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CHARACTERIZATION OF TAILINGS FROM THE PROCESSING OF ORNAMENTAL ROCKS

CARACTERIZAÇÃO DOS REJEITOS DO BENEFICIAMENTO DE ROCHAS ORNAMENTAIS

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ABSTRACT

The processing of ornamental rocks promotes the constant generation of abrasive sludge, which, in turn, causes different types of contamination. The possibility of reusing residues generated in industrial processes has economic advantages, therefore, it is necessary to know the physical, chemical and mineral properties of these residues. Thus, the objective of this research was to evaluate the possibilities of application of abrasive mud, generated in industries located in the city of Campina Grande - PB, based on its main characteristics. The methodology consisted of the following analyzes: X-ray fluorescence, X-ray diffraction, scanning electron microscopy, particle size analysis and physical-chemical analysis. Chemical analyzes revealed that the residues have interesting characteristics, mainly for use in civil construction. The mineralogical analysis indicated that the sample has an essential mineral composition for the cosmetic industry, which was confirmed by the detected elements present. The study of the particle size characterized the sample as material of irregular shape and with good micronization, confirming the potential for reuse as a cosmetic, while the morphological analysis showed the presence of iron as a limiting factor for certain applications. Physico-chemical tests confirmed the characteristics common to abrasive sludge. The characterized samples showed, in general, satisfactory properties with regard to its use as an alternative material in different segments.

RESUMO

O processamento de rochas ornamentais promove a geração constante de lama abrasiva que, por sua vez, ocasiona diferentes tipos de contaminação. A possibilidade de reutilização dos resíduos gerados em processos industriais apresenta vantagens econômicas, portanto, é necessário conhecer as propriedades físicas, químicas e minerais desses resíduos. Sendo assim, o objetivo dessa pesquisa foi avaliar as possibilidades de aplicação da lama abrasiva, geradas em indústrias localizadas na cidade de Campina Grande – PB, tomando como base suas características principais. A metodologia consistiu nas seguintes análises: fluorescência de raios X, difração de raios X, microscopia eletrônica de varredura, análise de tamanho de partículas e análise físico-química. Análises químicas revelaram que os resíduos possuem características interessantes, principalmente para uso na construção civil. A análise mineralógica indicou que a amostra possui uma composição mineral essencial para a indústria cosmética, o que foi confirmado pelos elementos detectados presentes. O estudo do tamanho das partículas caracterizou a amostra como material de forma irregular e com boa micronização, confirmando o potencial de reutilização como cosmético, enquanto a análise morfológica mostrou a presença de ferro como fator limitante para determinadas aplicações. Testes físico-químicos confirmaram as características comuns à lama abrasiva. As amostras caracterizadas apresentaram, de forma geral, propriedades satisfatórias no que diz respeito a sua utilização como material alternativo em diferentes segmentos.

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INTRODUCTION

Brazil is one of the largest producers and exporters in the world in the ornamental rock processing sector. The ornamental rock industry generates significant economic and social development in the country with the generation of thousands of direct and indirect jobs. On the other hand, it also generates huge amounts of polluting solid residues that are usually improperly deposited, putting the process at odds with sustainable principles and causing undesirable economic and environmental consequences (Manhães, Moreira & Holanda, 2009; Menezes, Ferreira, Neves & Ferreira, 2002; Torres, Fernandes, Agathopoulos, Tulyaganov & Ferreira, 2004).

The beneficiation of ornamental stones refers to the unfolding of raw materials, extracted in quarries in the form of blocks or, in some cases (quartzites and slates), as slabs. Blocks, with dimensions usually varying from $5m^3$ to $10m^3$, are mainly benefited by sawing (cutting process) in sheets, by looms and block cutters, for later finishing and square to their final dimension. The most common looms use abrasive sludge, whose main objectives are: lubricate and cool the blades, prevent the oxidation of the plates, serve as a vehicle to the abrasive (shot) and clean the channels between the plates, being distributed by showers on the block through pumping (Torres, Fernandes, Agathopoulos, Tulyaganov & Ferreira, 2004).

