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Article

# Characterization and Analysis of Iron Ore Tailings Sediments and Application Possibilities in Earthen Construction

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**Abstract:** Mineral extraction is one of the most important for the economies of different countries, and Brazil is one of the world's primary iron ore producers. Unfortunately, the dams are still the main problem, mainly in Minas Gerais, especially after the Fundão Dam rupture happened in 2015. Additionally, there is still a massive presence of buildings produced on earth throughout the Minas Gerais mining region, built from the 18th century to today. Investigating the potential of IOT to be incorporated into traditional earthen construction techniques in regions affected by dam ruptures presents a relevant and innovative research approach. In addition, the local reuse of these sediments should be the priority. Thus, the main objective of this work was to collect, characterize, and analyze the possibilities of application of these tailings to produce RE. XRD, XRF, granulometry, porosity, specific density, toxicity, and MEV/EDS were performed on the samples collected at three points. To analyze the soil-IOT compatibility, representative mixtures of RE were produced, and the specific mass, compaction, and compressive strength were performed. It was observed that the IOT samples have a high silica content and that the mixtures of IOT-soil, even without cement, reached compressive strength values above the current international standards.

**Keywords:** mining wastes; rammed earth; toxicity; mechanical analysis

## 1. Introduction

The mineral extraction activity is one of the most important for the economy of different countries. In this context, Brazil, Australia, and China are among the world's main iron ore producers [1]. According to data from IBRAM [2], Brazil's mining sector revenue reached R\$ 209 billion in 2020. Iron ore was responsible for 66% of total revenue, with R\$ 138 billion.

A significant portion of the occupation of Minas Gerais emerged in the 17th century due to gold exploration, which continued until the second half of the 18th century. The extraction of iron ore, starting in the 20th century, was the main factor that led industries such as Vale, Samarco, and Alcan to establish themselves in the region and intensely urbanize and occupy various municipalities.

For every ton of iron ore processing in Brazil, around 400 kg of tailings is generated [3]. One possible use of these tailings is the construction of mining dams, where these materials are disposed of as pulp or mud [4]. In Brazil, there are about 870 mining dams; of these, only 50% are included in the national dam safety policy [5].

Minas Gerais is a state in Brazil where the iron ore extraction industry is essential to economy. Starting in the 20th century, iron extraction in the state was the main factor that led industries such as Vale, Samarco, and Alcan to establish themselves in the region and intensely urbanize and occupy

various municipalities. As a result of the operation of these companies in Minas Gerais, there are several tailings' dams. Some dams have been uncharacterized in Minas Gerais since the last accident occurred in Brumadinho/MG, in 2019. Currently, 12 dams have already been uncharacterized [6].

### *1.1. Iron ore tailings*

Unfortunately, the dams are still the main problem which mining companies face, especially after the rupture of the Fundão Dam, happened in 2015, in Bento Rodrigues (a sub-district of the municipality of Mariana, MG). This rupture caused the displacement of tailings and mud through the Gualaxo do Norte, Carmo, and Doce Rivers for 663 km. Thirty-nine municipalities were affected, most in Minas Gerais and three in Espírito Santo state [7]. The mud splashed for nearly 17 days [8] until it reached the Atlantic Ocean. Tailings strongly change the rivers' sediment composition and can lead to long-term contamination of biological specimens. Even after seven years, it is important to quantify changes in the ecosystem by observing the environment and characterization of sediment and soil samples in places that had had contact with the mud.

A large part of the tailings displaced by the Rio Doce was retained in the Risoleta Neves hydroelectric plant, also known as Candonga, located 113 km away from the Fundão dam, thus paralyzing its operation. The process of dredging, compaction, and transport to the stacking areas has been carried out by the Renova Foundation, since 2016, in the Gualaxo do Norte River and Risoleta Neves hydroelectric plant [9]. However, only in February 2023 the hydroelectric plant activities were resumed in the testing phase and may be released for commercial functions after this phase has been concluded. Another part of the tailings accumulated where the mud passed has transformed the relief, landscape, and local soil composition.

In this scenario, the search for new ways of tailings disposal from the iron mining process has been the focus of different research. Some studies point to the feasibility of inserting these samples in construction materials [10], such as applying iron ore tailings (IOT) as a substitute for natural fine aggregate [11]. Iron ore tailings have also been applied as a partial substitute for cement in concrete [12-13], in colored mortars [14] in the production of pigments for paints [15], in the manufacture of bricks [16-17], and the production of micro concretes [18] and as a precursor in the synthesis of geopolymers [19-22].

