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Assessment of the effect of fiber percentage in glass fiber reinforced slag-based geopolymer

Lais Alves ^a, Nordine Leklou^b, Fabio de Souza^c and Silvio De Barros ^d

^aDepartment Of Mechanical Engineering, Federal Center of Technological Education (Cefet/rj), Rio De Janeiro, Brazil; ^bIut Saint-Nazaire, GeM, Cnrs Umr 6183, Research Institute in Civil Engineering and Mechanics, University of Nantes, Saint Nazaire, France; ^cDepartment Of Administration, Federal Center of Technological Education (Cefet/rj), Rio De Janeiro, Brazil; ^dLINEACT CESI EA 7527, Saint-Nazaire, France

ABSTRACT

Fibers can increase the ductility of geopolymer materials, acting as reinforcements to improve mechanical properties. These improvements depend on fiber content and quantity. This study evaluates the impacts of different percentages of glass fiber on a GBFS (ground blast furnace slag) geopolymer matrix. The glass fibers were used on the critical length of 20 mm, obtained by pull-out test in previous studies. Percentages of 0.25%, 0.50%, 0.75%, and 1.00% of glass fiber in mass were tested in fresh state, by flow measurement, and in hardened state, by variation of shrinkage, water porosity and response of composites under flexural and compressive load conditions at 7, 14, 28, and 90 days. The mixture resulted in a high porosity geopolymer paste. In addition, an analysis of variance (ANOVA) was performed to evaluate the statistical significance of fibers in compressive strength. The results show that increasing percentages glass fibers, until 0.75%, enhances mechanical properties. Percentage of 1.00% started to show accumulation of branch of multifilament fibers which contribute to higher drying shrinkage. The ANOVA test showed that the percentage of fibers can influence the compressive strength. With the results, the optimum percentage of fibers for his composite mixture was found at 0.75%.

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Ground blast furnace slag; sodium silicate activating solution; fiber percentage; glass fiber; reinforced geopolymer

1. Introduction

Although, a high volume of cement Portland (CP) production can be interpreted as high industrial development, it can also cause concern in the environmental aspect. The production process of CP consists in burning of raw materials, such as limestone and clays, at high temperatures causing high releases of CO₂, a gas that is the main cause of the greenhouse effect and rising earth temperature [1]. To obtain CP, there is a reaction between calcite and silica, and each ton produced generates 550 kg of CO₂. In addition, there is a need for combustion of carbon-based fuels, generating an additional 400 kg of CO₂ [2]. The production of geopolymer generates lower carbon dioxide emission compared to ordinary CP, because it is present only in obtaining raw materials and not in the reaction of the final production of the geopolymer [3]. The production does not need limestone calcination and fuel combustion for production.

Geopolymers are characterized by acquiring high values of resistance to compression in the early ages [4–8]. Metha and Siddique [9] found that the material acquired about 92% of its resistance in just three days and 97% in 7 days, while in conventional concrete the percentage of resistance acquired at seven days was only 70%. Certain geopolymer compositions can

achieve a compressive strength of approximately 20 MPa in just 4 hours after preparation, when cured at higher temperatures [10], while ordinary cement Portland has strengths ranging between 11 and 26 MPa at 3 days of age [11]. Low shrinkage rates when compared to CP also makes geopolymer a promising material for applications in several areas of civil construction [12].

Geopolymer matrix can be based on different precursor materials, such as fly ash, metakaolin, ground blast furnace slag, amongst other types of materials source of alumina and silica. The material is susceptible to deformations, having low tensile strength [13] and relatively low fracture energy, thus the need for reinforcements [7]. Incorporated into cementitious matrices, reinforcements supply an increase in tensile strength, ductility, toughness, and improve durability [14]. For fibrous reinforced composites, i.e. the function of the matrix is to guarantee the orientation and spacing of the fibers, to transmit shear forces between the fiber layers to resist torsional and bending forces, and to prevent superficial damage to the fibrous reinforcement [14].

Geopolymer composites can be reinforced with particulates [15–18] and fibers, such as sisal and pineapple leaf [19], jute [20], polypropylene [21], glass [22,23], carbon [24], steel [25,26], amongst other types of

fiber [14,27–31]. The interaction with the matrix for particulates reinforced composites is influenced by the size of the reinforcement [17]. Fibers can be arranged in line or randomly, and oriented continuously or discontinuously [28]. It is important to select a reinforcement that is not reactive with the constituents of the geopolymer during the curing process, and that is chemically resistant to the high alkalinity of the geopolymer paste [32]. It is also necessary to limit corrosion, especially on steel [33] and polypropylene fibers [34], which can cause deterioration of the composite due to the reaction between the matrix and the corrosion agent.

Due to high modulus of elasticity and high tensile strength, the fibers increase the ductility of the material and prevent abrupt rupture [31]. These effects will be directly proportional to the fiber-matrix interaction force. Ranjbar et al. [34] states that a proper bond between reinforcement and matrix also depends on the wettability of the matrix. The adhesion between the surface of the fiber and the surface of the composite, is directly proportional to the strength of the geopolymer matrix [35,36]. Fiber quantity superior to the wettability of the matrix can cause greater porosity which affects the strength of the geopolymer [34]. On the other hand, some precursor material require more water to obtain the desired workability, which also increases porosity [8].

An experimental analysis is necessary to analyze the adhesion between the fiber matrix and determine the critical embedded length. The critical length represents the optimum length capable of promoting the greater adhesion and better mechanical performance of the composite and can be assessed through the pull-out test [37]. It is important to achieve the appropriate amount and proportion of fibers [17] for an effective increase in the strength of the composite to occur with an efficient transfer of loads between fibers and matrix [38].