The tailings are basically fines (dust) produced during rock cutting, and may have other contaminants such as iron and lime. The effluent generated in conventional looms is the so-called high iron content abrasive sludge due to the use of iron or steel grit used in sawing granite blocks (Savazzini dos Reis & Engel de Alvarez, 2006; Silveira, Wilson, Vidal & Sênior, 2014). These residues can reach rivers, lakes, streams and even natural water reservoirs, as they are released into the ecosystem without any previous treatment. In addition, environmental waste in the form of mud aesthetically affects the landscape and requires large storage space, besides a high cost of collection and storage (Simsek, Karaca, Gemici & Gunduz, 2005). These residues when dried become a fine, non-biodegradable powder that causes damage to human health (Rego, Martínez, Quero, Blanco & Borque, 2001).

To help solve such problems, recycling and reuse operations are useful tools and are considered priority operations in solid waste management, as in most cases the reduction of generation is technically or economically unfeasible (Filho, Polivanov & Mothé, 2005; Torres, Fernandes, Olhero & Ferreira, 2009). Using these materials as raw materials in other production processes can turn waste into useful by-products, reducing the large amounts that are landfilled and minimizing environmental impacts (Manhães & De Holanda, 2008; Torres, Fernandes, Olhero & Ferreira, 2009).

The need to comply with the management and disposal requirements of solid waste generated in industrial activities has been imposed in the last two decades, either by environmental laws or ecological movements around the world, becoming a major challenge for production systems (Da Silva, 2014). The efficient management of the abrasive sludge waste from its generation to its final disposal, i.e., "cradle to grave", is fundamental. Even more important is the reduction of its generation. This is a crucial point in waste management, because when it comes to this issue, the first thing we hear is recycling, but we have to keep in mind that this is the third link in the chain. First, we have to reduce the waste generation, then we think about reuse and only if none of the previous options is feasible, we start recycling (Manhães & De Holanda, 2008).

The literature points to some recent studies, such as that of Allam, Bakhoum and Garas (2014), which discussed the compressive strength of green concrete mixtures produced by replacing sand and cement with abrasive mud. Ribeiro and Holanda (2014), on the other hand, obtained satisfactory results when studying the reuse of mud as an alternative raw material in a soil-cement brick mass, replacing the soil by up to 30% by weight. In 2015, Al Zboon and Al-Zou'by observed an increase in the mechanical properties of concrete when using abrasive mud to replace drinking water.

The characterization phase of the generated or accumulated residues is fundamental in the decision on the best solutions to be adopted regarding the treatment, disposal or reuse, depending on the characteristics of the material. It is essential to note that these residues can be used as integrating or substitute materials, reducing the pressure on natural resources. The residues from the ornamental rocks beneficiation process, for example, face problems with regard to an environmentally correct final destination due to the lack of characterization data

In view of the above, due to the growing and more effective inspection by the environmental agencies, it is essential to study, characterize and reuse these residues, called abrasive mud, resulting from the unfolding of ornamental rocks. The present work aims to evaluate the characteristics of the residues to later discuss the potential of reuse of them in the most different industries, in order to provide relevant data in the search for alternatives of reuse in the most diverse segments.

METHODOLOGY

To carry out the collection of samples of ornamental rock waste of abrasive mud, technical visits were made in granite splitting industries located in the city of Campina Grande – Paraíba, the geological context of the collection site is shown in Figure 1, where it is observed that the city in question is located in a predominantly granitoid range. The abrasive sludge that forms when the pulp blocks unfold after passing through the company's wastewater treatment plant filter press was exposed to air at room temperature for natural drying for 7 days. Waste samples were collected according to ABNT NBR 10007. Once collected, the solid residues were sprayed using mortar and pistil, and then sieved with a mesh of 0.074mm for 2 minutes, a method used to establish a standardization between the samples.

