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STUDY OF MECHANICAL AND DURABILITY PROPERTIES OF MORTARS USING SLATE WASTE

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ABSTRACT

This study was focused on the addition of slate waste to Portland cement to evaluate the potential utilization as a mineral addition. The physical characterization of the residues was done by granulometric analysis and X-ray diffraction. The pozzolanic activity was evaluated by electrical conductivity, modified Chapelle, Luxan method and thermogravimetric analysis. Cement pastes and mortars produced with only Portland cement and with 14% addition of slate residues were evaluated by: capillarity, water absorption, Le Chatelier expansion, porosity, accelerated carbonation, compressive and tensile strength tests, and modulus of elasticity. The results showed that the residues have a high level of SiO_2, however they do not present pozzolanic activity. Cement composites formulated with slate residues showed increase or maintenance of the mechanical properties and contributed to a significant increase in the parameters indicative of durability. It was also verified that such analyses have not yet been reported in the literature.

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INTRODUCTION

In the State of Minas Gerais, the mining and beneficiation of ornamental minerals produce, altogether, around 2.0 million tons/year (Chiodi Filho e Chiodi, 2014), a production that generates a high quantity of waste as mining and beneficiation processes have a production ratio (ratio of total production after beneficiation to the amount of waste generated) lower than 15%. According to Chiodi Filho and Chiodi (2014), in the municipality of Papagaios, a major slate producer, nearly 100 million tonnes of slate waste have been piled up in approximately 137 piles of over the past years. In addition to the need to reuse such huge quantity of slate waste, the importance of reducing the quantity of clinker consumed in the fabrication of cement can be clearly identified. Clinker is a major component in the composition of some types of cements commercialized in Brazil that are responsible for 8% (of worldwide) CO_2 emissions (John, 2000). Naik (2005) and Davidovits (2013) state that with the production of one ton of cement, 0.95 tonnes of carbon dioxide are dispersed into the atmosphere. In face of such scenery, alternatives towards reducing the consumption of clinker have been searched for over the past years. Among several different lines of research, the use of waste residues as an addition to cement has acquired special notability (Masuero, Dal Molin and Vilela, 2004; Della, Vilela and Hotza, 2011). These additions have provided for new types of Portland cements that can improve mechanical properties and durability of cementitious composites. Santos *et al.* (2015) pointed out the technical viability of adding up to 20% in weight of slate waste into coating mortars. Watson, Grant and Jones (1976) *apud* Labib and Eden (2005) realized that the cement mortars added with slate waste, in 10 and 20% in weight, presented greater compressive strength due to the filler effect of the addition. Therefore, the great potential of bringing improvements to the performance (mechanical and durability properties) of cements with the use of additions, the immense availability of slate waste and the increasing need of adopting alternatives to reduce the consumption of clinker indeed make it viable for this study to analyze the addition of slate waste to cement in the proportion of 14% to formulate cement mortars for structural purposes.

MATERIALS AND METHODS

The cement mortar used in specimens was made using high initial resistance cement with low level of additions (5% of limestone powder) and the regular IPT/SP sand, in the following granulometry fractions: coarse (#16); medium coarse (#30); medium fine (#50) and fine (#100); and potable water from the region of Belo Horizonte. The slate waste was collected with the company ArdominasLtda, located in the city of Papagaios, in Minas Gerais. The material collected consists of a grayish fine powder, classified as gray slate (Chiodi Filho and Chiodi, 2014). The experiments program (Table 1) was started with the characterization of the slate waste, followed by a study of its pozzolanic activity.