Considering strengthening and hardening of the matrix, it is desirable to have high concentration of incorporated fibers, in large proportions. However, there is a considerable reduction in workability with the increase of these two variables. The volumetric fraction of fibers in the composite also influences the stability of an adequate interaction between fiber and matrix [17]. To reduce cracking due to shrinkage, it is more common to use low volumetric fraction [7]. To significantly increase properties such as toughness, impact resistance and rupture modulus, the use of a moderate fraction is recommended [13].

This work seeks to verify how fresh and hardened properties of geopolymer composites, based on a pure ground blast furnace slag (GBFS)-based matrix and random synthetic glass fibers with 20 mm in length [39], are influenced by different glass fiber percentages. The specimens had 0.25%, 0.50%, 0.75%, and

1.00% of glass fiber content and were tested for compressive and flexural strength at 7, 14, 28, and 90 days, as well as water porosity at 28 days and shrinkage up to 270 days. It aims to examine the differences caused by fiber quantity in the specimens and to discover the optimum percentage of fibers for the composite mixture.

2. Materials and methods

2.1. Geopolymer precursor and activating solution characterization

GBFS was obtained by ECOCEM from France. Table 1 presents the chemical composition for the GBFS used in this research, according to the product's technical sheet. Median particle size for the GBFS from ECOCEM is $D_{50} = 11.8 \mu\text{m}$, with 95% of particles passing through $32 \mu\text{m}$ and apparent density of $0,8 \pm 0,1 \text{ g/cm}^3$.

Alkaline activating solution employed in the mixtures, in its proper proportions, consisted of mixing sodium hydroxide (NaOH) solution with sodium silicate (Na_2SiO_3) solution. NaOH was purchased from ALFA-AESAR prepared in pellet form, white colored, with 98% purity. Na_2SiO_3 was purchased from VWR in liquid form, of pH between 11 and 11.5, and density 1.35 g/cm^3 at 20°C . The chemical compositions for the mixture can be found in Alves et al [40]. Sodium silicate activator (Na-Si) was prepared by mixing 10 M NaOH and Na_2SiO_3 solution, with 2 $\text{Na}_2\text{SiO}_3/\text{NaOH}$ mass ratio. The produced Na-Si activator contained 66.7% water with 0.7 $\text{Na}_2\text{O}/\text{SiO}_2$ molar ratio. After preparation, the solution was kept for 24 hours in ambient conditions.

2.2. Fiber characterization

The glass fiber used was commercial S2-glass which have high tensile strength of about 3700 to 4300 MPa, without alkaline oxides, containing 65% SiO_2 , 10% MgO, and 25% Al_2O_3 . S2-glass fibers have a higher level of silica than standard glass fiber products, which results in improved physical properties such as tensile and compressive strength, high temperature resistance, and improved impact resistance. The diameter for a single fiber was measured around 17–20 μm , by Scanning Electronic Microscopy (SEM) using an electron microscope equipped with a secondary electron sensor and a backscattered electrons sensor. Strands of glass fibers measured around 1 mm in diameter and 20 mm in length. Fiber-matrix adhesion, shear stress, and relative displacement was assessed by fiber pull-out test in previous work by the authors [39]. The authors stated that fiber material

Table 1. Chemical composition of precursors (wt%).

CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Na ₂ O	SO ₃	TiO ₂	MnO	LOI
43.2	37.2	10.5	0.6	7.0	0.6	0.1	0.5	0.3	0.7

influences the efficiency of the reinforcement and applied force and greatest efficiency was obtained by glass fibers incorporated 20 mm in GBFS matrix. Differences can be explained by chemical interactions between fiber and matrix, where fibers with shorter lengths results in deficiencies of the transmission of the external loads and higher lengths are above the critical embedded length.

2.3. Glass fiber GBFS geopolymer composite preparation

The precursor material and the amount of glass fiber for the mixture was mixed for three minutes to have a more homogeneous mixture and avoid poor distribution. Fibers were randomly distributed. Research have shown the shrinkage of the composite is not affected by fiber orientation [41]. The activating solution mixed with the water was added to the dry mixture and blended for three minutes more. The percentage of water in the mixture was 12.4% and solid-to-liquid ratio 2.0. The material was immediately poured into 4 cm × 4 cm × 16 cm stainless steel molds and cured with a theoretical cycle proposed by Leklou et al [42]. The first phase consists in a pre-cure at 20°C for 1 h. At the second stage the temperature rises at a constant rate for 3 h until 40°C. The third stage maintains the temperature at 40°C for 10 h. And the last stage decreases at a constant rate for 10 h, until 20°C. After 24 hours, the specimens were demolded and kept in a chamber with an average temperature and humidity of 20°C ± 3°C and 50% ± 5%, respectively, until the testing day. The glass fiber content in geopolymer paste varied in the range of 0.25%, 0.5%, 0.75%, and 1.0%. Percentages higher than 1.0% resulted in accumulation of branch of multifilament fibers in particular place, nonuniformity, and agglomeration in the matrix. All the variations found in the development of the specimens were due to common causes (with small individual influence), with no variation due to special causes (great individual influence) being detected.

2.4. Experimental program

To evaluate workability, flow measurement tests were conducted in accordance with ASTM C1437 [43]. The geopolymer was poured into a truncated conical mold, measuring 50 mm in height, 100 mm diameter at top, and 70 mm of diameter at bottom, in two equal layers. After each layer, the paste was tamped 20 times for compaction. After one minute, the paste was demolded, as the conical mold was lifted, and the table with the specimen was dropped 25 times in 15 seconds. The flow is the percentage of the increase in base diameter of the paste in relation to the initial measurement of diameter.

Three specimens, of 4 cm × 4 cm × 16 cm, with fixed studs on the top and bottom, were tested for each formulation measuring shrinkage. The variation of axial expansion over time was measured with an extensometer with ± 1 μm of resolution, and weight, monitored using a digital scale with a resolution of ± 0.1 g.

