

Evaluation of Mechanical Properties of Sealing Mortar with Partial Replacement of Portland Cement by Stone Crusher Waste

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Civil construction is one segment responsible for major environmental and social impacts. Among its raw materials, Portland cement, which is produced by the burning and subsequent grinding of a composition of clays and limestones. This manufacturing process causes great environmental damage, both by the exploitation of deposits and by the process of clinkering (burning of raw materials), which emits a significant amount of CO₂ in the atmosphere. Therefore, there is a need for research on materials that reduce these impacts on the environment and that can be used by civil construction to the partial replacement of cement. Thus, this research had as objective to produce settlement mortar through the partial replacement of the Portland cement by filler powder, in different proportions and to study its mechanical properties. The results show that the partial replacement of the cement by the residue has acceptable behavior in the mortar.

Keywords: *Portland cement, basaltic filler, reuse of waste.*

1. Introduction

The cement industry has a high potential for contamination, generating pollution at almost all stages of the cement production process, ranging from the extraction, grinding and homogenization of raw materials, to clinker, cooling and grinding of the clinker, making the cement factory one of the major sources of carbon dioxide (CO₂) emissions, in addition to the high energy consumption demanded, especially in clinkerization¹⁻⁵. The cement industry consumes approximately 12-15% of the world's industrial energy and it's responsible for at least 5% of the world's CO₂ emissions^{6,7}.

Today gas emissions are great environmental concern in the cement industry, that to produce one tonne of clinker, approximately one tonne of CO₂ is released into the environment, where approximately 579 kg of CO₂ is emitted to one tonne of clinker, plus approximately 390 kg of CO₂ as a function of the use of fossil fuels during production⁸.

On the other hand, the construction industry has been of extreme importance for the development of the modern economy and with it, the demand for aggregates to supply the demand in the last decade had a big growth^{9,10}.

Mortar, being one of the main materials used by the construction industry, presents high potential to absorb alternative materials from industrial tailings and wastes from the construction itself. The adoption of practices for

a reuse and recycling of different type of residues to avoid environmental pollution is mostly desirable as it contributes to sustainable development¹¹. The civil construction industry appears as the best solution to overcome this environmental issue and the incorporation of residues in mortars can result not only technical advantages but also cost reduction of the fabricated products since the residues are considered waste materials for the industry¹².

Thus, several researchers studied technical feasibility for the incorporation of different residues in the production of mortars, aiming at the partial replacement of the cement, which is the most important agglomerant in the production of the mortar. Among them, they evaluated the use of marble residues^{13,11}, eggshell powder⁸, waste paper and cellulose, as well as construction and demolition industries for application in cement-based materials¹⁴, biomass residue from a pulp and paper mill¹⁵, heavy ash obtained from the combustion of mineral coal¹⁶, biomass ash¹⁷ and sewage sludge¹⁸. These articles aim to contribute to the sustainable development by making greater use of industrial residues in the civil construction as mortars¹² and concrete¹⁹.

An industrial residue that deserves attention is the rock powder, from rock crushing, also known as filler or gravel powder. During the exploration of the quarry, more specifically in the extraction, transport and crushing of rocks, large amounts of powder is generated. The handling and incorrect disposal of this residue can generate serious environmental problems, and its accumulation and dispersion

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directly contaminate the soil and surface waters, and may even deteriorate groundwater through the production of drainage¹⁰. Due to the fact that there is no commercialization and the commercial value involved is very low, this residue is often stored in the quarries, causing impacts such as air pollution due to the presence of particulate matter, pollution of water bodies caused by drainage of fine materials and also visual pollution of the site^{20,9,21}.

Researchers have been developing methods to properly dispose of these wastes generated during the crushing process, using them as feedstock to produce clinker or even as a mineral addition in Portland cements^{22,23}, but the use of basaltic filler replacing partially the cement, in the production of mortar, hasn't been evaluated yet. In view of this context, the objective of this study was to obtain settlement mortar through the partial replacement of the Portland cement by residue generated from the crushing process of basaltic rock, basaltic filler. The mortar obtained was characterized, through mechanical tests, with the intention to evaluate if it meets all the technical requirements of use foreseen in the standard norms of the civil construction.

