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RESEARCH PAPER

Fiber-Matrix Adhesion on Industrial Geopolymer

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Abstract: *The construction sector is responsible for relevant environmental impacts and one of its most crucial points is the use of concrete. Geopolymers represent the most promising green and ecological alternative for common Portland cement and cementitious materials, due to its proven durability, mechanical and thermal properties. This work presents an experimental and comparative study of adhesion at the fiber-matrix interface between glass fibers and carbon fibers added to the geopolymer matrix. This analysis was performed by pull-out test, whereby it was found that the greatest efficiency was obtained by reinforcing with the glass fibers, incorporated at 2 mm in the geopolymer matrix. As a result, the adhesion between the fibers and the geopolymer structure can be assessed, as well as the optimum length of application.*

Keywords: *Carbon fibers, Composites, Geopolymers, Glass fibers, Pull-out test.*

1. INTRODUCTION

The construction sector is responsible for relevant environmental impacts and one of its most crucial points is the use of concrete. Concrete is the most widely used construction material in the world and its production causes high levels of carbon dioxide (CO^2) in the atmosphere [1]. Although the CP is currently the most used binder to produce concrete and mortar, its use is less environmentally advantageous because its production causes damage due to the large release of carbon dioxide (CO^2). This impact becomes more significant since cement is fundamental for infrastructure construction, being the second most used material in the world, in volume, behind only to water. As a consequence, the cement industries together account for 5% of the world's CO^2 emissions, being among the most polluting industries [3, 4]

In recent decades, there has been a growing concern about the environment and its degradation, being the subject of several world conventions that sought solutions that improve the relationship between man, his activities, and the environment in which they live. Thus, in search of sustainable development, new technologies and materials began to be studied. A potential substitute for cement are alkaline activated materials (geopolymer binders) that appear to produce mechanical properties similar to Portland cement [5]. Although these materials are still in the early stages of development [6], geopolymers represent the most promising green and ecological alternative for common Portland cement and cementitious materials, thanks to their proven durability, mechanical and thermal properties.

A similar characteristic between the geopolymer cement and the CP, and one of the reasons that makes it a potential substitute, is the high compressive strength. But with low tensile strength and low deformation capacity, reinforcements are required. In addition to steel, used in reinforced concrete, an alternative is the use of fibers incorporated into cementitious matrices [7-10]. As reinforcements natural fibers, such as jute, or synthetic fibers, such as glass and carbon fibers, can be used. The use of natural fibers increases the advantage from the environmental point of view since they are composed of biodegradable materials and from renewable sources. The fibers act to prevent abrupt rupture of the material, increasing its ductility [11]. These effects will be directly proportional to the fiber-matrix interaction force: the composite will have greater strength the greater the adhesion between the surface of the geopolymer matrix and the surface of the fiber.

This work seeks to verify the interaction between a geopolymer matrix and synthetic glass and carbon fibers, comparing the adhesion between the fiber and matrix obtained by pullout test. In addition, it aims to examine the cracking pattern of the specimens and to discover the embedded length of the fiber, which represents the optimum length capable of promoting the greater adhesion and better mechanical performance of the composite.

2. EXPERIMENTAL PROCEDURE

The geopolymer matrix consisted of the geopolymer cement from the company Geo-Pol®, composed of a precursor powder and the activator liquid. Initially, with a precision digital scale, the required quantities of each material were separated to prepare 8 test specimens of the composite (4 for each type of fiber). These measurements were 42g of precursor powder and 58g of liquid activator, following manufacturer's instructions.

After weighing, the elements were mixed with a mechanical stirrer. The mixture was first stirred at a low speed (136 rpm) for 4 minutes. The stirrer was then turned off to clean the residue stuck to the walls of the vessel. The mixture was stirred again for 3 minutes at medium speed (281 rpm), following recommendations explained by Trindade [12]. Then, the homogeneity of the obtained geopolymer cement was verified.

