-0.015 +0.011	8.1	18.5	6.0	16.1	3.5	10.1	30.6	14.4
-0.011	6.0	23.6	5.7	10.0	5.9	6.9	27.9	45.9
Total	**7.0**	**20.6**	**6.3**	**13.1**	**4.9**	**8.0**	**29.6**	**100.0**

Source: Elaborated by the author

However, the high presence of ultrafine particles (fraction < 0.011 mm) makes it necessary to evaluate the reprocessing of this size fraction, given its significant mass contribution. Figure 2, which presents the metallurgical distribution of the chemical elements in the sample, highlights this condition by showing that approximately 40% of the total P_2O_5 is concentrated in the finest particle size fraction.

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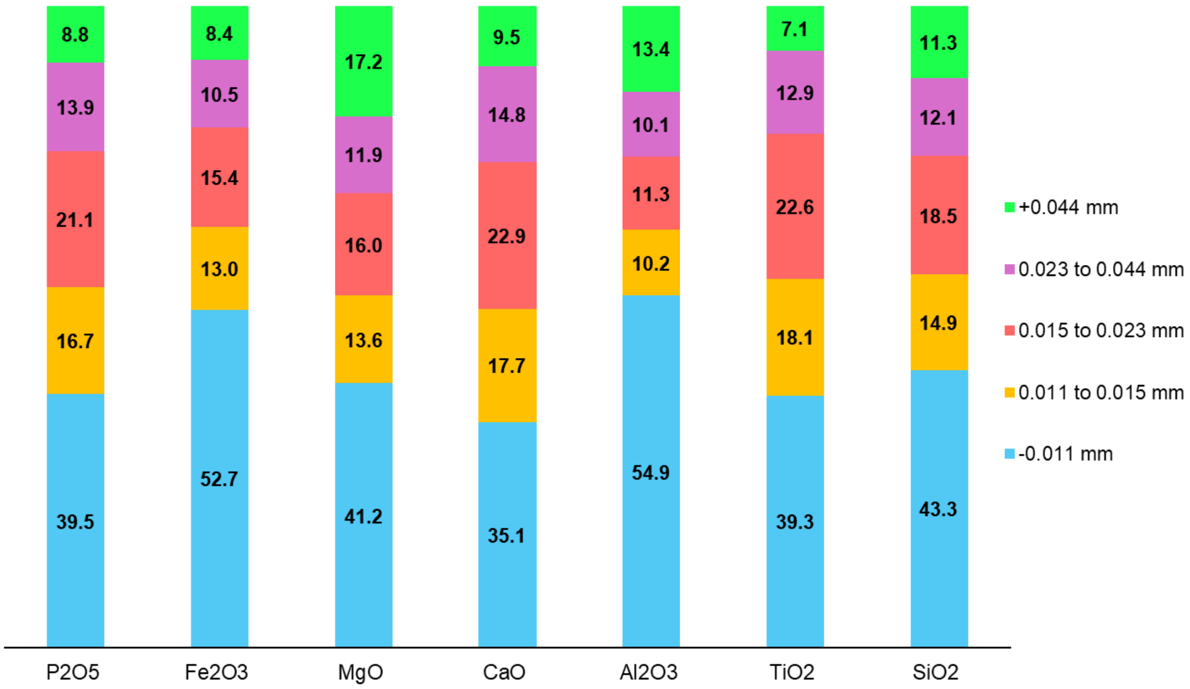
Figure 2 – Metallurgical distribution by particle size



Source: Elaborated by the author

Considering the metallurgical distribution presented, the assessment of the reprocessing potential of the phosphate tailings must necessarily consider the finest fraction of the sample. In this context, if desliming or classification stages were to be applied, most of the elements of interest would be discarded, significantly reducing the overall recovery of a new processing route.

Nevertheless, this finest particle size fraction tends to show a higher concentration of clay minerals, as evidenced by the increased proportion of Al_2O_3 relative to the other chemical constituents. The presence of clay minerals in the slurry increases system viscosity and adversely affects a potential flotation stage for P_2O_5 recovery, as it leads to higher reagent consumption and promotes the coating of apatite particles, thereby hindering their attachment to air bubbles. Furthermore, as flotation is a predominantly physical process, the transport of fine particles does not differentiate between hydrophilic and hydrophobic particles, resulting in increased gangue entrainment into the concentrate and a reduced probability of particle-bubble collision and attachment mechanisms that are

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fundamental to the selective collection achieved by flotation (Cheng, Zhang, and Lao, 2023; Subrahmanyam and Forssberg, 1988).

