

# Potential for Use of Iron Mining Tailings Calcined in a Flash Furnace as Pozzolanic Material

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## Abstract

This paper presents a study of the potential use of iron mining tailings as artificial pozzolan (metakaolin) after their submission to thermal treatment via calcination in a flash furnace. The research consists of the characterization of the tailings before and after calcination, chemical, mineralogical, thermogravimetric, and mechanical strength analyses were conducted. The results were compared with those for commonly used pozzolans, metakaolin, and similarities were identified. The study of the morphology of the particles before and after calcination was conducted through analyses of images obtained by scanning electronic microscope. The pozzolanic activity of the fine mining tailings calcined with flash technology was evaluated in uniaxial compression trials, which showed excellent results.

## Keywords

Mine Tailings, Flash Calcination, Pozzolanic Activity, Compressive Strength

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## 1. Introduction

Mining residues are rejects and tailings that are deposited and or removed to attain greater economic value. They are generally placed in piles and dams, which must be frequently monitored. According to the State Environmental Foundation, FEAM [1], in 2017 the state of Minas Gerais, Brazil alone generated more

than 470 million tons of tailings and residue. Therefore, there is a need to create viable and safer options for waste disposal.

The geology of the Iron Quadrangle region in the state shows that the material classified as fine mining tailings from iron ore extraction is constituted in part by weathered metabasic type rocks and phyllites. These are composed mostly of clayish minerals, such as kaolinite, and minerals of iron and silicon, with the presence of lateritic clayey soils that are formed by the actions of physical and chemical weathering [2].

The presence of these minerals becomes the tailings similar to the composition of clayish rocks that were studied to obtain metakaolin as a material with pozzolanic properties [3] [4] [5]. Reference [6] demonstrates that materials with pozzolanic properties can be obtained from thermal activation of lateritic tropical soils. Reference [7] shows that the presence of clayish minerals indicates that when submitted to heating, the material generates good pozzolanic products and also emphasized the importance of using X-ray diffraction to identify the minerals. Reference [8] shows that the minerals kaolinite and gibbsite found in lateritic soils are transformed into metakaolinite (amorphous alumina) when submitted to calcination between 750°C and 800°C, and acquire pozzolanic properties.

Reference [9] affirms that calcination results in damages and destruction to the crystalline structures of clays, and is a highly effective technique for activation of pozzolanic properties of clays. The calcination can be conducted in different types of furnaces, such as rotary kilns and fast burn ovens known as flash furnaces. The rotary form of calcination is the most commonly used to produce commercial metakaolin [4]. It is characterized by the lack of a standard in the calcined products, that is, particles are obtained that are poorly calcinated as well as particles that are highly burned [10]. Flash calcination is considered an ultra-fast process, in which solid particles of fine granulometries are suspended in gas and submitted to high temperatures and then cooled [11].

According to Brazilian standard NBR 12653 [12] pozzolanic materials are defined as siliceous or aluminous siliceous materials that alone have little or no binding property, but when finely divided and in the presence of water, react with calcium hydroxide at ambient temperature, forming compounds with binding properties. The pozzolanic activity is defined by the capacity of the material to react with the calcium hydroxide [13].

Reference [14] affirms that the use of pozzolans in cementitious materials offers technical improvements, such as a reduction in temperatures and improved durability and compressive strength of the material.

The main objective of this article is to show that fine mining tailings generated from iron mining have properties similar to kaolin, and clays rich in kaolinite, which are raw materials that when submitted to flash calcination result in products with pozzolanic characteristics of excellent quality, and can be used to substitute Portland cement, mortar for iron ore tailings, cold pelleting of dam tailings, and as flexible pavements for the base and subbase of roads. Other advantages include giving a valuable use to waste material and reducing manufactur-

ing of Portland cement, which can lead to lower CO<sub>2</sub> emissions.

## 2. Materials and Methods

### 2.1. Characterization of Raw Material

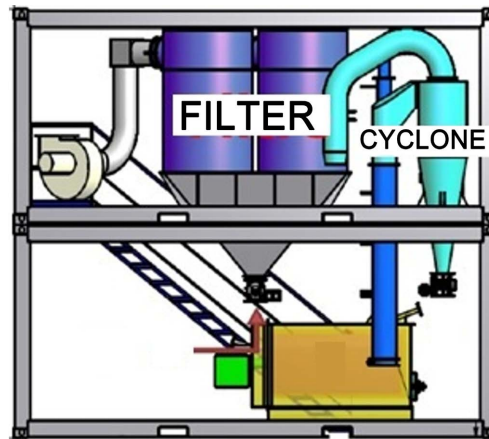
Samples were obtained of fine mining tailings, weathered metabase and phyllite coming from piles of iron mining tailings in the Iron Quadrangle region of Minas Gerais state in Brazil.

