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Contribution to the rheological study of cementitious pastes with addition of residues from the processing of ornamental rocks

Contribuição ao estudo reológico de pastas cimentícias com adição de resíduo do beneficiamento de rochas ornamentais



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Abstract

Brazil is one of the world's largest producers and exporters in the field of ornamental stones. On the other hand, the production and processing of ornamental stones result in a large volume of unused material in the form of sludge, usually discarded inappropriately in sedimentation ponds or landfills. Several researches have been carried out aiming the reuse of this material in cementitious matrixes. In the field of rheology, there are still incipient national studies that use the rheological parameters obtained experimentally to determine the behavior of cement matrix based on Portland cement. Thus, the objective of this work is to characterize rheologically the behavior of cementitious pastes with and without addition of ornamental rock processing residue (RBRO) in its natural condition. Cement pastes were prepared with three a/c ratios (0.45, 0.55, 0.65) and four residue addition contents (0%, 5%, 10%, 15%) and submitted to the flow test. In tests for characterization of the residue, the RBRO presented as a material of specific fineness and mass near the cement, having low reactive activity, indicating that the residue can be used as an inert mineral addition in the cementitious matrix. In the rheological characterization tests of the pulps studied, it was observed in the flow tests that the samples behaved as a non-Newtonian, pseudoplastic and thixotropic fluid.

Keywords: ornamental rock waste, cement pastes, rheology.

Resumo

O Brasil é um dos grandes produtores e exportadores mundiais no setor de rochas ornamentais. Em contrapartida, a produção e o beneficiamento de rochas ornamentais resultam em grande volume de material não aproveitado em forma de lama, geralmente descartados de maneira inadequada em lagoas de decantação ou aterros. Diversas pesquisas têm sido realizadas objetivando o reaproveitamento deste material em matrizes cimentícias. No campo da reologia, ainda são incipientes estudos nacionais que utilizem os parâmetros reológicos obtidos experimentalmente para determinar o comportamento de matrizes cimentícias a base de cimento Portland. Contudo, o objetivo desse trabalho é caracterizar reologicamente o comportamento de pastas cimentícias com e sem adição de resíduo do beneficiamento de rochas ornamentais (RBRO) em sua condição natural. Foram fabricadas pastas cimentícias com três relações a/c (0,45; 0,55; 0,65) e quatro teores de adição do resíduo (0%, 5%, 10%, 15%) e submetidas ao ensaio de fluxo. Em testes para caracterização do resíduo, o RBRO se apresentou como um material de finura e massa específica próximas do cimento, possuindo baixa atividade reativa, indicando que o resíduo pode ser utilizado como adição mineral inerte na matriz cimentícia. Já em ensaios de caracterização reológica das pastas estudadas, observou-se nos ensaios de fluxo que as amostras se comportaram como um fluido não newtoniano, pseudoplástico e tixotrópico.

Palavras-chave: resíduo de rochas ormamentais, pastas cimentícias, reologia.

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1. Introduction

The preliminary questions of this research revolve around the characterization and use of the ornamental stone residue (OSR) generated during the production and processing of ornamental rocks, named by the State Institute of the Environment and Water Resources (IEMA) as sludge from the processing of ornamental rocks, as alternative material in the production of concrete. According to the Brazilian standard for solid residues NBR 10004 [1], the solids from this mud were classified as class II A - non-inert residue.
Solid waste from this primary processing of ornamental rocks is generally discarded in decantation ponds or landfills and often without a treatment process to eliminate or reduce its constituents. Due to the large amount produced, environmentalists accuse industries of this sector as sources of contamination and / or pollution of the environment making these wastes an environmental problem [2].
Therefore, it is necessary to take advantage of the residue from ornamental stone processing (OSR) in Brazil, and especially in the state of Espírito Santo. In view of this, it is extremely important to be concerned with studies that make the ornamental stone sector more environmentally sustainable, giving a nobler destination to the rejects produced by it and also making it possible to use its waste materials from a technical point of view, guaranteeing the quality and durability of the product that will incorporate them.
Some studies have pointed out the reuse of OSR as an alternative material in several areas and applications, such as mortar [3], ceramic coating [2], interlocking floor [4], among others. Since much of this waste is recycled in the form of an industrial by-product, one way of contributing to the use of OSR is in concrete fabrication [5-12].
On the other hand, concrete is a material that requires special attention, from the specification phase to its curing time, since it often has a structural function. Through the Brazilian Association of Technical Standards (ABNT) we obtain the standard NBR 7212 [13], which determines the time limit to transport and pour concrete, which is 150 minutes between the start of mixing. If this time is exceeded, the concrete may present loss of workability due to water loss due to evaporation and start the setting time due to the hydration reactions of the cement with water.
For the technological control of the concrete and the verification of its workability in its fresh state, a commonly used and performed test is the Slump test, following the Brazilian standard NBR NM 67 [14]. However, some properties of the concrete (shear stress, yield stress and viscosity) are not possible to obtain through this test, being necessary the search of more information about these properties for a better understanding of its behavior in several applications.
In general, the properties of the concrete in its fresh state are directly related to the behavior of the cement paste [15]. One way of analyzing this behavior is through rheology, a branch of physics first suggested by Bingham [16] to describe the deformation and flow of materials when subjected to forces from external forces [17-20].
For the determination of the rheological parameters of the cementitious paste, such as yield stress, apparent viscosity, rheological profile of the material and also the consolidation effects of this material associated to the hydration of the cement, the literature proposes some tests such as, for example, flow test, oscillatory test (time sweep and strain scan) and vane test or Vane test [11; 21-25].
According to De Larrard et al. [26], the use of the rheometer is not only intended to measure as many parameters as possible, but also the physical quantities that may be scientifically related to the different stages of concrete use. As an example, one can cite the yield stress, which indicates the minimum tension required for the fresh concrete to deform. Reis [27] states that this tension is directly related to the slump test. The higher the yield stress of a cementitious matrix, the lower the slump obtained by the traditional slump test of