Regarding other contaminants, a higher Fe_2O_3 content is also observed in the finer size fractions. This behavior can be attributed to the magnetic separation stages in the mineral processing circuit from which the sample was collected, whereby coarser ferromagnetic mineral particles are removed prior to flotation, while finer particles are not efficiently captured due to their physical characteristics, being more strongly influenced by hydrodynamic drag forces than by the magnetic field of the separators.

These observations are corroborated by the X-ray diffraction analysis, the results of which are presented in Table 3. As previously discussed, the data highlight apatite as the predominant phosphorus-bearing mineral, associated with silicate minerals such as micas, quartz, and clay minerals, thereby justifying the elevated silicon and magnesium contents observed in the sample.

Table 3 – Tailing main mineralogical composition

Mineral	Group	Formula
Vermiculite	Silicate (mica)	$(MgFe,Al)_3(Al,Si)_4O_{10}(OH)_2 \cdot 4H_2O$
Goethite	Iron oxide	α-FeO(OH)
Diopside	Silicate	$Ca(Mg,Fe)Si_2O_6$
Augite	Silicate (pyroxene)	$(Ca,Na)(Mg,Fe,Al,Ti)(Si,Al)_2O_6$
Quartz	Silicate	SiO_2
Apatite	Phosphate	$Ca_5(PO_4)_3(F, Cl, OH)$
Anatase	Titanium oxide	TiO_2
Calcite	Carbonate	$CaCO_3$

Source: Elaborated by the author

The quantitative determination of the mineral phases was not feasible due to the significant presence of clay minerals, which introduce uncertainties in X-ray diffraction (XRD) quantification because of their low crystallinity, preferred

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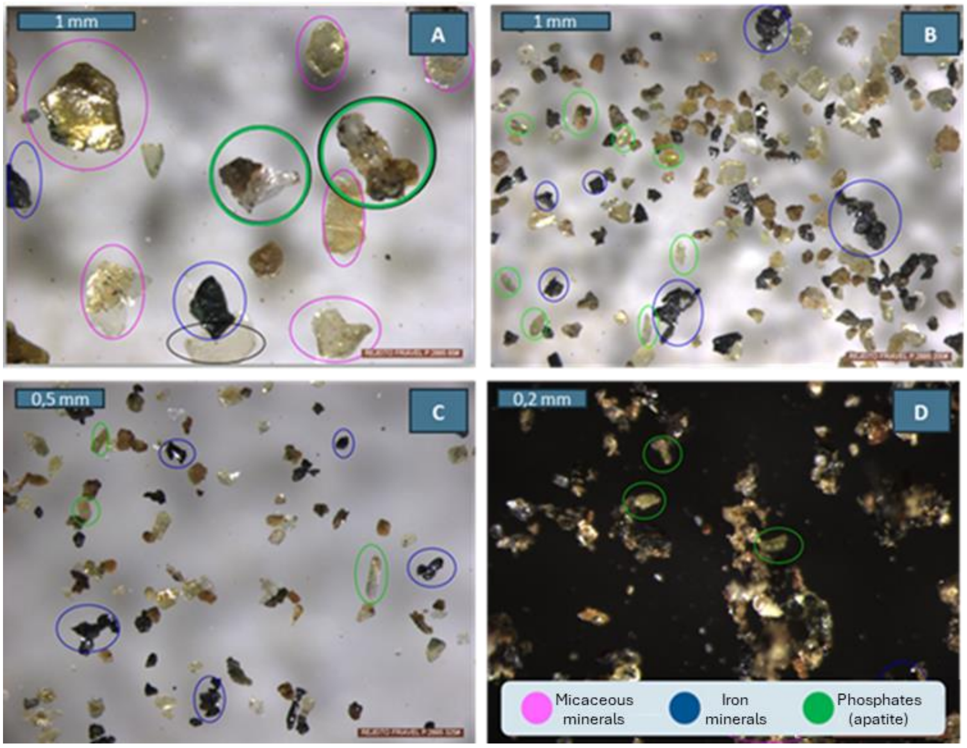
orientation, and structural variability. These minerals increase the so-called diffuse background, an effect that leads to peak overlap and reduced contrast between diffraction peaks, thereby hindering the accurate identification and quantification of the crystalline phases.