Water Porosity essay consisted of measuring the Buoyant mass of the saturated specimen in water, the saturated surface-dry mass, and the oven-dry mass at 105°C, porosity is then computed using three measured values, as stated by NF P18-459 [44]. Porosity was obtained at 28 days and is expressed as a function of the apparent density and the skeletal density.

The flexural and compressive strengths developments of the specimens were obtained after 7, 14, 28 and 90 days, according to the European Standard NF EN196-1 [45] in a Cyber-Plus Evolution testing machine. Specimens measuring 4 cm × 4 cm × 16 cm submitted to the 3-point bending test are broken into two half-prisms which are both tested to compressive resistance. SEM analysis was done using an electron microscope equipped with a secondary electron sensor and a backscattered electrons sensor for the samples.

Finally, an analysis of variance (ANOVA) was performed to verify if there is any evidence of a possible difference between the population means of the analyzed parameters that affect the strength of geopolymer. According to Levine [46], if this evidence of possible differences between the means is found by comparing the value of the F statistic, generated with the calculations performed and its results synthesized in the ANOVA table, with the tabulated value of this statistic considering a level of confidence for the test, it is possible to verify which population data differs from another, using the Tukey-Kramer test. Some hypotheses are intrinsic to the model, such as the randomness and independence of the data, the assumption that the sample size is of sufficient size to consider the Normal distribution, and equality of variance in the data of its populations [47].

3. Results and discussion

3.1. Fresh state properties

The strength of the structure depends on the density ratio and compaction, which depends directly on sufficient workability. An increase on the water-to-solid ratio indicates an increase in workability and increases porosity. Workability of pure geopolymer is relatively high and is expected to decrease as the percentage of fibers increases and offers higher shear resistance to flow [21]. The influence of glass fiber content on the flow reduction of

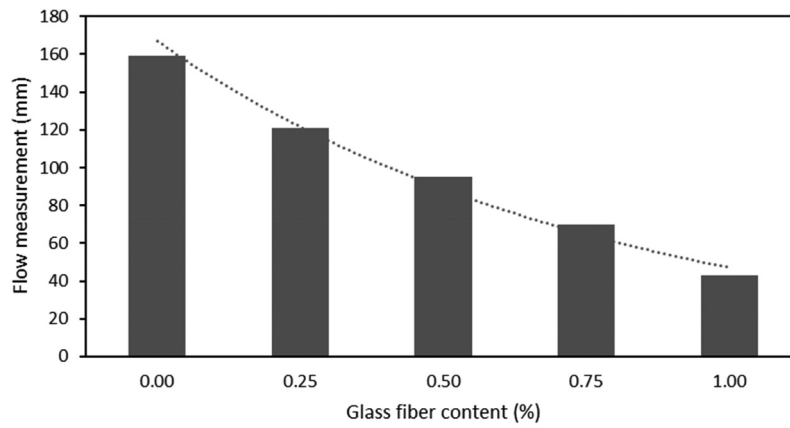


Figure 1. Workability by flow measurement tests by % of glass fiber on the geopolymer composite.

GBFS geopolymer composites in fresh state is shown on Figure 1. By adding 0.25%, 0.50%, 0.75%, and 1.00% of fibers, a reduction of 24%, 40%, 56%, and 73% in flow results, respectively, can be observed. Percentages higher than 1.00% presented very low workability and compaction, being discarded for use in the following stages of the study. Nematollahi et al. [48] also observed that addition of glass fibers into the geopolymer matrix generated a cohesive mixture and decreased the

workability. Higher volumetric replacement of glass fibers in the geopolymer concrete mix lowered the slump values significantly.

3.2. Effect of fiber percentage on mechanical properties

As indicated in Figure 2a, addition of 0.50% and 0.75% of glass fiber improves the flexural strength of the matrix. At 28 days, compressive strength was 15%

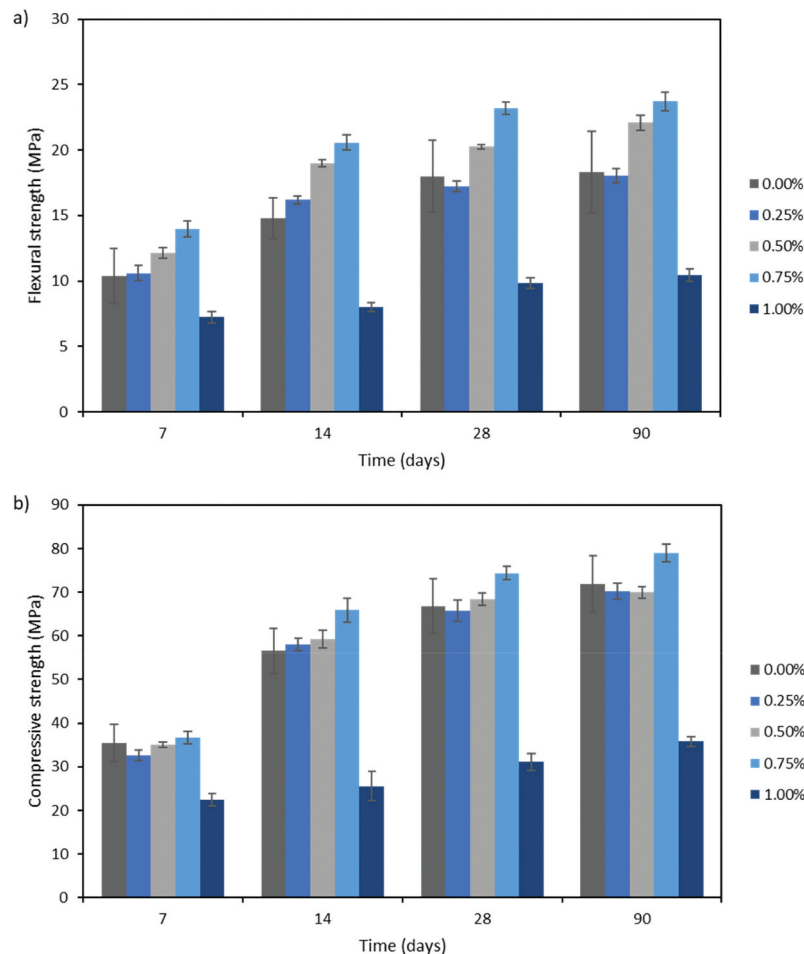


Figure 2. Maximum stress development over time by different fiber percentages for flexural strength (a) and compressive strength (b).