Thermogravimetric analysis (TG and DTG) was conducted in a nitrogen atmosphere at 50 ml/min, at a heating rate of 10 C/min until a temperature of 1000°C, to determine the best temperature for calcination of the material. X-ray diffraction (XRD) (Fig X) was performed to identify the minerals. The granulometric distribution was determined by laser granulometry (Cilas 1064) using sodium hexametaphosphate as a dispersant at a concentration of 0.05% in mass, in deionized water. The specific surface area was determined with the BET method (Brunauer, Emmett and Teller), by gaseous adsorption. The morphological study of the mineral particles of the raw material was conducted with a Scanning Electron Microscope (MEV) FEG, Quanta 200 FEI. First, confirm that you have the correct template for your paper size. This template has been tailored for output on the custom paper size (21 cm × 28.5 cm).

### 2.2. Preparation and Characterization of the Material

A schematic diagram of the flash calcination furnace located at the Geotechnology and Geomaterials Laboratory (LGG-UFMG) is shown in **Figure 1**. The furnace can be described as a calciner of particles smaller than 500 microns, that is, of powdery materials. The raw material with a granulometry below 80 microns and with maximum moisture content of 5% is led by a pneumatic feed to a U-shaped tube. An industrial ventilator creates negative pressure in the tube, generating a suction of the raw material. A combustion chamber is fed by a blower that feeds this chamber with air and fuel. With negative pressure in the tube the material is sucked through the tube at a speed of a few hundreds of a second and the fuel and air are also blown and sucked by the ventilator. In this tube the raw material suffers thermal shock, which transforms the original crystallography into amorphous material. The particles produced are selected in a cyclone, through the difference in density and shape. The ability to generate products depends on the raw material calcined. The products obtained from meta-basic rock are pozzolan (with an amorphous crystallography) silicon and iron oxide, the same products are obtained from phyllite rock, although with a lower quantity of iron oxide.

The feeding of the material begins after the furnace reaches the calcination temperature, which was previously determined with procedures mentioned in patent letter PI 9809941-8 held by Demeter Technologies and registered in Know How number 56 in 27 03 2020, CTIT, UFMG, preparation and calcination of raw materials for flash calcination.



**Figure 1.** Representation of flash calciner in pilot plant (Adapted from [2]).

When completed, the calcination of the products is separated by a cyclone that is responsible for separation of the calcined products at outputs a and b. In the output filters “a” the products have a finer granulometry and silicon and iron oxide are more abundant. At output “b”, we have the production of iron oxide and or metakaolin in a free form.

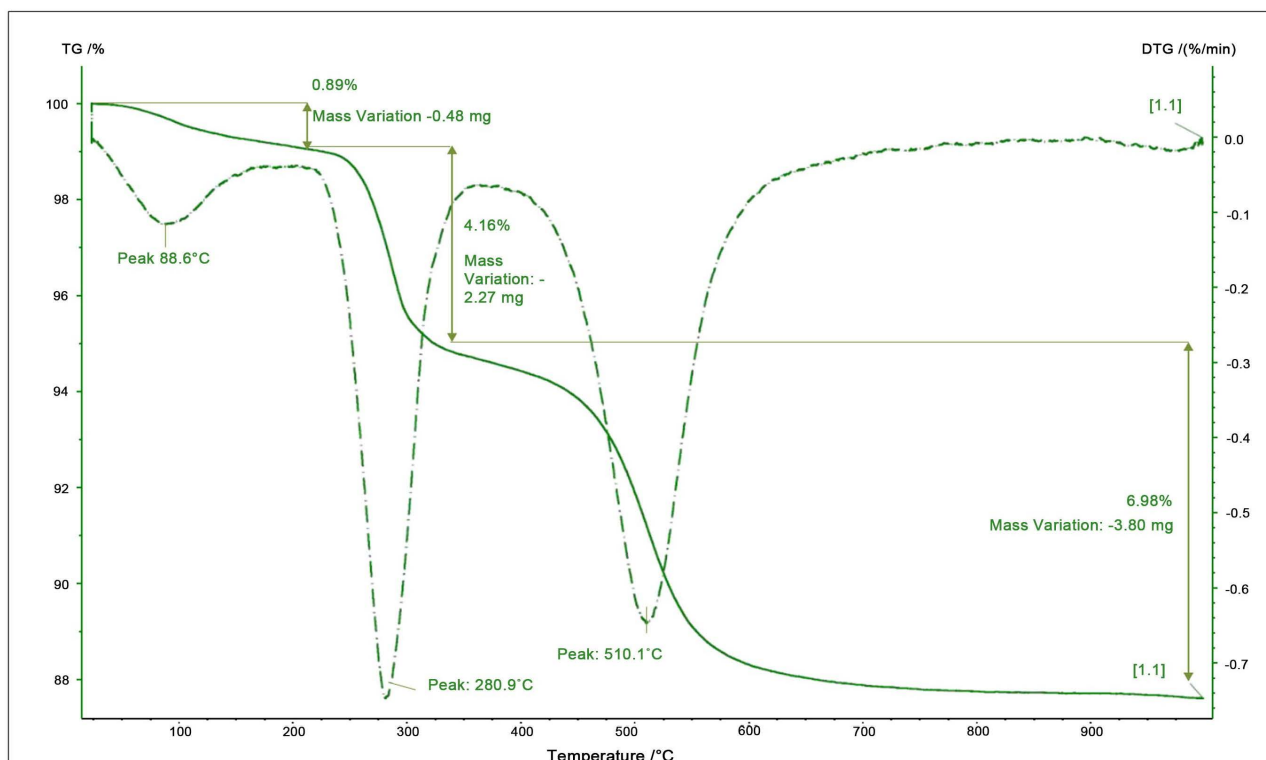
To feed the flash furnace, the raw material must have suitable granulometry and humidity. The humidity is adjusted in a dryer until the material reaches a maximum of 5% moisture, and 75% of the material should have granulometry below 200. When these specifications are reached, the raw material can be fed to the flash furnace. The calcination temperature was determined by the thermal analysis shown in **Figure 2**. The product of flash calcination will be identified as flash calcined tailings.