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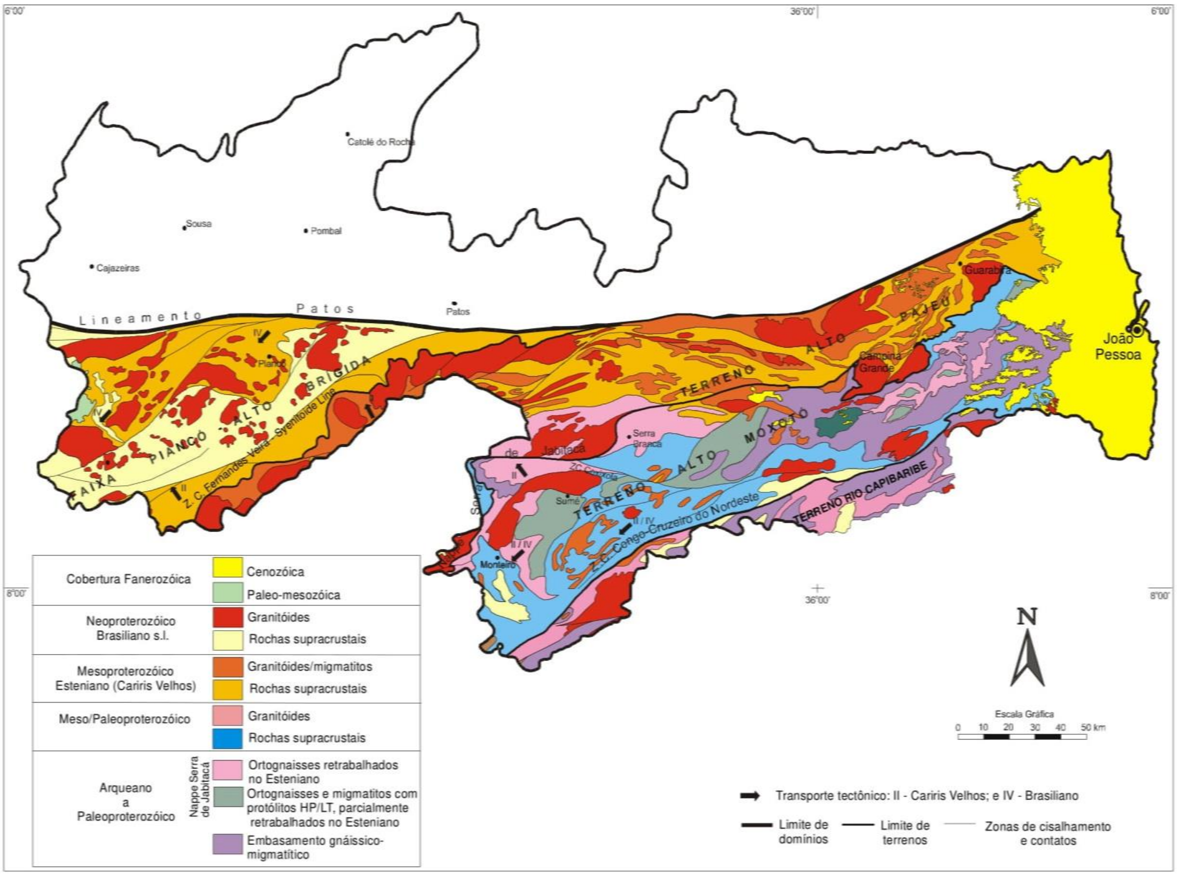
Figure 1. Geological map of the Transversal Domain of the State of Paraíba.



Source: Adapted from CPRM (2002).

Chemical analysis

The chemical analysis of the abrasive mud sample was carried out by means of X-ray fluorescence in SHIMADZU EDX-720 equipment to determine the elements present through the application of X-rays on the sample surface and the subsequent analysis of the emitted fluorescents, the generation of rays X is made through a tube with Rh target. An EDAX micro probe was used for X-ray spectrometric analysis with a lightweight element detector.

Scanning electron microscopy (SEM)

The analysis of the abrasive mud sample morphology was observed via scanning electron microscopy using a SHIMADZU SSX-550 scanning electron microscope, operating at 15kV, after covering the particles with a thin layer of gold.

Granulometric evaluation

For the granulometric analysis, it was necessary to carry out a different sample adequacy methodology the CEMP 109 standard was used to homogenize and pulverize the entire composition. The granulometric evaluation was carried out through wet screening using 8 screens with the following openings: 44µm, 53µm, 74µm, 105µm, 149µm, 177µm and 297µm.

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pH determination

To measure the pH of standardized samples, the solubilization method according to ABNT NBR 10006 was used. In this analysis, 20g of the sample, previously dried, were weighed and added to 100mL of distilled water, with the aid of a mechanical stirrer the sample was homogenized, in low rotation, for 5 minutes. The solution was then covered and left to stand for seven days at room temperature. As time passed, it was filtered using a vacuum filter, with the collection of the solubilized extract the pH was then measured using a conventional pH meter, previously calibrated.

