### Pozzolanic activity evaluation of slate waste

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# Abstract

This work had the intention of evaluate the pozzolanic activity of the residue generated by the slate cutting in order to determine the efficiency of its use as a mineral addition in cementitious materials. To perform this evaluation the residue was inicially subjected to the chemical and mineral characterization. The chemical analysis was done by a semi-quantitative technique of X-ray fluorescence and through the quantitative technique of X-ray diffraction the minaralogical characterization of the slate waste was obtained. In order to evaluate the pozzolanic activity of the slate residue was used the electrical conductivity method proposed by Luxan and the test of Pozzolanic Activity Index described by NBR 5752, 1992. The mineralogical and chemical analysis of the slate cutting residue reveled that the material has a large part consisted of silica in the form of quartz, indicating that the waste can present pozzolanic activity when added to the cement. The results in the Luxan test showed that the slate cutting residue has a moderate pozzolanic activity, but the mortar produced with the material didn't reach the pozzolanic activity index of 75%, which is the minimum physical requirement imposed by NBR 12653, 1992. However, the determined value of 70% is close to the required by the standards and further research is relevant in order to better understand the behavior of waste in cement materials and the feasibility of its use as an alternative mineral addition in the production of Portland cement mortars and concrete.

### Keywords

Slate, waste, pozzolanic, activity.

# 1. Introduction

Industrial processes of extraction and treatment of construction inputs generate waste accumulation, causing extensive economic damage related to the collection and disposal of such waste, and also ecological imbalances.

In this context, it is very important the searching for new technologies and the development of research contemplating the use of mineral waste in the production of building materials. The use of alternative materials in construction is increasingly used due the possibility of improve the durability and performance of concrete and mortar. Thus, the great need the use of waste and by-products makes necessary the study of their utilities, contributing as an ecological benefit, social and economic [1].

The biggest advantages of the use of mineral waste in the production of building materials are: its abundance, particularly at the regions where mining and mineral processing companies are concentrated; diversification of raw materials and the possibility of lower production costs [2]. The inserting of waste in the production cycle is in addition to an alternative to production cheapening, an option of recycling and reuse of these materials, minimization of non-renewable energy consumption and natural resources and creation an alternative to reduce the environmental impact on the surrounding of regions of slate processing.

Slate is widely used in construction. Brazil is the second largest producer and consumer with Minas Gerais accounting 95% of national production. In this state the slate production totals approximately 500 thousand tons / year. In value, these exports showed growth of 45.1% compared to 2003 and now account for 47.6% of total exports of Minas Gerais rocks, according to the Slate National Fair of 2006.

The extraction and rock cutting systems promote production of large amounts of waste in slurry form, composed primarily of water, lubricants and ground rock. The waste has little technological value and often ends up deposited in landfills, patios or reservoirs, resulting in problems of garbage relocation and silting of riverbeds. The Brazilian production of ornamental and coating rocks amounted about nine million tons in 2009. With this great production there is a large amount of waste generated in the exploitation of deposits. It is estimated that 7% of this production is slate and the waste generation is 25% of production, totaling  $1.5 \times 105$  tons of waste according to the Brazilian Association of Rocks Industry. In this context, there is a need to evaluate the potential use of slate waste as mineral admixture in mortars and concrete Portland, considering questions of durability, economy and ecology.

It is very important the searching for new Technologies and the development of research contemplating the use of new materials such as pozzolans that can be used as raw materials for use in the construction industry. The pozzolans have the ability to react and combine with calcium hydroxide forming stable compounds with binding power. These compounds added to the concrete produce benefits such as higher durability and workability of concrete, improvement in their mechanical properties and reduction of Portland cement consumption and energy used in its production. The purpose of this study was to investigate the pozzolanic activity of particulate waste generated by cutting the slate and determine the efficiency if its use as a mineral addition in cementitious materials.

### 2. Experimental

#### 2.1. Materials

The following materials were used:

- Slate wastes (from mining and cutting activities) from the Slate Province of Minas Gerais. It was donated by the MPM Slate mining company, located in the city of Pará de Minas, Minas Gerais state;
- Portland cement type CP III 40;
- Sand, for composing the mortar mix;
- Fluorite (CaF<sub>2</sub>) for x-ray diffraction internal standard;
- PA Calcium hydroxide for Luxan test solution;
- Distilled water for Luxan test solution;
- Mineral oil for NBR 5752, 1992.