The same tests conducted to characterize the raw material were also conducted to characterize the calcined product, to compare the changes caused by the flash calcination of iron mining tailings.

### 3. Experimental Results

#### 3.1. Characterization of Raw Metabasic, Flash Furnace Calcinated Metabasic; Raw Phyllite and Flash Furnace Calcinated Phyllite

The weatherized metabasic rock and phyllite, and the sum of the oxides of aluminum, silicon and iron total 80% of the composition of the sample, a result that is in keeping with the proportions of these elements found in studies (presented by [3]); and [4] of kaolin for the production of pozzolanic material. The difference between the results found is in the quantities of silicon and iron, because in the studies presented, there is more silicon than aluminum, followed by the presence of iron. In the analyses of the weatherized metabasic and phyllite, the three elements were found in similar proportions, with more aluminum, followed by silicon and iron (**Table 1** and **Table 2**).



**Figure 2.** Raw metabasic rock TG and DTG curves.

**Table 1.** Average chemical composition of metabasic and phyllite samples, raw and calcined.

(%)	PPC (1000°C)	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	MnO	CaO	MgO	PeO <sub>5</sub>	ZrO <sub>5</sub>	Na <sub>2</sub> O	K <sub>2</sub> O
Raw phyllite	8.25	23.75	42.37	2.52	19.3	0.05	0.51	0.04	<0.1	0.12	NR	<0.1	3.10
Cycloned phyllite 650°C	4.64	27.87	42.50	1.45	17.52	0.06	0.41	0.02	0.49	0.11	NR	0.17	4.73
Raw metabasic	8.77	18.66	28.17	3.45	40.53	0.05	0.06	0.06	<0.1	0.13	NR	<0.1	0.11
Cyclone metabasic 650°C	4.72	20.60	29.95	3.62	40.05	0.02	0.08	0.08	0.49	0.15	NR	<0.1	0.21

NR: Not Registered; Note that analysis of the chemical compositions did not find large variations between the raw and calcined elements.

**Table 2.** Average mineralogical composition, metabasic and phyllite, raw and calcined.

(%)	Muscovite	Kaolinite	Quartz	Gibbsite	Hematite.	Goethite	Anat/rut	Magnetite	Amorphous
Raw phyllite	26.3	22.6	20.0	7.3	7.1	14.2	2.5	0.0	0.0
Raw metabasic	0.9	38.2	10.1	4.9	24.1	16.3	3.5	2.0	0.0
(%)	Muscovite	Meta Kaolinite	Quartz	Gibbsite	Gibbsite	Goethite	<i>Anat/rut</i>	Magnetite	Amorphous
Cycloned phyllite 650°C	40.1	20.3	14.9	0.0	17.5	0.0	1.5	0.0	5.7
Cyclone metabasic 650°C	1.8	33.8	13.7	0.0	40.0	0.0	3.6	0.0	7.6

Note: The entrance of amorphous materials and also amorphous meta kaolinite are observed, and the transformation of goethite into hematite. The amorphous transformations and that of goethite for hematite occur by dehydroxylation.

The temperature of calcination can be determined through scaled loss-on-ignition studies and thermogravimetric analyses. Preferentially, the 2 types of analyses lead to very similar conclusions. Below we show the results of the thermogravimetrics for the metabasic rock.

Note that the change in the final phase occurs at 650°C and up to 1000°C and its derived DTG, from the sample of raw weatherized metabasic rock. Three changes in mass are identified. The first takes place at the beginning of the test, up to a temperature of 204.0°C, with a mass variation of approximately 0.89%; this step characterizes the loss of humidity, from the release of water that is not linked to crystalline structures in minerals in the sample. After the temperature of 204.0°C, up to 343.0°C, there is another change of mass corresponding to 4.16%, with a peak at 280.9°C, indicating that the highest rate of mass loss occurred at this temperature. At this interval, the loss of water is attributed to the thermal decomposition of the mineral gibbsite. The final interval of mass loss takes place between the temperatures of 343.0°C and about 630°C, with a rate of variation of mass of approximately 6.5% and a maximum peak of loss at the temperature of 510.1°C. These amounts are associated to the decomposition of goethite with the formation of hematite, and mainly the dehydroxylation of the kaolinite. The best temperature for calcination of the raw material was defined as 650°C, before the peak at 770°C.