2. Materials and Methods

For this study, the following materials were used: sand, classified as per ABNT (Brazilian Association of Technical Standards) n° 5/8" sieve (1.2 mm) and bulk density of 2.64 g cm⁻³; Portland cement, model CP II Z-32, of the Votoran, specific mass of 2.96 g cm⁻³; filer residues from the stone crusher from Cordilheira Alta, state of Santa Catarina, Brazil.

Initially the filer residues were physically characterized by specific mass for NBR NM 52²⁴, particle size in the laser diffraction analyser (Microtrac, S3500). It was also chemically characterized by fluorescence X-rays analysis (XRF) in a Panalytical spectrometer, Axios Max model. For the mineralogical characterization, X-ray diffraction (XRD) was performed in a Bruker diffractometer (Bruker, D8 model), with theta - theta goniometer. The x-ray radiation employed was K α - Copper generated under the conditions of 40 kV and 40 mA. The speed and scan interval of the goniometer used were 1 g of powder at 1 s to 0.02 ° of the goniometer from 2° to 72 ° 2 theta, respectively. TGA analysis was

also performed. The equipment used was a TGA Netzsch, model STA 409 EP, and the tests were carried out with a heating rate of 10 °C min⁻¹, starting at room temperature and ending at 910 °C. The cement was also characterized by XRF and XRD.

The mortars were produced according to NBR 13276²⁵. The dosage of the mixtures was established, in the proportions of 1: 3: 0.56 (wt%) of cement, sand and water, respectively. Before use, the sand was oven dried (QUIMIS, Q314M) at 105 °C for 24 h. The residues used were those how have passed through the sieve at 300 μ m and after were then oven dried for 24 h at 105 °C. The residues replace the cement in five mixes in different proportions, being 5%, 10%, 15%, 20% and 30% (wt%), maintaining the relationship between water and cement (w/c) adopted for the reference mix. The assays were completed in triplicate. All compositions were compared to the reference (control) sample without residues. Table 1 presents the composition of the investigated residues-cement mixtures.

For each mix, 36 specimens were produced, being that 18 were molded in the cylindrical dimensions of 5x10 cm³, according to NBR 7215²⁶ and 18 were molded in the prismatic dimensions of 4x4x16 cm³, according to NBR 13279²⁷. These 18 blocks 9 were molded to curing process over a period of 7 days and 9 to a period of 28 days. The curing process was performed in controlled temperature of 25 °C.

The consistency index and water absorption determination in mortars were determined according to Brazilian Standard NBR 13276²⁵ and NBR 13277²⁸, respectively.

It was determined, in the hardened state, the flexural tensile strength and the compressive strength through the Brazilian Standard NBR 13279²⁷. The mechanical tests were used a universal mechanical test press (Shimadzu, AG-X Plus) with capacity to 100 kN and speed of 50 \pm 10 N s⁻¹. According to the same standard, the axial compressive strength was obtained, where the same press was used, at a speed of 500 \pm 50 N s⁻¹. The test to tensile strength diametrical compression were done according to NBR 7222²⁹. It was used the same press, at a speed of 0.05 \pm 0.02 MPa s⁻¹. The mechanical tests were statistically analyzed using Statistica® 12.0 (StatSoft®, USA), using Tukey's test with 5% probability ($p \leq 0.05$) using analysis of variance (ANOVA).

Table 1. Compositions (w%) of the filer residues cement mixtures to production of mortars.