Goethite was identified as the iron-bearing mineral present in the highest proportion in the tailings. This is justified by the fact that goethite does not exhibit attraction to magnetic fields and, therefore, is not removed during magnetic separation stages, unlike minerals such as magnetite and hematite.

This behavior is corroborated by the microscopic analyses shown in Figure 3, which were performed on a representative sample of the coarser fraction of the sampled tailings. It should be noted that, due to inherent limitations of conventional microscopic analysis, the ultrafine fractions, which constitute the largest proportion of the studied material, were not directly evaluated using this technique, as they are predominantly composed of clay minerals that are difficult to individualize and interpret optically.

Figure 3 - Tailing sample microscopic analysis



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Thus, the images should be interpreted as qualitative evidence, intended to illustrate general mineralogical and textural trends of the tailings, and not as a replacement for the direct characterization of the ultrafine fraction, which was quantitatively assessed through the granulochemical analyses and X-ray diffraction presented in the previous sections.

For the analyzed material, a significant presence of iron-bearing mineral particles is observed across all investigated particle size ranges. This behavior indicates that simple particle size classification and the selective rejection of a given size fraction are not effective measures for the removal of this contaminant. Therefore, if a reduction in the iron content of the tailings is required, it becomes necessary to adopt concentration methods based on physicochemical or physicomechanical properties of the minerals, such as selective flotation.

In addition, as corroborated by the granulochemical analysis, a predominance of micaceous minerals is observed in the coarser fractions of the tailings. The layered (lamellar) structure of these minerals hinders their effective fragmentation during grinding stages. At the same time, this characteristic promotes a stronger hydrodynamic drag effect during desliming operations, resulting in low selectivity during classification. Consequently, mica minerals are found in virtually all process streams.

The images also reveal apatite crystals across the entire particle size range evaluated, indicating potential for reprocessing and recovery of P_2O_5. In this context, the study of the characteristics of pure mineral becomes valuable for defining potential processing routes and selecting reagents to be employed for its recovery.

4.2 Apatite pure sample characterization

The density of the pure apatite sample used in this study was determined, and the corresponding result is presented in Table 4. An average density of 3.3 g/cm³ was obtained, which is consistent with that expected for a pure mineral. Previous results indicated approximately 97% purity of the mineral under

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investigation. Therefore, this result validates the subsequent experimental tests and ensures the representativeness of the observed behaviors. The average specific surface area of the apatite was 2.42 m²/g.

Table 4 – Apatite specific mass

Tests	Sample 1	Sample 2
1	3.30	3.29
2	3.29	3.30
3	3.29	3.31
Average	3.29	3.30

Source: Elaborated by the author

Although the density value obtained differs from those of typical minerals such as magnetite (5.2 g/cm³) and quartz and silicates in general (2.7 g/cm³), it does not ensure satisfactory selectivity in potential gravity separation stages. Because the sample is predominantly composed of fine and ultrafine particles, the density contrasts among the constituent minerals are not sufficiently pronounced to overcome the effects of hydrodynamic drag and surface charges acting on the particles, causing all minerals to exhibit similar behavior in an aqueous medium. Therefore, to ensure the recovery of apatite from phosphate tailings, the need for physicochemical concentration processes, such as flotation, is once again evident.

One of the key factors governing flotation efficiency is related to the surface characteristics of the mineral of interest, as well as its behavior in contact with flotation collector reagents. Accordingly, the zeta potential results of pure apatite as a function of slurry pH are presented in Figure 4.