Table 1. Tests performed and reference standards

Test	Procedures	Standard
Characterization	Sedimentation granulometry	NBR 7181/1984
	Diffraction and fluorescence of X-rays	
Pozzolanic Activity	Electrical conductivity	LUXAN *et al.* (1989)
	Modified Chapelle	NBR 15895/2010
	Pozzolanic Index with Cement	ASTM C311/2016
	Thermogravimetry	
Durability	Water absorption by capillarity	NBR 9779/2012
	Water absorption by immersion and porosity	NBR 9778/2005
	Soundness/expansion by Le Chatelier	BS EM 196-3/2016
	Images by digital microscope	
	Accelerated carbonation	RILEM CPC-18
Mechanics	Compressive strength	ASTM C39-01/1999
	Tensile strength by flexural test	ASTM C78-15/2015
	Dynamic modulus of elasticity	ASTM 215/2008

Next, cement mortars were made with and without the addition of slate waste as described in Table 2. After curing the specimens in a saturated environment, the mechanical properties were evaluated and some durability indicators of the cementitious composites were obtained. The characterization was done with the verification of granulometry by sedimentation in water in conformity with standard NBR 7181 (ABNT, 1984).

Table 2. Mixture of mortar samples studied

	Cement CPV	Slate	Sand	Water
Slate	1.000	0.163	3.448	0.558
Reference	1.000	0.000	3.000	0.480

The mineralogical and chemical analysis was made using a Philips (Panalytical) X-ray diffractometer (XRD), X'Pert-APD system, PW 3710/31 controller, PW 1830/40 generator, PW 3020/00 goniometer using Cu-Kα1 radiation, operating at 40 mA and 40 kV, scanning from 8 to 60 ° angle 2θ and steps of 0.02 °, and counting time of 1.0 second, and a Philips (Panalytical) X-Ray fluorescence spectroscopy device (XRF) PW 2400. Four different methods were used to verify the pozzolanic activity of the slate waste: measuring the electrical conductivity as proposed by Luxan*et al.* (1989); modified Chapelle method (NBR 15895/2010); determination of pozzolanic index with cement (ASTM C311/2016); and thermogravimetric analysis (TGA). The method developed by Luxan *et al.* (1989) estimates the pozzolanic activity of the sample by the variation in conductivity of the saturated $Ca(OH)_2$ solution after adding the sample. In this test, 5 grams of slate were added to 200 ml of a saturated $Ca(OH)_2$ solution heated at 40°C. The electrical conductivity was measured before adding the sample and also 2 minutes after the addition using a portable conductivity meter. The method of Luxan *et al.* (1989) classifies the pozzolanic activity of the sample according to variations in electrical conductivity in the $Ca(OH)_2$ solution, determining the material as non-pozzolanic when values are found to be lower than 0.4 mS/cm. The pozzolanic index with cement is calculated by establishing a relation between the average compressive strength of a cement mortar with the addition of waste and the compressive strength of specimens with no addition. A direct pozzolanic activity measurement is obtained with compressive strength; however, the standard NBR 5752 (ABNT, 2014) highlights the need to compare this result with other tests as, for instance, the modified Chapelle method.

The modified Chapelle method, as commended by standard NBR 15895 (ABNT, 2010), provides for the evaluation of the pozzolanic activity measuring the fixed CaO content. This test basically consists in diluting 1g of waste with 2g of powdered CaO into 500ml of deionized water at 90ºC, keeping it in water bath for 16 hours. The same procedure is symmetrically adopted in the blank experiment using only 2g of powdered CaO. The volumes of hydrochloric acid, which are required to neutralize the mix where the material and the reference sample are being tested, are then established. Based on the difference between the two HCl solution volumes consumed, the level of CaO fixed by the material is obtained. However, this norm does not determine a reference value to classify a material as pozzolanic, which requires literature searching. According to Raverdy *et al.* (1980) a material presents pozzolanic activity when the consumption of CaO is equal to or greater than 339mg/g. The products acquired with the hydration reaction of the cement mortar with and without addition of the waste were indirectly evaluated by thermogravimetry (TGA). In this test, the mass variation was measured according to the temperature, ranging from 30 to 800ºC, of both normal consistency paste samples, one as a reference paste and the other with the addition. According to Rêgo (2006) *apud* Cordeiro (2009), the mass variation rate regarding specific temperatures provides information on the content of calcium hydroxide in the sample. This analysis is particularly important to study the pozzolanic activity of the material as samples added with pozzolanic material tend to present reduced mass loss due to the decomposing of $Ca(OH)_2$ (Cordeiro, 2009). Therefore, the mass loss due to $Ca(OH)_2$ was compared between the samples with and without addition of slate waste.