A deeper concern about their physicochemical properties is necessary before understanding how to use them and develop technologies using these wastes. The accumulated tailings in rivers, dredged and stocked, have been changed and can no longer be treated as dam tailings. It is important to understand that this material is a tailing sediment, so it is not possible to compare it with iron ore tailings taken from the dam [23].

Since the rupture that occurred in Mariana, characterization studies of the sedimented tailings have been carried out [24-30]. New characterization studies must be carried out because of the time that the material is in the environment, deposited in the soil and at the bottom of rivers as sediments. In addition, the local reuse of these sediments should be the priority, as it eliminates the environmental and economic impact of transporting this material and strengthens income generation with new products that can be consumed locally.

### *1.2. The use of wastes in earthen components*

Earth has been a building material for thousands of years due to its abundance and widespread availability. It is found worldwide and can be utilized in different forms and consistencies. In addition to traditional use, the earth is a promising material that can offer sustainable solutions due to significant reductions in environmental impact, as well as economic benefits for building construction, as material extraction and transportation costs are minimized due to wide availability at construction sites. Furthermore, building with earth presents environmental benefits by requiring less energy, producing less waste, and reducing fossil fuel burning during production, reducing greenhouse gas emissions such as carbon [31].

However, using earth as a building material may present challenges, especially regarding strength and durability. This is being overcome by incorporating other materials, which may be used

as stabilizers to improve the properties of earth components and not affect the environmental impact, such as cement. Concerning contemporary earthen construction techniques, rammed earth stands out internationally [32]. Rammed earth (RE) is a construction technique with the earth of a monolithic nature, which consists of setting up wooden forms called rammed earth, similar to what is done with concrete, where soil is placed and must be compressed with a pestle or with the help of feet. This mixing process aims to obtain greater dough consistency. It is also known that the soil chosen must have a mixture of sand and clay to obtain greater agglutination and less chance of disintegration of the material [31-32].

For this construction type, the earth must be stabilized using binders, fibers or through mechanical efforts. Soil stabilization, in the case of architecture, means improving structural parameters, such as compressive strength and durability of the building [33]. Traditionally, farmyard manure, clay, or animal hair mixtures were used to produce more significant binding to the mixture [31].

When rammed earth is not stabilized, the walls may suffer damage caused by erosion and water ingress, which can cause cracks if not adequately protected. On the other hand, incorporating additives into the earth mixture makes the material more resistant. Some of them are lime, cement, or biopolymers, reducing the need for maintenance and repairs [32].

Several residual materials have been researched as soil stabilizers to produce rammed earth [32, 34-35] and adobe [36, 37] but not yet a sediment of IOT. Cementitious stabilization in rammed earth has been widely employed in recent years to improve its durability. Adding cement or lime as stabilizers increases the rammed earth's strength and reduce shrinkage and wall disintegration [32]. This has also been found to be more effective in reinforced rammed earth walls [38] preventing corrosion of the structural reinforcement. According to Kariyawasam and Jayasinghe [39], contents above 4% of cement are desirable for rammed earth construction in tropical climates, and they also suggest that the use of up to 10% cement as a stabilizer still results in lower embodied energy compared to ceramic brick construction.

Rammed earth construction stabilized with cement achieves values of one-third of the embodied energy of conventional masonry construction and less than one-quarter of the embodied energy of reinforced concrete construction [40]. Thus, rammed earth construction with low cement addition is considered sustainable [39]. According to Arrigoni et al. [32], incorporating cement as a stabilizer has increased the durability of rammed earth. However, life cycle analysis can be used to measure the environmental impact of this construction technique, particularly with the addition of clinker to rammed earth. Therefore, the author recommends using alternative materials to cement as stabilizers for rammed earth to achieve even lower embodied energy values [32].

Regarding waste materials, some studies indicate that the environmental impacts can be similar between unstabilized rammed earth and rammed earth stabilized with waste materials when the local soil is not suitable on its own [32]. Kosarimovahhed and Toufigh [41] and Giuffrida, Camponetto, and Cuomo [42] pointed out the importance of using waste materials to stabilize soil for rammed earth construction so that the technique continues to be considered low impact. Moreover, the use of waste materials to improve the properties of rammed earth has already been presented by several authors, including fly ash, calcium carbide, and steel slag.

Investigating the potential of IOT sediment as a material to be incorporated into traditional earth construction techniques in regions affected by dam ruptures presents a relevant and innovative research approach. Such relevance is justified by the possibility of reconstructing damaged buildings using the same previous techniques, thus preserving the region's traditional construction.

The region of Mariana and Ouro Preto is connected to the origin of the occupation of Minas Gerais, and the architecture of the colonial period was widely based on earth constructions. Of the 20 buildings present in the architectural collection of the urban complex of Mariana, listed by IPHAN [43], at least 14 buildings were built with earthen techniques, with the most prevalent of them being adobe and rammed earth. Regarding the buildings damaged by the collapse of the Fundão dam, approximately 50% were constructed using earth as a building material, employing techniques such as rammed earth, wattle and daub, and adobe [44].

