AMANDA FERNANDES PEREIRA DA SILVA
(ORGANIZADORA)

# INGENIERÍA:
## INVESTIGACIÓN, DESARROLLO E INNOVACIÓN

Atena
Editora
Ano 2022

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# Ingeniería: investigación, desarrollo e innovación

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**Dados Internacionais de Catalogação na Publicação (CIP)**

I46 Ingeniería: investigación, desarrollo e innovación / Organizador Amanda Fernandes Pereira da Silva. – Ponta Grossa - PR: Atena, 2022.

Formato: PDF
Requisitos de sistema: Adobe Acrobat Reader
Modo de acesso: World Wide Web
Inclui bibliografia
ISBN 978-65-258-0862-8
DOI: https://doi.org/10.22533/at.ed.628220712

1. Ingeniería. I. Silva, Amanda Fernandes Pereira da (Organizador). II. Título.

CDD 620

**Elaborado por Bibliotecária Janaina Ramos – CRB-8/9166**

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Boa leitura!

Amanda Fernandes Pereira da Silva

SUMÁRIO

# CAPÍTULO 3

# CHEMICAL AND MICROSTRUCTURAL ANALYSIS OF TAILINGS AND WASTE ROCK FROM A PHOSPHATE MINING

*Data de submissão: 07/11/2022* *Data de aceite: 01/12/2022*

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**ABSTRACT:** Due to the complexity of the behavior of the tailings and waste rock disposal structures of mining and also to the recurrent accidents of rupture of tailings dams, the importance of using disposal techniques that guarantee greater safety, such as the co-disposal technique, which aims to mix tailings and waste rock to try to improve their geotechnical behavior. The objective of this work is to contribute to studies of the geotechnical behavior of tailings and waste in phosphate mining projects, based mainly on their microstructural and chemical characteristics to be used in co-disposition. For this, the geotechnical characterization of these materials was carried out, as well as chemical analyzes of the EDS, XRF and XRD type, which allowed a better understanding of the specific characteristics of these materials, especially the waste rock, regarding their granulometry and microstructural composition. It was possible to observe the presence of fine clays in the waste rock and the change in its behavior when using dispersants, which could influence the final behavior of the mixture and also a disposition structure designed with these materials.

**KEYWORDS:** Co-disposal, tailings, waste rock, chemical, microstructural.

## ANÁLISE QUÍMICA E MICROESTRUTURAL DE REJEITOS E ESTÉREIS DE UMA MINERAÇÃO DE FOSFATO

**RESUMO:** Devido à complexidade do comportamento das estruturas de disposição de rejeitos e estéreis de mineração e também aos recorrentes acidentes de ruptura de barragens de rejeitos, percebe-se a importância de utilizar técnicas de disposição que garantam maior segurança, como a técnica de co-disposição, que visa misturar rejeitos e estéreis para tentar melhorar seu comportamento geotécnico. O objetivo deste trabalho é contribuir para estudos de comportamento geotécnico de rejeitos e estéreis em projetos de mineração de fosfato, com base principalmente em suas características microestruturais e químicas para serem utilizados na co-disposição. Para isso, realizou-se a caracterização geotécnica desses materiais e também análises químicas do tipo EDS, XRF e XRD, que permitiram um melhor entendimento das características específicas dos materiais estudados, principalmente os estéreis quanto a sua granulometria e composição microestrutural. Foi possível observar a presença de argilas finas no estéril e a alteração do comportamento do mesmo quando da utilização de dispersantes, o que poderia influenciar o comportamento final da mistura e também de uma estrutura de disposição projetada com estes materiais.
**PALAVRAS-CHAVE:** Co-disposição, rejeitos, estéreis, química, microestrutural.

## 1 I INTRODUCTION

There is no doubting the relevance of mining in both Brazil and the rest of the world. According to economic data from the Brazilian Mining Institute (IBRAM, 2022), in 2021, the Brazilian mineral sector recorded a 62% increase in revenue compared to 2020, totaling BRL 339.1 billion (excluding oil and gas). The Financial Compensation for the Exploration of Mineral Resources (CFEM) produced revenues of almost BRL 10,3 billion. Brazilian mineral exports reached US$ 58 billion, an increase of 58.6% compared to 2020.

