MECHANICAL RESPONSE OF GEOPOLYMER COMPOSITES REINFORCED WITH DISTINCT TYPES OF FIBERS

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Abstract

Ceramic materials, such as geopolymers, have been increasingly used in civil construction as a compatible, green-friendly, and even more efficient alternative. They are obtained through the mixture of an aluminosilicate source, with an alkali solution, and its mechanical properties may vary according to its molar ratios, curing regimes and processing conditions. Geopolymers present compatible mechanical and durable responses, despite their characteristic brittle behaviour. Fiber reinforcement appear as an interesting solution to overcome this vulnerability. Particulate, discrete and textile forms of distinct fibers can be used as reinforcements, resulting in sustainable solutions (natural fibers) and even ultra-ductility performance (synthetic fibers). This study presents an experimental evaluation of distinct geopolymer composites reinforced with natural (sisal, jute and curauá) and synthetic fibers (PVA). The geopolymer mixture was produced with metakaolin in a sodium based solution. Compression, tensile and pull-out tests were performed. Scanning electron microscopy (SEM) was used to investigate the hardened geopolymer microstructure. Despite presenting distinct quantitative responses, all composites exhibited strain-hardening behaviour with multiple cracking formation.

1. INTRODUCTION

The lack of an adequate variability of construction materials, and the consequences caused by the exponential production of non-renewable cementitious sources, leads to a demand for compatible alternatives. One of these alternatives are the so-called geopolymers, firstly developed by Davidovits in early 70s in France, in a successful attempt to manufacture a material capable of withstanding elevated temperatures [1]. However, with the knowledge dissemination and further investigations performed by several research groups around the globe, additional features appear as advantages in the use of this technology, such as: high resistance in early ages [1]; improved durability behaviour [2]; and great adhesion performance with distinct types of fibers.

Geopolymers can be produced through the mixing of an aluminosilicate source, such as metakaolin or fly ash, with an alkaline solution (K, Na, or Cs based) [3]. The result is a polymeric inorganic network, with SiO_4 and AlO^-_4 linked by oxygen molecules [1,3]. Geopolymers, as well as any other ceramic materials, present fragile failure modes with low deformation capacity. Its mechanical response can be tailored with particulate (such as chamotte and sand) and/or fiber reinforcements [3]. These modifications can result in gains in tensile strength, ductility, toughness and even durability.

Recent studies demonstrate great improvements in the mechanical behavior of geopolymers with the use of reinforcements, such as: fique [4]; glass [5]; jute [2]; and steel fibers [6]. It is interesting, however, to notice that despite presenting distinct microstructural properties, it is possible to correlate the adhesion and stress transfer mechanisms of geopolymer and cementitious materials.

This work presents the results of an experimental investigation of a metakaolin-based geopolymer reinforced with distinct types of fibers: natural (jute, sisal and curauá), and synthetic (PVA) ones. The strength of the mixture is evaluated through compression tests. Its microstructure is investigated with the use SEM observations. The composites responses were studied through direct tensile tests. Additionally, pull-out tests were performed to try to demystify the fiber-matrix mechanisms.

2. EXPERIMENTAL PROGRAM

2.1 Materials and processing

The geopolymer mixture was manufactured through the combination of metakaolin ($Al_2O_3 \cdot 2SiO_2$), from Metacaulim do Brasil, and an alkaline solution based on sodium hydroxide and silicate, from Quimesp, resulting in a final composition of $Na_2O \cdot Al_2O_3 \cdot 3SiO_2 \cdot 11H_2O$. The latter was stablished following the results obtained in previous studies [7,8]. Sodium silicate and hydroxide solutions were mixed in appropriate proportions, and the final blend was cooled in a water bath for 10 min, before being added to the aluminosilicate source. A 5L capacity planetary mixer was used as follows: (i) 5 min at 136 rpm; (ii) removal of trapped solids on the walls of the container; (iii) final homogenization for 3 min at 281 rpm. River sand was incorporated as a natural aggregate, with density of 2.68 g/cm³ and maximum diameter of 1.18 mm.

Cylindrical specimens with 100 x 50 mm (height x diameter) were produced for compression tests. After the mixing process, the mixture was poured into steel molds protected by plastic papers (to avoid unwanted adhesion), alternating with medium vibration (to reduce voids content). The molds were sealed at room temperature for 24h to prevent crack formation due to dehydration during the curing process. After this period, the samples were withdrawn from the molds and stored in plastic bags for 6 more days, until mass constancy was obtained.