Thus, earth architecture has always been present in cities still linked to iron ore production. Furthermore, the use of IOT has not been analyzed yet in association with the earth, such that examining its characteristics to support its use in earth components remains a gap that researchers in the field of iron ore waste reuse have not filled. So, the present work aimed to characterize the tailings sediment collected around Mariana, Barra Longa and Rio Doce seven years after the failure of the Fundão dam.

## 2. Materials and Methods

To investigate the possible application of the IOT sediments in the production of rammed earth, the experimental design of this work was divided into three steps: i) collection of IOT sediment and soil samples; ii) characterization of the materials; and iii) soil-IOT compatibility analysis for rammed earth production.

### 2.1. Materials collection

The first step consisted of collecting IOT sediment samples in the three municipalities in Minas Gerais that suffered the worst effect from the Fundão dam rupture: Mariana, Barra Longa, and Rio Doce (Figure 1).



**Figure 1.** Location of the municipalities in Minas Gerais that had the greatest destructive effect due to the impact of the Fundão dam. Source: modified by the authors.

In Mariana, the samples (MA sample) were collected on the Gualaxo do Norte river's bank, just below the municipality of Bento Rodrigues, the first to be affected by the mud. The second collection was carried out in the municipality of Barra Longa (BL sample), where the mud arrived through the Gualaxo do Norte River and invaded part of this city. Part of this sediment was dredged and stored at Alta Floresta Farm, located on the river's bank, which became a surplus deposition and management area. The sediment was covered with a layer of soil for revegetation. Because of this, the samples were collected at a depth between 60 and 80 cm. The third collection point was in the Rio Doce (RD sample) municipality, where the mud reached the Carmo River (continuation of the Gualaxo do Norte) and the Risoleta Neves Hydroelectric Plant. The sediments dredged from that place were deposited at Floresta Farm, a surplus deposition and management area (Figure 2).



**Figure 2.** Location of collection points along the Rio Doce. Source: Google Maps (modified).

All three IOT samples were placed in closed containers for transport. Soil samples were collected in the same region of Minas Gerais to analyze soil-IOT mixtures for rammed earth production. Soil samples were taken from areas belonging to deposits of clayey soil, the same soil characteristic of the region where the sediment samples were collected. This soil was chosen because of its proximity to the sediment collection site. The intention is to understand the viability of rammed earth production with this sediment once the soil-IOT geographic proximity is an important aspect of the investigation. Before the analysis, the soil and IOT sediment samples were dried in an oven for 24 hours to remove moisture and quartered for better representation.

## 2.2. Materials characterization

In the second step, the samples of IOT sediment were analyzed by chemical, physical, and mineralogical tests to assess the sediment's characteristics, which is important to set after seven years of permanence in the environment.

The material samples' particle size was analyzed using the laser granulometry test to evaluate the behavior of IOT sediment samples in possible applications in earth components. The measurement range is from 0.04 to 2500  $\mu\text{m}$ , using a laser granulometer, model Cilas 1190 Particle Size Analyzer. The specific surface and porosity analyses were performed using the BET method (adsorption of  $\text{N}_2$  at 77 K), which consists of determining the volume of adsorbed gas from the physical adsorption isotherm, determined experimentally. The surface area was obtained from the nitrogen adsorption curve (BET method), while the distribution and pore size can be obtained from the desorption isotherm. The equipment used was a BET model Autosorb IQ Quantachrome. The unit weight and air-void content tests were also carried out according to the Brazilian technical standard NM 45 [45], the density according to the Brazilian technical standard NM 23 [46] and the water absorption, according to the Brazilian technical standard NM 30 [47].

The identification and quantification of minerals and chemical components in IOT sediment samples were performed by X-ray Fluorescence (XRF) with the WDS Spectrometer, Philips PANalytical, PW-2404, with a 4 kW Rh tube. Tests were carried out to determine the inorganic constituents and evaluate the toxicity in the raw material, in addition to the analysis of the leached and solubilized extracts, in accordance with the recommendations of the Brazilian technical standards 10004, 10005 and 10006 [48-50], respectively.

X-ray Diffraction (XRD) analysis was performed to identify the mineral phases in the sediment samples. The equipment used was a Philips Panalytical diffractometer, system 1710. The conditions used were:  $\text{CuK}\alpha$  radiation ( $\lambda = 1.54$ ), step of  $0.06^\circ/\text{s}$  between scanning ranges  $10^\circ$  to  $90^\circ$  at 2-theta angle ( $\theta$ ). The data obtained in X-ray Diffraction were processed using the Search Match software. In this software, the phases were identified, observing the characteristic diffraction patterns of each mineral and the relative intensity using the PDF-2 database of the International Center for Diffraction Data (ICDD).