Moisture content

The moisture content was determined using the sample of abrasive mud in its state of collection and in accordance with the requirements of the CEMP 105 standard. Weighed 10g of the sample, which was subsequently taken to the oven at a temperature of 120°C, for twenty-four (24) hours. After the time, the sample was transferred to the desiccator, where it was left to rest for 30 minutes. Soon after, the weighing was carried out and the moisture content was calculated using Equation (1).

$$\%U = \frac{Mu - Ms}{Ms} \; x \; 100 \qquad (1)$$

where U (%) is the moisture content, Mu (g) is the wet mass and Ms (g) is the dry mass.

Bulk density

For this analysis, about 25mL of the standardized sample was carefully added to a graduated cylinder, beating the cylinder three times against a hard, flat surface, at 2 second intervals between strokes. Soon afterwards, the volume obtained was read, thus, the filled beaker was weighed and the value obtained was subtracted from the weight of the beaker, obtaining the result corresponding to the sample mass. With the sample mass in grams (g) and the apparent volume in milliliters (mL), calculations were performed using Equation (2).

$$dap = \frac{m}{Vap} \qquad (2)$$

where dap (g/mL) is the bulk density, m(g) is the sample mass and Vap (mL) is the apparent volume.

Absolute density

Adapting Silva *et al*. (2009), an empty pycnometer was weighed, filling it with distilled water until it overflowed, drying the water present on the external surface of the pycnometer, and a second weighing was performed with the aid of a semi-analytical balance. After removal and drying of the water, the standardized sample was added, filling the maximum allowed, and a third weighing was performed. With the values of the three weighing, calculations were made using Equation (3).

$$D = \frac{m2 - m1}{m3 - m1} \qquad (3)$$

where D is the absolute density, m1 (g) is the empty pycnometer mass, m2 (g) is the pycnometer mass with the sample and m3 (g) is the pycnometer mass with water.

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RESULTS AND DISCUSSIONS

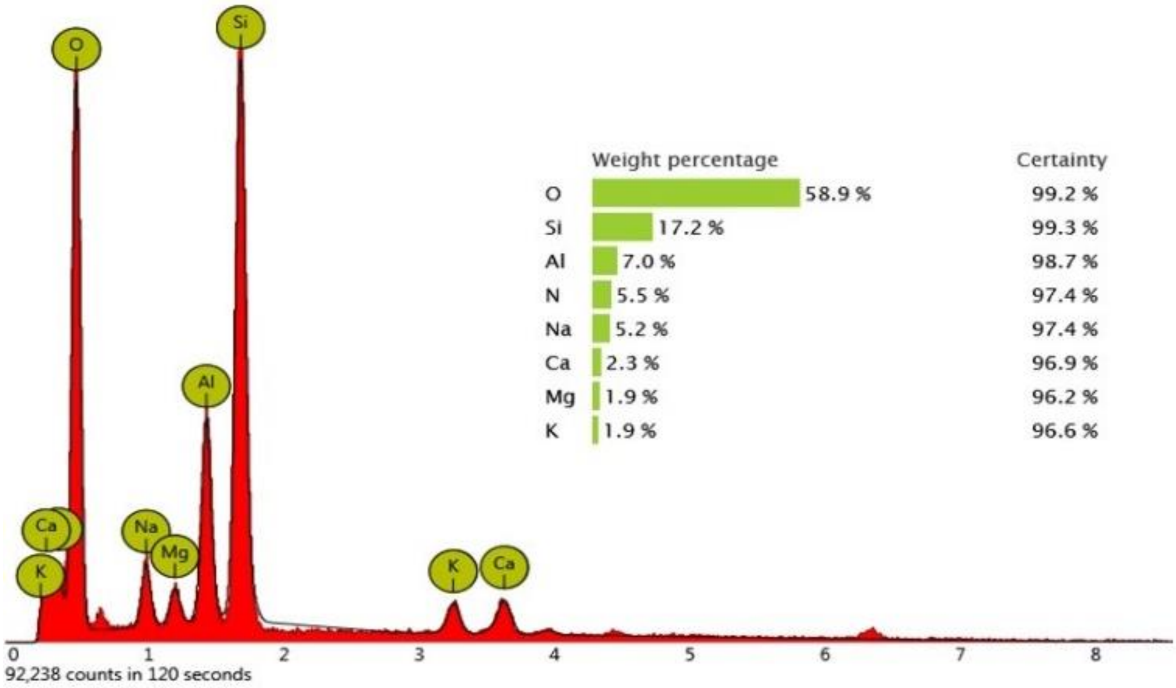
Chemical analysis

Figure 2 shows the result of the chemical analysis carried out by a micro probe with the use of X-rays and detection of light elements. The major presence of the elements silicon (Si) and oxygen (O) is observed, presenting, respectively, 58.9% and 17.2% of the total composition. These values are in accordance with the results presented in Table 1, making it possible to classify the sample as a high silica material.