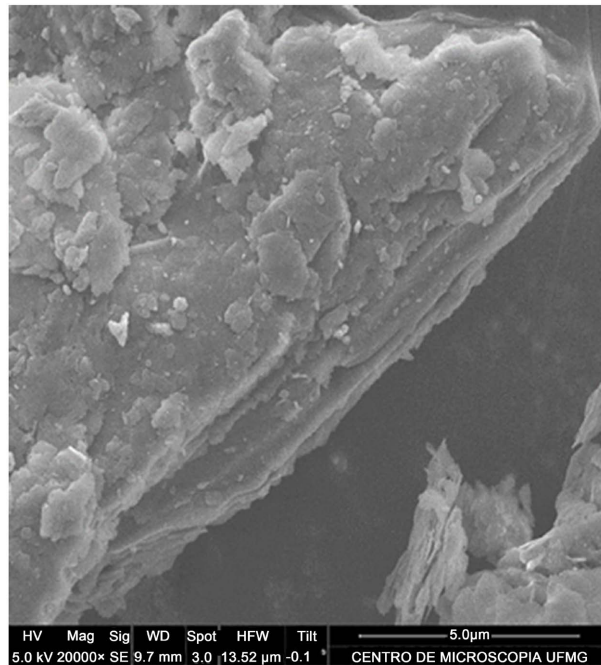
It is pertinent to highlight that the thermogravimetric analysis is coherent with the results found in the X-ray diffraction analysis, which indicated the presence of hydrated minerals cited in this thermal analysis. It is also important to show that the semiquantitative determination of the diffraction of X rays is in keeping with the rates of mass loss determined in the TG curve. As an example, we cite the abundant presence of the mineral kaolinite in the sample; this is confirmed by the high rate of mass loss that occurs at the interval from approximately 343.0°C up to 650°C.

The morphology of the particles that compose the tailings is seen in the image from the Scanning Electronic Microscope presented in **Figure 3**. The layered structures of the kaolinites are notable, with fine layers and the morphology of the flattened structures. **Figure 4** shows an image of kaolin obtained by [3], which indicates the kaolinite plates in well-defined hexagonal form, although it is not possible to determine the layered structure of the particles.

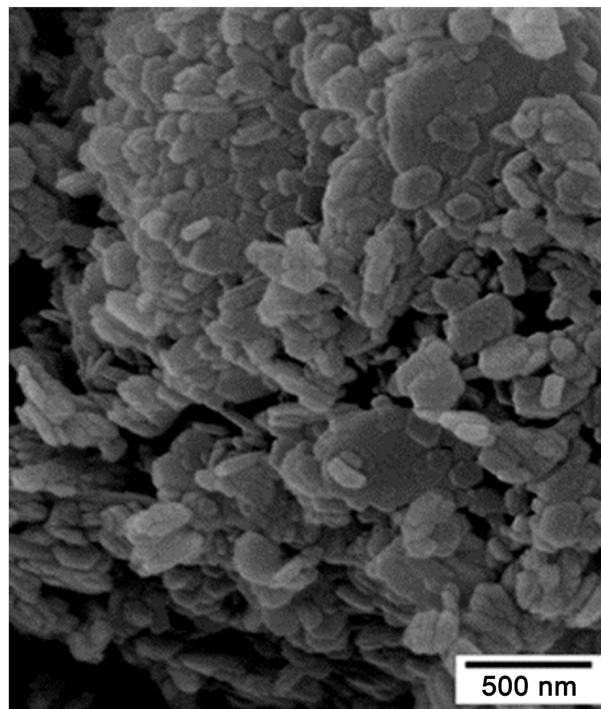
### 3.2. Characterization of the Raw and Calcined Material

The figures below are images of the raw and calcined weatherized metabasic samples, and raw and calcined phyllite and were realized in the scanning electron microscope (MEV-FEG), Quanta 200. The EDS results for micro-structure are presented below the images in **Figure 5** and **Figure 6** (EDS-X-ray spectrometer by energy dispersion, Bruker, software Esprit 2.1) [15].

**Figure 6** shows a larger lamellar particle with various other smaller particles above it. A microanalysis was conducted of this particle and the result is presented in the table. High levels of aluminum, silicon and oxygen are seen,