higher for 0.50% (20.3 MPa) and 30% higher for 0.75% (23.2 MPa) than the reference mixture with 0% (18.0 MPa). For the addition of 0.25%, the values were maintained approximately equal when comparing to 0% of fibers. At 28 days, a difference of 4% is presented for the mixture and the referred composite. The final strength of the matrix improved lightly (44%) compared to early strength for 1.00% fiber content, while for 0.75%, this difference was 70%. This behavior can be explained by the defect propagation observed in specimens for 1.00%.

Shrinkage cracks started to appear at 7 days curing, and accumulation of branch of multifilament fibers in particular places of the specimens could be observed. Nonuniformity in distribution can cause a higher volume of pores, which effect the flexural strength of the specimens. Flexural strength results were 45% lower (9.9 MPa) than reference for 28 days testing.

The compressive strength variation from 7 to 90 days is presented on Figure 2b, the same pattern explained in flexural strength can be observed. The addition of 0.50% and 0.75% of glass fiber improves the compressive strength of the matrix. At 28 days, compressive strength was 2% higher for 0.50% (68.5 MPa) and 11% higher for 0.75% (74.3 MPa) than the reference mixture with 0% (66.9 MPa). For the addition of 0.25%, the values were maintained approximately equal when comparing to 0% of fibers. At 28 days, a difference of 2% is presented for the mixture and the referred composite. For the addition of 1.00% of fibers, compressive strength results were 54% lower (31.1 MPa) than reference for 28 days testing. Thus, paste hardening is a dominant factor in compressive strength over time, improving about 115%, from 7 days to 90 days, for the 0.75% fiber content specimen.

3.3. Effect of fiber percentage on mechanical properties using analysis of variance

An analysis of variance (ANOVA) was performed to evaluate the application of fibers in the compressive strength. The results of the ANOVA statistical test, are structured in Table 2, indicating the *F-values* calculated, and the respective *p-values*. For the calculation of each *F-values*, the *MS* (mean square) of each factor, or the interaction between them, is divided by the *MS*

Table 2. Two-way ANOVA result for fiber percentage.

ANOVA	df	SS	MS	F-value	p-value
Time of essay (days)	3	4.95×10 ⁴	1.65×10 ⁴	9.41×10 ²	2.10×10 ⁻⁷⁴
Percentage of fibers (%)	4	4.75×10 ⁴	1.19×10 ⁴	6.77×10 ²	1.37×10 ⁻⁷²
Time of essay * Percentage of fibers	12	5.74×10 ³	4.78×10 ²	2.72×10 ¹	3.92×10 ⁻²⁶
Error	100	1.76×10 ³	1.75×10 ¹	-	-
Total	119	1.05×10 ⁵	-	-	-

of the errors. The *MS* of each factor, of the interaction and of the errors is obtained by dividing the *SS* (sum of squares) of each factor, of the interaction and of the errors, by the respective *df* (degrees of freedom).

Each *df* of the factors will be composed of the number of groups minus one. In the interaction, the *df* of each factor is multiplied, time of essay and percentage of fibers. The total *df* is the number of elements of the study minus one. Therefore, the *df* of the error consists of subtracting the total *df* by the *df* from each factor and the *df* for the interaction of the factors.

These calculated *F-values* are compared with the tabulated values of the statistic, and the null hypotheses are rejected if the calculated values are greater than the tabulated values before the level of significance defined for the tests. The first test available in this table, refers to the verification of possible interaction between the factors (in the third line of the table); there are also tests to analyze the possible effects of the factors separately (in the first and second lines of the table).

It is noticed that the null hypothesis for the interaction test between the data obtained with the date of essay and those recorded by the percentage of fibers is rejected, even considering a reduced level of significance of 1%, indicating that the compressive strength with some percentages of fibers is higher for certain curing times, and this fact compromises the analysis of the factors (curing time and percentage of fibers) separately. It is worth noting that the lower the level of significance, the greater the probability that population data will be recorded in its result [49].

Based on this observation, and following the artifice presented by Levine et al [46] for these cases, the values obtained in the tests are converted to different groups, regardless of the originating factor. Thus, if initially there were four groups of compressive strength by curing time (7, 14, 28, and 90 days) and five groups of fiber percentage (0%, 0.25%, 0.5%, 0.75%, and 1%), these converged to a one-way ANOVA with 20 levels of analysis. The results generated with these data are summarized in Table 3. The results of the ANOVA statistical test, are structured in Table 3, indicating the *F calculated*, and the respective *F critical* (tabulated). This calculated value is compared with the tabulated value, with the null hypothesis of the test for equality between the means being rejected if the calculated value is greater than the tabulated in the level of significance defined for the test.

Table 3. Result of one-factor ANOVA for compression with percentage of fibers.