The analysis also found the significant presence of the elements: Al (7.0%), N (5.5%) and Na (5.2%). Other elements appeared in a lower concentration, such as Ca (2.3%), Mg (1.9%) and K (1.9%). All these elements were found, in the form of oxides (shown in Table 1), in greater or lesser proportions, with the exception of nitrogen (N).

Bearing in mind that in the cosmetic area the application of clays is directly related to its chemical and mineralogical composition, it can be said that the ornamental rock residue meets, in a preliminary way, the needs of this industry. Clays with Fe, Si, Al, Ca, Ti and K percentage can be used for bactericidal, regenerative and antiseptic action contributing to cell renewal, adsorption of impurities, invigorating tissues and activation of blood circulation (Carretero & Pozo, 2010). Works such as de Oliveira, Queiroz and Ribeiro (2009) initially characterized the residues aiming at this application and managed to replace up to 40% by mass in the production of soaps.

Figure 2. Graph of the chemical analysis of the sample of abrasive mud carried out by a micro probe and percentage of the elements present.



The data presented in Table 1 show the main chemical components found in the abrasive sludge sample.

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Table 1. X-ray fluorescence chemical composition of the abrasive mud sample in percentage of oxides.

Analyte	Composition (%)
SiO_2	63.0
Al_2O_3	17.0
Fe_2O_3	6.1
K_2O	6.0
CaO	3.1
Na_2O	2.6
MgO	0.8
TiO_2	0.7
P_2O_5	0.4
MnO	0.01
ZrO_2	0.063
SrO	0.038
Rb_2O	0.014
ZnO	0.011
Y_2O_3	0.004
SUM	**99.84**

It can be seen that the residue consists mainly of silicon dioxide (SiO_2) and aluminum oxide (Al_2O_3), totaling about 80% of the analytes present in the sample. The origin of the silica can be related to several minerals, such as feldspars, micaceous minerals (biotite and muscovite mica) and free silica present in silicate rocks. While aluminum is mainly due to garnet, microline and albite (Anthony, Bideaux, Bladh & Nichols, 2003).

Samples with similar values of silica and alumina were successfully applied, according to Allam, Bakhoum and Garas (2014) in the production of green concrete, replacing 10% of the sand in the composition. This is an important aspect with regard to the reuse of the studied material, since glass wool has characteristics that meet the civil construction, industrial, automotive markets, among others (Alves, Espinosa & Tenório, 2009).

The residues that present about 80.980 - 95.316% of SiO_2 + Al_2O_3 + Fe_2O_3 have great potential to be used in the construction of mortars for civil construction. Other important qualities of this material include the non-propagation of flames, the non-emission of toxic fumes and high resistance to fire (Ding, Jalali & Niederegger, 2010; Marabini, Plescia, Maccari, Burragato & Pelino, 1998; Ueda, Murase, Takahashi & Matsumoto, 1999).

The still expressive values of the oxides of iron, potassium and calcium come from the processes of levigation (step necessary to make the plate less thick) and polishing. The presence of oxides in the residues is interesting, as it increases the potential of these residues for use as an alternative raw material in the formulation of ceramic masses for the manufacture of materials such as glazed floors and various porcelains (Carty & Senapati, 1998; Souza, Pinheiro & Holanda, 2010).

Scanning electron microscopy (SEM)

Figure 3 shows the morphology of the sample obtained via scanning electron microscopy. The sample of the ornamental rock residue showed typically irregular particles which is the type of shape specific to the process of cutting and processing ornamental rocks. The micrograph