Mix	residues	Amount (Kg)			
		sand	cement	residue	water
Reference/Control	0%	30.690	10.230	0.000	5.852
A	5%	30.690	9.718	0.512	5.852
B	10%	30.690	9.207	1.023	5.852
C	15%	30.690	8.695	1.535	5.852
D	20%	30.690	8.184	2.046	5.852
E	30%	30.690	7.161	3.069	5.852

Tests to available the pozzolanicity of the basaltic filler were made according NBR 5751³⁰ (2015). The technical standard suggests the production and tests of the mortar samples with $\text{Ca}(\text{OH})_2$ and the cure at 55 °C by 7 days. The mortar samples were submitted to compression tests using a universal mechanical test press (Shimadzu, AG-X Plus) with capacity to 100 kN and speed of $50 \pm 10 \text{ N s}^{-1}$. According to the same standard, the axial compressive strength was obtained, where the same press was used, at a speed of $500 \pm 50 \text{ N s}^{-1}$. The criteria considered to define a pozzolanic sample were the compression resistance values higher than 6 MPa, according NBR 12653³¹ (2015).

3. Results and Discussion

3.1 Characterization of cement and waste

Figure 1 shows the granulometric distribution of the filler residues and cement used. It is possible to observe that the amount of cement used for the mortar production presented to be a very fine material, where only 5.97% of material was retained in the 200 mesh sieve (aperture 0.074 mm), different from the residue, which presented larger particles, totaling 69.54% of the material retained in the same sieve. Only 10% of the cement particles had a diameter less than $3.27 \mu\text{m}$, while the residue had a diameter of less than $31.11 \mu\text{m}$ in 10% of the particles, thus showing the granulometric difference between the materials. The density of the filler residue was calculated $2.86 \pm 0.0122 \text{ g cm}^{-3}$.

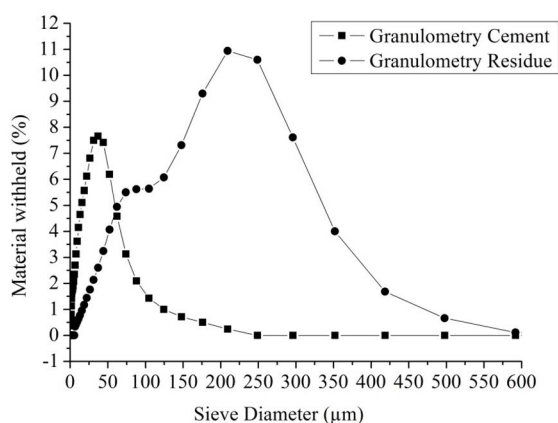


Figure 1. Filer residue and cement granulometric curve.

The optimization of the compactness in the cement paste is carried out using a material of higher particle size and one of smaller particle size in relation to the cement, so the filler used in this work can provide a better packaging of the materials and consequently a gain in the mechanical resistance, because the smaller particles tend to fit into the voids between the average particles, which in turn occupy the voids of the larger particles³².

Table 2 presents the XRF chemical analysis of the filler (crusher residue) and cement. It is found that the crusher residue is composed predominantly of SiO_2 , Al_2O_3 and Fe_2O_3 , totaling approximately 78.5% by mass. According to NBR 12653³¹, it can be said that this residue possesses a possible pozzolanic characteristic, being possible to fit it to the classification for pozzolanic materials. The residue sample shows chemical compatibility with the main raw materials used to produce cement clinker, which is alite and belite, that is, this similarity is due to the main oxides present in the residue (SiO_2 , Fe_2O_3 and Al_2O_3).

Table 2. Chemical composition (mass percent) by XRF of the filler and cement.

Chemical composition (oxides)	Cement (%)	Residue (%)
Al_2O_3	5.75	13.50
CaO	52.38	8.09
Fe_2O_3	3.10	13.04
K_2O	1.34	1.53
MgO	5.60	4.01
MnO	0.10	0.18
Na_2O	0.30	3.18
P_2O_5	0.13	0.47
SiO_2	23.35	51.91
TiO_2	0.32	3.00
B_2O_3	-	-
Li_2O	-	-
BaO	-	< 0.1
Co_2O_3	-	< 0.1
Cr_2O_3	-	< 0.1
PbO	-	< 0.1
SrO	0.28	< 0.1
ZnO	< 0.1	< 0.1
$\text{ZrO}_2 + \text{HfO}_2$	< 0.1	< 0.1
Loss on ignition	5.44	0.93

According to the chemical composition of the cement, presented in Table 2, it is possible to classify it, according to NBR 16697³³, as CP II-Z cement, because it meets the conditions established by the NBR in relation to the amount of magnesium oxide (MgO), less than 6.5% and less than 4.0% of sulfur trioxide (SO_3), which for the cement used was not registered. The presence of calcium oxide (CaO) and magnesium oxide (MgO) are characteristic compounds of carbonate rocks³⁴. For application in cements, the magnesium oxide content is generally limited to 5%, because amounts of this component in excess can cause slow reactions with water, which may lead to the destructive expansion of hardened concrete³⁵. For the case of the residue, the amount found was less than 5%, which allows its application in cements.