The following steps consists the manufacture of the composites from the geopolymer matrix incorporated to the glass fibers and carbon fibers. For this preparation, an iron mold was used, capable of producing 10 specimens of measurements equal to 15x10x10 mm [13]. Initially, the fibers, previously separated in equal parts in the mold, were positioned, with the aid of a pachymeter, in 4 different lengths of incorporation: 2, 4, 6 and 8 mm. The different lengths are justified since one of the objectives of the test was to find the critical embedded length.

Next, the mold was closed and filled with the previously prepared geopolymer mixture, taking care not to allow air pockets to form, which would compromise the integrity of the samples and consequently the test result. Excesses were then removed to facilitate demolding and the composites were reserved, for the curing time of the samples. After 24 h of curing, at room temperature, the composite samples reinforced with glass and carbon fibers (Fig. 1) were carefully demoulded so that they were undamaged and ready for the pullout test.

2. 1. Pull out test

A composite will be as resistant as the adhesion at the fiber-matrix interface, so it is fundamental to evaluate the interaction between these two elements. The analysis of this adhesion can be performed through the study of the materials involved, their geometries, the loads and the relative displacements, which provide the adhesion stress values, fundamental for the knowledge of the shear stress transfer between the fiber and the matrix. An efficient way to do this analysis is through the pull-out test [14].

A factor that influences the bond strength between the composite and, consequently, their strength and stiffness, is the length of the fiber used. The reinforcement efficiency depends on the effective transfer of the efforts. Thus, the fiber used must have a length equal to or greater than the critical embedded length (Lc). Fibers with shorter lengths results in deficiencies of the transmission of the external loads, and there may be slipping of the fiber through the matrix, even before its rupture, causing failures in the place that present lower

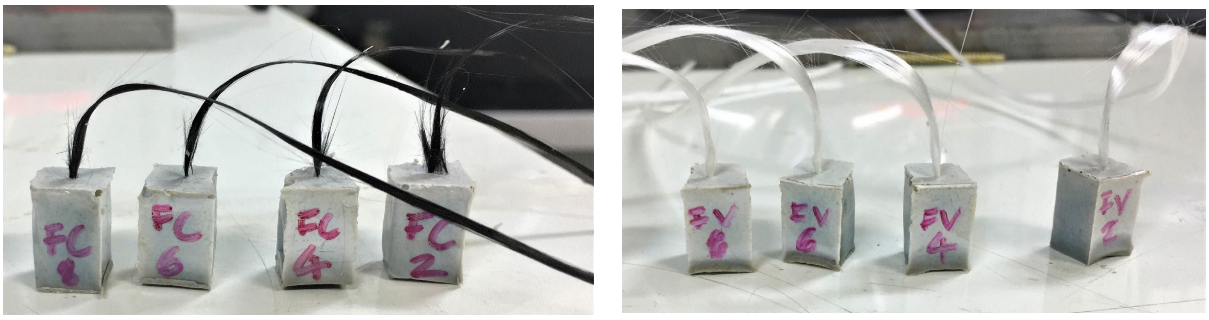

Fig. 1. Samples of geopolymer composites after demolding
(Glass fiber (FV) can be observed on the left and carbon fiber (FC) on the right)

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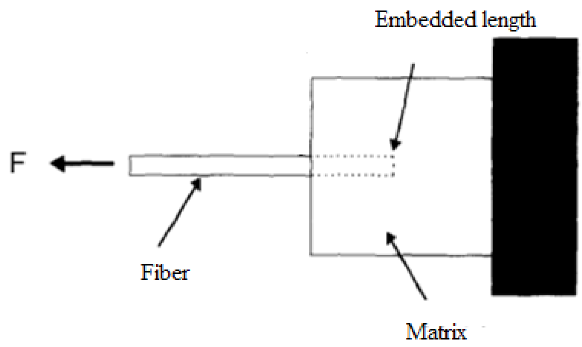

Fig. 2. Schematic drawing of the assembly of a pull-out test [16]

value of resistance, in the matrix or in the interface. The slip of the fiber, without breaking, is also called debonding or decoupling [15].