Table 1
Physical and chemical characterization of Portland Cement type CP V ARI

Property		Result	Standard method	Limits
Specific mass (g/cm³)		2.823	NBR NM 23 (ABNT, 2001)	N.E.
Finness	Blaine specific area (cm²/g)	4459	NBR NM 76 (ABNT, 1998)	≥ 3000
	Material retained in no 200 sieve (%)	0.1	NBR 9202 (ABNT, 1985)	N.E.
Setting time	Initial set (min)	128	NBR NM 65 (ABNT, 2003)	≥ 60
	Final set (min)	181	NBR NM 65 (ABNT, 2003)	≤ 600
Resistance to compression	1 day (MPa)	29.4	NBR 7215 (ABNT, 1997)	≥ 14
	3 days (MPa)	42.2	NBR 7215 (ABNT, 1997)	≥ 24
	7 days (MPa)	46.0	NBR 7215 (ABNT, 1997)	≥ 34
Chemical composition	Fire test, loss (%)	3.84	NBR NM 18 (ABNT, 2001)	≤ 4.5
	SiO_2 (%)	18.65	NBR 14656 (ABNT, 2001)	N.E.
	CaO (%)	63.72	NBR 14656 (ABNT, 2001)	N.E.
	MgO (%)	0.75	NBR 14656 (ABNT, 2001)	N.E.
	Al_2O_3 (%)	4.91	NBR 14656 (ABNT, 2001)	N.E.
	Fe_2O_3 (%)	2.97	NBR 14656 (ABNT, 2001)	N.E.
	K_2O (%)	0.80	NBR 14656 (ABNT, 2001)	N.E.
	SO_3 (%)	2.87	NBR 14656 (ABNT, 2001)	≤ 4.5
	C_3A teórico	7.66	NBR 14656 (ABNT, 2001)	N.E.
	Insoluble residue (%)	0.75	NBR NM 15 (ABNT, 2012)	≤ 1.0

Note: limits established based on NBR 5733 (ABNT, 1991); N.E. = not established.

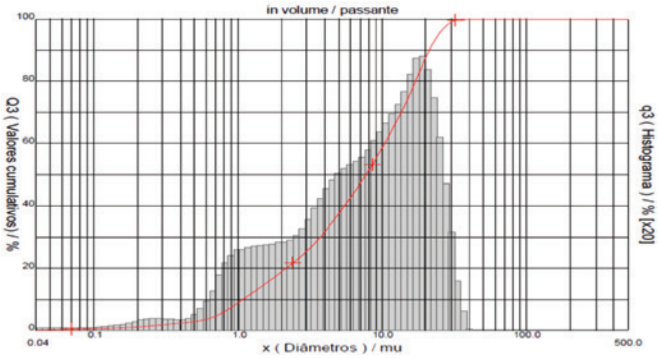

Figure 1
Size distribution of Portland Cement type CP V ARI

the concrete in its fresh state. Another rheological parameter is the apparent viscosity, which indicates the increase in tension that must be imposed to produce a given shear rate. Apparent viscosity is in practice associated with the application, pumping, segregation and finishing capabilities, making it easier to distinguish a workable concrete from a "sticky" one, with difficulties in pumping and voids on the surface of the element when the form is removed [15].
Despite the increasing number of studies aiming to incorporate the ornamental stone residue, most of these studies focus on the mechanical behavior of concrete. The study of the rheological behavior with addition of these wastes is still incipient.
Thus, in general, this work seeks to make feasible the use and use of the RBRO in civil construction, incorporating it in concrete as an addition to minimize the environmental impacts caused by the ornamental stone industry and also to reduce the costs due to the possible reduction in the volume of Portland cement required in the fabrication of concrete.

2. Objectives

The present work aims to use rheology concepts to investigate the influence of the incorporation of ornamental stone residues on the rheological properties of cement pastes, analyzing the interactions that occur between this waste and the other constituents of the pastes (cement and water, in this study). Above all, it is investigated how the filler effect of this waste material can alter the analyzed rheological parameters, since it becomes a high fineness powder after its preparation process (drying, dewatering, quarrying and grinding).

3. Materials and methods

3.1 Cement

Portland cement classified as high initial strength Portland Cement (CP V ARI) according to the Brazilian standard NBR 5733 [28] was used in this research. The cement used was donated by a company located in the municipality of Serra - ES. The chemical characterization of the batch of cement used was provided by the manufacturer and its physical characterization was performed in the laboratory, as presented in Table 1.
The particle size distribution of the cement was performed using a laser diffraction particle sizing equipment model 1064 - CILAS. Figure 1 shows the particle size distribution curve of the cement.
Through the particle size distribution results, it is possible to observe that the cement grains have D10 values in the order of 1.08µm, D50 in the range of 7.68µm, D90 with 21.15µm, 100% is below 36.00µm, with average value for grain size in the order of 9.64µm.

3.2 Ornamental Stone Residue (OSR)

The residue from the processing of ornamental rocks is supplied in the form of mud, without previous treatment, and it was donated by a company located in Serra - ES. The residue comes from the processing of ornamental stone blocks using diamond wire looms and from the polishing of the rock steps. The material was collected in different warehouses of company, following the Brazilian standard NBR 10007 [29], and stored in 20-liter plastic tanks to transport to the laboratory.
The material presented high humidity (38.3%), requiring air drying for 48 hours prior its use as an addition and/or substitution in the studied cement pastes, for better sampling and obtaining a homogeneous mixture. After drying, the residue passed through a sampling procedure according to the Brazilian standard NBR 26 [30]. After collection, the samples were distributed in trays and dried in an oven for another 24 hours.