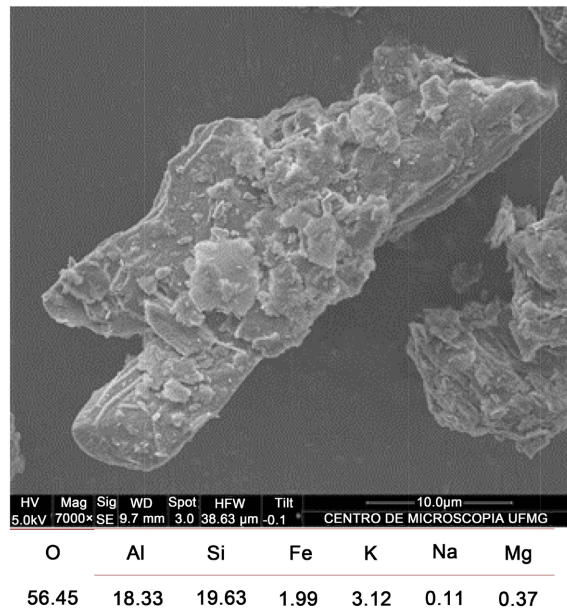


**Figure 3.** SEM Image kaolin Brazil (Source: [3]).



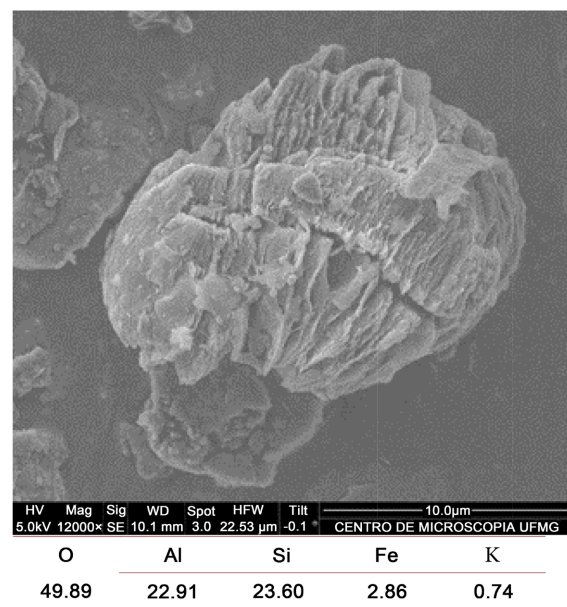
**Figure 4.** SEM Image kaolin Brazil (Source: [3]).

confirming that the lamellar particles are truly from mineral kaolinite. **Figure 7** shows the presence of metakaolinite in the sample; this is confirmed by the morphology of the particle, in which the lamellas are distanced (foliation) and with damaged edges, which is characteristic of metakaolinite. The microanalysis shown in the table below indicates higher levels of aluminum, silicon, and



Mineral: Kaolinite.

**Figure 5.** Secondary electron images from the weatherized metabasic sample raw (10.0 μm).



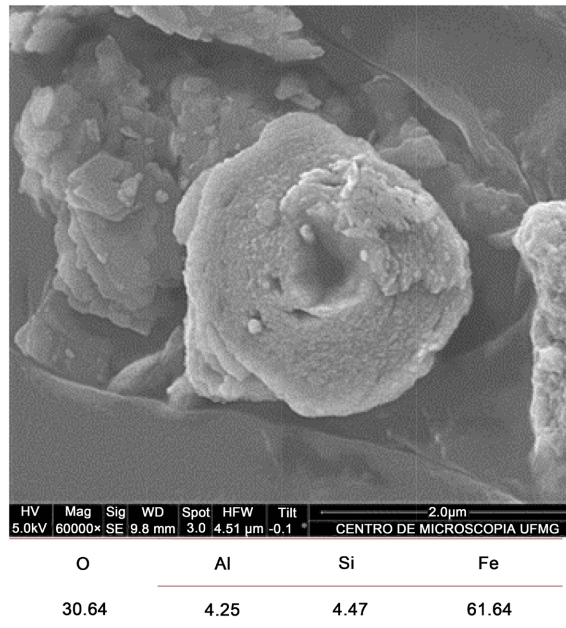
Mineral: Meta Kaolinite.

**Figure 6.** Secondary electron images from the weatherized metabasic sample calcined at 650°C (10.0 μm).

oxygen, confirming that the particle in question is metakaolinite, a product that originated from the kaolinite found in the raw sample.

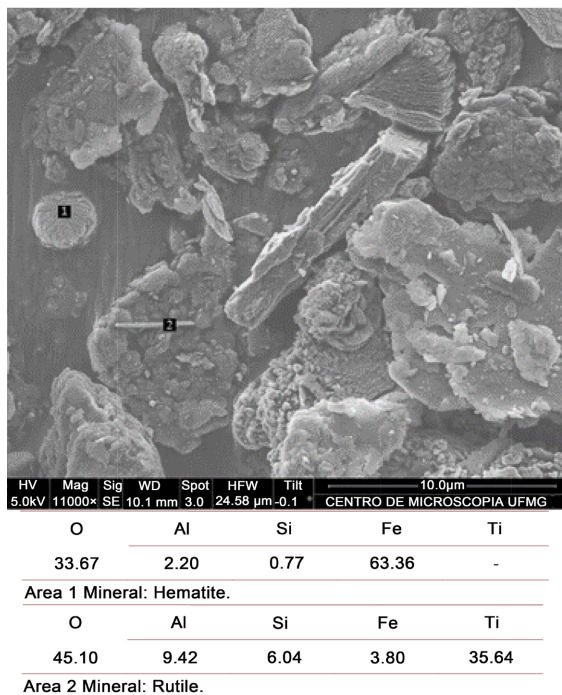
**Figure 7** shows a particle of the mineral goethite, which is demonstrated by the microanalysis presented in the table below that shows high levels of iron and oxygen. In addition, the morphology of the mineral is also characteristic of botryoidal goethite, which appears here to be similar to a doughnut. In **Figure 8**, a





Mineral: Goethite.