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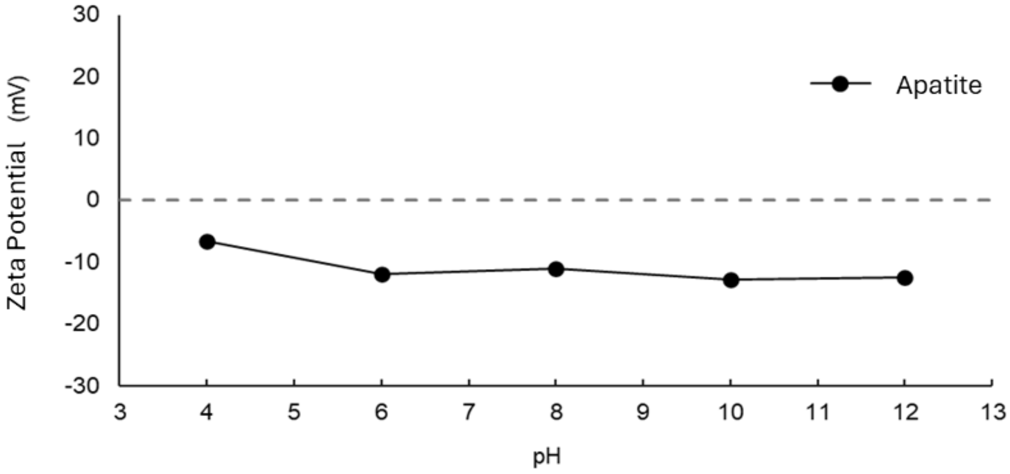
Figure 4 – Apatite Zeta potential



Source: Elaborated by the author

It is observed that throughout the entire pH range investigated, apatite exhibited a slightly negative surface charge, without significant variations in its electrostatic characteristics. Due to difficulties in achieving electrophoretic stability for measurement, resulting from the excess of H^+ ions required for pH adjustment, it was not possible to determine the zeta potential in the more acidic pH range.

Considering the negative surface charge of apatite and the use of fatty acids as the main collectors employed in phosphate flotation, reagent adsorption onto the mineral surface occurs predominantly through chemical mechanisms. This behavior arises because the anionic collector also exhibits a negative charge, leading to electrostatic repulsion between the reagent and the mineral surface.

Therefore, the determination of the optimal pH condition for enhanced collector adsorption onto the apatite surface is governed by the dissociation behavior of the reagent in the medium. Figure 5 presents the species distribution diagram of oleic acid, one of the most used fatty acids in flotation processes. Analysis of this diagram, which depicts the concentration of each species as a function of pH, indicates that under alkaline conditions above pH 8.5, the fatty acid molecules predominantly exist in their ionic form. In contrast, under more acidic pH conditions, the molecular form of the reagent prevails, inhibiting chemical interaction with the apatite surface.

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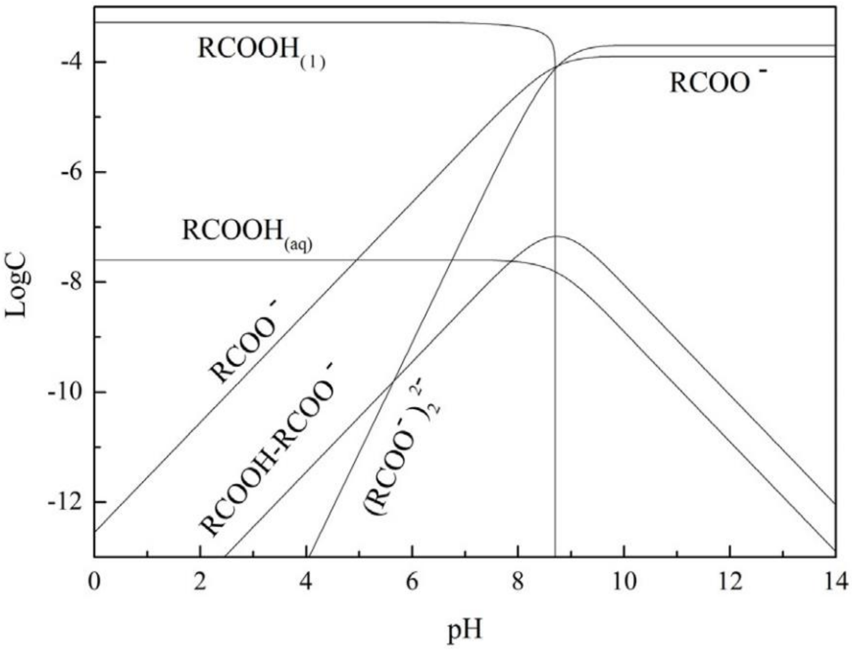
Figure 5 – Sodium oleate species diagram