3.2.1 OSR characterization

a) Physical characterization
The tests were carried out to determine the specific mass, unit mass, specific surface, fineness and grain size distribution of the ornamental stone residue. The results are described in Table 2.

Table 2
Physical characterization of OSR

Test method	Material property	Results	Limits
NBR NM 23 (ABNT,2001)	Specific mass (g/cm³)	2.5253	N.E
NBR 12127 (ABNT, 1991)	Unity mass (g/cm³)	0.766	N.E
NBR NM 24 (ABNT, 2002)	Initial moisture content (%)	38.27	N.E
NBR NM 24 (ABNT, 2002)	Used OSR mositure content in this study (%)	0.04	≤ 3.0
NBR NM 76 (ABNT, 1998)	Blaine specific area (cm²/g)	6179.3	N.E.
NBR 11579 (ABNT, 2012)	Material retained in n° 200 sieve (%)	3.08	N.E.
NBR 9202 (ABNT,1985)	Material retained in n° 325 sieve (%)	7.0	≤ 34

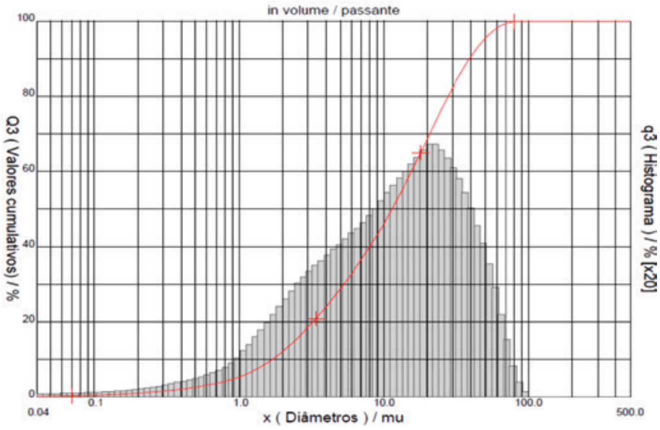

Figure 2
OSR size distribution

The specific mass of the RBRO, 2.525 g/cm³, is 10.5% less than the specific mass of the CP V ARI cement (2.823 g/cm³).
The specific area is an important material characteristic for mineral additions. When comparing materials of the same chemical composition, the larger the surface area of the material, the greater the possibility of reaction with the calcium hydroxide of the cement paste due to its contact area with the reagents [31]. The specific area of the RBRO (6179 cm²/g) is larger than that of the CP V ARI cement (4459 cm²/g), which indicates that the residue has finer particles than the cement.
The size distribution is shown in Figure 2, the grain size equivalent to D10 (diameter below which 10% of the particles are located) in the order of 1.7 µm, D50 in the order of 11.0 µm and D90 in the order of 40 µm. According to Gonçalves [10], the fine aggregate usually has a mean diameter between 50 and 150 µm and fills the voids left by the discontinuity of the small aggregate. The residue with potential filler effect, on the other hand, has an average diameter of less than 50µm and acts to fill the pores left by the cement hydration products [10]. It is possible to verify that the residue OSR is a pulverulent material, thinner than the cement, that can have filler effect in cement matrices.

b) Chemical characterization
Chemical characterization of OSR were performed by means of the fire tests and X-ray fluorescence spectrometry (FRX), as shown in Table 3. The OSR is composed predominantly of silica quartz (SiO_2) and by feldspar alumina (Al_2O_3) being, therefore, a silico-aluminous material. From the X-ray fluorescence results, the predominance of silica quartz (SiO_2) and, secondarily, of the feldspar alumina (Al_2O_3) is evident, evidencing the silica-alumina residue. At lower levels, minerals from the group of micas (Na_2O and K_2O) are presented, which are present in granitic rocks. The presence of magnesium oxide (MgO) is possibly due to the wear of polishing shoes which are made from sorel cement, formed by magnesium and chlorine oxides typically used in the manufacture of abrasive crowns [32]. As for the fire loss, the value found can be related to the polishing materials, such as waxes and resins, since the residue sample studied has both the mud coming from the cut of the blocks and the slurry of the polishing of the stone sheets.

c) Pozzolanic activity index
NBR 12653 [33] defines as pozzolan a silica or siliceous-ammonium material that alone has little or no cementitious property. However, at room temperature and in the presence of moisture, it can react with calcium hydroxide to form compounds with cementitious properties. Uliana [34] and Soares [35] found that the OSR does not meet the requirements of NBR 12653 [33] in its aspect of verifying pozzolanicity in mortars, both with hydrated lime and Portland cement. In this way, OSR can be used as a filler in mortars and concretes, and it can employ a physical effect of filling voids and densification of the mixtures.

3.3 Studied cement pastes

A total of twelve (12) cement pastes were studied, varying the percentage of residue added in relation to the cement mass and the w/c ratio. A nomenclature was defined for each sample to be studied in order to distinguish more easily, starting with the cement paste without residue (reference paste), followed by another three percentages of addition of the residue with respect to the cement mass (5%, 10%; 15%) and three different w/c ratios

Table 3
OSR chemical composition

Propriedades		RBRO	Limites
Chemical composition	SiO_2 (%)	66.80	N.E.
	CaO (%)	3.44	N.E.
	MgO (%)	0.93	N.E.
	Al_2O_3 (%)	13.50	N.E.
	Fe_2O_3 (%)	3.79	N.E.
	K_2O (%)	3.83	N.E.
	Na_2O (%)	3.50	N.E.
	C (%)	1.11	N.E.
	TiO_2 (%)	0.16	N.E.
	SO_3 (%)	0.06	N.E.
	$SiO_2 + Al_2O_3 + Fe_2O_3$ (%)	85.08	N.E.