#### 2.2. Sample preparation

The slate cutting residue is a thin material, removed from the decantation tank in slurry form that after drying at room temperature, is presented in the form of lumps. To use it were necessary drying and grinding processes. The residue was subject to heat treatment, being dried in an oven for 24 hours at a temperature of approximately  $110 \degree$  C, resulting in a very thin material in the form of lumps. After drying, the material was ground on a horizontal mill for 20 minutes. The purpose of the milling processing is the reduction of particle size of the solid material, promoting an increase in specific surface and enabling better reactivity with other materials.

### 2.3. Characterization techniques

### 2.3.1. Particle size analysis

It was performed laser granulometry test intended to determine the granulometric characteristics of the material, that is, grain size and size distribution. It was used a Bettersize laser granulometer.

# 2.3.2. X-ray fluorescence

Through this technique it was possible to perform a qualitative chemical analysis of RCA, showing its main elements. It is important to note that this analysis is semi-quantitative, not reflecting the actual percentages of each chemical component. A Shimadzu EDX7000 model spectrometer was used.

#### 2.3.3. X-ray diffraction

The residue was physically characterized by X-ray Diffraction technique used to determine the phase (minerals) present. The analysis was performed on diffractometer Empyrean of PANalytical with X'Celerator detector, and the diffraction pattern was collected with CuK $\alpha$  radiation. The scan was performed from 4 ° to 70 ° and the results refined by the Rietveld method. Each crystalline compound has a characteristic pattern of diffractometry and the pattern overlapping of all the component minerals form the XRD pattern of the material analyzed. According the literature, mineral quartz, muscovite and chlorite are part of the mineralogical composition of the slate [3].

# 2.4. Pozzolanic activity evaluation

#### 2.4.1. Luxan method

This method consists in measuring the pozzolanicity of the slate cutting waste by varying the electrical conductivity of a saturated solution of calcium hydroxide, before the pozzolan be added and after 2 minutes of continuous mixing of the solution.

The saturated solution preparation procedure consists of initially maintain in heating distilled water in a beaker, stirring constantly through magnetic stirrer. Reached the stabilization of temperature of 40  $^{\circ}$  C was added pure calcium hydroxide. After complete dissolution of the Ca (OH)<sub>2</sub> was introduced in the solution a conductivity sensor and the initial measurement of the electrical conductivity was performed in mS / cm. Then, was added to the solution the material to be evaluated. 120 seconds after the adding, the final reading of the conductivity was performed.

The pozzolanic activity index was then calculated as the ratio of the electrical conductivity before and after two minutes of the RCA addition. According to the method proposed by Luxan the pozzolanic material can be classified according to the conductivity variation. There are three groups where natural pozzolan are divided according to their reactivity: no pozzolanicity, with moderate pozzolanicity and good pozzolanicity.

# 2.4.2. Compressive strength test – NBR 5752, 1992

The evaluation of the pozzolanic activity of slate waste followed the guidelines of the brazilian standard NBR 5752, 1992 [4]. The standard determines the pozzolanic activity index (PAI) in Portland cement with a mineral addition. The PAI is the ratio between the resistance values of the compression mortar with 65% of cement and 35% pozzolan and a reference mortar produced with 100% of cement as a binder.

For mechanical testing were mechanically mixed two mortar traces. The first mortar was admitted as a reference, using only cement, standard sand and water, while at the second mortar, 35% of the cement was replaced by pozzolana to be analyzed. After the curing period of the specimens, the determination of the mechanical property of compressive strength was performed on a uniaxial compression testing machine EMIC, model DL20000.

#### 3. Results and discussion

#### 3.1. Characterization techniques

The curves obtained from the laser granulometric testing are shown in Fig. 1. The particle size distribution was represented by the cumulative percentages corresponding to 10, 50 and 90%. It was obtained that 10% of the waste is below 1,153  $\mu$ m, 50% below 6,211  $\mu$ m and 90% below 59,23  $\mu$ m.