The critical length can be analyzed through the pull-out test. The test is done by embedding the fiber up to a certain length into a specimen of the matrix material, in this study geopolymer. Then, the two ends are attached, the test body and the tip of the fiber, thereafter applying a tensile force F on the fiber, as shown in Fig. 2. If the length of the fiber is equal to or greater than Lc, the fiber will break. Otherwise, the fiber slips from within the matrix without breaking [15].

By pull-out test it is also possible to study the influence of fiber-matrix bond strength (τ), shear stress (μ) and shrinkage of the matrix by the pressure on the fiber (P0) on the mechanical properties of the composite. When a composite has a high adhesive force at the fiber-matrix interface, it exhibits high strength. This is due to a greater efficiency of the tension undergone by the matrix to the fibers. On the other hand, a high value of τ causes the system to have a low tenacity, since the energy spent during the crack propagation is low, thus the failure of the matrix will propagate through the fiber-matrix interface. By analogy, it can be inferred that low values of τ result on low resistance, by the ineffective transfer of the tension to the fibers, and a high tenacity, since a high energy value would be expended not only by the cracks, but by the decoupling of the fiber . These implications, as well as the effects of μ and P0, also interfere with the fiber extraction curve resulting from the tear test [16].

The Instron equipment, model 5966, with a load cell of 10 kN, was used in the laboratory of composites and adhesives (LADES) of CEFET/RJ. This machine needed to be adapted with a Restrained Top Constrain (RTC) clamp for anchoring the specimens and performing the pull-out test (Fig. 3). In addition to assisting in fixing the block of the test piece, the clamp has a sufficient opening for the passage of the fiber. The use of the RTC

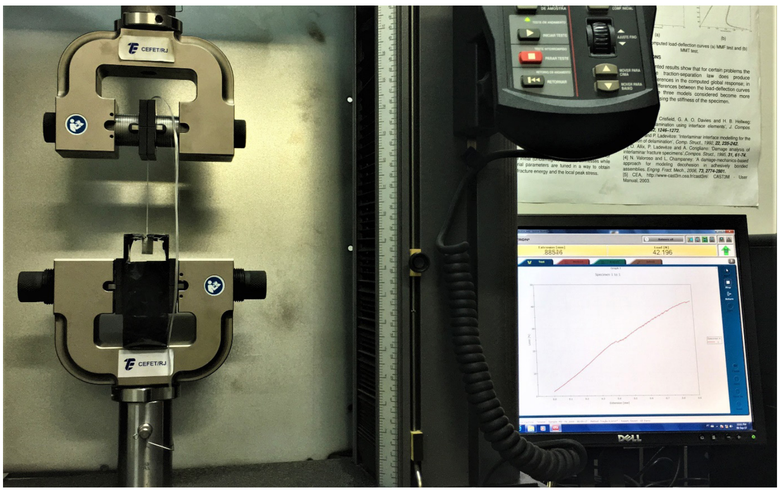

Fig. 3. Instron machine and RTC clamp

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clamp is justified since it was desired that the applied force was aligned with the efforts on the specimen and the fiber as well as in the same direction of the fiber [13].

After the preparation was done, the test was started by applying force at a rate of 0.5 mm/min to the fiber [17]. While the force was applied, the behavior at the fiber-matrix interface was monitored through the graph generated. When the fiber underwent the total pull-out of the matrix, the test was finalized.

To analyse the test results, it is importante to notice that the pull-out test can be divided into three phases: the initial phase, found in region I of the graph, is characterized by being an elastic-linear section, which corresponds to the beginning of the detachment and slip of the fiber, where the load is constantly increased until reaching a nonlinear stretch, corresponding to region II, qualified as the region where the extraction force reaches the maximum value (Fmax), which will be as high as the resistance value, and decohesion becomes partial. After this phase, there is a constant drop in the load, corresponding to region III. This region is controlled by the friction resistance of the interface and continues until the fiber is extracted and completely withdrawn [16].