Note: limits established based on Pozolana Class E data described in NBR 12653: Pozzolanic Materials - Requirements (ABNT, 2014); N.E. = not established.

Table 4
Sample's nomenclature

Sample	OSR percentage (%)	w/c ratio
C REF 45	0	0.45
C R5 45	5	
C R10 45	10	
C R15 45	15	
C REF 55	0	0.55
C R5 55	5	
C R10 55	10	
C R15 55	15	
C REF 65	0	0.65
C R5 65	5	
C R10 65	10	
C R15 65	15	

(0.45, 0.55, 0.65). Table 4 presents the nomenclatures adopted, where C means cement, R is the residue followed by the percentage number added and the last number is the w/c ratio.
The residual and w/c ratios used were based on the work of Soares [35], who studied the influence of the addition of residues on the workability and mechanical properties of concrete in the fresh and hardened state with emphasis on the analysis of concrete durability. In order to correlate the slump testing results found by Soares [35] with those presented in this study, the w/c ratio and percentage of residues were kept the same.

3.4 Determination of the mixing procedure for the production of cementt pastes for rheological testing

It is critical to define a mixing procedure to be used in tests using the Dynamic Shear Rheometer (DSR) so that the sample is homogeneous before performing the rheological tests. According to some researchers, the mixing time is one of the factors that influence the paste homogenization. França et al. [36,37] have shown in their research that samples with longer mixing times demonstrate more efficiency, producing more fluid and stable mortars. The cement paste with higher viscosity, lower w/c ratio and higher percentage of OSR residue (C R15 45) were chosen for the analysis to define mixing process, as it would be the paste with greater difficulty to homogenize.
Then, several tests were performed to analyze and define the best mixing procedure for sample preparation. It was based on a modification of the blend method developed by the Texas Transportation Institute, based on the study conducted by the Portland Cement Association and later by the National Institute of Standards and Technology [38]. After this process, the samples were submitted to the 3-cycle flow test on the rheometer and analyzed.
A high-shear mechanical mixer (14-speed, 450-watt 6855 Oster mixer) was used in this research as it was done by [38]. Features stainless steel blades, heat-proof glass cup tested to withstand extreme temperature changes. The masses of the materials were established in a precision electronic digital scale of 0.01g.

3.4.1 Determination of mixing time for sample fabrication

The first factor to be defined in the sample preparation procedure was the mixing time in the mixer. In this study, two effective mixing times were analyzed: (a) 100 seconds and (b) 300 seconds. The mixing procedure to be followed was the same for the two times, the only difference was the time the mixer was turned on and using only the first two speeds of the mixer. Firstly, the amount of water, cement and residue were weighed separately to the nearest 1.0 g. Then, the cement and the residue were placed in the mixer first and then the water was added. After mixing the materials, the mixer was turned on at the first speed for (a) 30 seconds or (b) 90 seconds depending on the actual mixing times of 100 seconds or 300 seconds, respectively, and then the equipment was turned off for 60 seconds to manually clean the walls of the mixer cup. Then the appliance was turned back on at the second speed for (a) 30 seconds or (b) 90 seconds and then off for another 60 seconds to clean the mixer bowl. Finally, the cement paste was mixed for (a) 40 seconds or (b) 120 seconds at the second speed so as to obtain a homogeneous paste.
The total time of the mixing process was (a) 3 minutes and 40 seconds for the procedure with 100 seconds of effective mixing and (b) 7 minutes for the procedure with 300 seconds of effective mixing (Table 5).

3.4.2 Cycles flow tests

Immediately after each mixing process (a) and (b), the flow test was performed with 3 consecutive cycles (acceleration from 0 to $100s^{-1}$ for 2 minutes and deceleration from 100 to $0s^{-1}$ for a further 2 minutes, totaling 12 minutes of testing, as shown in Figure 3. What is expected is that after each cycle, the same result will be obtained for the rheological parameters thus showing the efficiency of the mixing procedure for dispersion of the particles. Otherwise, this dispersion would occur during the test, which is not desired.
Through the stress versus shear rate graph generated by the test performed on the rheometer, it is possible to calculate the hysteresis area (stress difference between the acceleration curve and the deceleration curve), which indicates the dispersion state of the

Table 5
Mixing procedure for 100 e 300 seconds

Mixing time	Low speed mixing (V1)	Stopping and cleaning	Mixing at low speed (V2)	Stopping and cleaning	Mixing at low speed (V2)	Total time
100 seconds	30s	60s	30s	60s	40s	3 minutes and 40 seconds
300 seconds	90s	60s	90s	60s	120s	7 minutes

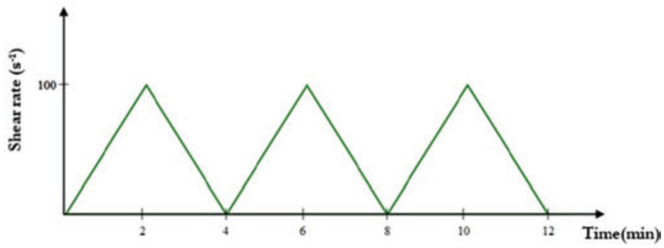

Figure 3
Acceleration and deceleration ramp for 3-cycles flow test

sample. The results of this initial analysis for determination of the mixing time are shown in Figure 4 and Table 6.
It was observed that, although the shear stress values for samples mixed in 100 seconds were greater than those mixed with 300 seconds, the hysteresis area found for the two mixing times did not vary significantly. Thus, the mixing time adopted in this study was 100 seconds. However, even with these mixing times, it was observed that the values found in each cycle were still dispersed, not observing a constancy of values of hysteresis areas. It was then adopted, in addition to the mixing time of 100s, a pre-shear executed on the rheometer, as proposed by Williams et al. [39] to break down the structures of the particles before the test. According to these authors, the pre-shear works as a high-shear mixer, which makes the paste more homogeneous.

