Heat treatment of processing sludge of ornamental rocks: application as pozzolan in cement matrices

Tratamento térmico da lama do beneficiamento de rochas ornamentais: aplicação como pozolana em matrizes cimentícias

Abstract

The sector of ornamental rocks produces significant volume of waste during the sawing of the blocks and demand to find ways to recycle, given its environmental impact. Considering the possibilities of use of industrial by-products as mineral admixtures, aiming at sustainable development in the construction industry, this paper aims to study the performance of the processing sludge of ornamental rocks and grinding after heat treatment, based on their potential application as partial substitute for cement. The residue was characterized, cast and milled to produce glassy material. Was analyzed the mechanical performance and pozzolanic activity with partial replacement of cement by waste in natural condition and after heat treatment in mortars for comparison. The results were promising, so it was possible to verify that after heat treatment, the treated waste is presented as a material with pozzolanic characteristics.

Keywords: heat treatment, pozzolanicity, sludge, waste, ornamental rocks.

Resumo

O setor de rochas ornamentais produz volume significativo de resíduo durante a serragem dos blocos e demanda encontrar formas de reciclagem, dado seu impacto ambiental. Considerando as possibilidades de utilização de subprodutos industriais como adições minerais, visando o desenvolvimento sustentável na construção civil, este artigo tem por objetivo estudar o desempenho da lama do beneficiamento de rochas ornamentais após tratamento térmico e moagem, baseando-se no seu potencial de aplicação como substituto parcial do cimento. O resíduo foi caracterizado, fundido e molido para produção de material vítreo. Analisou-se o desempenho mecânico e a atividade pozolânica com substituição parcial do cimento pelo resíduo na condição natural e após tratamento térmico em argamassas para comparação. Os resultados foram promissores, tendo sido possível verificar que após tratamento térmico, o resíduo tratado se apresentou como um material com características de pozolanicidade.

Palavras-chave: tratamento térmico, pozolanicidade, lama, resíduos, rochas ornamentais.

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1. Introduction

Sustainable development has become a global concern, and there has been a collective effort towards the improvement of processes and products, seeking the optimisation of natural resource use in the context of sustainability. Because civil construction places one of the largest demands on these resources for the production of its construction materials and buildings in general [1], improvements have been sought to minimise its impact on the planet by making use of both planning and management techniques, which can avoid reworking and waste, and by reusing the waste that is generated and recycling products. The incorporation of waste into construction materials has been indicated as a strategy to preserve natural raw materials, save energy, reduce pollutant emissions, and, in some cases, eliminate landfill costs. Fly ash, active silica, and rice husk ash are examples of waste materials already established as pozzolans [2].

The ornamental stone sector has been emphasised among the industrial sectors with broad waste generation. This industry is considered one of the most important areas of the mineral-industrial business, and Brazil is one of the five main countries that produce blocks and slabs of marble and granite [3]. The processing stage involves splitting blocks into slabs and treating their surfaces. Cutting is performed by metal blade looms and abrasive pulp (rock dust, grit, and lime) or diamond wire looms, with water aspersion to avoid suspension of the dust [4,5]. In this stage, approximately 25% of each of the cut blocks is converted into waste. The volume generated in Brazil is approximately 1.8 million tonnes annually [6] and is known as sludge from ornamental stone processing (SOSP). Rock dust is mixed with water and separated for discharge according to its composition: waste with grit, produced by cutting with traditional looms and referred to in this study as SOSP G; and waste without grit, produced by cutting with diamond wires and from polishing, known as SOSP D. Given the costs of this waste’s correct transportation and discharge and the environmental impact that can be caused by the large volume produced, studies have been performed examining its potential reuse in civil construction [5]. In its natural state, the waste has a moisture level between 20 and 30%, but after a drying process is performed, it has broad potential for application in construction materials given its fine powder condition. Promising results have been identified when the waste is added to matrices of ceramics [7-11], bitumen [12-16], and cement [17-28] with the objective of increasing the final material’s durability, reducing the use of aggregates or agglomerates, improving mechanical behaviour based on the physical performance that the inert materials exert when they are in a reduced granulometric range, acting as an element for filling pores, and improving the system’s packing. Using the waste’s chemical composition, elevated potential has also been identified for its use in the fabrication of glasses due to the significant presence of glass network-forming oxide (\( \text{SiO}_2 \)) and other oxide components (\( \text{Al}_2\text{O}_3 \), CaO, K₂O, Na₂O, and MgO). Studies have indicated the technical viability of producing soda-lime glass [29] and borosilicate glass [30] from a thermal treatment application following proper complementing of the chemical composition. Additionally, glass wastes have potential applications as a pozzolan given their reactive characteristics when finely ground [31-34].