The Figure 2 shows the X-ray diffractogram of the cement and Figure 3 shows the X-ray diffractogram of filler. It is possible to note the typical peaks of quartz, expressed by the number “1”, alite, indicated by the number “2” and belite, number “3”, which are constituents of the Portland cement^{36,37}. This result confirms the data presented by the chemical analysis of the material in Table 2.

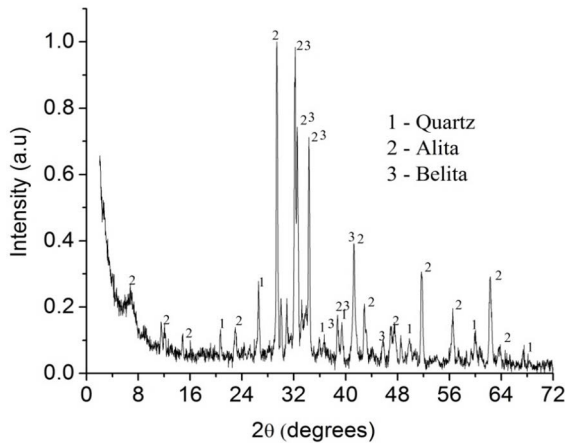


Figure 2. X-ray diffractogram of cement.

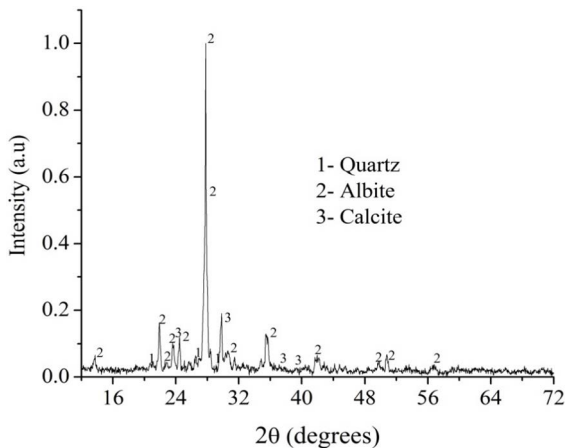


Figure 3. Diffractogram of the crusher residue used in the production of mortars.

Table 3 presents a mineralogical composition obtained through the XRD analysis of the cement. The cement presents 83.60% alite in its composition. This result was expected, because the alite must be the main constituent of the clinker after the processing, because it is its hydration reaction that confers the mechanical resistance to the cured cement, generating hydration products with high initial mechanical strength, hardening, and as it is more reactive than the belite, participates in the initial phases of hydration, being responsible for a great amount of the released heat. While the belite forms cements known as low energy, however

with a slower increase of mechanical resistance^{33,38-40}. The presence of quartz in the composition was expected, due to the presence of SiO_2 of the kaolite clay in clinker production. The quartz when in high contents (above 10% in relation to the cement mass) can reduce the quality of cement, either by its inert character or by the high hardness⁴¹.

Table 3. Mineralogical composition obtained through the XRD analysis of cement.

Mineralogical Phase	Percentage in the sample (%)
Quartz	5.47
Alite	83.60
Belite	10.93

Through Figure 3, which presents the diffractogram for the residue (filler), it is possible to identify the crystalline phases corresponding to the quartz (number “1”) albite ($\text{NaAlSi}_3\text{O}_8$) (number “2”) and calcite (number “3”). The Table 4 presents the mineralogical composition obtained through the filler XRD analysis.

Table 4. Mineralogical composition obtained by the XRD analysis of the crusher residue.