**Figure 7.** Secondary electron images of the goethite sample in the raw (2.0 μm).



**Figure 8.** Secondary electron images of the hematite mineral in the sample that was flash calcined at 650 °C (10.0 μm).

significant presence of iron was found in area 1, in particles with a morphology similar to that seen in the original raw sample, in which was identified the mineral goethite. However, considering that the sample analyzed was submitted to calcination at a temperature of 650 °C, and at this temperature the mineral goe-

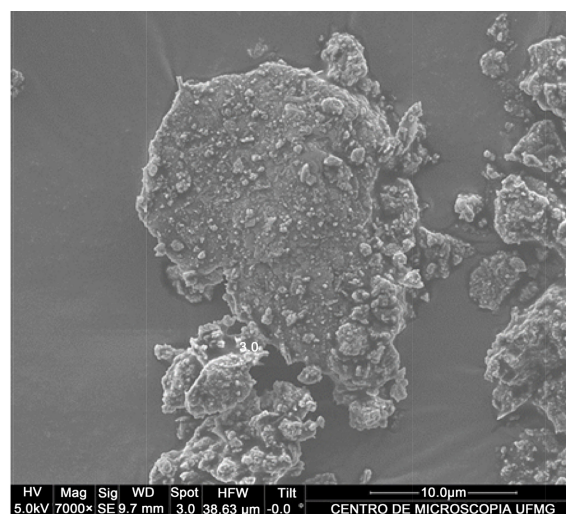
thite lost its hydroxyl, becoming a dehydrated mineral, it is coherent to affirm that after the thermal treatment, the mineral comes to be hematite. Area 2 has a high level of titanium and in keeping with the morphology of the particle it can be affirmed that the mineral in question is rutile, showing that after calcination the mineral in question still remained.

**Figure 9** and **Figure 10**, are amplified by 7000, in them can be observed, respectively, particles with a plate morphology, which indicates the mineral kaolinite. An amplification of the large plate is seen in **Figure 9**. A microanalysis was conducted in this area whose results are found in **Figure 10**. It is concluded that the area in question presents the mineral kaolinite and a minority mineral of iron, possibly hematite.

In **Figure 11** two highlighted particles are seen, identified as point 1 and point 2. With the EDS microanalysis it is pertinent to affirm that particle 1 is composed by hematite associated to a mineral of manganese, given that there are higher levels of the elements iron and manganese. Particle 2 suggests the occurrence of pseudo morphism of the quartz mineral, following the kaolinite, a fact that is justified by the high level of the element silicon in the particle and by the characteristic morphology of mineral kaolinite. **Figure 12** shows a particle with subhedral morphology, characteristic of mineral quartz. This is confirmed by the EDS microanalysis, which indicates a high level of the element silicon in the particle. Below are the results of the EDS microanalysis.

### 3.3. Resistance to Uniaxial Compression Following Norm ABNT NBR 5752/2014

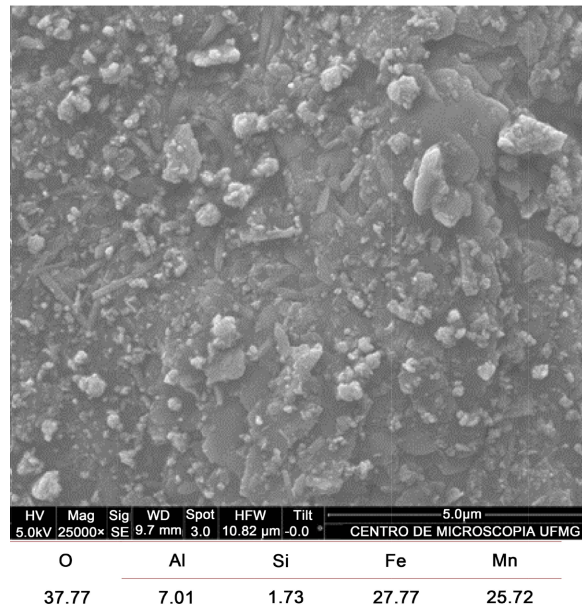
**Table 3** and **Table 4** show the results of the trials for performance index with Portland cement for the metabasic and phyllite calcined in the flash furnace,