Table 6
Hysteresis area of mixing samples of 100 and 300 seconds

Mixing time (s)	100	300
Cycle 1	303.31	3,549.91
Cycle 2	468.65	1,410.36
Cycle 3	1,482.37	1,076.52

3.4.3 Determination of the pre-shear rate on the rheometer

The second definition was the use of a pre-shear made by the rheometer before each test. The actual 100-second blend was used in the mixer and before each 3-cycle flow test the sample was subjected to a pre-shear. Several pre-shear mixing procedures (PM) were performed, varying the time and shear rate for a more detailed analysis.
Through the tests, the shear stress versus shear rate curves were analyzed and it was possible to calculate the hysteresis area of each cycle as well as the yield stress of the first cycle of each. The hysteresis area values, and each cycle and for each pre-shear rate are shown in Table 7 and Figure 5.

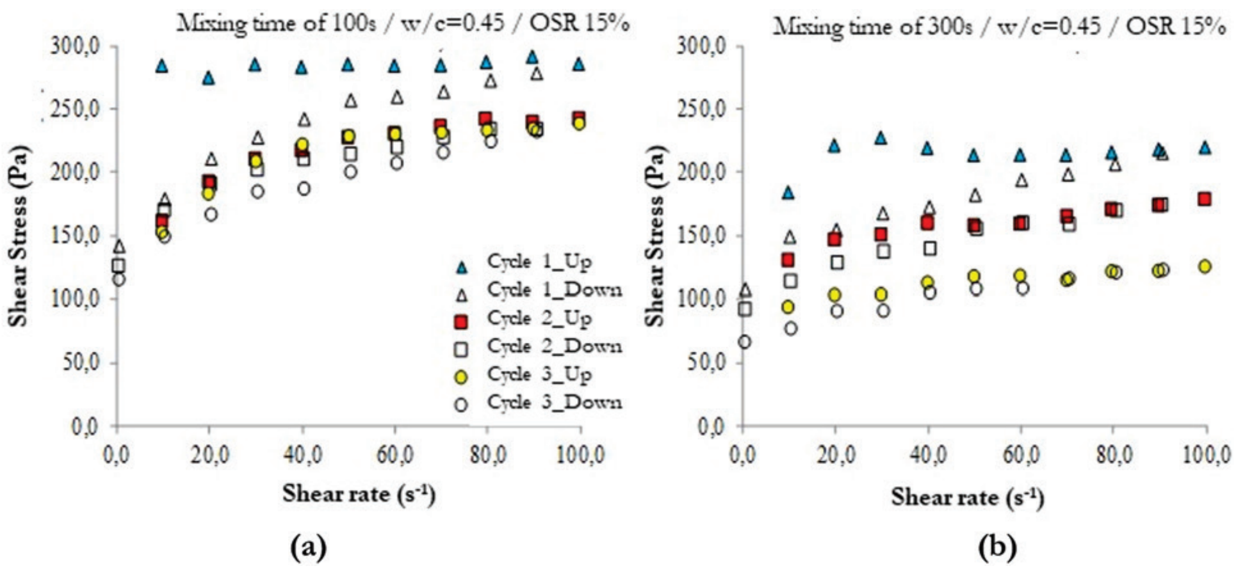

Figure 4
Shear stress vs. shear rate of the mixing times of (a) 100 seconds and (b) 300 seconds

Table 7
Hysteresis area of the mixing processes for different pre-shear tests performed by the rheometer

Mixing procedure	Pre-shear	Cyclo 1	Cyclo 2	Cyclo 3
PM 1	–	3,035.31	468.65	1,482.37
PM 2	50s-1 / 1min	1,864.28	1,059.67	170.91
PM 3	100s-1 / 1min	794.31	89.07	777.06
PM 4	100s-1 / 2min	1,150.42	265.07	205.40

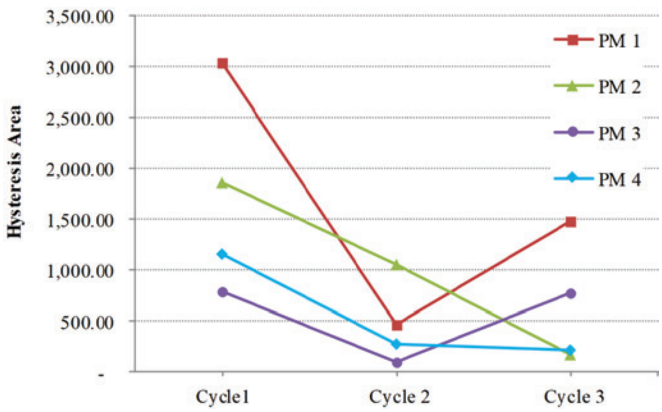

Figure 5
Hysteresis area values of each mixing process

Looking at the graph, it is noticed that the last mixing process (PM 4) presents more constant results between the three loading cycles with a smaller area of hysteresis. This means that the mixture was effective enough to break all the agglomerates at this stage, after a longer time and a higher shear rate, presenting greater homogenization [39].
Thus, it was defined as mixing procedure for the production of cementitious pastes the mixing time for 100 seconds with a pre-shear rate of 100s-1 mixer for 2 minutes, pre-set in the rheometer program to be run before each test takes place.