ANOVA	df	SS	MS	F	F critical
Between groups	19	1.03×10 ⁵	5.41×10 ³	3.09×10 ²	2.35
Within groups	100	1.75×10 ³	1.75×10 ¹	-	-
Total	119	1.05×10 ⁵	-	-	-

The result of the *F* statistic obtained in Table 3 will be compared with the tabulated value of the statistic, considering the level of significance proposed for the test, the null hypothesis that all arithmetic means of the studied populations are equal. If this hypothesis is rejected, it appears that there is evidence that at least one of the population averages is different from the other. And based on this perception, the Tukey-Kramer test can be performed to see between which averages there is this possible evidence of differentiation.

Considering the level of significance, $\alpha = 1\%$, the value of the critical *F* statistic for 15 degrees of freedom in the numerator and 60 degrees of freedom in the denominator is 2.35 [46]. The critical *F* statistic is tabulated and represents the frontier to reject the null hypothesis of the equality between means. And since the *F*-value found with the study data is 308.19, higher than the *F* critical, the null hypothesis is rejected and at least one of the means differs from the other.

When performing the Tukey-Kramer test to verify which average(s) differ(s) from other(s), the critical value found for the analysis was 43.67 and, considering the 190 possible difference results between the 20 groups analyzed, statistically significant differences were found between 78 groups.

Table 4 presents the quartile of the most significant different found between the 78 groups. Considering the data presented, a statistically significant difference between the means of the compression strength results were obtained between the values of compression strength at 7 days, and the values 28 and 90 days of compression, regardless of the percentage of fibers

used can be observed. Considering, e.g. the first difference recorded in this table [(7d.0.25% – 90d.0.75%)] it reads “the module of the difference between the result obtained with 7 days of curing and 0.25% addition of fibers and the result generated with 90 days of curing and 0.75% addition of fibers.” A statistically significant difference is also perceived between the means of the compression strength results obtained between the values of compression strength at 14 days, and the values of compression strength at 28 and 90 days, regardless of the percentage of fibers used.

Results also indicate a statistically significant difference between the means of the compression strength results obtained between the values of compression strength at 28 days with the results obtained of compression strength at 90 days, regardless of the percentage of fibers used. It is worth noting that the processing of results obtained with 1% fiber content have a statistically significant difference with values generated with all other percentages, even when analyzed on the same category of date of essay, considering 7, 14, 28, or 90 days. It is also observed that 55% differences (in module) of the quartile, are defined by the fiber percentage of 0.75%. Thus, validating the discussion of the results presented previously.

3.4. Effect of fiber percentage on drying shrinkage

As can be observed in Figure 3, the drying shrinkage reduced with the addition of glass fibers, from 0.25% to 0.75% in geopolymer composites. As the % in fiber content increased, it can be expected that fibers located parallel to the main axis of the specimen are able to overcome shrinkage stress due to the interface contact area, as well as act prevent abrupt failure due to linkage in microcracks that appeared [21]. The magnitude of the porosity, size, shape, and the continuity of the capillary system in the mixture are the main factors that influence the values for drying shrinkage [41]. For these percentages, the specimens underwent

Table 4. Significant differences between groups.

Parameter	Diff.	Parameter	Diff.
[(7d.0% – 90d.0.75%)]	69.48	[(7d.1% – 90d.0.5%)]	76.11
[(7d.0.25% – 90d.0.75%)]	74.19	[(7d.1% – 90d.0.75%)]	90.45
[(7d.0.5% – 90d.0.75%)]	70.22	[(14d.1% – 28d.0.75%)]	78.08
[(7d.1% – 14d.0.75%)]	69.66	[(14d.1% – 90d.0%)]	74.11
[(7d.1% – 28d.0%)]	71.12	[(14d.1% – 90d.0.25%)]	71.53
[(7d.1% – 28d.0.25%)]	69.35	[(14d.1% – 90d.0.5%)]	71.08
[(7d.1% – 28d.0.5%)]	73.68	[(14d.1% – 90d.0.75%)]	85.42
[(7d.1% – 28d.0.75%)]	83.12	[(28d.0.75% – 28d.0.1%)]	69.22
[(7d.1% – 90d.0%)]	79.15	[(28d.1% – 90d.0.75%)]	76.55
[(7d.1% – 90d.0.25%)]	76.57	[(90d.0.75% – 90d.1%)]	69.06

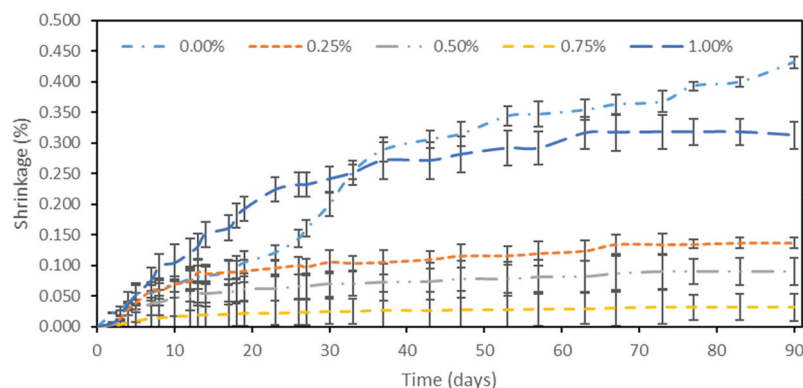


Figure 3. Glass Fiber content effect on controlling the shrinkage of fly ash-based geopolymer.

shrinkage strain for about 10 days, and then remained practically unchanged. Percentage of 1.00% started to show accumulation of branch of multifilament fibers in particular places, and non-uniformity in distribution, which can have caused the increased shrinkage due to the pores trapped among clusters of fibers. Silva et al [41] states that the increase in matrix porosity, caused by the addition of fibers above 1.00% in volume, result in retention of moisture which contributes to the higher drying shrinkage.