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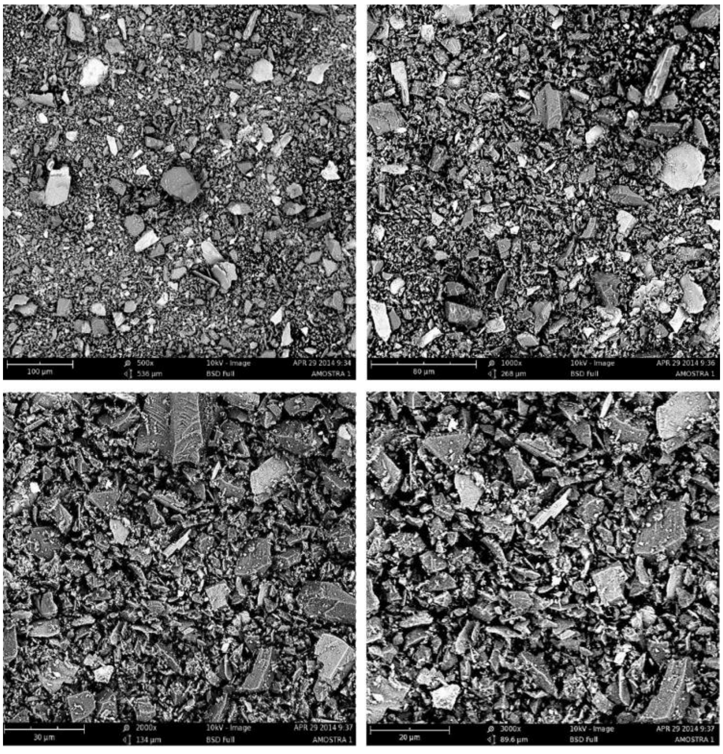
also points to a wide distribution of particle sizes for the abrasive mud sample, which corroborates with the granulometric data (Manhães & De Holanda, 2008).

The residue is basically presented in three distinct phases: white, light gray and dark gray, as shown in Figure 3. The white phase is characterized by the presence of the element iron (93%) and other elements Si (5%), Ca and Al in lesser quantity, this phase constitutes the metallic fraction of the residue and consists of particles of irregular morphology and rounded surfaces. The light gray phase is characterized as being made up of other components of the abrasive mixture (lime and residue from the rock itself, Si (40%) have regular morphology and rounded corners. The dark gray phase is characterized by the presence of elements from the granitic rock itself, with the same morphology as the previous phase (Da Silva, 2014).

The significant presence of iron largely explained by the iron shot used in the cutting process, although beneficial for some applications, as previously discussed, presents as a limiting factor for other typical applications of these waste, such as: mortars, asphalt concrete, ceramic blocks and coverings, red ceramics and tiles. The iron present in the mud is harmful when it is in metallic form and it oxidizes, which can cause an expansion within the material and consequently the appearance of cracks in the finished parts (Costa Borges, Gadioli, Pinto & Oliveira, 2012; Da Silva, 2014; Menezes, Ferreira, Neves, & Ferreira, 2002; Vieira & Monteiro, 2009).

Magnetic separation is an option for removing iron depending on the proposed application, since according to Zichella *et al*. (2020), magnetic separation can also attract part of paramagnetic minerals, such as biotites. They were able to observe in the magnetic fraction studied by them the presence of minerals such as quartz and feldspar.

Figure 3. SEM micrographs of the abrasive mud sample obtained by scanning electron microscopy.



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Granulometric evaluation

The results of the granulometric evaluation by wet sieving the sample of abrasive mud, shown in Table 2 and Figure 4, show a performance close to the micronization of the particles, since the sample presented a percentage of bypass slightly higher than 62% for the 44µm aperture which characterizes a significantly fine particle size.