Table 1 – Chemical composition and physical properties of the wastes and Portland cement

<table>
<thead>
<tr>
<th>Chemical property</th>
<th>SOSP G(1)</th>
<th>SOSP D(1)</th>
<th>CP V ARI(2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \text{SiO}_2 ) (%)</td>
<td>63.75</td>
<td>66.80</td>
<td>18.65</td>
</tr>
<tr>
<td>( \text{CaO} ) (%)</td>
<td>3.72</td>
<td>3.44</td>
<td>63.72</td>
</tr>
<tr>
<td>( \text{MgO} ) (%)</td>
<td>0.31</td>
<td>0.93</td>
<td>0.75</td>
</tr>
<tr>
<td>( \text{Al}_2\text{O}_3 ) (%)</td>
<td>14.55</td>
<td>13.50</td>
<td>4.91</td>
</tr>
<tr>
<td>( \text{Fe}_2\text{O}_3 ) (%)</td>
<td>7.57</td>
<td>3.79</td>
<td>2.97</td>
</tr>
<tr>
<td>( \text{K}_2\text{O} ) (%)</td>
<td>5.01</td>
<td>3.83</td>
<td>0.80</td>
</tr>
<tr>
<td>( \text{Na}_2\text{O} ) (%)</td>
<td>3.58</td>
<td>3.50</td>
<td>–</td>
</tr>
<tr>
<td>C (%)</td>
<td>0.39</td>
<td>1.11</td>
<td>–</td>
</tr>
<tr>
<td>Loss on ignition (%)</td>
<td>0.69</td>
<td>3.50</td>
<td>3.84</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Physical property</th>
<th>SOSP G</th>
<th>SOSP D</th>
<th>CP V ARI</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/cm³)</td>
<td>63.75</td>
<td>66.80</td>
<td>18.65</td>
</tr>
<tr>
<td>Blaine specific surface (cm²/g)</td>
<td>3.72</td>
<td>3.44</td>
<td>63.72</td>
</tr>
<tr>
<td>Material retained in sieve no. 200 (%)</td>
<td>0.31</td>
<td>0.93</td>
<td>0.75</td>
</tr>
</tbody>
</table>

Notes
(1) The chemical composition of the glass waste was determined using the x-ray fluorescence technique (XRF) using a Shimadzu EDX720 device.
(2) The data on the chemical composition of the cement CP V ARI were provided by the manufacturer and corresponded to the cement lot used.
Despite the previously identified potential uses for SOSP and considering the large volume of waste generated that remains unconsumed, this study sought to harness the vitreous potential of SOSP to develop a pozzolan, identifying the benefits of this additive in cement materials to reduce agglomerate use and improve their mechanical properties. This study was also based on the understanding that scientific institutions must work on challenging subjects at the forefront of the field, expecting to achieve a balance among the sustainable development triad of economics, the environment, and social aspects in the long term.

2. Material and methods

The experimental program was divided into two stages: (1) Part 1: thermal treatment of the waste and (2) Part 2: Application of thermally treated waste in mortars.

In Part 1, based on an initial study of the characteristics of SOSP G and SOSP D, four thermal treatment types were applied, and the resulting characteristics were identified in an effort to use the thermally treated material as a pozzolan.

After choosing one of these researched treatments, the wastes were melted in larger quantities and used as a substitute for cement in mortars. Hence, in Part 2 of this research scope was possible to evaluate the influence of the different percentages on the resulting mechanical properties.