Subsequently, the mechanical properties and durability of cement mortars and pastes were evaluated. The mechanical feature was evaluated according to compressive strength (ASTM C39-01/1999), and tensile strength measured by flexural test (ASTM C78-15/2015) and dynamic modulus of elasticity by forced resonance (ASTM 215/2008). A 2-ton-load Instron Universal Test Machine was used in the first two tests. In the final test the Erudite MKII Resonant Frequency Test System equipment was used on a specimen of 10 centimeters in diameter and 20 centimeters in height. The durability was evaluated by porosity and capillarity (NBR 9779/2012 and NBR 9778/2005) and the carbonation of the cement mortars, while also testing the pastes in expansion by Le Chatelier method (BS EM 196-3/2016) which provides for the verification of expansion due to free lime. The carbonation test was made using a Thermo Fisher Scientific, RCO 3000 T-5 VBC model incubator. Three specimens with and without slate waste with dimensions of 4x4x16cm were utilized under dried conditions. The samples were kept for 55 days in the incubator under humidity of 100%, temperature of 25ºC and 5% of CO_2.

After 55 days the samples were longitudinally sectioned and aspersions were made with a solution composed of 70% absolute alcohol, 29% of distilled water and 1% of phenophtalein. Subsequently, a caliper was used to measure the four edges of each surface. Water absorption, porosity and absorption by capillarity were measured by obtaining the quantity of water that entered in the samples and verified with 0.001g scales. A 1000x digital microscope was also used to analyze surface images of cement mortars with and without slate waste to better express the behavior of the matrix and aggregates in these composites.

RESULTS

Characterization of the slate waste: Figure 1 and table 3 show the results of the granulometry analysis of sedimentation of the slate waste. It was verified that approximately 53% of the particle sizes of the material analysed are smaller than 10 µm, as found in the study of Catarino*et al.* (2003), in which the slate is shown to be adequate for the fabrication of ceramic roof tiles and, also, in the studies of Gonçalves (2000), who analysed waste residues of rocks as filler in concrete and mortars. Additionally, the slate waste granulometry found is like the granulometry of the cement being used (average diameter of nearly 12 µm).The data displayed in table 4 ND Figure 2 demonstrates that the waste has high level of SiO_2, a result similar to that described by Chiodi Filho, Rodrigues and Artur (2003).

Table 3. Diameter below which 20% (D_{20}), 50% (D_{50}) and 80% (D_{80}) of slate residues were found