3.5 Flow test

The flow tests were carried out in a steady state, that is, the properties in a given section of the flow did not change over time. As the cement is in constant hydration and heat release, a stage was chosen in which a low rate of heat release called induction period or dormancy was observed, lasting approximately 30 minutes to 3 hours. This period of induction is observed after the first 20 minutes of cement hydration. For this reason the hydration time of 20 minutes counted from the contact of the cement with the water was chosen for the tests.
A rotational rheometer model AR 2000ex (TA Instruments) was used to perform the flow tests. The geometry of parallel plates, having a diameter of 40 mm and spaced 1 mm, was chosen. To avoid evaporation of the water and maintain the sample temperature at 23 ° C during the test, a peltier plate was used. To prevent slipping of the sample, a textured adhesive consisting of an acrylic adhesive and No. 60 grain (aluminum oxide) abrasives applied to a polyester film and protected by a siliconized paper coating was affixed to the surface of each plate.

In the flow test, the sample was subjected to a first increasing shear rate variation of 0 to 100 s-1 (acceleration curve) for 2 minutes and then immediately decelerated to 0 in a further 2 minutes. The tensile curves versus shear rate and viscosity versus shear rate were plotted and thus the rheological properties of the material, such as viscosity and yield stress, can be determined. Besides the effect of the w/c ratio on rheological properties, the effect of cement hydration on rheological behavior was also verified.

4. Results and discussions

4.1 Analysis of rheological parameters

In Figure 6 the results of flow tests are presented for the 12 cement pastes studied herein.
In an initial analysis, it is observed that the samples behave as a non-Newtonian fluid, since the shear stress varies non-linearly with the shear rate. A behavior of a pseudoplastic fluid is observed, showing an apparent viscosity decrease when there is an increase in shear rate or stress.
It is also noticeable that all cement pastes have a thixotropic behavior, in which the acceleration curve presents shear stress values higher than the shear stress values of the deceleration curve.
It is possible to observe in all the graphs that the residue C R15 (15% of residue) presents the highest values of shear stress and viscosity. This is due to the fineness of the OSR, filling the intergranular pores, which difficult the initial flow (higher yield stress) of the fluid.
In Figure 7, it is notable that the pastes produced with w/c ratio of 0.65 showed the smallest area of hysteresis in all residue contents. This means that the agglomerates were broken, presenting greater homogenization.
After observing the figures, it was possible to calculate the hysteresis area of each cycle (Table 8), which consists of the area between the acceleration and deceleration curves of the shear stress versus shear rate graph.
In Table 9 and Figure 8 the results of the yield stress values obtained are presented. It is possible to note that the pastes have a flow point fluid behavior with high yield stress values because this type of fluid requires a minimum shear stress to start flowing.
Analyzing the effect of the w/c ratio, it is verified that the yield stress is practically constant for w/c equal to 0.65, showing its homogeneity even with the addition of the OSR residue, as shown in Figure 9. Thus, even for higher percentage of residue addition used (i.e., 15%), the workability of the mixture is not affected if w/c ratio of 0.65 is used. For smaller w/c ratios, a reduction in workability

Table 8
Values of the hysteresis area of the flow test

Residue content	w/c = 0.45	w/c = 0.55	w/c = 0.65
0%	614.96	192.20	121.35
5%	632.49	241.57	125.77
10%	685.72	349.23	157.32
15%	1,284.39	432.48	210.86

Table 9
Yied stress values from flow tests

Residue content	Yield stress (Pa)		
	w/c = 0.45	w/c = 0.55	w/c = 0.65
0%	41.56	10.63	6.91
5%	58.37	15.43	7.79
10%	69.68	27.09	11.52
15%	119.86	41.50	15.29