2.1 Materials

High early strength Portland cement, or HES PC V (according to the NBR 5733 standard for high early strength Portland cement) [35], was used to fabricate mortars, using the lowest additive concentration among those available on the market, seeking to avoid combined effects. The fine aggregate was normal Brazilian sand, a natural quartz material obtained from the Institute for Technological Research (IPT) according to the requirements established in NBR 7214 (normal sand for cement tests) [36]. The sand was used in four grain size ranges corresponding to the standardised dimensions: 1.20, 0.60, 0.30, and 0.15 mm, added in equal proportions of 25% for each grain size range according to the standard adopted by NBR 7215 (Portland cement: determination of compressive strength) [37].

The wastes used in this study, SOSP G and SOSP D, came from a mill located in the Brazilian state of Espírito Santo. This mill splits blocks of marble, granite, and other rocks from various Brazilian states into slabs.

The waste underwent drying, lump breaking, and homogenisation before being characterised. The physical properties and chemical compositions of these materials and the cement are shown in Table 1. The predominance of silicon dioxide (SiO$_2$) and aluminium oxide (Al$_2$O$_3$) can be observed in both waste types, these being materials characterised as aluminium silicates, with the sum of the SiO$_2$, Al$_2$O$_3$, and Fe$_2$O$_3$ percentages greater than 50% in both. The pozzolanic activity index when combined with the cement indicated that these materials did not show reactive activity in the cement matrix but could...
be used as an inert mineral additive given that their fineness is higher than that of the cement, promoting a filler effect. Micrographs of the waste types in Figure 1 show the fragments of steel grit in the SOSP G sample and demonstrate that the SOSP G actually had smaller grains than the SOSP D, which was justified by the recirculation of the sludge in the traditional looms used to cut various blocks until it lost its abrasive capacity.

The morphological phases present in the waste types are shown in the x-ray diffractograms of Figure 2, in which it is possible to identify well-defined peaks attributed primarily to silica (quartz), a typical characteristic of chemically stable crystalline compounds indicating a low possibility of reactive activity.

To understand the behaviour of the wastes at high temperatures, a heating simulation was performed based on their chemical compositions using ThermoCalc® software. According to this simulation, the probable fusion temperature of SOSP G with the complete formation of slag (molten mass) was approximately 1125°C and that of SOSP D was 1090°C. To evaluate viscosity during the heating process, a computational simulation was also performed based on the materials’ chemical compositions using SlagViscosityModel software from the company Magnesita®, which indicated high viscosity even at high temperatures, on the order of 104 Poise (P) for a temperature of 1450°C. This viscosity hindered the material’s conformation and prevented fluidity, meaning that the melted mass could not be poured. Glasses in fusion show a viscosity of approximately 100 P [38].

2.2 Methods

2.2.1 Part 1

The methods used for the application of thermal treatment (TT) were intended to identify the temperature and ideal condition of the glassy material from the SOSP G and SOSP D wastes, generating an amorphous composite with the possibility for application as a pozzolan. This step in the experiment consisted of executing and evaluating the thermal treatments.

In addition to the drying and lump breaking condition (TT0), four other treatments were executed at temperatures varying from 1200°C to 1500°C, according to Table 2.

The treatment performed in a chamber-type muffle oven was

<table>
<thead>
<tr>
<th>ID</th>
<th>Description</th>
<th>Temp. (°C)</th>
<th>Cooling</th>
<th>Oven</th>
<th>Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>TT0</td>
<td>No thermal treatment</td>
<td>100</td>
<td>–</td>
<td>Oven</td>
<td>Drying and lump breaking</td>
</tr>
<tr>
<td>TTI</td>
<td>Thermal treatment I</td>
<td>1200</td>
<td>Slow</td>
<td>Muffle</td>
<td></td>
</tr>
<tr>
<td>TTI</td>
<td>Thermal treatment II</td>
<td>1300</td>
<td>Rapid</td>
<td>Tubular</td>
<td>Addition of CaO</td>
</tr>
<tr>
<td>TIII</td>
<td>Thermal treatment III</td>
<td>1400</td>
<td>Rapid</td>
<td>Tubular</td>
<td>Use of refractory paint in the crucible</td>
</tr>
<tr>
<td>TTV</td>
<td>Thermal treatment IV</td>
<td>1500</td>
<td>Rapid</td>
<td>Tubular</td>
<td></td>
</tr>
</tbody>
</table>

Table 2 – Treatments applied to the residues

The dosages of the produced mortars (by mass) are shown in Table 3.