D_{20}	D_{50}	D_{80}
2.40 µm	9.15 µm	61.53 µm

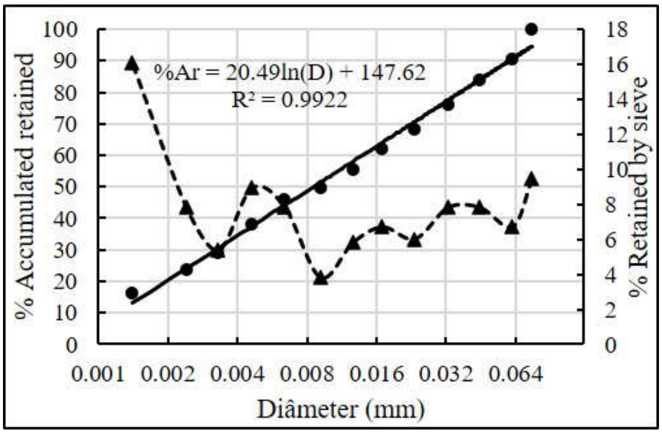

Figure 1. Slate sample granulometry distribution

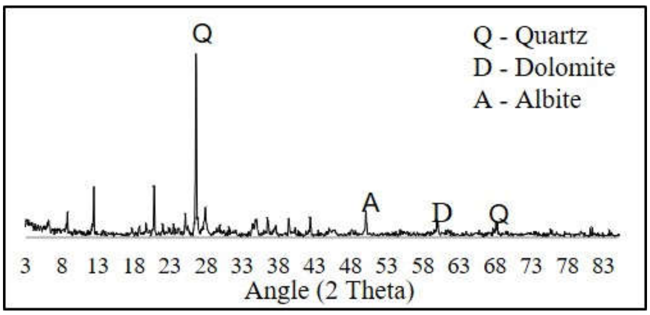

Figure 2. XDR - X-ray diffraction of slate samples

Evaluation of the pozzolanic activity: The results of the pozzolanic activity tests carried out on the slate are shown in Table 5. The values obtained by the Modified Chapelle method and the method proposed by Luxan*et al.* (1989) suggest that the sample analyzed presents no pozzolanic activity.

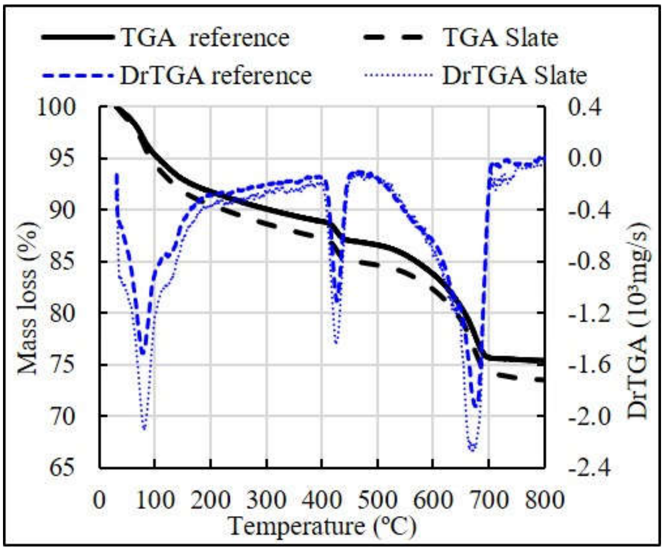

Figure 3. Curve of mass loss in function of time and its derivative

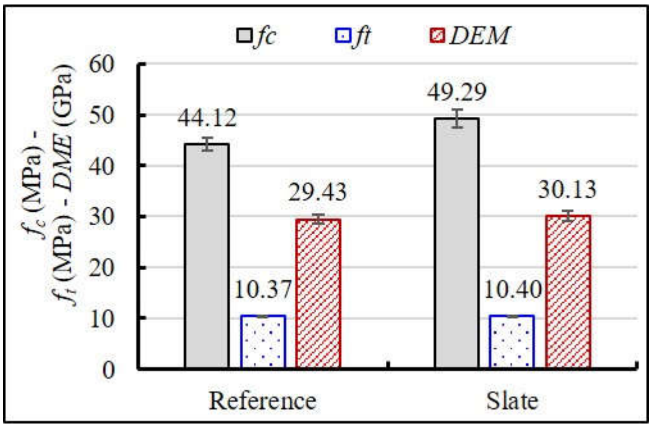

Figure 4. Results of mean values and standard deviation of tensile strength (f_t) and compressive strength (f_c) and dynamic modulus of elasticity (*DME*) tested at 28 days

In fact, Fríaset *al.* (2014) verified that the slate residue presents good pozzolanic activity only when it is activated at temperatures ranging from 800 to 1100°C. The gains in strength verified by the pozzolanic index test might indicate that it is a pozzolanic material. However, based on these data, this compressive strength should be related to the filler effect promoted by this material, which would make the cementitious matrix more compact and resistant; or also act as a nucleating agent, which would provide for an increase in compressive strength (Labib and Eden, 2005). The reactivity of the waste residues with calcium hydroxide was also measured by thermogravimetric analysis (TGA). The TGA results, jointly with the derivative data (DrTGA), are presented in figure 3. Table 6 contains the data from the analysis made on mass loss percentages. As shown in Figure 3, the curves are similar to those described in thermal analyses on the hydration of Portland cement pastes and Portland cement additions (Karen *et al.*, 2015; Zhutovsky and Kovle, 2013) found in the literature. Thus, the presence of the slate waste caused no alterations in the chemical composition of the cementitious composites.