O	Al	Si	Fe	Mg
50.77	22.54	23.29	2.49	0.92

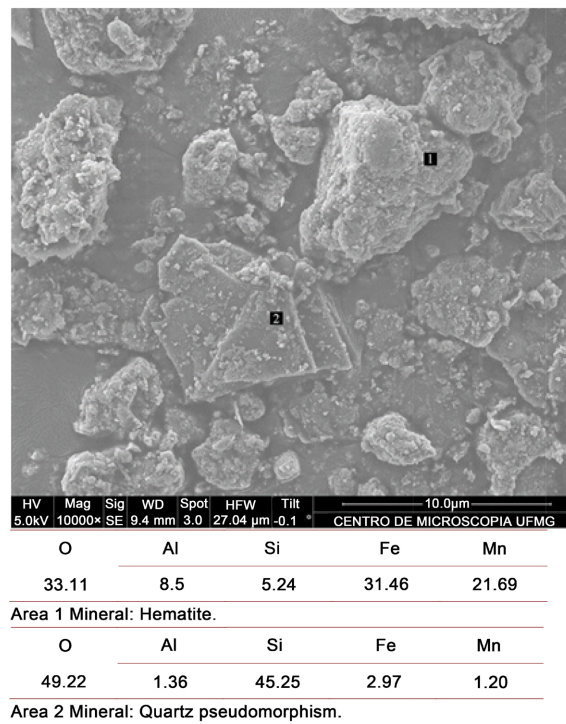
Mineral: Kaolinite.

**Figure 9.** Secondary electron images of the sample of raw phyllite, mineral kaolinite (10.0 μm).



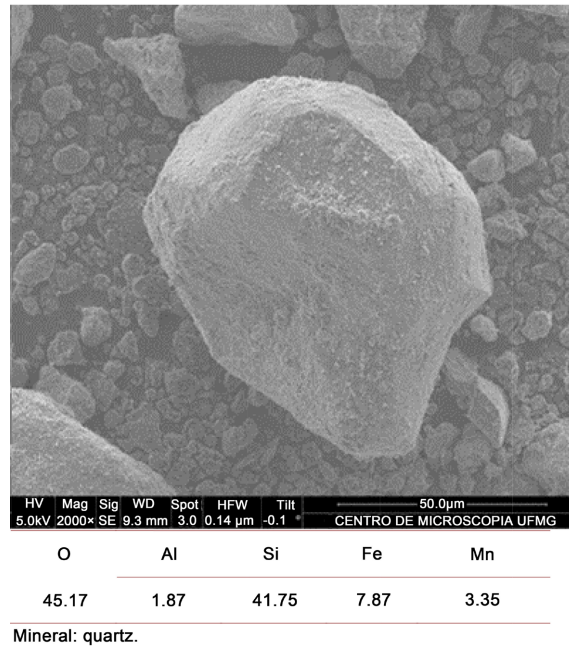
Mineral: Kaolinite and hematite.

**Figure 10.** Secondary electron images of the sample of raw phyllite, mineral kaolinite, hematite (5.0 μm).



**Figure 11.** Secondary electron images of the sample of phyllite flash calcined at 650 °C (10.0 μm).

and the calculation of the cement performance index (Icimento). It can be seen that the two calcined rocks have a favorable performance index, the flash calcined metabasic had an index of 113.94% after 7 days of cure in relation to the reference, while calcined phyllite had an index of 90.46% after 7 days of cure.



**Figure 12.** Secondary electron images of the sample of phyllite flash calcined at 650°C (50.0 μm).

**Table 3.** Comparative results [12] for Metabasic.

Time (days)	Flash Metabasic 770°C -Pressure (Mpa)	Reference CP II 32 F -Pressure (Mpa)	Icimento (%)
7	29.793	26.148	113.94
28	41.431	39.880	103.89

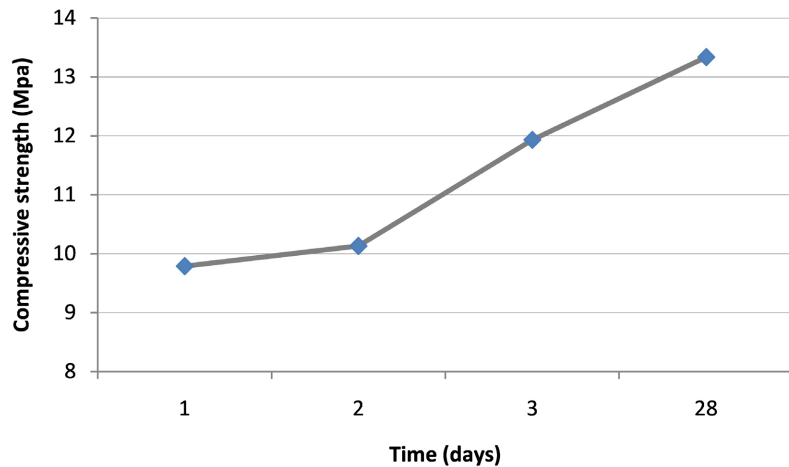
**Table 4.** Comparative results [12] for Phyllite.

Time (days)	Flash Phyllite 750°C -Pressure(Mpa)	Reference CP II 32 F F - Pressure (Mpa)	Icimento %
7	24.200	26.750	90.460
28	37.400	39.860	93.820

**Table 4** presents the results of the performance index trials with Portland cement for the phyllite flash calcined at 650°C. It can be seen that the flash phyllite reached strength values above the reference.