3. RESULTS AND DISCUSSION

Through the data obtained from the fiber pull-out test, it is possible to determine the transferred loads from the shear stress and the relative displacements occurring at the fiber-matrix interface, and hence the relative adhesion stress and the strength effectiveness conferred by the fibrous reinforcement.

In the test performed, the bond failure mode, the value of the average bond strength and the adhesion and slip curves were obtained. These results guided the discussion and made possible the evaluation of the effect of embedded length on the final resistance of the composite and the performance of the adhesion of the fiber on the geopolymer matrix.

Glass fiber reinforced geopolymer matrix. The results of the computed values of the applied force (F), the resulting stress (τ) and the displacement (δ) generated, separated by the length of embedding of the reinforcing fiber, are shown in Table 1.

Table 1. Compiled values from the pullout test on glass fiber reinforced composites

FV (mm)	F (N)	τ (MPa)	δ (mm)
2	58,86	0,39	2,02
4	27,56	0,18	1,64
6	35,72	0,24	1,35
8	27,03	0,18	1,43

The behavior of the fiber-matrix adhesion during the test can be observed in Fig. 4. From this graph it is possible to compare the performance of each sample, according to the length of insertion of the fiber, and identify the points

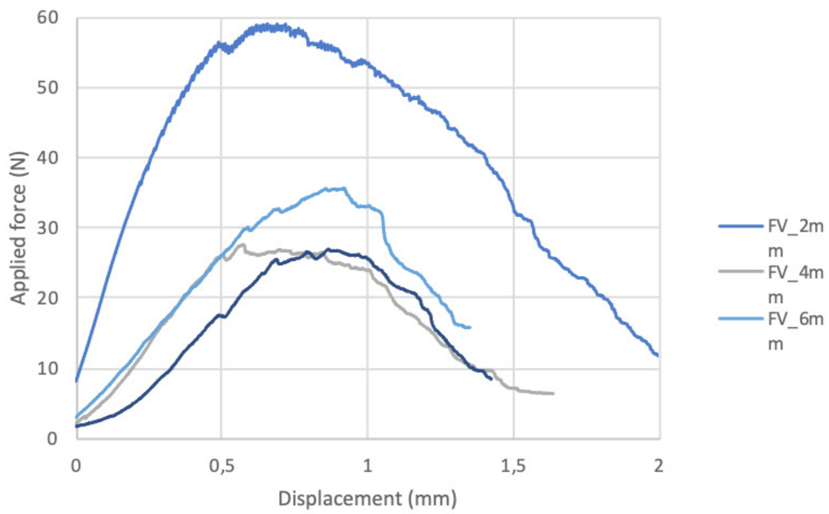

Fig. 4. Behavior of glass fiber reinforced composites

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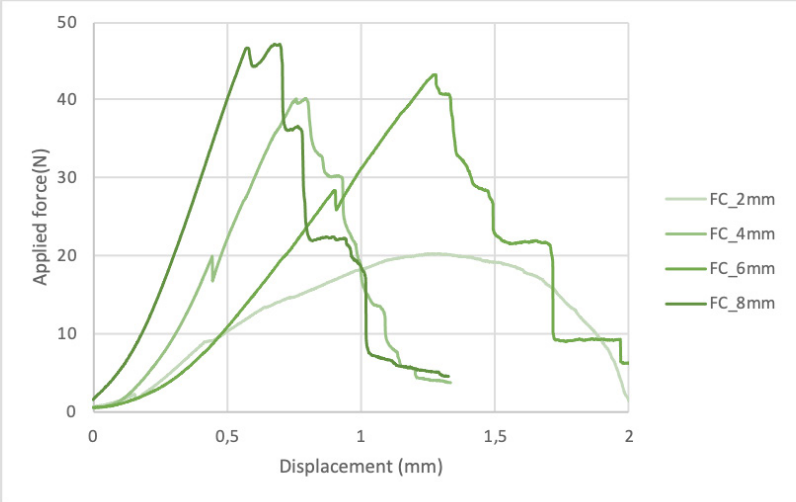

Fig. 5. Behavior of carbon fiber reinforced composites

of greatest force applied and the moment of fiber extraction.