Table 4. Semi-quantitative chemical composition of the slate waste

XRF		XRD		
		Mineral	Composition	Content
		Dolomite	$CaMg(CO_3)_2$; trigonal	Abundant
Element	Content	Quartz	SiO_2; trigonal	Medium
Si, O	High	Albite	$NaAlSi_3O_8$; triclinic	Medium
Al, Fe, K	Medium	Orthoclase	$KAlSi_3O_8$; monoclinic	Medium-low
Ti, Ca, Na	Low	Muscovite	$KAl_2Si_3AlO_{10}(OH)_2$; monoclinic	Medium-low
Mg, P, S, Cu, Ce, Zr, Rb, Sr	Mixture	Calcite	$CaCO_3$; trigonal	Medium-low

Table 5. Results of the pozzolanic activity tests and reference values

Testing	Results	Reference values
Modified Chapelle	83.76 mg/g	> 330 mg/g (RAVEDRY *et al.,* 1980)
Method of electrical conductivity	0.392 mS/cm	> 0.4 mS/cm (LUXÁN *et al.*, 1989)
Pozzolanic Index	84%	>75% (NBR 5752/2014)

Table 6. Percentage of mass loss by the thermogravimetric analysis

Mass loss (%)	Temperature (ºC)			
	30-105 (H_2O free)	110-180 (C-S-H)	410-580 ($Ca(OH)_2$)	520-900 ($CaCO_3$)
Reference	5	3	2	5
Slate	6	4	2	5

As the pozzolanic reaction is characterized by the formation of C-S-H upon the addition of $Ca(OH)_2$, samples with the addition of pozzolanic materials are expected to confirm greater mass loss relatively to C-S-H dehydration intervals and also reduced mass loss in ranges where the decomposition of portlandite occurs (Alonso and Fernandez, 2004; Rodrigues, Beraldoand Savastano Jr, 2010; Alarcon-Ruiz *et al.,* 2005; Mitchell and Margeson, 2003). Table 6 clearly demonstrates that the mass loss respectively to both C-S-H and Ca(OH) in the reference paste is equivalent to that found in the paste added with slate, thus proving one more time that the slate waste presents no pozzolanic effect. Some studies (Frias*et al.,* 2014; Barluenga and Hernández-Olivares, 2010 and Campos*et al.,* 2004) analyzed the activation of slate waste as addition in concretes, mortars or for the fabrication of roof tiles, finding that the material can possibly have a pozzolanic activity at 1000ºC. In this study, we opted for the material found in the waste storage piles, without any changes, using only the coarse fraction (larger than 0.075mm) retained on sieve (approximately 10% of the mass of the material). Therefore, the use of this material produces no cost (material and energy costs) increases.

Mechanical and durability analysis of cementitious composites with addition of slate waste: Figure 4 displays the results (mean values and standard deviation) of tensile strength, compressive strength and dynamic modulus of elasticity tests performed with the reference cement mortars and also with those added with slate waste residues. The data obtained with compressive strength tests demonstrate that the cement mortar with addition presented an average increase of 11.8% in relation to the reference samples (with no addition). In the tensile strength measured by flexural tests both mortars presented the same mean values, which indicates that despite the increase in compressive strength the addition of the slate waste residue produces no alterations in flexural strength. Based on these data and on the indication that the slate waste residue presents no pozzolanic activity, an inference of the filler effect of the waste can be made (Pan and Weng, 2012; Peng, Hu and Ding, 2009; Kadri and Duval, 2002; Sahmaran*et al*., 2009).