3.5. Effect of fiber percentage on water porosity

The results displayed in Figure 4 show the relationship between the fiber-reinforced geopolymer total porosity and maximum compressive strength, both

measured at 28 days. Porosity directly affects compressive strengths [50]. The addition of the glass fibers at 1.00% increased the matrix porosity of the geopolymer composites in approximately 19%, compared to the reference geopolymer, and a decrease in compressive strength can be noted (see also Figure 2b). This can be explained by the fiber quantity higher than wettability of the matrix, which can cause an increase in porosity and impair the transfer of efforts as a result from the agglomeration of fibers [51]. Porosity results varied from 1% to 5% for samples from 0.25% to 0.75% which did not affect negatively mechanical properties results.

SEM analysis was carried out after the flexural and compressive essays, to observe fiber distribution in the cross section of each mixture. In Figure 5 the

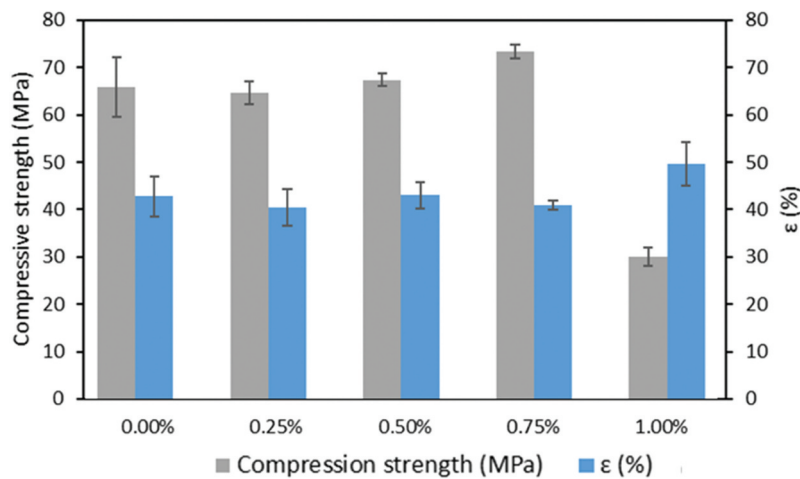


Figure 4. Relationship between concrete porosity (ϵ) and compressive strength for different fiber percentages in mass.

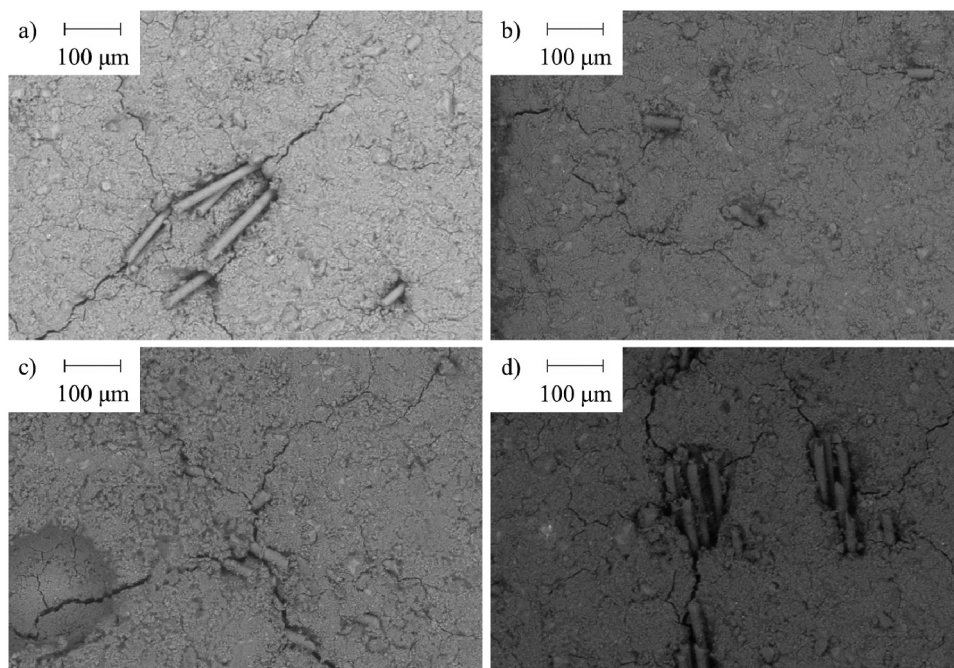


Figure 5. Scanning electron microscopy (SEM) analysis for the different sets of mixtures (a) 0.25%, (b) 0.50%, (c) 0.75%, and (d) 1.00% of glass fiber addition.

images for the different mixtures can be observed. As seen in previous results, 1,00% fiber content specimens showed a decrease in durability and mechanical properties. This can be caused by the high volume of fibers, which started to show accumulation of branch of multifilament fibers in a particular place, nonuniformity, and agglomeration of the matrix, as showed in Figure 5d, which is not observed at other percentages. Some research has found the same loss in properties after reaching the optimum content of natural fiber in the matrix [52–54].

4. Conclusions

This study evaluated GBFS-based geopolymer reinforced by glass fiber from the aspects of shrinkage variation, fresh and mechanical properties. The workability of the composite reduced by increasing percentages of fiber content due to higher shear resistance to flow.

Shrinkage of the composite can be controlled based on the fiber content and showed best results for 0.75% addition of fiber into the geopolymer matrix. It can be concluded that shrinkage variation significantly affects the mechanical properties of glass fiber-reinforced GBFS geopolymer. Water porosity was not significantly affected by addition of fibers and had a small variation as the percentage ranged from 0.25% to 0.75%. For 1.00% the porosity was higher, and compressive strength decreased, showing the percentage was above the optimum value.