<table>
<thead>
<tr>
<th>ID</th>
<th>Cement</th>
<th>SOSP</th>
<th>Sand</th>
<th>Water</th>
<th>Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>AREF</td>
<td>1,00</td>
<td>0,00</td>
<td>3,00</td>
<td>0,50</td>
<td>No waste</td>
</tr>
<tr>
<td>ALG 5</td>
<td>0,95</td>
<td>0,04</td>
<td>3,00</td>
<td>0,50</td>
<td>SOSP G - TT0</td>
</tr>
<tr>
<td>ALG 15</td>
<td>0,85</td>
<td>0,13</td>
<td>3,00</td>
<td>0,50</td>
<td>SOSP G - TT0</td>
</tr>
<tr>
<td>ALD 5</td>
<td>0,95</td>
<td>0,04</td>
<td>3,00</td>
<td>0,50</td>
<td>SOSP D - TT0</td>
</tr>
<tr>
<td>ALD 15</td>
<td>0,85</td>
<td>0,13</td>
<td>3,00</td>
<td>0,50</td>
<td>SOSP D - TT0</td>
</tr>
<tr>
<td>ALGF 5</td>
<td>0,95</td>
<td>0,04</td>
<td>3,00</td>
<td>0,50</td>
<td>SOSP Gf - Ttselection</td>
</tr>
<tr>
<td>ALGF 10</td>
<td>0,90</td>
<td>0,08</td>
<td>3,00</td>
<td>0,50</td>
<td>SOSP Gf - Ttselection</td>
</tr>
<tr>
<td>ALGF 15</td>
<td>0,85</td>
<td>0,12</td>
<td>3,00</td>
<td>0,50</td>
<td>SOSP Gf - Ttselection</td>
</tr>
<tr>
<td>ALGF 20</td>
<td>0,80</td>
<td>0,16</td>
<td>3,00</td>
<td>0,50</td>
<td>SOSP Gf - Ttselection</td>
</tr>
<tr>
<td>ALDF 5</td>
<td>0,95</td>
<td>0,04</td>
<td>3,00</td>
<td>0,50</td>
<td>SOSP Df - Ttselection</td>
</tr>
<tr>
<td>ALDF 10</td>
<td>0,90</td>
<td>0,08</td>
<td>3,00</td>
<td>0,50</td>
<td>SOSP Df - Ttselection</td>
</tr>
<tr>
<td>ALDF 15</td>
<td>0,85</td>
<td>0,12</td>
<td>3,00</td>
<td>0,50</td>
<td>SOSP Df - Ttselection</td>
</tr>
<tr>
<td>ALDF 20</td>
<td>0,80</td>
<td>0,16</td>
<td>3,00</td>
<td>0,50</td>
<td>SOSP Df - Ttselection</td>
</tr>
</tbody>
</table>

Table 3 – Dosages of the produced mortars (by mass)
2.2.2 Part 2

Mortars were produced with a proportion of 1:3:0.5 by mass (agglomerate:sand:water). The agglomerate concentration varied with the use of waste substitution for cement. To verify whether the wastes had comparable performance to the pozzolans after thermal treatment, substitution concentrations of 5, 10, 15, and 20% cement were adopted for each treated waste (ALG and ALD) along with traces of mortar substituting waste without thermal treatment (ALG and ALD) at concentrations of 5 and 15%, in addition to the reference mortar (MREF) for comparison. The dosage of mortars was established by mass based on the appropriate volume compensation to replacement cement by waste according to the relation between the specific gravities. The dosages and each age. On these dates, the specimens were examined to check their modulus of elasticity and axial compressive strength. Determination of the dynamic modulus of elasticity was conducted at 28, 63, and 91 days using the velocity obtained from an ultrasonic pulse, a Pundit Lab model from Proceq, according to NBR 15630 [44], while the axial compressive strength was determined at the same ages according to NBR 5739 [45].