The dynamic modulus of elasticity values of the reference sample, which was calculated by measuring the resonant frequency, was similar to those found by Sales *et al.* (2010).

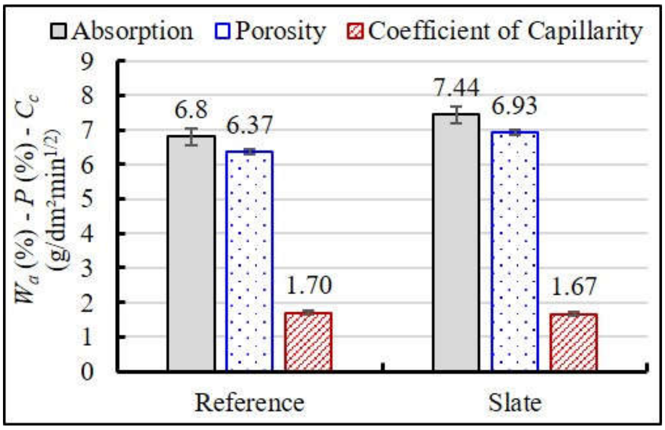

Figure 5. Results of water absorption by immersion (W_a), surface porosity (P) and coefficient of capillarity (C_c) of reference mortars and with addition of slate residues

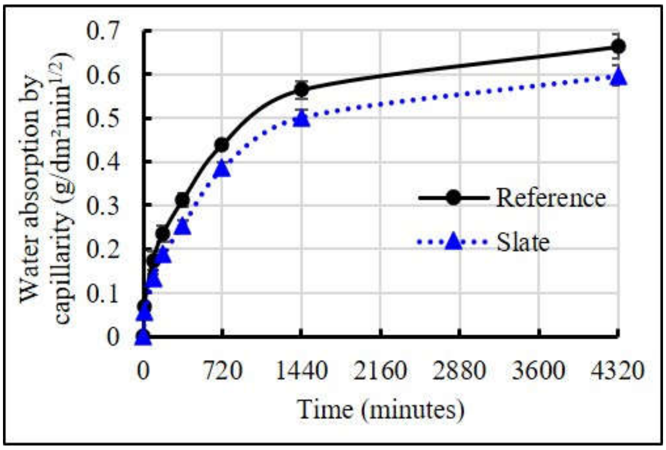

Figure 6. Result of water absorption by capillarity of reference mortars and addition of slate residue in function of time

With the expansion tests made with the Le Chatelier needles, it was found that the openings of three needles were inferior to