Despite the mining sector's contribution to the economy, in recent years, accidents with mining tailings dams and mine waste dumps have risen, with countless losses in terms of human lives and social and environmental impacts. After the rise in accident cases, the world has followed an increasing concern regarding managing risks associated with tailings dams and other residues in the mining sector (IBRAM, 2019).

The structures constructed by the disposal of mine waste rock, known as mine waste dumps, also have a high potential for negative environmental effects when improperly managed. It is primarily because they have the potential to generate acids, contaminate soil and groundwater, and require large construction sites.

Alternative management techniques based on increasing the tailings solids contents have been studied and applied to minimize the possibility of instability of tailings disposal structures constructed using conventional techniques (MEND, 2017; CARNEIRO & FOURIE, 2018). Among the alternative methods, co-disposal technique occupies a special place when the aim is to reduce the disposal area of the generated wastes. Still, it is crucial to understand the behavior and interactions between the mixed materials.

As in most igneous phosphate deposits, in low-grade Brazilian reserves, different reagents are used in the flotation stage to beneficiate apatite and increase the phosphate recovery. The use of these reagents tends to chemically alter the mineral and gangue particles present to facilitate the mineral concentration process, which can influence the geotechnical behavior of tailings and waste rock when disposed separately or co-disposed. Therefore, it is essential to carry out a chemical and microstructural study to understand the structure and composition of these materials.

This article aims to contribute to tailings and waste rock geotechnical behavior studies in mining projects, based mainly on their microstructural and chemical characteristics. It focuses on minimizing the environmental impacts resulting from the mining process, having as a reference the waste rock and tailing generated by phosphate mining and processing.

## 2 I MATERIALS AND METHODS

Tailings and waste rock from phosphate mining and processing were used in this study. The samples were composed of tailings generated in the flotation stage of mineral processing, sampled on the mining tailings dam, and waste rock material generated in the mining and sampled in the waste rock dump.

The samples of materials used in this research were geotechnically characterized according to the requirements of the American Society for Testing and Materials (ASTM) standards, and the tests were carried out at the Geotechnics Laboratory of the Graduate Program in Geotechnics of the Faculty of Technology of University of Brasília (UnB), in the Geotechnics Laboratory of the Engineering Faculty of the Federal University of Catalão (UFCAT) and in the Multiuser Laboratory in Renewable Energies (LAMER) of Chemical Engineering at the Federal University of Uberlândia (UFU).

The samples were submitted to the tests of: specific gravity - ASTM D854-14 (ASTM, 2014); limits of consistency - ASTM D4318-17e1 (ASTM, 2017), and; sieving and sedimentation - ASTM D6913-17e1 (ASTM, 2017) and ASTM D7928-21e1 (ASTM, 2021). The specific gravity was also obtained using an automatic density analyzer, brand Quantachrome Instruments and model PENTAPYC 5200e. It is worth mentioning that for the penta-pycnometer test, the waste rock passing through the 2.0 mm sieve was considered, while in the pycnometer method (ASTM D854, 2014), the material passed through the 4.75 mm sieve was considered.

The granulometric curves of the materials were obtained considering coarse and fine sieving tests initially, without the use of dispersant and without agitation with a dispersion cup. Later the joint sieving and sedimentation test was carried out with and without the use of a dispersant (sodium hexametaphosphate). The tests aimed to evaluate if there would be any alteration in the granulometric curves due to the adhesion of the silts and clays in the waste rock material, a behavior observed in a tactile-visual analysis. The material was classified according to the Unified Soil Classification System (USCS) as per ASTM D2487-17e1 (ASTM, 2017).

Chemical and microstructural analyzes were carried out on the tailings and waste rock samples. The objective of the analysis was due to the significant influence of silts and clays on the granulometric curves of the waste rock. Therefore, chemical analyzes were performed using the Energy Dispersion Spectroscopy (EDS) method, with an X-ray detector, evaluating the main elements present in the materials. Part of the EDS analyzes was carried out at the UFCAT, and they were performed from two samples of each material.