Geopolymer composites were produced with distinct types of reinforcements: natural (jute, sisal and curauá) and synthetic ones (PVA). Jute fibers are extracted from the stem of the plant *Corchorus capsularis,* while sisal and curauá fibers are extracted from the leafs of the plants *Agave sisalanais* and *Ananas erectfolius*, respectively. Jute was incorporated in a geopolymer material as a plain weave fabric (as received), while sisal and curauá were positioned in aligned unidirectional forms. All the natural reinforcements were used with 10% in weight content. Synthetic PVA fibers, with 12 mm in length, from Kuraray Co. (REC 15), were used as disperse reinforcement in a volume fraction of 2%. All fibers properties are presented in Table 1.

Table 1: Mechanical and physical properties of jute, sisal, curauá [9] and PVA fibers [10].

Properties	Jute	Sisal	Curauá	PVA
Tensile Strength (MPa)	104	447.2	632.1	1600
Strain-to-failure (mm/mm)	0.01	0.03	0.02	0.06
Young's modulus (GPa)	5.7	20.16	38.1	41
Average diameter (mm)	0.483	0.023	0.008	0.040

Plate specimens of geopolymer material reinforced with the four types of fibers mentioned before, with 450 x 60 x 12 mm (length x width x thickness), were produced for tensile tests. Cylindrical specimens with 25 x 20 mm (height x diameter) were produced for pull-out tests, where filaments of each fiber were embedded in 25 mm (natural fibers) and 4 mm (PVA) lengths into the geopolymer material. All specimens were prepared and cured following the indications previously mentioned for the cylindrical specimens.

3. TESTING METHODS

The flow table test was carried out with the fresh geopolymer mixture following the instructions presented in ASTM C143 [11]. Compression tests were performed on a MTS universal testing machine, model 810, using a load cell with maximum capacity of 500 kN, following the indications on ASTM C39 [12]. Three cylindrical specimens were tested using a displacement controlled rate of 0.5 mm/min. The upper and lower surfaces of the samples were regularized, in order to obtain an adequate mechanical response. The axial displacement was measured through the readings of two LVDTs, with 70 mm of length, coupled to acrylic rings positioned around the specimens.

A scanning electron microscope (SEM), model FEI Quanta 400, operated at 20 kV, was used to evaluate the microstructure of the hardened geopolymer. The samples were polished and thinned using a semiautomatic Struers equipment (Tegramin). For a better resolution (result of the amount of conductivity inside the equipment) a gold layer was applied on the sample for 20 seconds in a vacuum media.

Pull-out and tensile tests were carried out on a MTS testing equipment, model 810, with a load cell of 250 kN. For the pull-out tests, a load cell with maximum capacity of 1 kN was adapted for a better measurement. The displacement rate used was equal to 0.1 mm/min. For the pull-out tests, one LVDT (70 mm) was placed between the upper and lower grips, and 10 specimens were tested for each variation. However, for the tensile tests, two LVDTs were positioned on the sides of the specimens with 250 mm of gauge length. The tensile tests were performed following the recomendations described on ASTM 1275 [13], in 3 specimens for each reinforcement. The specimens were fixed in steel plates, and a 10 N.m torque was applied to each screw. Figure 1 presents the tests setups.

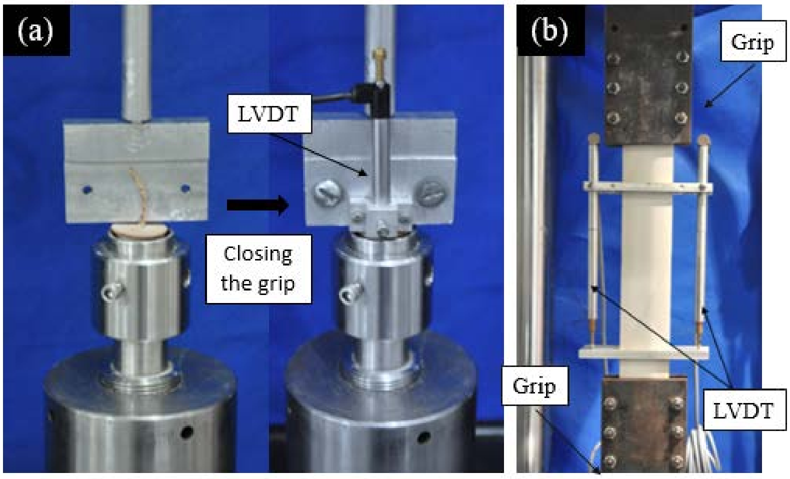

Figure 1: (a) Pull-out; and (b) tensile tests setups.

4. RESULTS AND DISCUSSION

The flow table tests results presented an average value of 127.5 mm for standard consistency, evidencing an intermediate fluidity for fresh geopolymer mixtures, according to the parameters proposed by previous studies [14]. This result is justified by the great H_2O absorption capacity of metakaolin based materials.