3. Results and discussion

3.1 Part 1: evaluation of the applied thermal treatments

In terms of production, TTI offered the best conditions because it presented minimal risk to the operator and had the production performed by heating the sample at a rate of 10°C/min to the defined temperature, which was maintained for 2 hours and then slowly cooled according to the oven’s inertia. The treatments performed in the tubular oven involved preheating the oven to the temperature of the TT and subsequent insertion of the samples, which were rapidly heated and maintained at the standard temperature for two hours and then cooled by removing the graphite crucible from the oven with the appropriate necessary safety apparatus and immersing it in water at room temperature. Abrupt cooling is ideal for glass production [38], but given the high viscosity of the slag produced in the fusion, it was not possible to pour it, which hindered the cooling process. The vitreous materials resulting from the thermal treatments were subjected to a grinding process using a ring mill and sieved in a 75-μm sieve (no. 200). The materials were evaluated according to three aspects, production, mineralogy, and pozzolanicity, by the Lúxan method [39], which is a rapid test on a 5.0-g sample of material that evaluates the pozzolanic activity through the conductivity variation of a saturated solution of Ca(OH)₂.

From this procedure, the TT with the best results was selected for production in larger quantities and application in mortar. These samples were named SOSP Gf and SOSP Df and characterised with regard to their physical properties, chemical compositions, and pozzolanic activity [40] according to the requirements established in NBR 12653 (Pozzolanic materials specification [41]).

### Table 4 – Results for the pozzolanicity tests performed using the Lúxan method

<table>
<thead>
<tr>
<th>Waste type</th>
<th>Thermal sample treatment</th>
<th>Initial reading (mS/cm)</th>
<th>Final reading (mS/cm)</th>
<th>Conductivity (mS/cm)</th>
<th>Pozzolanicity classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>SOSP D</td>
<td>TT0</td>
<td>4.86</td>
<td>4.84</td>
<td>0.02</td>
<td>Not pozzolanic</td>
</tr>
<tr>
<td>SOSP D</td>
<td>TT1</td>
<td>4.89</td>
<td>4.45</td>
<td>0.44</td>
<td>Variable pozzolanicity</td>
</tr>
<tr>
<td>SOSP D</td>
<td>TTII</td>
<td>4.88</td>
<td>4.63</td>
<td>0.25</td>
<td>Not pozzolanic</td>
</tr>
<tr>
<td>SOSP D</td>
<td>TTIII</td>
<td>4.87</td>
<td>4.65</td>
<td>0.22</td>
<td>Not pozzolanic</td>
</tr>
<tr>
<td>SOSP D</td>
<td>TTIV</td>
<td>4.85</td>
<td>4.12</td>
<td>0.73</td>
<td>Variable pozzolanicity</td>
</tr>
<tr>
<td>SOSP G</td>
<td>TT0</td>
<td>4.86</td>
<td>4.83</td>
<td>0.03</td>
<td>Not pozzolanic</td>
</tr>
<tr>
<td>SOSP G</td>
<td>TT1</td>
<td>4.88</td>
<td>4.60</td>
<td>0.28</td>
<td>Not pozzolanic</td>
</tr>
<tr>
<td>SOSP G</td>
<td>TTIV</td>
<td>4.89</td>
<td>4.59</td>
<td>0.30</td>
<td>Not pozzolanic</td>
</tr>
</tbody>
</table>