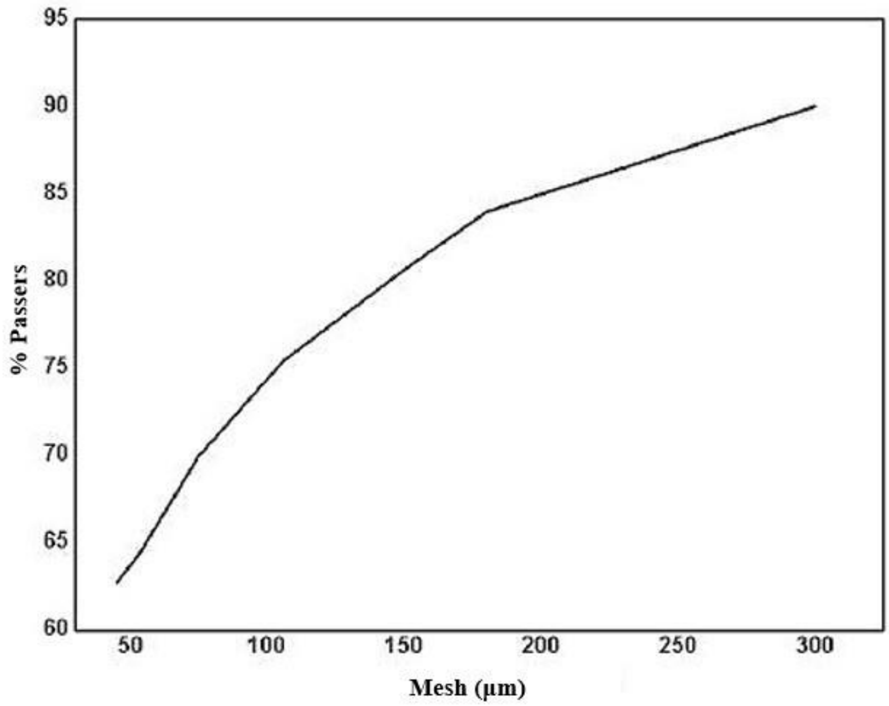
A high percentage of silica and aluminum explains the healing, anti-inflammatory, antipyretic and absorbent properties found in several clays, as well as particles with a particle size less than 0.063mm can have an effect anti-inflammatory and can assist in skin hydration, retaining moisture due to the high adherence to the skin (Dário *et al*., 2014; López-Galindo & Viseras, 2004). Therefore, the particle size range, combined with the high percentage of these elements present in the sample, increases the possibility use of abrasive mud in the cosmetic industry.

Table 2. Results of particle size analysis that identifies the percentage of retained and passers of the abrasive mud sample.

Opening (µm)	297	177	149	105	74	53	44
Retained (%)	9.97	16.01	19.37	24.52	10.01	35.61	37.29
Passers (%)	90.03	83.99	80.63	75.48	69.99	64.39	62.17

In Figure 3, observed the granulometric distribution curve of the percentage of passers in the abrasive sample sieves. The curve indicates that the waste be well graded material, there is also a loss of uniformity in the curve, due to the variable granulometric distribution of sample.

Figure 4. Percentage curve of passers by opening (µm) obtained by wet granulometric evaluation of the abrasive mud sample.



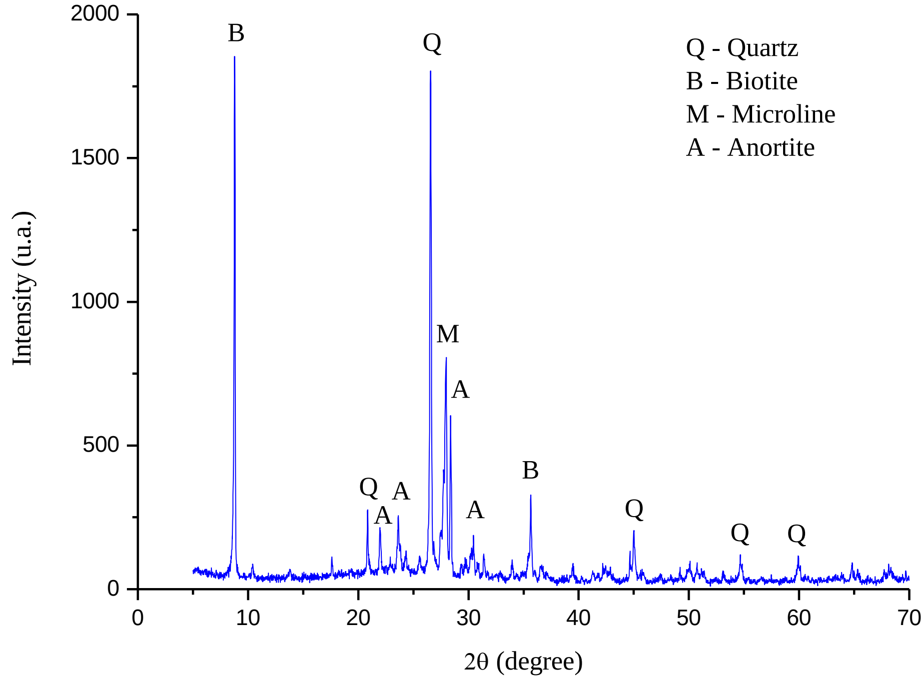
The result of the granulometric analysis expressed in Table 2 and corroborated in Figure 3 indicates, according to, that the particle size distribution of the residues generated in the sawing process of the blocks on looms is partially homogeneous. It is necessary to draw attention to the fact that the samples analyzed are of different types of rocks. One of the positive points regarding the incorporation of ornamental rock residues from a multi-wire loom in ceramic mass is its fine granulometry (Pizetta & Gadioli, 2014). As studied by Ribeiro and Holanda (2014), samples that presented a similar granulometric curve were characterized and subsequently successfully replaced in the production of soil-cement bricks.