The changes in pozzolanic activity were studied by measuring the uniaxial compression strength of mixtures of flash calcinated metabasic and calcium hydroxide. The ratio of the calcinated material to lime was 1.5. The breaks of the test bodies were conducted successively in the first 3 days, to evaluate the initial change in pozzolanic activity, other breaks at 28 days were conducted to simulate the pozzolanic activity over the period.

The results obtained from the breaks are presented in graph form in **Figure 13**. The average break at 1 day of curing showed high values for MPa, with a small change for breaks conducted at 2 days of cure. After 3 days of cure, the



**Figure 13.** Compressive strength of mortars.

uniaxial compression reached 12 Mpa, showing a good evolution in pozzolanic activity. The breaks at 28 days were on the order of 13 MPa. It can be seen that the higher rate of increase of pozzolanic activity took place between the breaks on days 2 and 3 of cure, by 28 days there was a rise of pozzolanic activity in relation to previous breaks, but with a lower rate of increase.

#### 4. Conclusions

The flash calcination of materials considered to be waste from iron mining generates products with reactivity, which can be considered to be artificial pozzolans, proved by mechanical strength tests conducted according to norms.

The analyses of the X-ray diffraction show that the weathered metabasic rock is characterized by greater abundance of mineral kaolinite than the phyllite rock. This contributes to the better performance of flash metabasic in strength tests. In conjunction with the X-ray diffraction, the results of the thermogravimetric trials identified the thermal transformations that the minerals that compose the sample underwent.

The images obtained by MEV show the effect of flash calcination on the morphology of minerals, and the EDS microanalysis allowed chemically identifying the minerals. In the raw samples were found particles with morphology similar to the sample of pure kaolin. After flash calcination, there were deformations in the lamellas that compose the particles, revealing that there was a transformation from kaolinite to metakaolinite.

Finally, we can conclude that the weatherized metabasic rocks and phyllite that compose part of the tailings from iron mines in the Iron Quadrangle of Minas Gerais state acquired pozzolanic activity after undergoing flash calcination, and present good results for the index of pozzolanic activity, when reacted with calcium hydroxide. Thus, they proved to be a good option for use as an additive to Portland cement, contributing to the reduction of greenhouse gases and giving a valuable use to the masses of materials that compose tailings at mining sties.

The circular economy shows that the insertion of mining residues in the chain of products of other segments such as for cement and highway pavings is an opportunity to decrease or even attain zero stocking of waste at dams and or piles of tailings, simply from the fact that after water, cement products are those most consumed by humanity.

The Brazilian cement industry maintains a prerogative without technical justification for reddish or even pink pozzolan, to indicate low mechanical strength. This article shows that to the contrary, the insertion of iron in the ionic bonds between aluminum, iron, and quartz, effectively increases the mechanical strength. Brazilian and global norms do not make determinations based on the color of cement. The discrimination against red or pink cement is a popular habit that impedes the use of mining tailings and rejects. In the twenty-first century when architects are requesting colored concrete, there is no technical justification for impeding or depreciating cements that do not have the common color, this is only an old and baseless habit.

The samples of iron mining tailings were characterized and later underwent flash calcination at a temperature of 770°C, the characterization of the samples demonstrated that:

- The chemical analyses show that the sum of the amounts of  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$  and  $\text{Fe}_2\text{O}_3$  is similar in the tailings and kaolins, which indicates the potential for use of the tailings as raw material for the production of artificial pozzolan;
- (The X-ray diffraction) showed the presence of kaolinite in the tailings, which is an important majority mineral in the kaolin samples;
- The thermal analyses of TG and DTG were important to determine the temperature of flash calcination of the tailing material;
- After undergoing flash calcination, the tailings had changes in their physical parameters. Their specific surface area dropped 18.57  $\text{m}^2/\text{g}$ , and D50 varied 3.51  $\mu\text{m}$ ;
- The pozzolanic activity of the calcined tailings was determined by uniaxial compression strength tests, which showed high rates of increased strength between the breaks at 2 to 3 days of cure. The breaks at 28 days conclusively affirmed that the pozzolanic activity of the calcined tailings tends to increase after a larger time interval. The procedure to determine the pozzolanic activity used was French norm NF P 98-104. This procedure is more suitable because it uses the pozzolanic product obtained from the pure flash calcination to which calcium hydroxide is added. The pozzolanic activity is based on the absorption of calcium hydroxide. The Brazilian norm to determine the pozzolanicity index uses Portland CF II-F32 composed of a carbonate filler. In the author's master's dissertation, this procedure with Portland cement was also used. In this article, however, the objective was to use norms accepted in other countries and not just in Brazil.
- (The X-ray diffraction) showed the presence of kaolinite in the flash calcined tailings, although the results for uniaxial compression strength were satisfactory, on the order of 13 MPa, suggesting that the presence of minerals such as

gibbsite, which after the thermal process came to have an amorphous structure, and goethite, which upon losing the hydroxyl became hematite, provide greater reactivity to the calcined product, improving the mechanical performance of the test cores.

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## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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