For microstructural characterization, a Scanning Electron Microscope (SEM) model Zeiss EVO MA10 was used. The analysis was performed at the Multiuser Laboratory of Scanning Electronic Microscopy (LAMEV) of Chemical Engineering at the Federal University of Uberlândia (UFU). Square samples of 1 cm x 1 cm were prepared from the coating/impregnation with gold in a Leica EM SCD050 equipment to become the samples conductive and allow analysis by SEM.

Quantitative chemical analyzes of oxides were also performed by X-Ray Fluorescence Spectrometry (FRX). The analyzes were carried out at the Multiuser Laboratory in Renewable Energies (LAMER) of Chemical Engineering at the Federal University of Uberlândia (UFU) with a Bruker X-Ray Fluorescence Spectrometer, model S8 Tiger. The samples were prepared by melting the tailings and waste rock into pellets, presenting better homogeneity and results than the pressing technique. For casting the pastilles, a Vulcan 4 MA melting machine was used.

The X-Ray Diffractometry (XRD) technique was used to identify, characterize, and quantify the minerals phases. The research was carried out in a Rigaku diffractometer, model Geigerflex D/MAX-2A/C, from the Ray Diffractometry Laboratory at UnB.

## 3 I ANALYSIS AND RESULTS

In Table 1 and Table 2, there is a summary of the results of the characterization tests. With the results of consistency indices tests from Table 1 (liquidity and plasticity limits), it was evidenced that the tailings present a non-plastic behavior, not having consistency parameters. The specific gravity was calculated using the pycnometer method and an automatic density analyzer - PENTAPYC 5200e. The penta-pycnometer obtained a $\rho_s$ of 3.15 g/cm$^3$ for the tailings and 3.47 g/cm$^3$ for the waste rock.

| Samples | $\rho_s$ (g/cm$^3$) | LL (%) | LP (%) | IP (%) |
|---|---|---|---|---|
| Tailing | 3,14 | NL | NP | - |
| Waste rock | 3,68 | 38 | 24 | 14 |

Table 1 – Summary of the results of the characterization tests.

The granulometric test presented in Table 1 was performed considering the percentages of gravel, sand, silt and clay (USCS scale) in the fractions of materials submitted to the test.

| Samples | Granulometry (%) | | | | | Classification USCS |
|---|---|---|---|---|---|---|
| | Clay and silt | Sand | | | Gravel | |
| | | Fine | Medium | Coarse | | |
| Tailing – Sieving (WD)* | 12,49 | 83,06 | 4,45 | - | - | SM |
| Tailings – Sieving and sedimentation (WD)** | 14,01 | 82,00 | 3,99 | - | - | SM |
| Tailings – Sieving and sedimentation (WTD) | 13,2 | 82,74 | 4,06 | - | - | SM |
| Waste rock – Sieving (WTD) | 14,59 | 22,71 | 17,85 | 22,60 | 22,25 | SC |
| Waste rock – Sieving and sedimentation (WTD) | 30,59 | 11,8 | 11,70 | 23,00 | 22,92 | SC |
| Waste rock SP1 – Sieving and sedimentation (WD) | 33,88 | 8,23 | 11,52 | 23,25 | 23,13 | SC |
| Waste rock SP2 – Sieving and sedimentation (WD) | 32,2 | 8,12 | 13,77 | 23,02 | 22,90 | SC |

* WD – with use of dispersant.
** WTD – no use of dispersant.
SP1 and SP2 – specimen 1 and 2.
Note: there was no agitation in the dispersion cup in the tests with sieving only.

Table 2 – Summary of the results of the characterization tests.

The results of the granulometric analysis indicate a high presence of non-plastic fines in the tailings, probably due to the tailings processing method. Using a dispersant does not change the tailing’s granulometric distribution.

The influence of the dispersant in the characterization was verified only in the waste rock samples, whose in situ condition must have a predominant granulometry closer to the result obtained without the use of dispersant and without the agitation process in the dispersion cup. There was a very marked distinction between the curves obtained with and without the use of the dispersant and considering the agitation or not with the dispersion cup before sieving the fine fraction (Figure 1). It is observed that the presence of silts and clays influences the granulometric curves of these materials, with a tendency for the finer particles to aggregate, both around themselves and adhering to larger particles as the sand fraction, considering the material that did not undergo agitation before of the sieving.