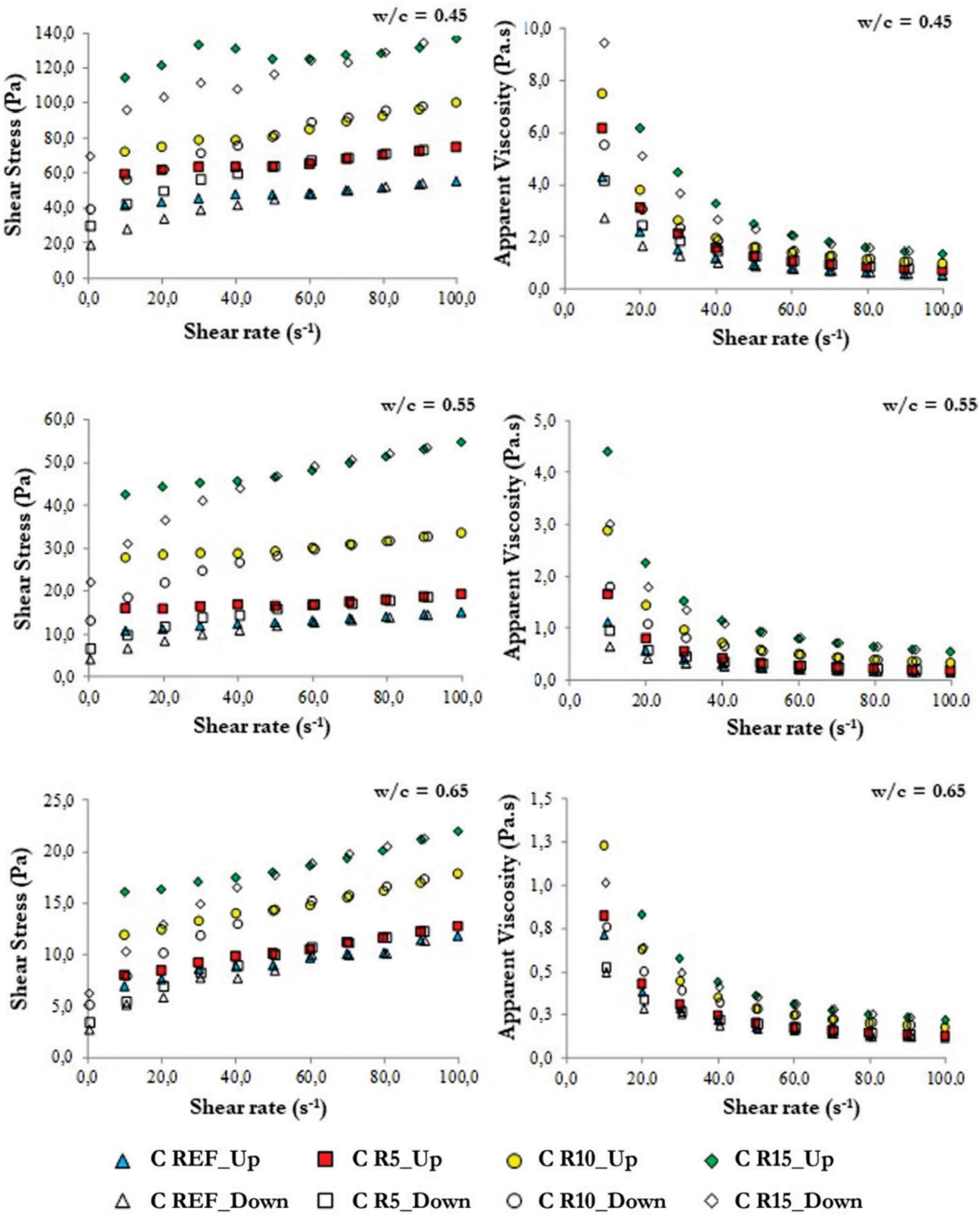

Figure 6
Shear stress vs. shear rate varying the water / cement ratio and the OSR residue content

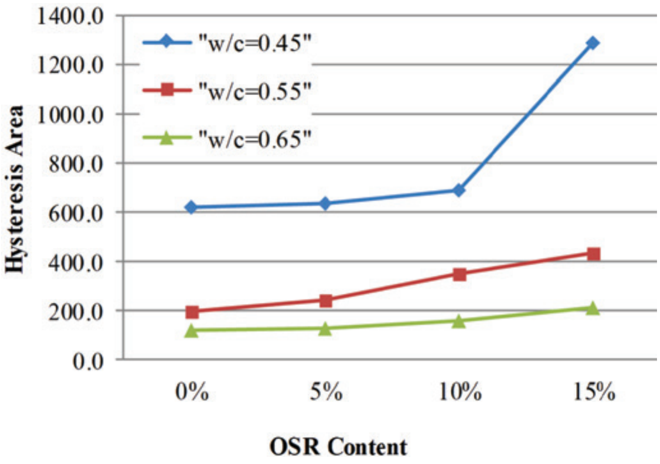

Figure 7
Hysteresis area *vs.* OSR content

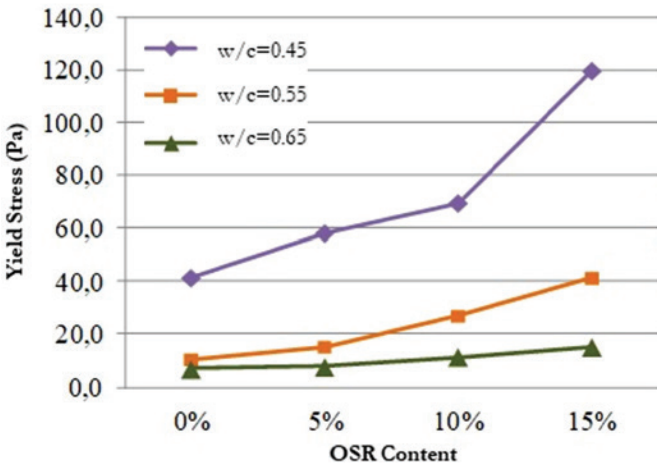

Figure 8
Yield stress *vs.* OSR content

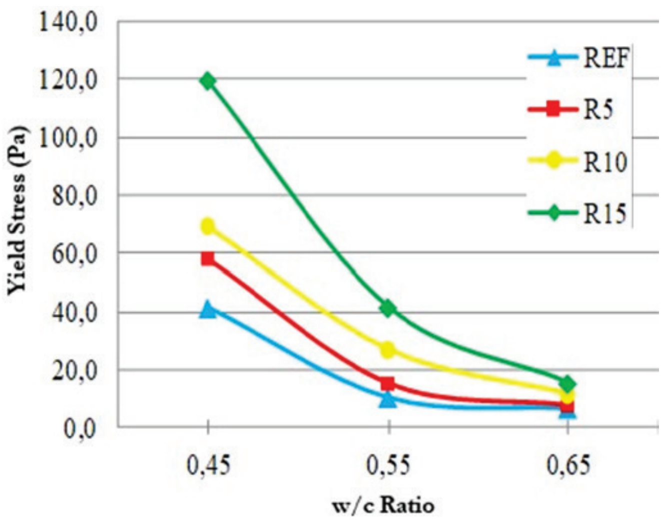

Figure 9
Yield stress *vs.* w/c ratio

Table 10
Slump test results obtained by Soares [35] related to the yield stress of the flow test

Trace	Slump	Yield stress
C REF 45	85	41.56
C R5 45	70	58.37
C R10 45	60	69.68
C R15 45	35	119.86
C REF 55	80	10.63
C R5 55	70	15.43
C R10 55	45	27.09
C R15 55	40	41.50
C REF 65	70	6.91
C R5 65	100	7.79
C R10 65	90	11.52
C R15 65	55	15.29

is expected because of a greater agglomeration of the particles. There is a significant drop in flow rate when the w/c ratio is increased. This occurred by increasing the amount of water added in the paste, presenting a less viscous fluid, with greater workability and fluidity.