Mechanical properties increased with the addition of glass fiber into the matrix, until the optimum percentage of fibers of 0.75%. Specimens of 1.00% fiber content in the geopolymer matrix showed accumulation of branch of multifilament fibers in particular place, nonuniformity, and agglomeration of the matrix which decreased durability and mechanical properties due to pores trapped among clusters of fibers.

The application of the ANOVA statistical tool found evidence of an interaction between the results obtained with the percentage of fiber and the compressive strength at different ages of the tested geopolymer mixture. Two findings were highlighted: (1) the data with fiber content of 1% proved to be, on average, statistically different from all other percentages of concentration, regardless of the date of the compressive strength test, and (2) that almost half of the highest, the values of the average differences recorded were in specimens with 0.75% of fiber content in their composition, highlighting this percentage of the other percentages of fiber used in the test.

In conclusion, the addition of fibers from 0.25% to 0.75% in GBFS geopolymer matrix improved durability and mechanical properties. Percentages higher showed a decrease in the results for the composite

studied. A composite mixture of GBFS reinforced with glass fiber, with smaller shrinkage and good mechanical results can be produced by adding 0.75% of fiber to the reference mixture.

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ORCID

Lais Alves  <http://orcid.org/0000-0003-0543-2374>

Silvio De Barros  <http://orcid.org/0000-0002-2520-569X>

References

- [1] Cement Sustainability Initiative (CSI). Guidelines for emissions monitoring and reporting in the cement industry. Switzerland: World Business Council for Sustainable Development (WBCSD); 2012 (Report; ISBN:2-940240-77-9).
- [2] Davidovits J From ancient concrete to geopolymers. *Arts et Metiers Magazine*. 1993;180:8–16.
- [3] Davidovits J Properties of geopolymer cements. Kiev, Ukraine. In Proceedings of the First International Conference on Alkali Cements and Concretes. 1994.
- [4] Bakharev T. Geopolymeric materials prepared using class F fly ash and elevated temperature curing. *Cem Concr Res*. 2005;35(6):1224–1232.
- [5] Davidovits J. Geopolymer chemistry and applications. Saint-Quentin: Institut Geopolymere; 2020.
- [6] Tchadjjié L, Djobo J, Ranjbar N, et al. Potential of using granite waste as raw material for geopolymer synthesis. *Ceram Int*. 2016;42(2):3046–3055.
- [7] Sakulich AR. Reinforced geopolymer composites for enhanced material greenness and durability. *Sustain Cities Soc*. 2011;1(4):195–210.
- [8] Duxson P, Fernández-Jiménez A, Provis JL, et al. Geopolymer technology: the current state of the art. *J Mater Sci*. 2007;42(9):2917–2933.
- [9] Mehta A, Siddique R. Strength, permeability and micro-structural characteristics of low-calcium fly ash based geopolymers. *Constr Build Mater*. 2017;141:325–334.
- [10] Davidovits J. Geopolymer cement: a review. *Geopolym Sci Technics*. Saint-Quentin: Geopolymer Institute Library; 2013 (Technical Paper; no. 21