The surface morphology of the IOT sediment samples was visualized by scanning electron microscopy (SEM) using the scanning electron microscope used as an FEG with FIB Nanofabrication System - Quanta FEG 3D FEI. Energy Dispersive X-ray spectrometry (EDS) was used to identify the elements present. For this, the samples were fixed on a carbon tape.

Soil samples were characterized using Atterberg limits tests (plasticity index), X-ray Diffraction, X-ray Fluorescence, and granulometry. For the determination of the soil liquidity limit, the parameters of the Brazilian NBR 6459 standard [51] were used, and the determination of the limit and the plasticity index followed the NBR 7180 [52] both with the previous drying of the samples in an oven.

In the particle size test carried out by simple sieving, it was observed that the maximum characteristic dimension was 2.40 mm and a fineness modulus of 2.90 mm. For particles smaller than 0.5 mm, a laser analysis was carried out using the equipment of a particle size analyzer at the Nuclear Technology Development Center at the Federal University of Minas Gerais. The laser granulometry results showed that the soil contains 3.95% clay and silt, with 80.65% sand. According to NBR 17014 [53] the values found are lower than ideal. However, a new granulometric analysis was carried out by sedimentation, following the NBR 7181 [54] through the combination of the sieving and sedimentation methods. First, 1.5 kg of each sample was separated, passed through a 2.0 mm sieve, washed, and dried in an oven at 105 °C. After that, 70 g of the soil sample was taken for the test. The deflocculant used in this test was sodium hexametaphosphate.

### 2.3. Soil-IOT compatibility analysis

The IOT collected in Barra Longa (BL sample) was used to produce mixtures of soil-IOT aiming to produce RE. The BL sample was chosen because of its availability and the ease of collection, once this material is available to be used in any applications. Eight mixes were produced to analyze the mixture of IOT sediment and soil in the mixture for the rammed earth: one mix only with soil (T0-0); three mixes with soil and IOT sediment; and four mixes with cement CP II E (Portland composite cement, like CEM-III/A [55]) and IOT sediment. Cement was used in a fixed proportion of 5% in addition to the total mass.

The reference is related than the recommendations in the literature [56-58] which present the contents of 4% to 10% of cement as the most suitable for the stabilization of rammed earth or soil-cement blocks. Thus, it was possible to analyze mixtures with and without a binder addition. The sediment replaced the soil in groups G1 and G2. The water content varied due to the natural soil and sediment moisture and the ideal moulding consistency (Table 1).

The mixtures were moulded in cylindrical moulds with 10 cm in diameter and 20 cm in height, adapted to increase the height to obtain a greater number of layers, in a proportion of 1:2 (diameter/height). Therefore, the number of layers applied to mould the specimens was five. After preliminary analysis, 10 blows per layer were used for correct compaction, a value adapted from NBR 7182 [59] which presents 12 as the number of blows for the large cylindrical mould (proctor). All layers were scarified before starting the next layer.

The metal socket was adapted from NBR 12024 [60] and NBR 7182 [59] to standardize the compaction energy. The mass of the socket was  $2500 \pm 10$  g in a height control device of drop (guide) of  $305 \pm 2$  mm. After weighing, the mixture was carried out in a 60 L industrial mortar mixer. Water was added gradually, initially starting from an amount referring to the immediately previous moulding (with less sediment content), visually and tactilely analyzing the mixture until it reached the optimum humidity for moulding. After moulding, the specimens were immediately demoulded and placed on shelves in the open air, in a laboratory environment (open shed), under real conditions of local temperature and humidity (outdoors, only protected from rain). The specimens were not taken to chambers or greenhouses to not alter the analysis and to be as close to the curing of rammed earth under real production conditions [61].

**Table 1.** Mixes and IOT content.

Group	Mix	Soil (%)	IOT sediment (%)	Portland Cement (%)
G1	T0-0	100.0	-	
	T10-0	90.0	10.0	
	T20-0	80.0	20.0	-
	T40-0	60.0	40.0	
G2	T0-5	100.0	-	
	T10-5	90.0	10.0	
	T20-5	80.0	20.0	5.0
	T40-5	60.0	40.0	

\* PC - Portland Cement.