Mineralogical Phase	Percentage in the sample (%)
Quartz	1.58
Albite	87.36
Calcite	11.07

Through Table 4, it is verified that the major compound present in the filler was the albite, with 87.36%, followed by calcite (11.07%) and quartz (1.58%). The albite is a common mineral in the feldspar, typical of alkaline and acidic magmatic rocks belonging to the group of plagioclases, that the more calcics, end up providing losses of calcium and aluminum in its composition, thus generating the albite. Its presence in feldspar is explained because it is the last mineral to crystallize in molten rock^{42,43}. The albite is presented as a mineral that is widely used by the industry in the production of refractory artifacts due to its high resistance to heat⁴⁴. In relation to calcite and quartz, they are very common minerals in the Earth’s crust, because they form part of the mineralogical composition of igneous and metamorphic rocks, corresponding approximately 95% of the terrestrial layer⁴⁵.

By analyzing the results of the XRD, it can be said that none of the minerals that constitute the crusher residue could hinder the use of this material as a component of the mortar.

The results of the thermogravimetric analysis (TG) and its respective derivative (DTG) performed for the crusher residue are shown in Figure 4. The thermogram indicates the loss of 1% mass with the heat treatment up to 100 °C, the remaining mass constant until temperature of 750 °C, where there is a small loss (less than 1%). The first mass

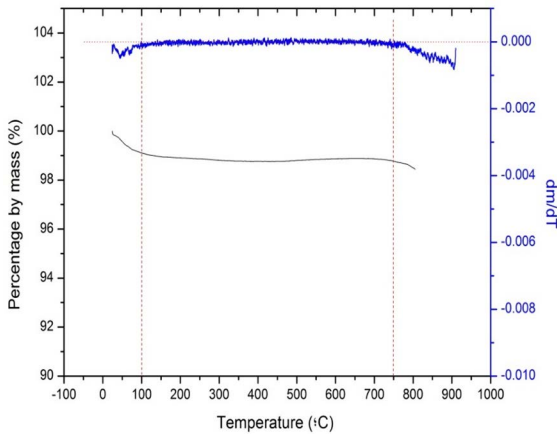


Figure 4. Thermogram of crusher residue and Differential TGA thermogram for crusher residue.

loss is possibly due to the volatilization of water molecules present on the surface of the material.

From the DTG thermogram it is possible to identify a constant behavior in the mass of the material, that is, without loss of mass until the temperature of 750 °C, where it is observed a loss of mass of 2.4%, due to a possible thermal decomposition of the calcite, promoted by the gas release carbon dioxide (CO₂). The thermal decomposition of the calcite (CaCO₃) occurs from 700 °C, with phase transformation at 850 °C, resulting in the formation of lime (CaO) and CO₂ release and the relationship between the calcite mass loss (CaCO₃) and the decomposition of the organic matter occurs between the temperatures of 350 °C and 800 °C^{14,45,46}.

By the TGA analysis, a percentage of 96.7% of residue was obtained up to 900 °C, therefore, the residue presented a thermal characteristic behavior with very low mass loss (2.4%). This result confirms too, the loss on ignition obtained by the chemical analysis, indicating a low number of contaminants.

3.2 Characterization of the mortar in the fresh state

The results of the consistency (workability) of the six mortars obtained are shown in Figure 5 and the water retention results are expressed in Figure 6. Which one sample is the reference and the others are the in different proportions of residue, 5%, 10%, 15%, 20% and 30%, replacing the cement. The results are expressed as mean values of the tests, and the averages followed by the same letter in the column do not differ from each other by the Tukey test at 5% probability.