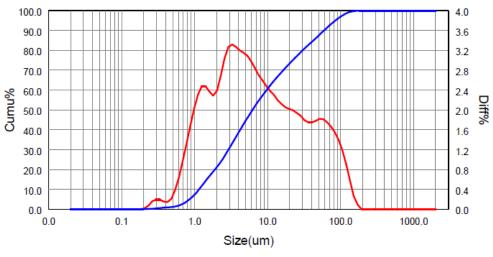


Fig. 1. Particle size distribution curves

The X-ray fluorescence showed, according to Table 1:

- high levels of Si and Fe;
- average levels of Al, K and Ca;
- low levels of Ti and S;
- traces of Mg, Mn, Sr, Zr, Zn, V, Y and Rb.

# Table 1 - Result of X-ray fluorescence

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Analyte	Result	
Si	37.652	8
Fe	25.983	8
Al	12.491	8
К	9.884	8
Ca	8.250	8
Ti	1.999	8
S	1.934	8
Mg	0.713	8
Mn	0.544	8
Sr	0.222	8
Zr	0.131	8
Zn	0.076	8
V	0.070	8
У	0.037	e S
Rb	0.013	80

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The high silica and aluminum content observed indicates that the material may exhibit pozzolanic activity when added to cementitious materials, since the main characteristic of pozzolan is the presence of silicates and aluminates in its composition.

The X-ray diffraction analysis revealed that the waste is formed largely of silica  $(SiO_2)$  in the form of quartz, indicating that this residue when crushed and added to the cement may exhibit pozzolanic activity. The minerals muscovite, albite and chlorite were also identified.

#### 3.2. Pozzolanic activity evaluation

According to the proposed Luxan method an initial conductivity reading of 8.99 mS/cm was performed and a final reading of 8.69 mS/cm. Thus, the pozzolanic activity index is 1.03, showing that the material exhibits moderate pozzolanicity. It was observed formation of precipitate material in the solution, suggesting the reaction between amorphous silica present at the residue powder and the free calcium ions ( $Ca^{2+}$ ), decreasing the concentration thereof and promoting the decrease in conductivity.

The compression test of NBR 5752, 1992 [4] resulted in an average values of compressive strength of the reference specimens and of the specimens with slate replacement of 15.37 MPa and 10.78 MPa, respectively. Thus, the pozzolanic activity index found is 70.1%. This shows that the mortar has not reached the pozzolanic activity index of 75%, not meeting minimum physical requirement imposed by the NBR 12653, 1992 [5]. However, the obtained value is close to the required.

### 4. Conclusions

The mineralogical and chemical analysis of the slate cutting residue showed that the material has a high percentage of silica and alumina in their composition, indicating that the material could exhibit pozzolanic activity if it is used in Portland cement-based products. However, pozzolanic evaluation results showed some diverging. By the Luxan method the material presented pozzolanic activity, however, by NBR 5752, 1992 [4] this property was not identified in the material. The difference in results may be explained by the possibility that in the Luxan test the calcium ions not effectively reacted with the material, only being attracted to the surface thereof. This implies that the decrease in conductivity is linked to the surface area of the material analyzed and not necessarily with the residue reactivity with the ion solution, reducing the reliability of the method. The results of this assay should be treated with caution in order not to misinterpret the conductivity decrease of the mixture.

The use of slate waste did not impair the final properties of the mortars, instead, replacing 35% of Portland cement for the recycled residue it was reached 70% of the reference mortar resistance. With this substitution, besides saving a significant amount of cement, reduce the production cost and volume of waste destined for landfills, it is

guaranteed up to 70% of the strength obtained without the addition. This value determined in this study was close to that required by the standard. So it is recommended deepening research in order to better understand the residue behavior in cementitious materials and the feasibility of its use as an alternative of mineral addition in the production of mortars and concretes with Portland cement.

The work has several advantages, since the potential for reuse and recycling of this waste is presented as a form of contribution to sustainable development, minimization of environmental damage, preservation of natural resources and obtainment of low-cost materials.

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