The mortars were produced according to the procedures defined in NBR 7215 [37] and their characteristics verified in the fresh state, with tests performed to determine the consistency index by spreading a truncated cone on the table, performed according to NBR 13276 [42], and to verify the density of the mortars according to NBR 13278 [43]. To perform tests on the samples in their hardened states, it was necessary to mould cylindrical specimens measuring 50 x 100 mm, for which the methodology described in NBR 7215 was adopted [37]. The specimens were kept inside the moulds in a humid chamber and removed from the moulds after 24 hours and then identified and subjected to immersed curing in water saturated with lime until the date designated for performing tests in their hardened states. The evaluation occurred at 28, 63, and 91 days. A total of 234 specimens were moulded for this purpose, with 18 for each trace and each age. On these dates, the specimens were examined to check their modulus of elasticity and axial compressive strength. Determination of the dynamic modulus of elasticity was conducted at 28, 63, and 91 days using the velocity obtained from an ultrasonic pulse, a Pundit Lab model from Proceq, according to NBR 15630 [44], while the axial compressive strength was determined at the same ages according to NBR 5739 [45].

2.3 Statistical analysis

The results obtained in the tests performed on the mortars in their hardened states were subjected to a statistical analysis for variance verification, with the goal of comparing the influence of each controllable factor (thermal treatment, waste type, substitution concentration, and age) on the response variables (modulus of elasticity and compressive strength). Analysis of variance (ANOVA) was employed, considering a confidence level of 95%, using the Statistica 7.0 program [46].
capacity for a large quantity of material despite the need for a high energy demand to heat the oven’s entire internal volume. The treatments performed in the tubular oven (TTII, TTIII, and TTIV) show high operational risk and limitations in terms of volume produced. The treatments had a low energy demand for heating, but pouring the molten mass was impossible, and it was difficult to obtain an adequate recipient.

In TTII, the addition of CaO was not effective at reducing the viscosity, but the sample remained porous, facilitating forced removal. In TTIII, the use of refractory paint aided slightly in the removal of the sample after cooling, but the paint interacted with the material on its edges. It was difficult to remove the molten material from TTIV after cooling.

Regarding the mineralogy of the samples produced by the applied thermal treatments, a trend of disorganisation in the crystalline network, with the appearance of an amorphous halo, was noted in all cases, even with the occurrence of crystalline peaks related to SiO₂ in the form of quartz because this phase was stable and its fusion temperature higher than the temperatures of the applied treatments. The samples’ pozzolanicity results obtained from an analysis of the Luxan method are shown in Table 4.

The samples’ pozzolanicity results obtained from an analysis of the Luxan method are shown in Table 4. The samples of SOSP D from TTII, performed in the muffle oven at up to 1200°C with slow cooling, and TTIV, performed in the tubular oven at 1500°C, were classified as having variable pozzolanicity. It was expected that the samples with the highest fusion temperatures and highest cooling velocities would have the largest variations in conductivity due to the material’s greater reactivity, as occurred with the sample of SOSP D during TTIV. However, the sample also achieved an index to be classified as a pozzolan during TTI, indicating that it was possible to obtain a pozzolan from SOSP at a temperature of 1200°C and even with slow cooling. This reactivity may be related to the high viscosity of the material during the heating process, which, after the disorganisation of the atomic arrangement, makes difficult the reorganization of the material, thus generating a material with an amorphous halo even without abrupt cooling.

With the criteria used to evaluate the thermal treatments, it was judged that the use of TT was most appropriate for the task. This treatment presented the lowest production risks, with the possibility of producing larger quantities. The samples’ crystalline networks showed disorganisation and the appearance of an amorphous halo. TTI also had the lowest limit temperature of the treatments with indications of pozzolanicity.

### 3.2 Characterisation of the wastes after TT selection

The physical and chemical properties of the waste types used after the thermal treatment (TTI) was selected, known as SOSP Gf and SOSP Df, are shown in Table 5. The data shown demonstrate that the wastes showed similar characteristics after the fusion and grinding processes, with the SOSP Gf waste having a slightly higher density due to the presence of traces of steel grit, which are denser than the remainder of the material. Even with a particle size similar to cement, the SOSP Gf and SOSP Df wastes showed higher specific surface areas, which was attributed to the grains’ angularity, a characteristic of vitreous
Heat treatment of processing sludge of ornamental rocks: application as pozzolan in cement matrices

After fusion, the wastes remained predominantly aluminium silicates with a concentration of SiO$_2$, Al$_2$O$_3$, and Fe$_2$O$_3$ higher than 50%, as established in the requirements of NBR 12653 [38] for Class E pozzolanic materials.