From Figure 5, it can be observed that for sample A, with a 5% substitution of the cement by crusher residue, the consistency increased by 12.41% in relation to the reference sample, showing that there is statistically significant difference when compared to the sample reference. However, this behavior is not observed for samples B, C and D, which

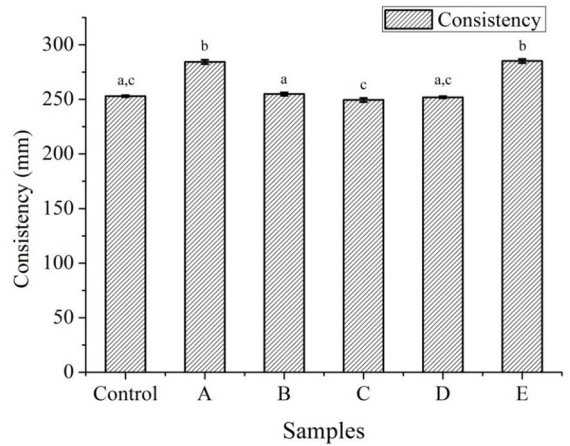


Figure 5. Results of the consistency of the reference mortar and the mortar with 5%, 10%, 15%, 20% and 30% of residual in partial replacement of the cement.

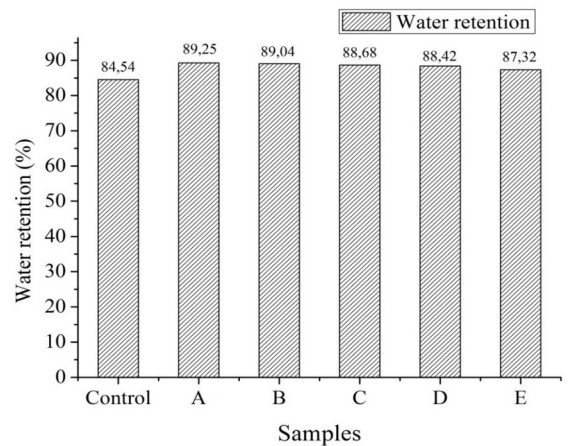


Figure 6. Results of the water retention test on the reference mortar and on the mortar with 5%, 10%, 15%, 20% and 30% of residue in partial replacement of the cement.

did not show a significant difference when compared to the reference sample. According to NBR 13276²⁵, for a mortar presenting good workability it must have a standard consistency of 255±10 mm, so the samples with 10%, 15% and 20% of residue comply with that standard. Therefore, it can be said that samples B, C and D show good workability, that is, they adhere well to the trowel, slide smoothly, and easily deform under loads. While the samples A and E were outside the range of values stipulated by the standard, possibly because the particles of the residue are immersed inside the binder, without internal cohesion and with tendency to deposit by segregation, in this way, the mortar spreads without allowing the proper execution of the work.

Figure 6 shows that the addition of the residue in the mortar and, consequently, the reduction of the cement, generated a higher percentage of water retention, with a maximum value of 5.57% in relation to the reference sample. The values presented in Figure 6 show that all mortars with

residuals have a normal water retention, according to NBR 13277²⁸, which standardizes as normal 80 to 90% retention and as high above 90%. The mortar must retain the settling water which serves both to lubricate the dry materials and to ensure cement hydration⁴⁷. The retention of water influences some characteristics of the mortar coating, not only in the fresh state, but also in the hardened state, as the evolution of the hardened⁴⁸.

3.3 Characterization of the mortar in the hardened state

The obtained mortars were characterized in the hardened state as the tensile strength in flexion, resistance to compression in the prism and the tensile strength in diametrical compression, with a curing time of 7 and 28 days. The results presented are the average of each test with its respective standard deviation and the averages followed by the same lowercase letter in the 7-day column and the averages followed by the same capital letter in the 28-day column do not differ from each other by the Tukey test at 5% probability.

Figure 7 presents the results of the flexural tensile strength test of the mortar samples obtained with different proportions of the residue and the reference mortar in the hardened state at 7 and 28 days.

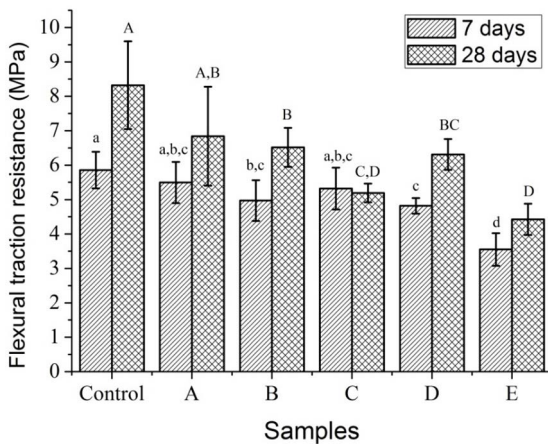


Figure 7. Results of tensile strength in flexion of mortar samples obtained with different proportions of the residue, in the hardened state at 7 and 28 days.