When analyzing the graph, it is possible to observe that the 4 samples have similar behavior. This fact can be explained because the materials involved are the same, with the same chemical composition and, thus, have the same adhesion and adsorption behavior caused by the chemical reactions that occur on the surface of the matrix and the fiber.

From these data, it can also be concluded that the sample that has the best adhesion between the fiber and the matrix is the one with an embedded length of 2 mm, since it has the maximum applied force equal to 58.86 N, greater than the other samples. Isolating the curve referring to this model, it is possible to identify the areas of the three phases of the pullout test. Zone I present elastic-linear behavior, with beginning of fiber detachment, some microcracks and fiber and matrix working in a linear pattern. With the continuous increase of the load, it approaches to zone II, where the maximum force is reached and the fiber decohesion is partial. Afterwards, it starts decreasing to zone III and the total detachment of the fiber can be observed whether with rupture or not of the body and fiber fracture.

Carbon fiber reinforced geopolymer matrix. As for the composites that were made using carbon fibers as reinforcement, the compiled values of the data generated during the test applied force (F), the resulting stress (τ) and the displacement (δ), separated by the degree of embedding the reinforcing fiber are shown in Table 2.

Table 2. Compiled values from the pullout test on carbon fiber reinforced composites

FC (mm)	F (N)	τ (MPa)	δ (mm)
2	20,27	0,14	3,74
4	40,22	0,27	1,33
6	43,25	0,29	2,02
8	47,18	0,31	1,33

The behavior of the carbon fiber reinforced samples during the test can be analyzed by a graph of applied force x displacement (Fig. 5), comparing the performance of each sample, according to the embedded length of the fiber and identifying the points of greatest force applied, as well as the moment of fiber extraction.

By analyzing the data contained in the table and in the graph, it can be inferred that the sample with the most efficient adhesion between the fiber and the matrix has an embedded length equal to 8 mm, since it presented maximum applied force equal to 47.18 N, the highest value among all samples. As stated earlier, the greater the maximum value of the applied force, the greater the adhesion at the fiber-matrix interface.

As in the graph corresponding to the glass fiber reinforced composites, the curve for the higher-adhesion reinforced model can be isolated to identify the three areas corresponding to each phase of the test. A linear elastic behavior, with beginning of fiber detachment, some microcracks and with fiber and matrix working linearly, corresponding to zone I of the pull-out test, is

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verified at the beginning of the test. As the tensile load on the fiber increases, it reaches zone II, region that includes the maximum point of force applied and where the fiber decohesion becomes partial. In the next post-peak zone, the applied force decreases until the total pullout of the fiber is obtained, which can occur with breaking or not of the specimen, as well as with or without fracture of the fiber.

Another analysis made from the graphs is the behavioral similarity between 3 of the 4 samples. In the composites that presented similar behavior, this fact can be explained because the materials involved are the same, with the same chemical composition and, therefore, have the same adhesion and adsorption behavior caused by the chemical reactions that occur on the surface of the matrix and fiber. However, in the sample with fiber insertion equal to 2mm, there was a behavioral differentiation, even with the same materials and manufacture as the others. Therefore, this performance is not caused by chemical adhesion. In fact, by analyzing the graph it is possible to notice that the fiber displacement has higher values than the other samples with the same applied force, characterizing that there was a slip of the fiber. The maximum strength applied also presents a much lower value when compared to the other composites, resulting in a lower reinforcement efficiency in this material. This problem may have occurred due to a failure in fiber anchoring, causing a transfer inefficiency between the stresses sustained from the fiber to the matrix.