Source: Dhar, Thornhill e Kota (2020)

Under alkaline pH conditions, the oleate ion ($RCOO^-$) predominates in solution. This ion is primarily responsible for forming complexes with Ca^{2+} ions present on the apatite surface, resulting in the formation of calcium oleate, $Ca(RCOO)_2$, and promoting the hydrophobization of the mineral surface. Therefore, it can be inferred that the application of anionic collectors based on fatty acids is potentially suitable for slurries operating at pH values above 8.5, a hypothesis that must be confirmed through experimental flotation tests.

4.3 Implications for future processing routes

Based on the mineralogical, granulometric, chemical, and electrokinetic characterization performed in this study, it is evident that the reprocessing of the analyzed phosphate tailing is strongly constrained by the predominance of fine and ultrafine particles and by the complex mineralogical associations present. The granulochemical distribution indicates that a significant fraction of the total P_2O_5 is concentrated in the finest particle-size classes, which also represent the largest

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mass proportion of the material. As a result, processing strategies based solely on size classification or selective desliming are unlikely to be effective, since they would lead to substantial losses of the mineral of interest.

The electrokinetic behavior obtained from pure apatite samples suggests surface characteristics compatible with the use of anionic collectors under alkaline conditions, particularly at pH values above 8.5, which favor chemical interactions with calcium sites on the mineral surface. These findings support flotation as a technically plausible concentration route for phosphate tailings, if it is specifically adapted to deal with ultrafine particles and with the interference effects associated with clay minerals, micas, and iron-bearing phases.

Nevertheless, the technological implications discussed herein must be interpreted within the limits of the experimental scope of the present study. No direct beneficiation or reprocessing tests, such as flotation, selective aggregation, or leaching, were carried out on the tailing, which prevents any direct evaluation of metallurgical performance or operational efficiency. In addition, fundamental physicochemical data were obtained from pure apatite samples rather than from apatite contained within the tailing itself, and mineralogical characterization was predominantly qualitative, with microscopic observations restricted to the coarser fractions of the material.

In this context, the processing routes outlined should be regarded as technically grounded indications rather than validated process solutions. Future investigations should focus on direct flotation testing of the tailing, the assessment of more selective collectors and reagent schemes, and the evaluation of complementary approaches as selective aggregation or hydrometallurgical routes particularly for the treatment of ultrafine fractions where conventional flotation performance is expected to be limited.

5. Conclusion

This study provided a detailed mineralogical and technological characterization of a phosphate tailing generated during the beneficiation of a

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carbonatitic phosphate ore, aiming to support future investigations on its potential reprocessing. The results demonstrated that the tailing is predominantly composed of fine and ultrafine particles, with a substantial portion of the total P_2O_5 associated with these size fractions, which poses significant challenges for recovery using conventional mineral processing routes.

The chemical and mineralogical analyses revealed that apatite is distributed across all particle-size classes investigated and is closely associated with iron-bearing minerals, micas, and clay minerals. This combination of ultrafine granulometry and complex mineralogical associations tends to limit the selectivity of purely physical separation methods and reinforces the need for process strategies based on physicochemical principles.

Fundamental studies carried out on pure apatite samples provided relevant insight into the intrinsic surface properties of the mineral, indicating electrokinetic behavior compatible with the use of fatty-acid-based anionic collectors under alkaline conditions. Although these results do not directly represent the behavior of apatite within the tailing system, they offer valuable guidance for the selection of reagents and operating conditions in subsequent experimental studies.

Overall, the present work should be regarded as a diagnostic and foundational contribution, delineating the main constraints and technical indicators associated with phosphorus recovery from phosphate tailings. By clarifying the key limiting factors and identifying plausible directions for future processing strategies, this study contributes to guiding further experimental research aimed at the sustainable utilization of phosphate mine tailings and the reduction of this environmental liability.

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