It can be seen from Figure 7 that for the 7-day samples, a tendency was observed in the reduction of tensile strength in flexion as the cement was replaced by the residue. This behavior was expected because the introduced residue behaves as an inert material, and the reactive material, in this case the cement, is withdrawn in the same proportion. This perception is visualized for the D and E mixes, where they presented a significant reduction in tensile strength in flexion, of 17.75 and 36.52%, respectively, when compared with the control sample. However, samples A, B and C (5%, 10% and 15%) did not differ significantly from the control sample. Mix C, with 15% of residue, presented the best performance among

the mortars with residue, presenting a decrease of resistance of 9.21% in relation to the reference sample.

For mortars aged 28 days, Figure 7, it was also observed that there was a decrease in the tensile strength in the flexion when comparing the mix reference to the other mortars. Sample A was the one that presented better performance in relation to mixes with substitution, presenting no statistically significant difference when compared to the reference mix. The mixes C and E showed the greatest decrease in tensile strength, being 37.62 and 46.75%, respectively. When comparing tensile strength in flexion at the age of 7 and 28 days, it is verified that for higher age, there is an increase in resistance, since it is believed that there was not enough time for hydration in the mortar mass.

The variation of the compressive strength of the mortars containing the residue and the reference mortar, after 7 and 28 days of cure, is shown in Figure 8. For mortars at the age of 7 days, a reduction in compressive strength was observed as the percentage of residue increased, except for the mix C which presented a 13.91% increase in resistance when compared to the reference mix. In the mix E was observed the least resistance, being 40.99% smaller than the reference mix. However, for mortars with 28 days of age, there was a small decrease in resistance to mix A, B and C, when compared to the reference mix, with no statistically significant difference. When comparing the reference sample with the D and E mixes, these showed a significant difference. The mix C, replacing 15% of the residue with cement, was the one that presented better performance with the replacement of cement, having only a decrease of 6.27% in the resistance.

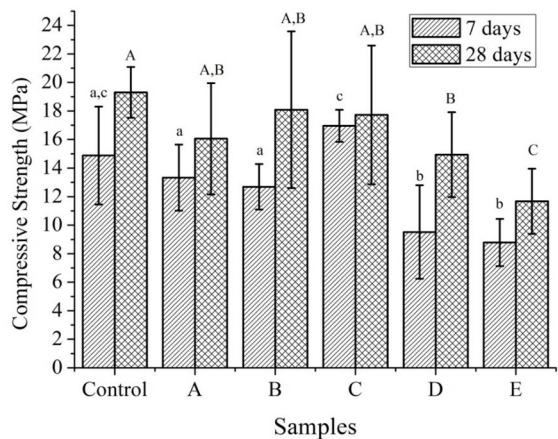


Figure 8. Results of the compressive strength of mortars obtained with different proportions of the residue, in the hardened state at 7 and 28 days.

The fact that the compressive strength did not suffer significant interference in samples A, B and C is possibly due to the microfiller effect of the powder, where the smaller particles of the dust fit into the voids of the particles of the aggregate and binder, filling the pores of the mortar and

making them discontinuous. For the other mixes, D and E, the decrease can be the removal of a larger quantity of cement and the introduction of the inert material, as already mentioned. As with tensile strength in flexion, the increase in compressive strength may have occurred until the closure of the sizing package and decrease in the sequence, with increase in the contents of substitutions and reduction in the quantity of cement, results also observed by Canova⁴⁹. The decrease in resistance, in this case, can be explained by De Larrard⁵⁰, which states that after full completion is reached, any fine grain that is added to the mixture can produce the removal effect of the larger grains, that is, the fine particles are added and do not fit perfectly between the apertures of the smaller particles damaging the densification. Thus, an increase in pore volume and a decrease in packing density occurs, which may adversely affect the strength of the mortar.