Comparison of results. The compiled set of results, for both glass and carbon fiber reinforced geopolymer is presented on Fig. 6. By evaluating the graph curves for the specimens during the test, it can be inferred that the material not only influences the efficiency of the reinforcement and the applied force to remove the fiber, but also interferes on the behavior of the displacement related to the force applied. In the samples with fiberglass, for each applied force there is a relative displacement. In the samples with the carbon fiber, there is the presence of constant force levels, that is, at certain points for a given force there is a linear evolution of the displacement. This difference can be explained by the different chemical interactions between the reinforcement material and the matrix material that influence the degree of the applied force as well as the affinity between the materials. This affinity is one of the factors responsible for determining the mode in which the transfer of loads at the fiber-matrix interface will occur.

Another possible confrontation, of fundamental importance, that can be done through the graphically identified data is that, among all the models of composites studied, the one that needed to apply a greater value of force for the pulling of the fiber is the composite made with fiber of glass embedded at 2 mm.

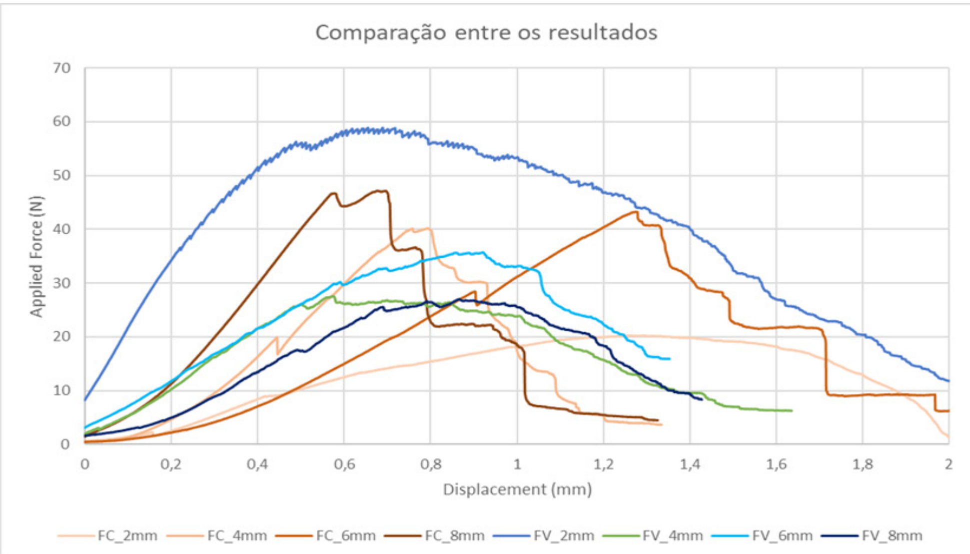

Fig. 6. Comparison of the results of the different samples [17]

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4. CONCLUSIONS

The work developed sought to present as an alternative of using the geopolymer the composite form, with the use of fibrous reinforcements. The parameter used was the adhesion of the fibers to the geopolymer matrix, evaluating the performance of the interaction between the materials at the fiber-matrix interface. This study is fundamental to determine which material can be considered more efficient and makes the geopolymer more resistant to the tensile and deformation strength. In addition to the different behaviors noticed due to the different materials used, another factor analyzed was the influence of the fiber insertion behavior in the matrix, which changes the load transfers and, therefore, the reinforcement efficiency.

The research method used was the pull-out test, which provided results of the force applied to each composite for the pulling or sliding of the fiber, the failure mode in the fiber-to-matrix and the adhesion and slip curves. It is possible, through these data, to infer which material and the fiber insertion length confer greater resistance to the composite.

It was concluded that the fiber that would best solve the problem of resistance of the geopolymer cement is glass fibers, with 2 mm embedded length, presenting bond strength and interaction in the fiber-matrix interface superior to the others. A possible explanation for the fact that the glass fiber inserted at 2 mm has better results than when it was inserted at longer lengths is the critical length. As well as below the critical length there is deficiency in the transfer of loads - the fiber undergoes sliding - above it there are also failures, since under these conditions there is a greater interaction between the fibers, causing entanglement and decrease of the effective length.

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