The compressions tests resulted in an average strength of 72.7 MPa and 14.26 GPa of Young's Modulus, with a fragile failure mode. The micrograph of the cured sample is presented in Figure 3. It is possible to distinguish unreacted MK particles and aggregates (distinct grey scales). It is, however, important to notice a significant presence of large pores interconnected by microcracks through the surface. Those are direct results from the dehydration occurring during the first hours of curing, where the water restrained inside the material looks for a way out, creating undesired internal pressures, resulting in microstructural defects (cracks and pores). The presence of aggregates is very important, since it creates difficulties during the crack formation process, resulting in strengthening mechanisms. Another method to improve the mechanical performance of fragile materials is the incorporation of fibers, which will be discussed below.

The tensile tests average responses for the geopolymer composites are presented in Figure 3 and Table 2. Weibull statistics was used for the ultimate stress values, mostly to determine the reliability of the results. Following the indications presented in previous studies [5,16], the parameters were obtained by plotting the following relation:

$$\ln\left[\ln\left[\frac{N+1}{N+1-i}\right]\right] = m \ln\left(\frac{\sigma}{\sigma_0}\right)$$

where N corresponds to the number of tested samples; i corresponds to the order of the stress (σ) related to each sample (from lowest to highest); m represents the Weibull modulus; and σ_0 is the characteristic strength in a scale parameter. The higher the Weibull modulus the lower is the dispersion of failure stresses.

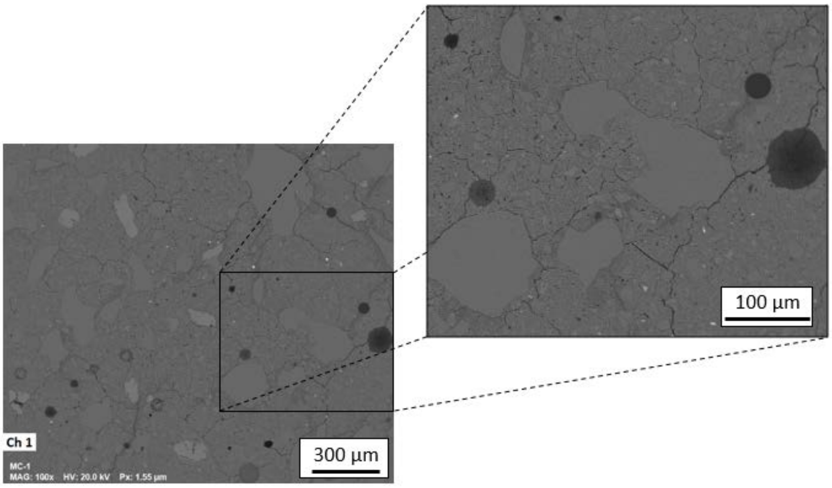

Figure 2: SEM micrograph of the hardened sodium based geopolymer.

It is possible to affirm that all composites (natural or synthetic fiber reinforced) exhibited a strain-hardening behaviour with multiple cracking formation during the tests. They all reached similar low responses in first cracking stresses (σ_{1f}), with greater results in ultimate average tensile stresses for curauá and sisal fiber reinforced ones, reaching 14.33 and 11.53 MPa, respectivelly. The latter, despite showing a small reduction in strength, exhibited a more ductile response than the previous one. A very distinct behaviour, regarding cracking formation, was observed for jute and PVA fibers reinforced geopolymers, with greater deformation capacities and lower crack formation, possibly due to insufficient fiber-matrix adhesion. Jute and PVA geopolymer composites reached ultimate stresses of 6.31 and 5.56 MPa, respectively. All composites exhibited great reliability, with a lower Weibull modulus, equal to 6.8, for PVA reinforced geopolymers. It is important to notice, however, that the content of PVA fibers was equal to 2% in mass, against 10% for the other fibers, resulting in an interesting mechanical behaviour due to its greater strength (1600 MPa) and Young's modulus (41 GPa).

Table 2: Results of direct tensile tests with geopolymer composites reinforced with: curauá, sisal, jute and PVA fibers.

Composite	σ_{1f} (MPa)	m	σ_0 (MPa)	σ_u (MPa)	S.D. (MPa)	95% C.I. (MPa)
Curauá GC	4.83	13.1	14.89	14.33	1.25	(13.9, 14.8)
Sisal GC	4.37	15.3	11.92	11.53	0.86	(11.2, 11.8)
Jute GC	4.13	9.2	6.65	6.31	0.78	(6.0, 6.6)
PVA GC	3.58	6.8	5.94	5.56	0.94	(5.2, 5.9)

σ_{1f} = first crack stress; m = Weibull modulus; σ_0 = ultimate stress (scale parameter); σ_u = ultimate stress (average); S.D. = standard deviation; 95% C.I. = 95% confidence interval.