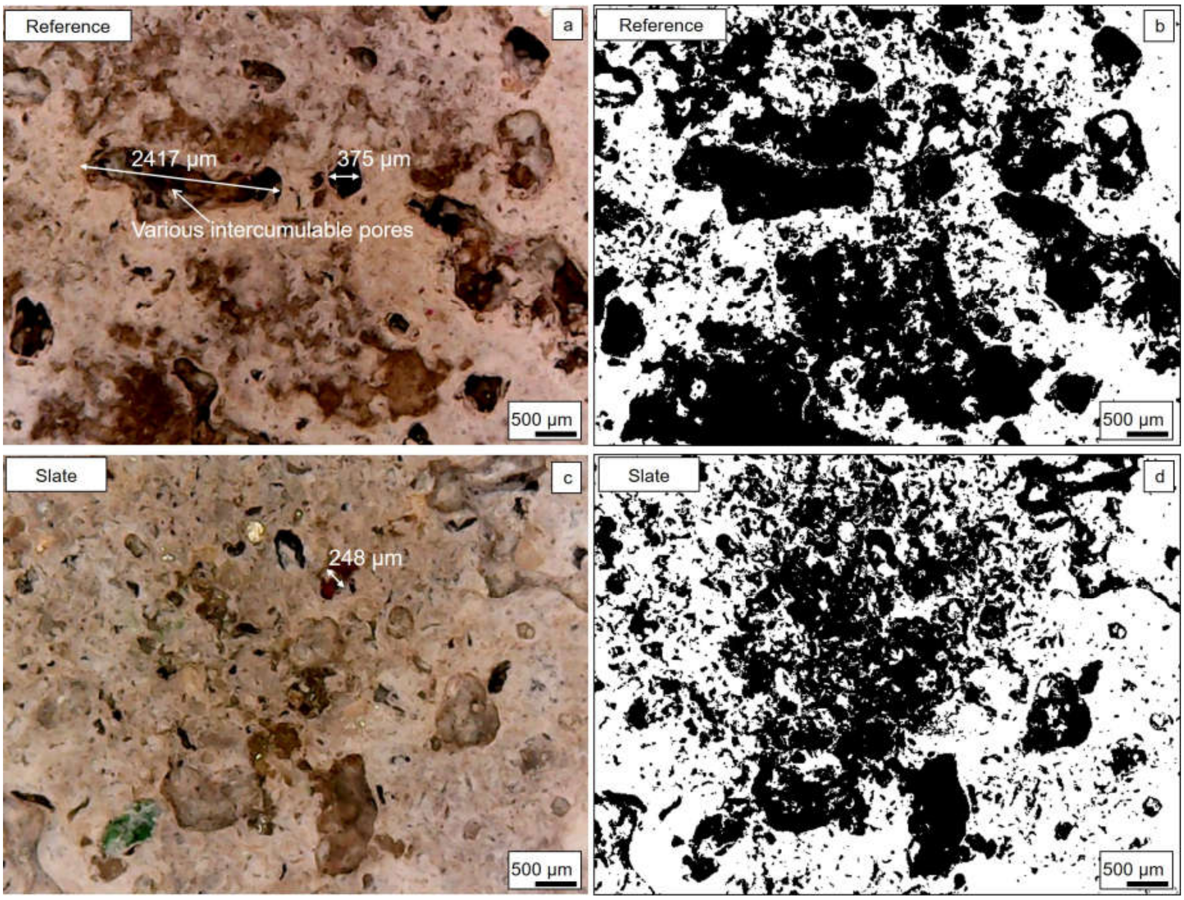

Figure 7. Digital microscope image of (a) reference mortars and (c) with addition of slate residue and (b and d) image treatment to enhance pores

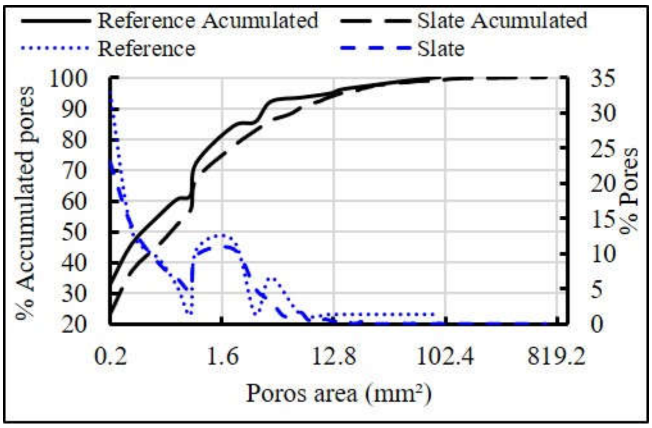

Figure 8. Digital microscopic image of reference mortars and with addition of slate residue

1mm in both samples, which demonstrates that the cement with or without this addition complies with the standards established in NBR 11582 (ABNT, 2012). The results of water absorption by immersion, and porosity and capillarity tests are presented in figures 5 and 6. In figure 5, it can be noted that water absorption by immersion in the reference cement mortars was slightly lower (8.6%) than in the mortars added with slate waste. Even though this difference is not significant (test *t*), it demonstrates that the similarity in size between the cement particles and the slate waste particles provide for a similar conformation of the cementitious matrix and of its interface with the aggregate, which generates similar values for this property. The number of pores and their distribution are relative to the proportion of mixture, the cure and the presence of mineral additions (Kim *et al.*, 2014; Kabay*et al.,* 2015; Kumar and Bhattacharjee, 2003).