X-ray diffractograms of the wastes after thermal treatment are shown in Figure 3.

A reduction and broadening of the crystalline peaks was observed, indicating a trend toward disorganisation in the network. This disorganisation is a marked characteristic in glasses that favours the material’s pozzolanic action. The presence of silica in its quartz phase (SiO$_2$) was also noted, which, despite heating, did not reach its fusion temperature of approximately 1700°C.

To verify the pozzolanic action capacity of the treated wastes in the cement matrix, they were evaluated in terms of their Pozzolanic Activity Index with the cement (PAI-cem) [40]. NBR 12653 [41] establishes that the minimum PAI-cem value needed for a material to be considered pozzolanic is 75% and that the maximum water required is 110%. The PAI-cem of SOSP Gf was 94.7% and that of SOSP Df was 97.3%. Thus, both wastes exhibited proven pozzolanic activity with the cement. Using the chemical and physical requirements after thermal treatment, the materials could be classified as Class E pozzolans.

3.3 Part 2: application of thermally treated waste in mortars

3.3.1 Fresh state

The a/c relationship of all of the mortars prepared was maintained, the additives were substituted for cement, and the quantity of water was standard. The consistency index (CI) varied from 211.5 mm in MREF to 226.0 mm in ALDf 5, 10, and 15. The maximum variation was 6.9% relative to MREF. The CI was within the interval inherent to the test type’s variability, and it was impossible to state whether these results were effectively different.

There was a reduction in the density of the mortars in their fresh state with the increase in the substitution concentration, which was justified by the lower densities of the wastes compared to that of the cement.

3.3.2 Hardened state

As shown in Figure 4, and analysing in terms of absolute values, the mortars with waste substituted for cement had lower axial compressive strengths than those from MREF. The ALGf and ALDf samples had the closest strength values to that of the MREF. By analysing the samples at 91 days, it was verified that the equivalent percentages relative to MREF varied from 74.4% to 95.7%, with the mortars ALG 5, ALGf 5, and ALGf 10 having values closest to that of the MREF. This result could be explained by the filler effect with a low substitution percentage in ALG 5, and the
The performance of the ALGfs was attributed to the pozzolanic activity of the wastes with the cement that received thermal treatment. Although it could not be proven in this study, an increasing trend was expected in the axial compressive strength of mortars using wastes subjected to thermal treatment, such that it would be possible to achieve strengths very close or equal to those of the MREF (primarily with lower substitution concentrations, considering the slow effect of the pozzolanic reactions, which was a consequence of the production of calcium silicate hydrate (C-S-H) in these phases [1]).

The analysis of variance of these results occurred separately, using the data set obtained for the mortars that used wastes without thermal treatment and the mortars that were prepared with wastes subjected to thermal treatment and grinding. The type of waste (SOSP G or SOSP D) was not a significant variable in the results for the axial compressive strength of mortars produced using wastes without thermal treatment, considering a confidence interval of 95% and a significance level of 5%. Age and substitution concentration were significant as was the interaction between them, as expected.

The mortars produced with the thermally treated wastes, SOSP Gf and SOSP Df, had compressive strength results that were in agreement with the conclusions of different authors studying additives in cement matrices, knowing that they had similar characteristics to glasses. Researchers have identified that mortars containing finely ground glass waste possess lower but satisfactory strengths than reference mortar, primarily at later ages, associated with the material’s pozzolanic action [33-34,47].

The analysis of variance of this group of mortars was performed on a confidence interval of 95% and at a significance level of 5% and indicated that all of the controllable factors (type of waste, substitution concentration, and age) interfered independently with the compressive strength in a significant manner. The interaction between these factors was significant only between the type of waste and substitution concentration and between the percentage and age, which was differentiated from the occurrence of significance in the mortars with waste that was not thermally treated. The similar behavior to that of the reference mortar in this group can be observed in Figure 5.