The results obtained of the tensile strength in the diametrical compression for the mortars obtained using the residue and for the reference mortar are presented in Figure 9, for 7 and 28 days of cure.

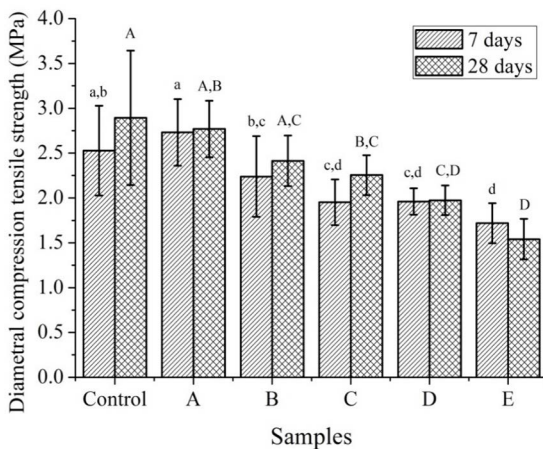


Figure 9. Result of tensile strength with diametrical compression in mortars, in the hardened state of 7 and 28 days.

It can be verified for mortars A, B and C, aged 7 days, there was an increase in mix A, B and C, and a statistically significant reduction in the D and E mixes compared to the reference mix. Mix A presented a 1.10% increase in tensile strength when compared to the reference mix. It is observed that the mix E presented a reduction of 36.30% in tensile strength with diametral compression, when compared to the reference mix. It has been found that the tendency is to reduce the tensile strength of the mortar as the proportion of residue increases. However, for the proportions of 5% and 10% of residue in substitution to the cement, there is no significant difference in the tensile strength when compared to the resistance of the control mix. For sample E, with 30% residual and 30% less cement, the decrease in compressive strength was 46.71% in relation to the reference

mix, proving to be statistically significant the difference between these mixes.

3.4 Tests for the pozzolanicity of the mortar

The mortar samples were submitted to pozzolanicity tests according NBR 5751³⁰ (2015) with simple compression tests, Table 5. The results shown that not is possible to define the basaltic fillers as a pozzolanic material because the values of the medium maximum values of the compression resistance of the mortar samples containing the basaltic filler is 0.28 MPa, lower than 6.00 MPa, defined by the NBR 12653³¹ (2015). So, the effect of the basaltic filler in the mortar is only to substitute the cement and to compensate the mechanical resistances.

Table 5. Medium maximum values of the compression resistance of the mortar samples containing the basaltic filler.

Sample	Rupture stress (MPa)
1	0.27
2	0.30
Average	0.28

4. Conclusion

From the laser granulometric analysis of the residue it was possible to observe that its particles were larger than the particles of the cement, providing a better sizing between the aggregate/binder. Through the FRX in the residue, it was possible to verify a percentage of 78.45% SiO₂, Al₂O₃ and Fe₂O₃, which according to NBR 12563²³, the material has pozzolanic characteristics.

It is possible to observe that the higher the substitution of cement by residue, the lower its resistance, this effect may be related to the substitution of the reactive material by a material with low reactivity, and may also be related to the a/c factor that was not altered as the cement was replaced by the residue, implying an increase in porosity and consequently the decrease of the resistance.

It is possible to highlight the mortar with partial replacement of 10% of Portland cement by crusher residue, where it presented behavior close to the reference mix. The good results may be related to the ideal particle size closure, improving the packaging and consequently gains in the resistances. The retention of water in this mix was within the expected, also favoring its application. Although the mix did not reach the value of the reference mortar in the tests, the difference did not make it impossible to apply ceramic blocks.

Finally, it can be stated that there is technical feasibility of the use of crusher residues in mortars and the substitution content of 10% of cement was the most adequate among the percentages tested, being this the mix that presented

characteristics close to the reference in relation the other substitutions.

5. Acknowledgment

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