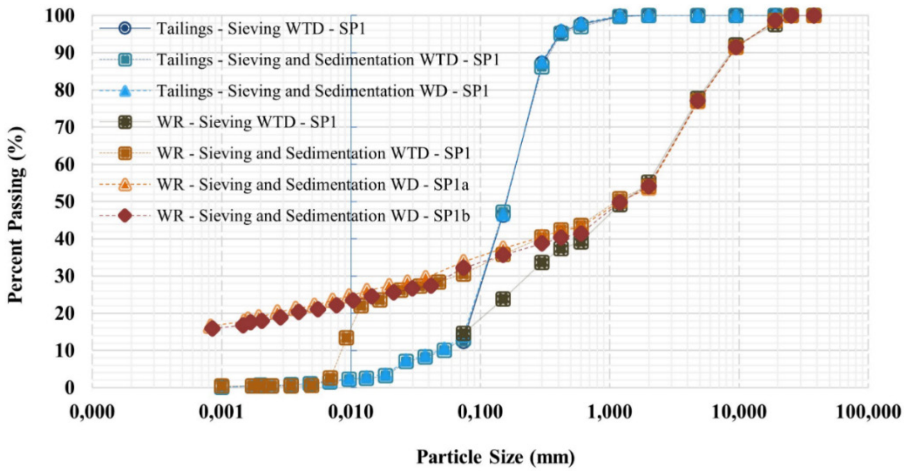


Figure 1 – Particle size analysis. Where, *WR* is waste rock, *WD* is with use of dispersant, *WTD* is no use of dispersant and SP is specimen number.

Due to the change in the granulometric curve with dispersants in the case of waste rock, it is important to consider the influence of mining processing water before co-disposing. It uses several products in mineral beneficiation to change the properties of the materials to concentrate the mineral of interest, which may change the in situ condition of the waste rock, where finer fractions could influence the entire mechanical and hydraulic behavior of the mixture.

Table 3 presents the results of chemical analyzes performed by the Energy Dispersion Spectroscopy method (EDS), identifying the main elements present in the materials. It can be seen from the results of the chemical analysis that the mining tailings are basically composed of the iron element (where the minerals related to it are removed in the mineral processing, mainly in the magnetic separation stage), silica and titanium, in addition to other elements of lesser weight, such as calcium, phosphorus and some rare earth elements. Waste rock is also composed primarily of iron, silica and aluminum. However, the element iron, in this case, makes up more than 60% of the sample, while silica and aluminum make up approximately 10.6% to 12%.

| Samples | | | | | | | |
|---|---|---|---|---|---|---|---|
| AM1 - Tailings | | AM2 - Tailings | | AM3 - Waste rock | | AM4 – Waste rock | |
| **Elements** | **%** | **Elements** | **%** | **Elements** | **%** | **Elements** | **%** |
| **Fe** | 37,672 | **Fe** | 38,595 | **Fe** | 61,9 | **Fe** | 61,636 |
| **Si** | 30,592 | **Si** | 29,458 | **Si** | 12,051 | **Si** | 11,363 |
| **Ti** | 10,748 | **Ti** | 10,877 | **Al** | 10,646 | **Al** | 10,932 |
| **Ca** | 8,559 | **Ca** | 8,419 | **Ti** | 4,755 | **Ba** | 5,38 |
| **P** | 5,011 | **P** | 5,138 | **Ba** | 4,603 | **Ti** | 4,306 |
| **Ce** | 1,859 | **Ce** | 2,116 | **P** | 2,285 | **P** | 2,285 |
| **Mn** | 1,185 | **Mn** | 1,07 | **Sr** | 1,322 | **Sr** | 1,261 |
| **Zr** | 0,849 | **Zr** | 1,047 | **Zr** | 0,728 | **Zr** | 1,071 |
| **Sr** | 0,813 | **Sr** | 0,783 | **Ca** | 0,559 | **Ca** | 0,518 |
| **Mg** | 0,65 | **Mg** | 0,771 | **Mn** | 0,386 | **Nb** | 0,402 |
| **Nb** | 0,641 | **Nb** | 0,679 | **Nb** | 0,304 | **Mn** | 0,385 |
| **Al** | 0,567 | **Al** | 0,425 | **K** | 0,171 | **K** | 0,126 |
| **K** | 0,274 | **K** | 0,284 | **S** | 0,088 | **S** | 0,098 |
| **V** | 0,178 | **Cr** | 0,161 | **Cu** | 0,087 | **Cu** | 0,088 |
| **Br** | 0,172 | **Zn** | 0,15 | **Zn** | 0,073 | **Zn** | 0,073 |
| **Zn** | 0,157 | **Y** | 0,028 | **Y** | 0,042 | **Y** | 0,043 |
| **Ni** | 0,043 | | | | | **Br** | 0,036 |
| **Y** | 0,031 | | | | | | |