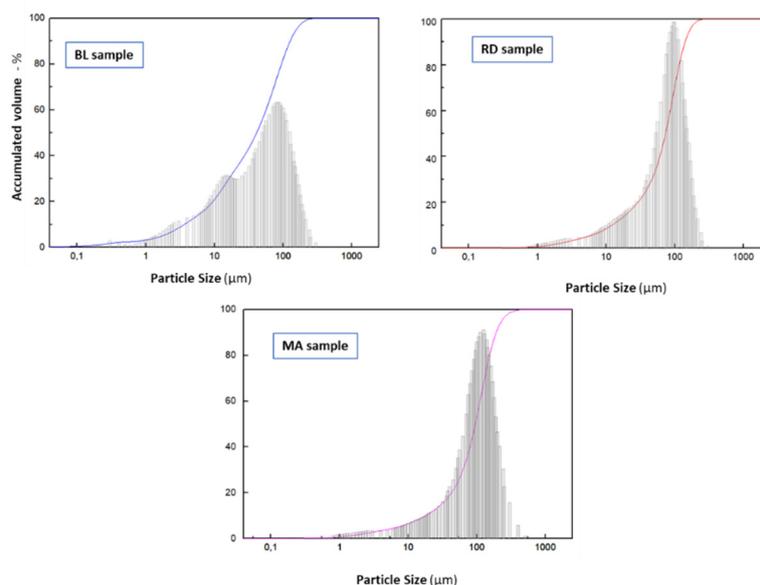
Six specimens were moulded for each mixture, and the curing period was 28 days. At 21 days, the specimens had their mass measured and were capped with a homogeneous mixture of cement and water to obtain smooth and uniform tops for the compressive strength test, according to NBR 12025 [62]. It was carried out with a semi-automatic hydraulic press, with a loading speed of up to 1,00 KN/min. The analysis of results was based on the Brazilian technical standard NBR 7215 [63]. The average of the individual strength values of the specimens was calculated in MPa. After finding an average, it was necessary to calculate the maximum relative deviation (MRD). When it is greater than 6%, it is necessary to calculate a new average, disregarding the outlier and persisting the calculated value until reaching the MRD value  $\leq 6\%$  [63].

### 3. Results

#### 3.1. IOT samples characterization and analysis

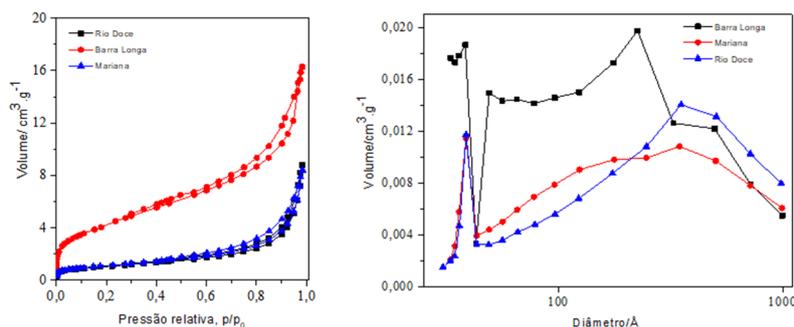
In this item, the results of the sample characterization test. Figure 3 shows the granulometric curves of the IOT sediment samples from Mariana (MA sample), Rio Doce (RD Sample), and Barra Longa (BL Sample).

Through the curves obtained, it is possible to observe that both samples have sandy characteristics with an insignificant portion of clay. According to Figueiredo et al. [28], this fact is associated with the composition of the tailings from the Fundão Dam, which was a mixture of sandy tailings and mud. The diameters varied between 1 and 200  $\mu\text{m}$ , with greater predominance in a range close to 15  $\mu\text{m}$  and 100  $\mu\text{m}$ . Such values are consistent with those obtained in other works of characterization of the tailings from the rupture of the Fundão dam carried out by other authors [24, 25, 28].



**Figure 3.** Granulometric curves of sedimented tailings samples.

Figure 4 shows the isotherm curves of the sedimented tailings samples, obtained by the analysis of nitrogen adsorption and desorption. According to other works found in the literature, the isotherms obtained have a type II profile. According to IUPAC (International Union of Pure and Applied Chemistry), these curves are characteristics of non-porous or macroporous materials [64].



**Figure 4.** N<sub>2</sub> sorption isotherms for the sedimented tailings samples.

It is possible to see the presence of narrow and inclined hysteresis, classified as H3 type, indicative of non-rigid aggregates of lamellar particles, mainly mesopores/macropores in the form of slits or parallel plates [65, 66]. Table 2 presents the specific surface area and porosimetry and it is possible to observe all the samples are similar regarding the pore volume and diameter values. As expected for this type of sample, the specific surface area values were low and similar. Such results are compatible with other studies carried out with ore tailings. Almeida et al. [25] found specific surface area (SSA), and silt and clay size values, of 5.25, 5.66 and 20.77 m<sup>2</sup> g<sup>-1</sup>, respectively.