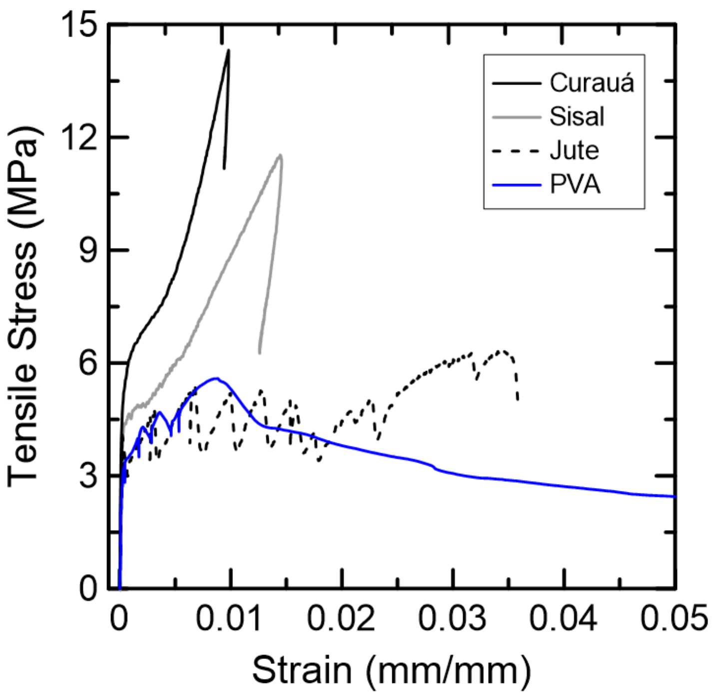

Figure 3: Tensile Stress x Strain curves for geopolymer composites reinforced with: curauá, sisal, jute and PVA fibers.

An additional evaluation of the fiber-matrix interface is presented in Figure 4, and Table 3, through the pull-out tests results. Its efficiency depends mainly on its chemical-physical bond, frictional adhesion and mechanical anchoring [17]. Among all fibers, it can be stated that sisal and curauá present the better adhesion (justifying its improved mechanical behaviour in the composite), followed by jute and PVA. This analysis indicates that the higher the fiber matrix adhesion, the greater it is the composite strength and the lower it is its deformation capacity. The amount of fibers incorporated in the geopolymer material also present significant changes in its mechanical capacities.

Table 3: Pull-out tests results for: curauá, sisal, jute and PVA fibers embedded in a geopolymer material.

Fiber	Embedded length (mm)	P_a (N)	D_a (mm)	Stiffness (N/mm)
Curauá	25	7.14	1.55	4.61
Sisal	25	8.47	2.12	3.99
Jute	25	4.84	1.83	2.64
PVA	4	0.65	0.74	2.91

P_a = adhesional force; D_a = adhesional displacement.

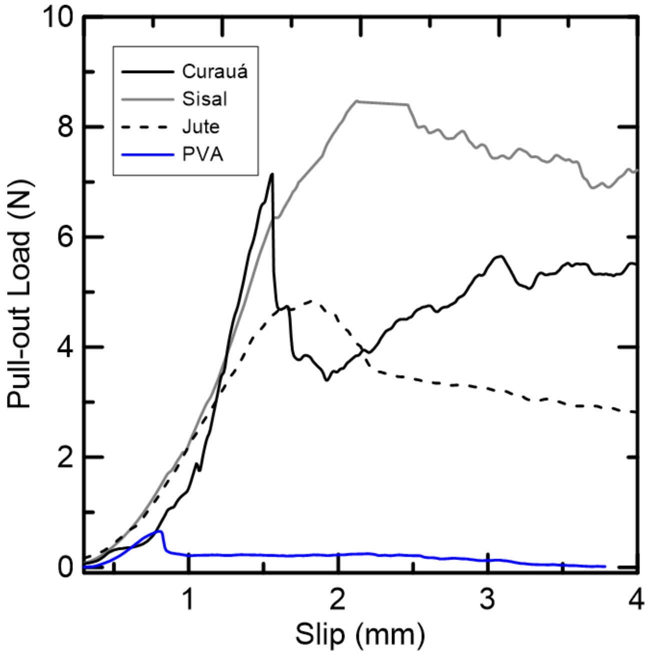

Figure 4: Pull-out load x Slip curves for: curauá, sisal, jute and PVA fibers embedded in a geopolymer material.

5. CONCLUSIONS

The geopolymer material, when incorporated with the natural aggregate sand, achieved 72.7 MPa in compression strength. All composites, reinforced with distinct types of fibers (natural and synthetic ones), achieved multiple cracking formation in a strain hardening behaviour. The curauá and sisal fibers presented the higher stresses when compared to the lower results obtained for jute and PVA fibers. The latter, despite being used in smaller contents (2% in weight), was still capable to present interesting mechanical responses and an improved deformation capacity (>5% in strain). It was possible to notice an increase in stress in a better fiber-matrix adhesion environment (evidenced by the pull-out tests), while weak adhesions presented greater deformation capacities.

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