4.2 Correlation between rheological parameters of the cement paste and the workability of the concrete in the fresh state

Correlating the results of the concrete slump test with addition of fresh residue obtained by Soares [36] and the yield stress of cement pastes obtained in present study, Table 10 and Figure 10 were established. It is important to mention here that the correlation is done using the results of slump tests obtained by Soares [36] for concrete in the fresh state while the present study shows values of yield stress of the cement phase of these concretes, ie, only water mixed with cement and OSR residue, using the same type of cement and w/c ratio for the two studies.

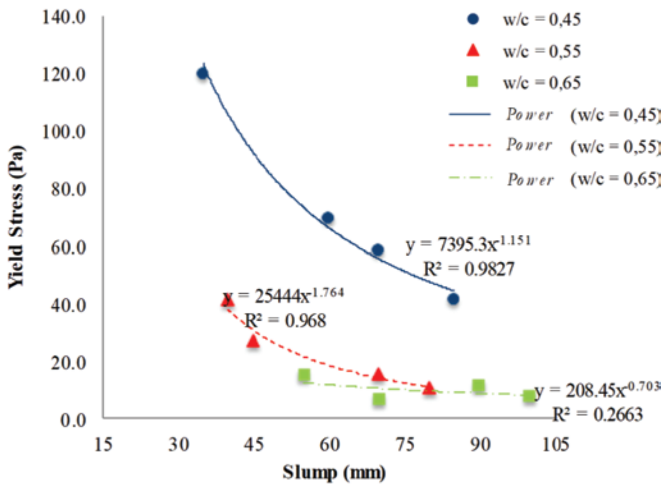

Figure 10
Yield stress from this study *vs.* slump values obtained by Soares [35]

Analyzing the data, it was observed that the values of yield stress and the slump values of the respective traces are inversely proportional. With increasing OSR content, the slump decreases and the yield stress increases. This confirms that the residue behaved like a filler, making the sample more viscous and less workable, as it fills the inter-granular voids in the matrix.
It is possible to perceive a tendency of correlation between the parameters analyzed using a power function. The determination coefficients (R2) of 0.98 and 0.97 for cement pastes with w/c of 0.45 and 0.55, respectively, show a strong negative correlation between the yield stress with slump testing results.
For mixtures with higher flowability, less viscous, this correlation presented the same trend, but with a low correlation (R2 of 0.27) between the values of the rheological tests proposed in this study and those of slump test. It is believed that the use of this type of correlation, based on analysis of rheological parameters can lead to measures of degree of workability in a more scientific way.

5. Conclusions

From the experimental program proposed in this work, results were obtained presenting the OSR as a material of fineness and specific mass near the cement, having low reactive activity. This indicates that the residue can be used as an inert mineral addition in the cementitious matrix. The addition content of OSR that gave satisfactory performance in the studied properties was 5%, in relation to the mass of cement.
In relation to the characterization of the rheological behavior, it was verified that the cementitious pastes present a non-Newtonian, pseudoplastic and thixotropic behavior.
Therefore, it can be concluded in general that the use of this residue presents benefits to the environment, providing a useful and plausible destination for the material that is discarded in the environment by the Brazilian companies of ornamental stone processing.
The ornamental rock residue studied presented the physical characterization of a powdery material with a filler effect in cementitious matrixes. In the chemical characterization, it was shown silica-aluminous formed by crystalline compounds with low reactive activity, indicating chemical stability. It is concluded, therefore, that the residue of ornamental stone processing, in its natural state, can be considered as a mineral filling addition (filler) of the inter-granular pores because it has no chemical activity and has a high specific surface area.
The hysteresis area found for the two mixing times (100 and 300 seconds) did not change significantly and the values found in each cycle were still dispersed. It was then adopted, in addition to the mixing time of 100s, a pre-shear performed on the rheometer and it was observed that among the different times and shear rates the pre-shear mixing process $100s^{-1}$ for 2 minutes (PM 4) presented results, with a lower area of hysteresis and lower yield stress. This indicates that the mixture was effective enough to break all the clusters and make the sample more homogeneous than the others. Therefore, the PM 4 mixing process was adopted on the rheometer.
After the flow tests were carried out, it was observed that the samples behave as a non-Newtonian, pseudoplastic and thixotropic fluid.
It is possible to observe that the residue C R 15 (15% of residue) presents the highest values of shear stress and viscosity. This is due to the fineness of the OSR, filling the inter-granular pores, which difficult the initial flow (higher yield stress) of the fluid. When the w/c ratio is increased, there is a significant drop in yield stress. This occurred by increasing the amount of water added in the cement paste, presenting a less viscous fluid, with greater workability and fluidity. However, in the w/c ratio of 0.65, the yield stress is practically constant, showing its homogeneity even with addition of the residue.
It has also been observed that the yield stress values and the consistency values of the respective features are inversely proportional. With increasing OSR content, the slump value decreases and the yield stress increases. This confirms that the residue behaved like a filler, making the sample more viscous and less workable, as it fills the inter-granular voids in the matrix. It is believed that the use of this type of correlation, based on analysis of rheological parameters can lead to measures of degree of workability in a more scientific way.

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