Table 3 shows the results obtained in the specific mass, unit mass, void index and water absorption tests. Comparing the specific mass values found with the other results [67, 68] it is possible to attest that they are compatible with silica-rich tailings with lower concentrations of iron due to the density closest to sand, which is 2.65 g/cm<sup>3</sup>. These results are consistent with those found in the mineralogical analysis, which showed quartz as the most present mineral in the samples, directly interfering with the specific mass results. The values obtained for the void ratio were similar. Relating to the characteristic granulometry of iron ore tailings, which presents a high concentration of particles of the same size, representing a uniform granulometric curve, a relatively high void volume index might be expected.

**Table 2.** Specific surface area and porosimetry of sedimented tailings samples.

Samples	Specific surface area (m <sup>2</sup> /g)	Pore volume (cm <sup>3</sup> /g)	Pore diameter (mm)
MA Sample	3.60	0.013	3.864
RD Sample	3.26	0.013	3.873
BL Sample	7.28	0.020	3.300

The values obtained in the water absorption test showed that the tailings from Mariana (MA sample) and Rio Doce (RD sample) have a low rate of water absorption, which is expected for tailings composed mainly of silica. The Barra Longa tailings (BL sample) showed a higher water absorption rate than the other samples, which may be related to clay minerals in the sample. Additionally, the BL sample showed a lower value of specific mass, which attests to the higher presence of clay minerals in the sample. According to Taiz et al. [69], clayey soils retain a higher water content than sandy soils due to the larger surface area and smaller pores between particles. This statement can be confirmed by the specific surface area results obtained and shown in Table 2.

**Table 3.** Specific mass, unit mass, void ratio, and water absorption of sedimented tailings samples.

Samples	Physical tests			
	Specific mass (g/cm <sup>3</sup> )	Unit mass (kg/m <sup>3</sup> )	Ev (%)	Water absorption (%)
MA Sample	2.93	1526.50	66.9	2.97
RD Sample	2.82	1498.70	64.5	3.33
BL Sample	2.79	1368.47	64.2	7.52

Table 4 presents the results obtained from the XRF analyses. The oxides found in the samples match the mineralogical phases obtained by XRD. Furthermore, among the oxides found in the sedimentary tailings samples, the highest concentrations are quartz (SiO<sub>2</sub>) and hematite (α-Fe<sub>2</sub>O<sub>3</sub>). According to other authors [29, 70], quartz and hematite are the main mineral components of tailings from the Fundão dam. Traces of manganese oxide, zinc, chromium, and sodium were also found.

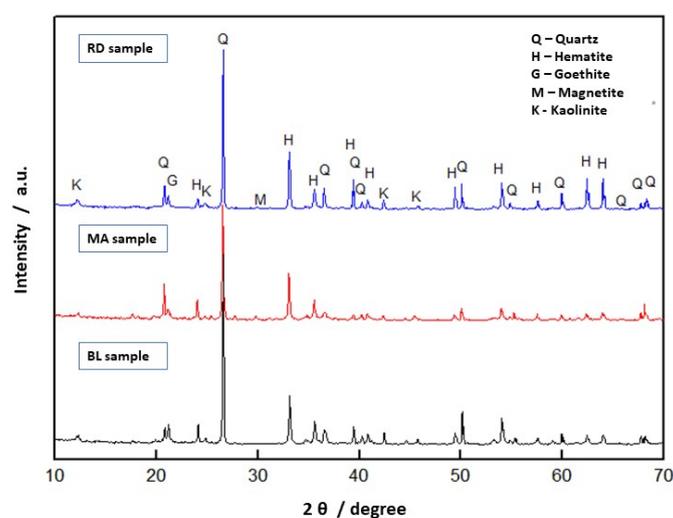
**Table 4.** Chemical analysis of samples by X-ray Fluorescence.

Samples	Composition (%)						
	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	K <sub>2</sub> O	MnO	CaO
MA Sample	78.13	8.88	1.09	0.21	0.20	0.28	0.04
RD Sample	76.85	8.46	2.49	0.27	0.34	0.06	0.24
BL Sample	67.05	9.57	6.39	0.67	0.62	0.07	0.21

Due to the possibility of contamination of these samples in the environment, since the rivers Gualaxo do Norte, Carmo, and Doce have been used, over the decades, for gold mining during the colonial period in Brazil, assays for the determination of inorganic constituents in the raw material, in addition to analysis of the leached and solubilized extracts are shown to be extremely important. The possibility of contamination was raised at the time of the accident [8, 24, 71] but after five years, the pollutant load of these rivers may have been concentrated or dispersed, which may have impacted on the composition of these sediments.