Table 3 – Summary of chemical analysis results performed on 4 samples.

Through the microscopic characterization of the tailings and waste rock using Scanning Electron Microscopy (SEM), it was possible to observe the arrangement, shape and possible adhesion of the fine particles in the waste rock and tailings.

Figure 2 shows the tailings sample observed with different resolutions by SEM, considering an increase ranging from 50x (Figure 2a) to 700x (Figure 2d). Because the materials have been oven dried, it is observed that there is no cohesive aspect between the particles, which tends to be purely due to an apparent cohesion when water is present. It can also be noted that the particles are quite angular for the most part.

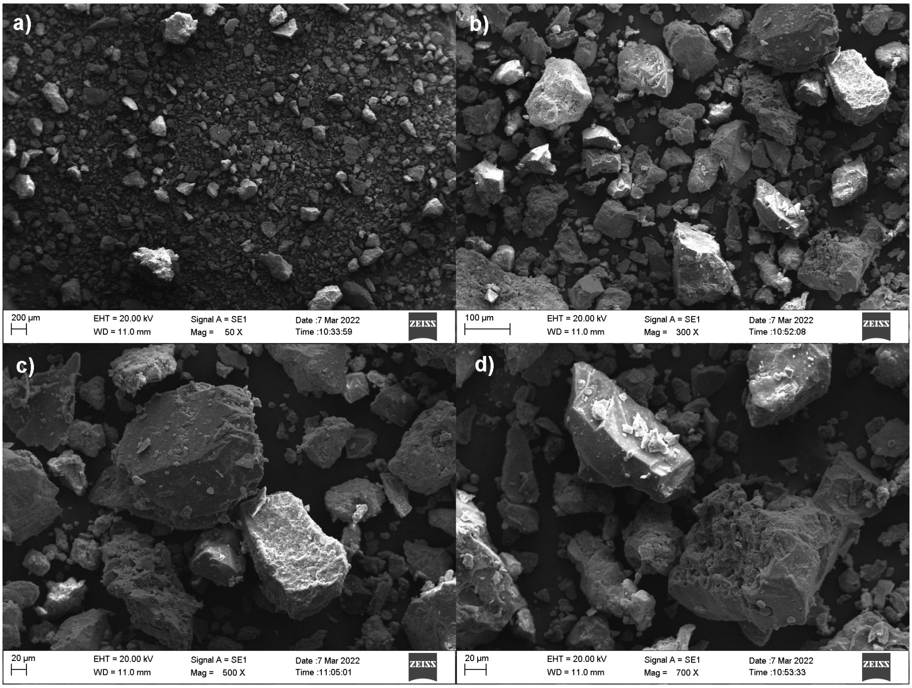


Figure 2 – Scanning electron microscopy in the tailings: a) 50x magnification; b) 300x magnification; c) 500x magnification; d) 700x magnification.

In Figure 3, it is possible to observe the waste rock sample for different resolutions in the SEM, considering an increase of 50x, 300x, and 6,500x. Due to the previous stage of drying in an oven, it is noticed that the larger particles are loose. Still, it is possible to see that these are formed either by agglomeration of smaller particles or by the adhesion of these smaller particles on the mineral’s surface. The shape of the particles is more rounded and without angulations. It is interesting to note in Figure 3c the adhesion of the silt and clay particles on the minerals’ surface. This aspect was also observed in the tactile-visual analysis, where the finer material tends to adhere fully to the surface of certain particles, being removed only after washing with agitation or using a dispersant.