An analysis of variance of the results was also performed for the compressive strength of mortars ALG, ALD, ALGf, and ALDf, with substitution percentages of 5 and 15%, to evaluate the influence of the wastes’ thermal treatment on the strength results. The source of variation was included to determine whether thermal treatment of the waste was performed. The question of whether the waste was thermally treated was significant to the mortars’ axial compressive strength and the other variables being studied.

In terms of the difference between the waste’s behavior with and without thermal treatment, (i.e., pozzolanic or non-pozzolanic), the investigation identified a reduction in strength with higher concentrations when non-pozzolanic material was used, and the pozzolanic material yielded results closer to the reference mortar, as can be observed in Figure 6.
As observed in Figure 7, the mortars with substitution had lower moduli of elasticity, in absolute values, than the MREF, as with the compressive strength. ALGf and ALDf showed modulus of elasticity values closer to the MREF values. At 91 days, the equivalent percentages compared to MREF varied from 87.7% to 99.6%, with the ALGf 5, ALGf 10, and ALDf 10 mortars having values closest to MREF. This behaviour could have been related to the higher specific gravity in the matrices of the mortars with wastes subjected to fusion due to the pozzolanic activity of these materials.

An analysis of variance was performed according to the compressive strength, initially in two groups (i.e., mortars produced with wastes in the TT0 condition and after the TTI treatment) and then in a joint manner for concentrations of 5% and 15%.

The results of the analysis were similar to those for compressive strength in that the type of waste was not significant in the mortars with wastes not subjected to thermal treatment, but it was significant in the other cases. Increasing age increased the modulus of elasticity in all of the experiments, and the increase in substitution concentration generally decreased the value of the modulus. During the joint analysis of the results, the performance of the wastes’ thermal treatment had a significant influence on the mortars’ moduli of elasticity. The photos in Figure 8 demonstrate that the wastes with thermal treatment generated higher modulus results, with a less accentuated reduction as the substitution concentration increased.

Observation of the microstructure indicated dense matrices with the formation of portlandite plates in the mortars containing wastes subjected to thermal treatment, possibly from the pozzolanic reaction.

It was possible to observe the presence of pores with late crystal-line formations of calcium aluminate hydrates at 91 days in mortar ALD 15, which had the lowest mechanical performance of the mortars evaluated. These pores are shown in Figure 9.

The mortar with 5% substitution of SOSP Gf for cement had a similar mechanical performance to the reference mortar. Figure 10 shows that piles of portlandite plates and C-S-H crystals were formed at 91 days. These crystals filled the pores previously occupied by water during the cement hydration, refining the matrix and making the mortar less permeable, which is a typical behaviour observed with the pozzolanic effect.
4. Conclusions

- The sludge produced by processing ornamental stones (SOSP), with and without grit, is an aluminium silicate with broad potential for use in construction materials.
- The application of thermal treatment to SOSP can produce a pozzolan, as observed in this study, and new studies are needed to obtain better results by seeking appropriate techniques for abrupt cooling, which results in a predominantly amorphous material.
- SOSP can act as a filler in cement matrices, substituting for cement and maintaining a compressive strength close to reference values at low substitution concentrations (5%). There is no significant difference between the residue action with and without grit in this condition.
- The performance of the SOSP after thermal treatment and grinding indicated that this material could be used in cement matrices as a substitute for cement, generally achieving higher axial compressive strength and modulus of elasticity values than those observed with wastes that were not thermally treated.
The production of pozzolanic material from sludge produced by ornamental stone processing can be established as an alternative to recycling of waste, and for this it is necessary to structure ways to optimize the production, whereas this research was conducted in order to verify the technical feasibility of the product and demand an analysis of sustainability of the material.

In conclusion, the application of sludge produced by ornamental stone processing, after thermal treatment and grinding, was deemed promising as a partial pozzolanic substitute for cement.

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6. References