Through the data obtained by the tests of determination of heavy metals in raw material and obtaining of leached and solubilized extracts, it was observed that the leaching and solubilization tests of metals (inorganic) presented results with values below the maximum limits prescribed by NBR 10004 standard [48]. The leaching and solubilization tests of volatile and semi-volatile organics showed null results, i.e., below the detection limits of the technique adopted and below the maximum limits prescribed by NBR 10004 [48]. Because of that, the data indicates that all the collected sediment tailings samples can be classified as Class II B - Non-Hazardous Inert [48].

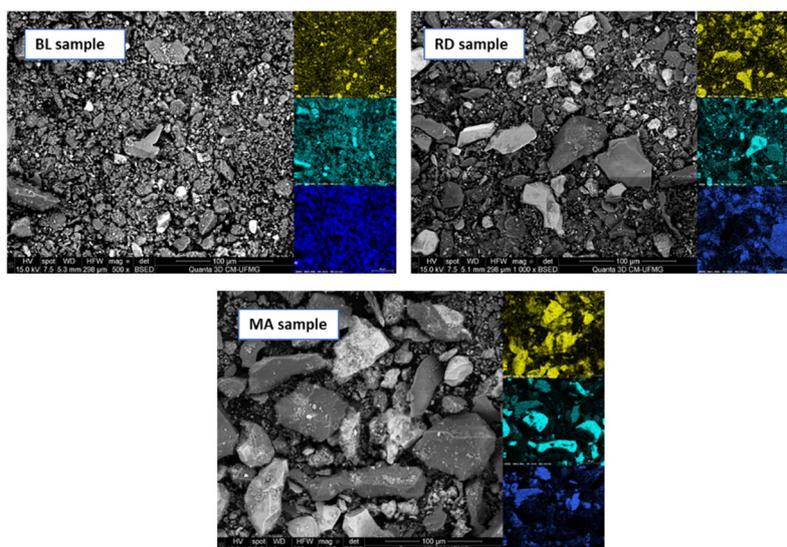
The X-ray diffractograms obtained from the sedimented tailings samples can be seen in Figure 5. It was possible to observe the great similarity between the diffractograms of the analysed samples, with a predominant presence of Quartz phases, SiO<sub>2</sub> (ICDD - 46-1045); Hematite,  $\alpha$  - Fe<sub>2</sub>O<sub>3</sub> (ICDD - 33-664); Goethite,  $\alpha$  - FeO(OH) (ICDD - 74-2195); and Kaolinite, Al<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub> (ICDD - 80-885). Although the intensity of the peaks of each mineral does not represent the amount, it can indicate which mineral is more present in the sample.



**Figure 5.** X-ray diffractograms of the sedimented tailings samples.

This result is consistent with those found in the literature [25, 29, 67, 72-74] confirming the results obtained in the X-ray Fluorescence analysis. XRD analysis also allows to evaluate the amorphicity of the samples, which would make this material suitable for use as a pozzolanic mineral addition, which would increase the durability of cementitious matrices [75] or as a precursor in alkali-activated materials [19, 22, 76]. A crystalline material, as shown in the sedimented tailings samples, however, may have its reactivity improved through grinding and calcination [77-79] depending on its use.

The images obtained through scanning electron microscopy (SEM) analysis may be seen in Figure 6. Through the images, it was possible to observe that the particle size is compatible with the results obtained by laser granulometry, with a maximum diameter close to 200  $\mu$ m. These results agree with other researchers [24-25] who found these same characteristics for sedimented tailings from the Fundão dam failure.



**Figure 6.** Scanning electron microscopy images of sedimented tailings samples.

It is also possible to identify the presence of prismatic particles with sharp edges, presenting edges and vertices, and the presence of tabular and granular particles. According to Shettima et al. [12] and Zhao et al. [80], the particles of the first type may be associated with quartz particles. Particles of the second type may indicate the presence of hematite particles [72, 75]. According to Dedavid et al. [81], the contrast of the images is related to the atomic number of the elements present in the sample. Thus, through the images obtained, it can be affirmed that quartz particles have larger granulometry when compared to ferrous mineral particles.

In the picture generated for the samples from Rio Doce and Mariana, it is possible to observe particles with rough and angular surfaces, which is expected for a material obtained during mineral processing, with comminution steps [28]. In BL sample, it was possible to perceive a smaller number of particles of ferrous minerals represented by the light gray color. This can also be seen with the help of EDS images. It also shows predominantly dark-colored particles characteristic of quartz.

In general, the sedimented tailings samples were similar. Traditionally, however, Brazilian IOT, collected in dams, still has a high amount of iron [72, 75] due to the still inefficient extraction processes practiced 30-40 years ago.