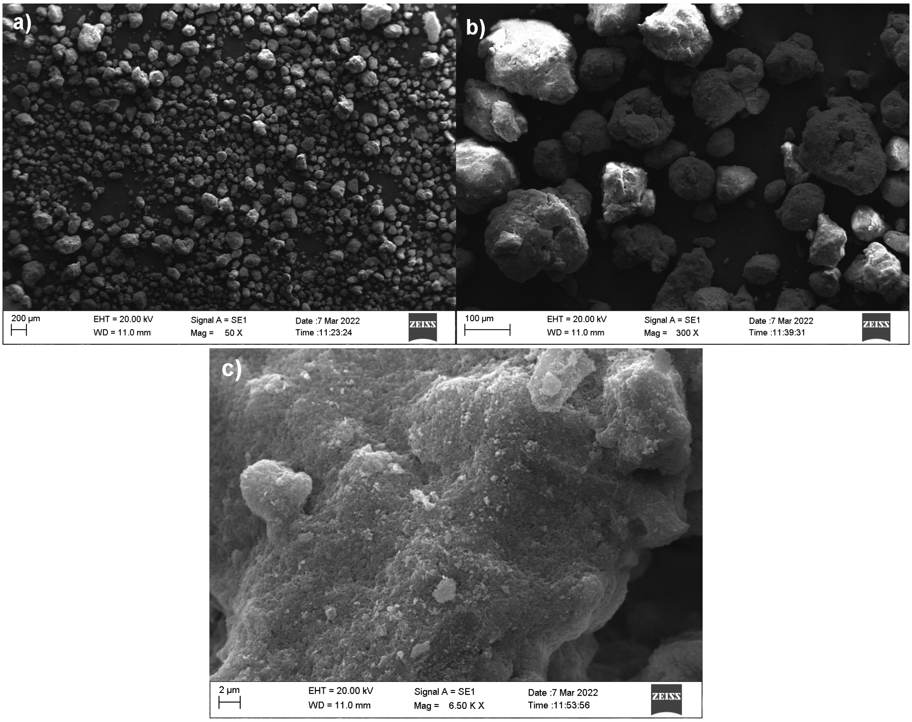


Figure 3 – Scanning electron microscopy in the waste rock: a) 50x magnification; b) 300x magnification; c) 6500x magnification.

X-Ray Fluorescence (XRF) made it possible to determine the principal oxides in the waste rock and tailings samples (Figure 4). For the tailings samples, the principal oxides were $SIO_2$, $Fe_2O_3$, $TiO_2$, CAO, and $P_2O_5$.

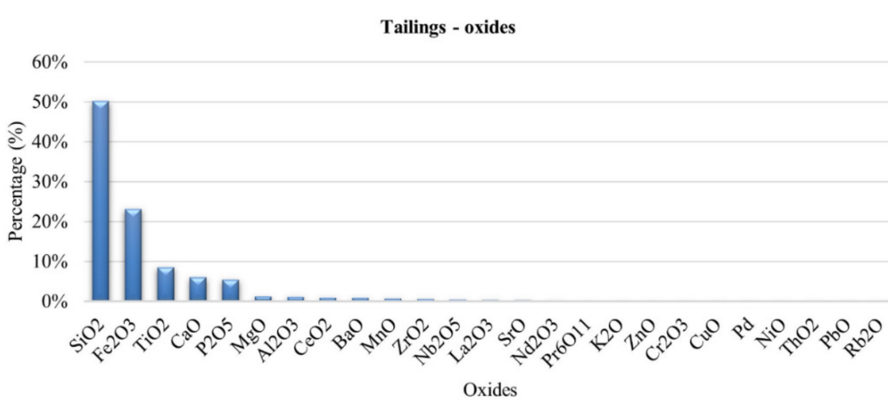


Figure 4 – Results of XRF analyzes on tailings samples.

The principal oxides present in the waste rock samples (Figure 5) were $Fe_2O_3$, $SIO_2$,

$Al_2O_3$, $TiO_2$ and $P_2O_5$. In this case, most oxides come from iron minerals, such as magnetite. In contrast, the other oxides appear in much smaller amounts, thus explaining the greater density of the waste rock.