In this study, it was found that the addition produced a cement mortar 8.8% more porous. However, it was also found that such fact did not significantly influence that property (test *t*). When the coefficient of capillarity is analysed, an inversion of the results can be noted as the reference mortar coefficient is found greater (1.8%) than that of the mortar with addition. It can be observed that the pores are slightly smaller in the mortar without addition, and that their distribution provides for a greater continuity of the pores, which consequently produces an improvement in this property of cement. Figure 7, which presents macrostructure images of mortar, corroborates the preceding analysis indicating that in the reference mortar the pores tend to have a greater number of intercumulable pores, with sizes and distribution that contribute more effectively to water intrusion by capillarity, as shown in Figures 7 ((a), (b),

(c) and (d)) and 8. The images provide for a visual analysis of the morphology of the pores (irregular) and the paste-aggregate arrangement through digital microscopic images. The images can be analysed and worked on the ImageJ Software, which furnishes results of the pore size distribution (Figure 8). The macrostructure data reassure porosity and water absorption results: the pore sizes in the samples with and without addition are similar. However, the reference mix presents a larger pore network (several pores close to each other, forming conducts), which explains the augmented capillarity. An analysis of figures 7 ((a), (b), (c) and (d)) and 8 shows that the use of addition refines the pores and reduces their conductivity and that, as already shown in figure 6, porosity and water absorption remain equal, while also reducing capillarity and producing a cement mortar with prospects of longer durability, thus corroborating bibliography (Andrade and Tutikian, 2011; Gonçalves, 2000). The results acquired in the accelerated carbonation test are very similar: carbonated layers with depths of 1.5±0.2 mm and 1.4±0.3 mm, respectively for the reference mix and the mix with slate waste addition. It is worth noting that a large variability of results was obtained, even though the values remained at low levels when compared to those of 25 mm listed in the norm NBR 6118/2014 and by Neville and Brooks (2013), who state that the carbonation rate depends on the permeability of the composite, on its humidity and CO_2 levels and on relative humidity in the environment. Cascudo and Carasek (2011) understand that the greater the addition content employed, the greater is the depth of the carbonated layer, which is explained by a drop in the alkaline reserve due to a reduction in the content of cement. This study verified that the values remain the same, regardless of the addition, which shows that the cement mortar with addition of slate waste is of good quality and durability.

Conclusions

The results demonstrate that the waste – with the characteristics of the waste found in the region of Papagaios – presents a similar granulometry to that of the cement, and also has a potentiality to be used as a filler-type addition to Portland cement. Mechanical tests and durability tests to validate this product were made on cementitious composites.

The results obtained provide for the following conclusions:

- The slate addedand the cement have similar granulometry;
- The slate waste under analysis presents no pozzolanic activity, as shown by the results obtained from the modified Chapelle test, the electrical conductivity test proposed by Luxan*et al.*(1989), and the thermogravimetric analysis.
- The addition of waste led to an increase in compressive strength, while not significantly impacting the tensile strength or the modulus of elasticity of the cementitious composites.
- The expansion assessed by the Le Chatelier test faced no different effects with the addition of slate waste.
- No significant difference in water absorption by immersion, capillarity or porosity was found. The slight differences identified due to the refining of pores and to nucleation promoted by the addition lead to improvements in the performance of cementitious composites.
- The results indicate that the addition of slate waste enhances a filler effect in the composite.

Therefore, the 14% addition of slate in a cement mortar did not weaken any of the mechanical properties, or the durability of the cementitious composites, inclusively producing small improvements to some of these properties. So, the addition of slate waste to cement mortar contributes to reductions in environmental impacts by reducing the production of clinker and by utilizing materials that are an environmental liability, such as the slate waste. It is important to note that this addition enables the formulation of environmentally friendly cement and provides economic benefits with the use of low cost materials that require no processing to be used.

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