Through the characterization, it can be observed that this material, when exposed to the weather, underwent significant changes over time. Even so, it is a material stored for future use, as its permanence in the places where it is found changes the landscapes, the soil, and the waters, as reported by Brazilian researchers in post-accident publications [8, 29, 30, 70, 82].

### 3.2. Soil-IOT compatibility to produce RE

Table 5 shows the soil samples characteristics. It was found that the clay contents and sand were quite different, with soil with a high clay content and fine particles. In this way, the need to correct the soil with the IOT-S became even more evident.

As it was carried out before the launch of the Brazilian rammed earth standard [53] the results of the physical characterization of the soil were based on the parameters of the soil-cement standards and adobe, as well as recommendations from scientific literature, which determine values between 35% and 45% for the liquidity limit and between 7% and 30% for the plasticity index [83]. In this sense, the results demonstrate that the soil analysis could benefit from stabilization since its value for the liquidity limit exceeds the recommendation.

**Table 5.** Soil characterization.

Specific mass (kg/m <sup>3</sup> )	2340	Liquidity Limit (%)	53
Unit mass (kg/m <sup>3</sup> )	1012	Plasticity Limit (%)	31
Volume of voids (%)	57.50	Plasticity Index (%)	22
Granulometry (%)			
Clay	55.5		
Sand	28.0		
Average diameter (µm)	26.4		

The results of the compressive strength test are presented in Table 6. From the values obtained, it is possible to infer that the addition of 5% cement to the soil and the stabilization with IOT proved to be adequate in comparison to the reference values (T0-0), which do not have the addition of any component. For the mixtures without the addition of cement, only with IOT, it was observed that the compressive strength values increase as there is an increase in the replacement of soil by sedimented tailing, in proportions of 10%, 20%, and 40%.

**Table 6.** Compressive strength – Soil-IOT samples.

Mixture	Compressive strength (MPa)	Standard deviation	Coefficient of variation (%)
T0-0	0.72	0.08	11.55
T10-0	1.12	0.13	11.90
T20-0	1.24	0.05	4.07
T40-0	1.80	0.15	8.53
T0-5	1.35	0.14	10.63
T10-5	1.07	0.11	9.91
T20-5	1.27	0.15	12.20
T40-5	1.57	0.08	4.98

Regarding the compressive strength values found in the literature, it is important to say that the reference trace (T0-0), only with unstabilized soil, is outside the parameters of what is considered satisfactory for rammed earth, while the values of samples stabilized with IOT are within the recommended range. The adopted values range from 1.0 MPa to 2.0 MPa [61, 84] for the compressive strength of rammed earth and earth constructions. In other research [85] the results were between 1.0 and 2.5 MPa, consistent with the present study.

With the addition of cement, Eusébio [86] used a content of 7% of the stabilizing material and obtained compressive strength greater than 2.0 MPa. Jayasinghe & Kamaladasa [87] analyzed the compressive strength of rammed earth walls stabilized with cement contents in the range of 6.8 and 10% and observed that the stabilizing action of cement is more effective for sandy soils. However, they still obtained high resistance values for clayey soils.

It is important to point out that the highest values were for the mix with the highest amount of IOT (T40-0), which reached 1.80 MPa, followed by the mix with IOT and cement (T40-5). Because of that, there is a tendency to adequately stabilize with only IOT, without the addition of cement, which has been more effective, especially at higher levels of IOT addition (40%).

Finally, the Peruvian technical standard E.080 [88] and the NZS 4297 standard [89] present values of 1.0 MPa and 0.50 MPa, respectively, for compressive strength. In other words, the results found in this research are superior to the recommendations, including the Brazilian standard [53] even though this study was developed before its launch.

The study showed a positive interaction between the soil, collected at MRBH, and the IOT collected in the Mariana region. This is probably due to the rocky origin common to both materials. The advantages can be seen, especially concerning the compressive strength of the rammed earth stabilized with IOT, without cement, compared to the rammed earth cylindrical specimens without any stabilization.

#### 4. Discussion

Characterizing iron mining waste disposed in the environment is of fundamental importance for its correct use and recovery. The analysis carried out with samples of sedimented tailings from the three cities most impacted by the accident made it possible to see that the IOT is a crystalline material with a high silica content. The quartz content and specific surface area/grain size vary depending on the collection site. The compressive strength initial results indicate the possibility of using this material as a physical stabilizer to produce earthen components, as RE, without cement or lime addition, which may contribute to a less environmental impact. Furthermore, since the samples collected were classified as non-hazardous materials, IOT can be used by communities to reconstruct their buildings. Using IOT as an inert material to produce traditional earthen constructions can be aligned with social sustainability and innovation of the earth's construction techniques.

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