Considering that oxides such as $Fe_2O_3$, MgO, $Al_2O_3$ and $SiO_2$ are some of the main constituents of phyllosilicates, and that the chemical composition of the primary Brazilian phosphate deposits has these minerals, it is possible to suggest that the waste rock presented here has minerals from the group of phyllosilicates, which can be: vermiculite, biotite, tetraferriphlogopite, phlogopite, muscovite, and vermiculite. Biotite and phlogopite have iron and aluminum oxides and magnesium in their composition.

Another mineral that could be present in tailings and waste rock is magnetite, a common iron ore that tends to be disposed of separately in the form of piles after being removed from the beneficiation circuit in the magnetic separation stages in the phosphate plants.

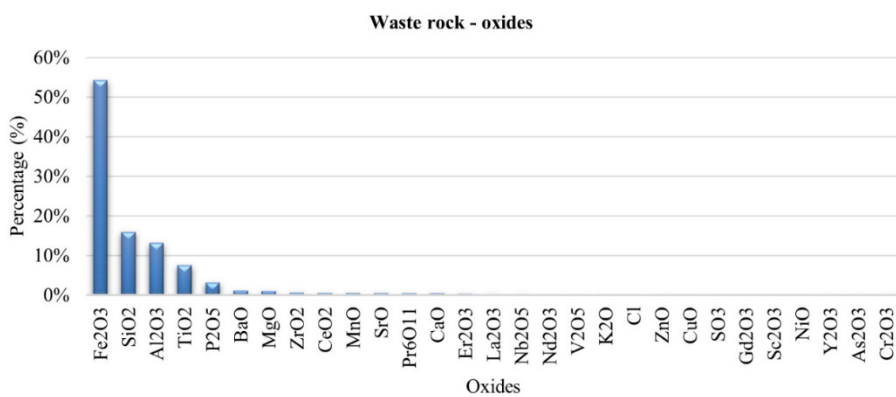


Figure 5 – Results of XRF analyzes on waste rock samples

The X-Ray Diffractometry (XRD) was used to evaluate the samples' constituent minerals. The tailings diffractograms showed in Figure 6 the following mineral constituents: quartz, anatase, stilpnomelan, vermiculite, ilmenite, siderite, pyrochlore, antigorite. Considering the x-ray fluorescence analysis and the main constituent minerals of the phosphate deposits, possibly the most appropriate would be the identification of magnetite instead of ilmenite and siderite. The analysis is performed by software containing an extensive database, which tends to identify minerals with similar peaks and constitutions.

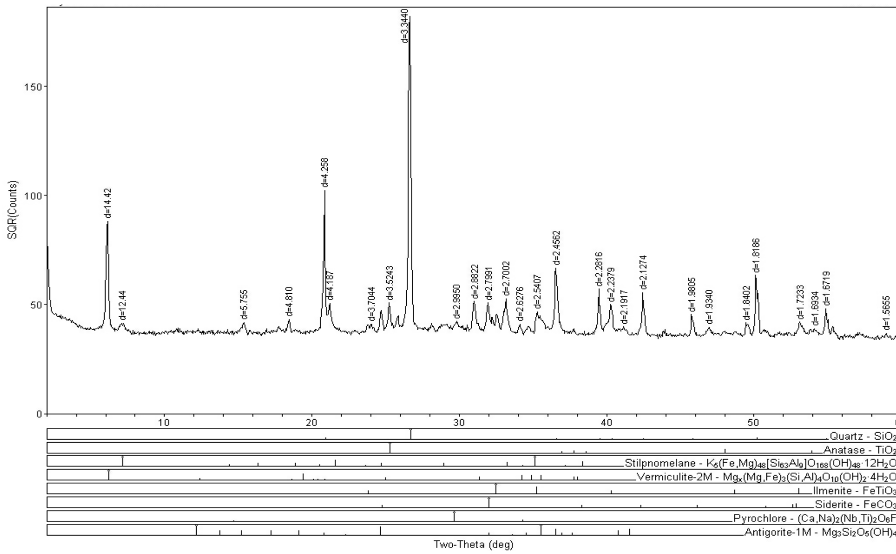


Figure 6 – Results of XRD analysis on tailings samples.