- [11] Zhang H. Building materials in civil engineering. In: Woodhead Publishing Series in Civil and Structural Engineering. Amsterdam: Woodhead Publishing; 2011.
- [12] Bigno IC Geopolímeros à base de resíduos agrícolas e agroindustriais [Geopolymers based on agricultural and agro-industrial waste]. [dissertation]. Rio de Janeiro: Military Engineering Institute (IME) 2008. Portuguese.
- [13] Bentur A, Mindess S. Fibre reinforced cementitious composites. Ohio (OH): CRC Press; 2007.
- [14] Vickers L, Van Riessen A, William DA. Fire resistant geopolymers: role of fibres and fillers to enhance thermal properties. Berlin: Springer; 2015.
- [15] Mano EB. Polímeros como materiais de engenharia [Polymers as engineering materials]. São Paulo: Edgard Blücher; 1991. (Portuguese).
- [16] Natali A, Manzi S, Bignozzi MC. Novel fiber-reinforced composite materials based on sustainable geopolymer matrix. *Procedia Eng.* 2011;21:1124–1131.
- [17] Metha PK, Monteiro PJML. Concrete: microstructure, properties, and materials. Pennsylvania (PA). McGraw-Hill Publishing; 2006.
- [18] He PG, Jia DC, Lin TS. Effects of high-temperature heat treatment on the mechanical properties of unidirectional carbon fiber reinforced geopolymer composites. *Ceram Int.* 2010;36(4):1447–1453.
- [19] Correia EA, Torres SM, Alexandre MEO, et al. Mechanical performance of natural fibers reinforced geopolymer composites. *Mater Sci Forum.* 2013;758:139–145.
- [20] Lopes G, Paes V, Dos Santos J, et al. Study of the compression resistance of a geopolymer based composite with added jute fiber. *Res Dev Material Sci.* 2019;12(3):1276–1280
- [21] Ranjbar N, Mehrali M, Behnia A, et al. A comprehensive study of the polypropylene fiber reinforced fly ash based geopolymer. *PLoS ONE.* 2016;11(1):e0147546.
- [22] Alomayri T. Effect of glass microfibre addition on the mechanical performances of fly ash-based geopolymer composites. *J Asian Ceram Soc.* 2017;5:334–340.
- [23] Vijai K, Kumuthaa R, Vishnuramb BG. Properties of glass fiber reinforced geopolymer concrete composites. *Asian J Civ Eng.* 2012;13(4):511–520.
- [24] Yan S, He P, Jia D, et al. Effect of fiber content on the microstructure and mechanical properties of carbon fiber felt reinforced geopolymer composites. *Ceram Int.* 2016;42:7837–7843.
- [25] Eik M, Antonova A, Puttonen J. Phase tomography to study near-field effects of polypropylene fibres on hardened cement paste. *Cem Concr Compos.* 2020;114:103800.
- [26] Eik M, Antonova A, Puttonen J. Roughness of steel fibre and composition of cement paste close to fibre surface. *J Adv Concr Technol.* 2019;17:628–638.
- [27] Silva FJ, Thaumaturgo C. Fibre reinforcement and fracture response in geopolymeric mortars. *Fatigue Fract Eng Mater Struct.* 2003;26:167–172.
- [28] Zhang YS, Sun W, Li ZJ. Impact behavior and microstructural characteristics of PVA fiber reinforced fly ash-geopolymer boards prepared by extrusion technique. *J Mater Sci.* 2006;41(10):2787–2794.
- [29] Lin TS, Jia DC, He PG. Thermo-mechanical and microstructural characterization of geopolymers with α -Al₂O₃ particle filler. *Int J Thermophys.* 2003;30(5):1568–1577.
- [30] Alomayri T, Shaikh FUA, Low IM. Mechanical and thermal properties of ambient cured cotton fabric-reinforced fly ash-based geopolymer composites. *Ceram Int.* 2014;40(9):14019–14028.
- [31] Silva F, Mobasher B, Tolhedo Filho RD. Cracking mechanisms in durable sisal fiber reinforced cement composites. *Cement Concret Comp.* 2009;31(10):721–730.
- [32] Musil S, Kriven W, Biernacki J. In Situ Mechanical Properties of Chamotte Particulate Reinforced, Potassium Geopolymer. *J Am Ceram Soc.* 2014;97(3):907–915.
- [33] Ranjbar N, Zhang M. Fiber-reinforced geopolymer composites: a review. *Cem Concr Compos.* 2020;107:103498.
- [34] Ranjbar N, Talebian S, Mehrali M, et al. Mechanisms of interfacial bond in steel and polypropylene fiber reinforced geopolymer composites. *Compos Sci Technol.* 2016;122:73–81.
- [35] Kessler A, Bledzki AK. Correlation between interphase-relevant tests and the impact-damage resistance of glass/epoxy laminates with different surface treatments. *Compos Sci Technol.* 2000;60(1):125–130.
- [36] DiBenedetto AT. Tailoring of interfaces in glass fiber-reinforced polymer composites: a review. *Mater Sci Eng A.* 2001;302(1):74–82.
- [37] Callister WD, Rethwisch DG. Fundamentals of materials science and engineering: an integrated approach. New York (NY): Wiley; 2013.
- [38] Nogueira A, De Barros S, Alves LA. Fiber-matrix adhesion on industrial geopolymer. *Iran J Mat Sci Eng.* 2020;17(3):85–91.
- [39] Alves L, Leklou N, Casari P, et al. Fiber-matrix bond strength by pull-out tests on slag-based geopolymer with embedded glass and carbon fibers. *J Adhes Sci Technol.* 2021;1–11. DOI:10.1080/01694243.2020.1870322
- [40] Alves L, Leklou N, De Barros S. A comparative study on the effect of different activating solutions and formulations on the early stage geopolymerization process. *MATEC Web of Conferences.* 2020;322:01039.
- [41] FdA S, Filho RDT, JdAM F, et al. Physical and mechanical properties of durable sisal fiber—cement composites. *Constr Build Mater.* 2010;24(5):777–785.
- [42] Leklou N, Aubert J-E EG. Microscopic observations of samples affected by the delayed ettringite formation (DEF). *Mater Struct.* 2009;42(10):1369–1378.
- [43] American Society for Testing and Materials (ASTM). Standard test method for flow of hydraulic cement mortar. Pennsylvania (PA): ASTM International; 2015. Standard No. C1437-15: 2015.
- [44] Association Française de Normalisation (AFNOR). Test for hardened concrete - porosity and density test. Ile-de-France: AFNOR Editions. 2010. Standard No. NF P18-459: 2010
- [45] Association Française de Normalisation (AFNOR). Methods of testing cement - part 1: determination of strength. Ile-de-France: AFNOR Editions; 2016. Standard No. NF EN196-1: 2016.
- [46] Levine DM, Stephan DF, Szabat KA. Statistics for managers using microsoft excel. London: Pearson; 2020.
- [47] Montgomery DC. Design and analysis of experiments. New York (NY): Wiley; 2020.

- [48] Nematollahi B, Sanjayan J, Xia Hui Chai J, et al. Properties of fresh and hardened glass fiber reinforced fly ash based geopolymer Concrete. *Key Eng Mater.* [2013](#);594-595:629–633.
- [49] Krishnan T, Purushothaman R. Optimization and influence of parameter affecting the compressive strength of geopolymer concrete containing recycled concrete aggregate: using full factorial design approach. *Conf. Ser.: Earth Environ. Sci.* [2017](#);80:012013
- [50] Farhana ZF, Kamarudin H, Rahmat A, et al. A study on relationship between porosity and compressive strength for geopolymer paste. *Key Eng Mater.* [2014](#);594-595:1112–1116.
- [51] Marvila TM, Azevedo ARG, Cecchin D, et al. Durability of coating mortars containing açai fibers. *Case Stud Constr Mater.* [2020](#);13:e00406.
- [52] Saloni SA, Sandhu V, Jatin P. Effects of alccofine and curing conditions on properties of low calcium fly ash-based geopolymer concrete. *Mater Today Proc.* [2020](#);32(4):620–625.
- [53] Chakraborty S, Kundu SP, Roy A, et al. Improvement of the mechanical properties of jute fibre reinforced cement mortar: a statistical approach. *Constr Build Mater.* [2013](#);38:776–784.
- [54] Momoh EO, Osofero AI. Behaviour of oil palm broom fibres (OPBF) reinforced concrete. *Constr Build Mater.* [2019](#);221:745–761.