X-ray diffraction (XRD)

Figure 5 presents the result of the mineralogical analysis with identification of the phases of the sample of the abrasive mud, which confirms the basic constitution of the following minerals: quartz, anortite (calcium feldspar) and biotite (mica), with a low intensity peak of microline ($K(AlSi_3O_8)$). The quartz peaks (SiO_2) indicates that its presence in the sample is due to the sawdust of the stone block, resulting in a mineral residue in the abrasive mud, known as stone powder.

Biotite ($K(Mg,Fe)_3(OH,F)_2(Al,Fe)Si_3O_{10}$), which presents another expressive peak, with greater intensity, is a common mineral of the silicate class, group of micas (present in the granite formation), while anorthite ($CaAl_2Si_2O_8$) compose one of the mineral groups abundant in the earth's crust, as well as quartz, also appeared in the sample due to sawdust (Menezes, Ferreira, Neves, & Ferreira, 2002).

Figure 5. X-ray diffractogram of the abrasive mud sample.



The basic composition of the abrasive mud characterizes it as a versatile material for use in the most different industries, mainly due to the different oxides present. When combined with mineral forms, such as quartz, feldspar, mica and calcite, ornamental rocks residues also become interesting as a low-cost raw material for the manufacture of red ceramics and glazes (Manhães, Moreira, & Holanda, 2009). The alkaline oxides present, coming from the microline, act as melting agents in the ceramic formulations, however the small amount of Fe_2O_3, possibly from crystalline phases such as biotite, behaves as a limiting factor for the reuse of the residue in this segment (Menezes, Ferreira, Neves & Ferreira, 2002).

Determination of physical-chemical proprieties

The results presented below refer to the arithmetic mean of the triplicates performed for each parameter. Table 3 presents the results obtained in the analysis of pH, moisture, bulk density and absolute density.

Table 3. Results of determination of the following physical-chemical properties of the abrasive mud sample: pH, moisture, bulk density and absolute density.

Parameter	pH	Moisture (%)	Bulk density (g/cm^3)	Absolute density (g/cm^3)
Result	9.34	6.50	1.18	3.57

From the results obtained, it is possible to see a tendency to alkaline character of the abrasive sludge due to the chemical composition of the analyzed sample and lime addition during the rock unfolding process. Regarding the moisture analysis, a relevant value is observed, confirming the presence of water that remains as moisture in the accumulated tailings after disposal. It can also be said that the filtration step carried out on the abrasive sludge cannot extract all the water.

It is noteworthy that this moisture value tends to decline over the days of exposure, causing detachment of fine and ultrafine tailings with low particle size, which can cause damage to health. When in aqueous solution, the abrasive sludge samples analyzed tend to decant naturally, a result confirmed by the density analysis. These values reflect the chemical and mineralogical composition of the waste (Manhães & De Holanda, 2008; Rego, Martínez, Quero, Blanco & Borque, 2001).

CONCLUSION

The experimental results obtained indicate several ways of reusing the ornamental rock residue, the abrasive mud. The chemical and mineralogical analysis of the sample classified it as silico-aluminous material, pointing out the possibility of reusing the residue in civil construction and in the manufacture of ceramics. The morphological analysis confirmed the irregular shape common to the ornamental stone beneficiation process and pointed out the significant presence of iron that can behave as a limitation for typical waste applications, although magnetic separation may be used in some cases.

The investigation of the physical-chemical characteristics showed us a basic character of the sample due to the nature of the rock and the addition of chemical additives such as lime, during the stage of cutting the ornamental rock. The moisture test confirmed that not all the water from the processing of ornamental rocks evaporates or seeps into the soil after the waste is discarded. The parameters that can be changed according to the need, such as humidity and pH, characterize the sample, but do not behave as limiting factors in the potential of the waste.

For future work, the importance of specific analyzes for possible applications is emphasized in order to enrich the related literature and enable better targeted discussions, as well as large-scale applications.

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