The main mineral constituents of the waste rock sample were: quartz, kaolinite, montmorilinite, anatase, goyazite and goethite (Figure 7). According to the characteristics of the deposit, the most relevant results, in this case, would be the identification of apatite instead of goyazite, and magnetite instead of goethite.

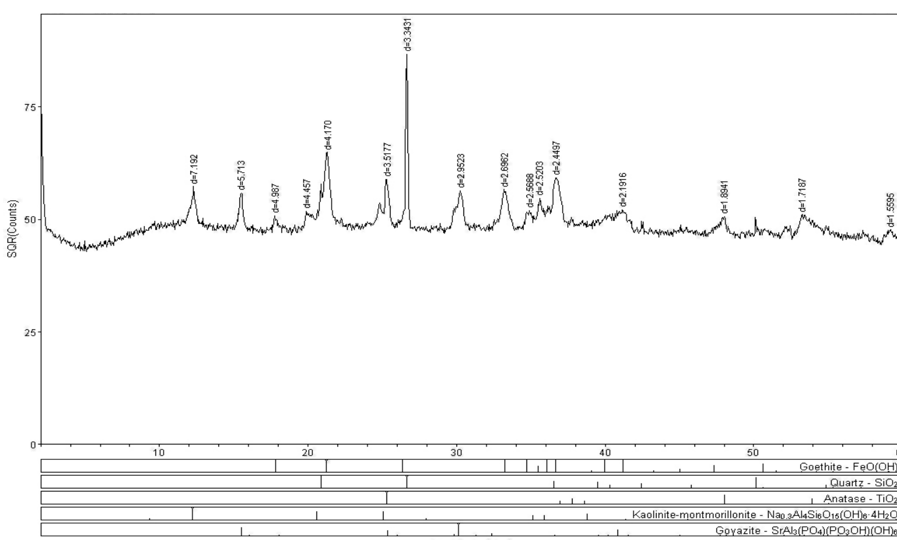


Figure 7 – Results of XRD analysis on waste rock samples.

In general, some mineral constituents that are indicated in other chemical analyses performed (EDS and XRF) were probably not identified. Still, it was possible to perceive the presence of clay minerals, mainly in the waste rock samples, which strongly influence the behavior of the materials and mixtures, both in mechanical and hydraulic properties.

## 4 I CONCLUSION

From the results obtained from the geotechnical characterization of waste rock and tailings, it can be seen that the waste rock has a greater presence of fines than other materials generally studied for co-disposal, such as iron ore waste. It is observed that there is a smaller proportion between the average size of the grains, $D_{50wr}$:$D_{50tailings}$ = 7.5, while for other mining materials, this proportion tends to be equal to or greater than 20 according to Furnas (1928) and Wickland et al. (2006).

As the phosphate mining and processing waste rock and tailings is produced through very specific beneficiation steps, such as froth flotation and the use of different types of reagents, such as collectors and dispersants, there is a need to carry out chemical and microstructural tests, mainly using the processing water of the tailings, what was not possible in this study.

The chemical analyses allowed a better understanding of the specific characteristics of these materials, mainly of the waste rock regarding its granulometry and microstructural composition, where they are formed by agglomeration of smaller grains among themselves and also adhesion of these on the surface of larger particles, being more rounded, without angulations.

Considering the analysis of EDS, XRF and XRD, it can be seen that these smaller particles tend to be fine clays, such as montmorillonite and kaolinite, which can greatly influence the mechanical and hydraulic behavior of a possible mixture between tailings and waste rock.

Because the tailings have more angular materials, a possible mixture may reduce these angles due to the presence of the clays, which may influence the mixture's friction parameters and global cohesion.

An important factor is the behavior of the mixed materials when in contact with the mineral processing water, which can act as a dispersant and deflocculate the fines in the waste rock and mixture, generating a behavior similar to that observed in the granulometric curves, where the fines are naturally adhered forming larger particles. Therefore, it is essential to mechanically test the mixtures under different scenarios to understand the real behavior of the materials.

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