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# Fiber-matrix bond strength by pull-out tests on slag-based geopolymer with embedded glass and carbon fibers

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**Keywords:** blast furnace slag, cement Portland, sodium silicate activating solution, pull-out test,  
glass fiber, carbon fiber

**Abstract.** The reinforcement efficiency on a composite depends on the effective transfer of the stress between matrix and fiber. This work presents an experimental and comparative study of fiber-matrix bond strength for fiber-matrix interface between glass fibers and carbon fibers added to the slag-based geopolymer matrix. This analysis was performed by pull-out test. A total of 18 tests have been conducted, three for each type of fiber at each embedded length of 10 mm, 20 mm and 30 mm. The critical embedded length and the maximum interfacial shear (bond) strength were analyzed, and SEM observations were carried out for the cross-section of each fiber to measure diameter and observe the interface. It was found that the greatest efficiency was obtained by reinforcing with the glass fibers, incorporated at 20 mm in the slag-based matrix.

## 21        **1. Introduction**

22        The geopolymer began to be engendered in the 1970s by Davidovits, emerging as a new class of  
23 material derived from rocks and not from oil, as organic polymers [1]. It consists of inorganic  
24 polymers obtained by the alkaline activation of materials rich in silica ( $\text{SiO}_2$ ) and alumina ( $\text{Al}_2\text{O}_3$ ) [2]  
25 and have similar physical and mechanical characteristics as Cement Portland (CP) [3]. CP is the most  
26 used binder to produce cement and mortar, and the second most used material in the world, behind  
27 only water [4]. But the production process classifies it as the third-largest source of anthropogenic  
28 emissions of carbon dioxide ( $\text{CO}_2$ ) [5]. In 2018, global process emissions reached  $1.50 \pm 0.12$  Gt  
29  $\text{CO}_2$ , which corresponds to about 4% of emissions from fossil fuels and cumulative emissions from  
30 1928 to 2018 result in  $38.3 \pm 2.4$  Gt  $\text{CO}_2$ , from which 71% occurred after 1990 [5]. Thus, the need to  
31 find an alternative material for construction processes arises.

32        Compared to CP, geopolymer emissions of  $\text{CO}_2$  are approximately 43% less. Previous studies on  
33 these materials show that values for compressive strength are comparable [6 - 9], with values  
34 depending on the precursor material and activator solution. Correia et al. [10] found that geopolymers  
35 maintain considerable mechanical properties at temperatures up to  $1000^\circ\text{C}$ , whereas the application  
36 of most polymer resins is often limited to temperatures below  $400^\circ\text{C}$ . The material also presents high  
37 durability due to low apparent porosity, that results in low water permeability [11]. The main  
38 precursor materials are clays such as metakaolin [12 - 14] which is an artificially calcined kaolinite  
39 clay, and industrial waste such as blast furnace slag [14, 15] and fly ash [16 - 20].

40        Geopolymers have low tensile strength and low deformation capacity, which require the use of  
41 reinforcements [21]. Geopolymer matrix composites reinforced with particulates and fibers can be  
42 considered a solution to improve flexural strength and compressive strength [22 - 28]. The fibers  
43 increase the ductility of the material, preventing abrupt rupture [29]. Natural fibers, such as jute, and  
44 synthetic fibers, including glass and carbon fibers, can be used to reinforcement the geopolymer.  
45 There is a direct proportional relationship between the fiber-matrix interaction force, the strength, and  
46 the adhesion between the surface of the geopolymer matrix and the surface of the fiber [30 - 32].

47 The mechanical properties of fiber-reinforced composite materials highly depend by how efficient  
48 the load transfer through the interface between the fiber and the matrix [31, 33 - 36]. When a  
49 composite has a high adhesive force at the fiber-matrix interface, it exhibits high strength. This is due  
50 to a greater efficiency of the tension undergone by the matrix to the fibers. On the other hand, a high  
51 value of resulting stress ( $\tau$ ) causes the system to have a low tenacity, since the energy spent during  
52 the crack propagation is low, thus the failure of the matrix will propagate through the fiber-matrix  
53 interface. By analogy, it can be inferred that low values of  $\tau$  result on low resistance, by the ineffective  
54 transfer of the tension to the fibers, and a high tenacity, since a high energy value would be expended  
55 not only by the cracks, but by the decoupling of the fiber [37].

56 A factor that influences the bond strength between the composite and the fiber is the critical  
57 embedded length ( $L_c$ ) [4]. Fibers with shorter lengths results in deficiencies of the transmission of the  
58 external loads, besides causing debonding or decoupling. It can also be led to failures in regions that  
59 present lower values of resistance, either in the matrix or in the interface [38]. The critical length can  
60 be analyzed through the pull-out test and represents the optimum length capable of promoting the  
61 greater adhesion and better mechanical performance of the composite.

62 The analysis of the fiber-matrix adhesion can be performed through the study of the materials  
63 involved, their geometries, the loads, and the relative displacements, which provide the adhesion  
64 stress values [39], fundamental for the knowledge of the shear stress transfer between the fiber and  
65 the matrix. Different types of tests to study this interface have been developed, namely pull-out tests  
66 [40, 41], push-out tests [42, 43], micro-bond tests [44, 45] and fiber fragmentation tests [43, 46, 47].  
67 The main difference between these tests is the test geometry [48]. However, the results analyzed by  
68 Pitkethly et. al [49] from different tests or from the same test applied by different researchers showed  
69 that the scatter was high.

70 To evaluate the bond quality at the fiber-matrix interface and the ability of stress transfer between  
71 the fiber and the matrix, the fiber pull-out test is one of the most important test methods developed  
72 [34]. The pull-out test is done by embedding the fiber up to a certain length into a specimen of the

73 matrix material. Then, the two ends are attached, the test body and the tip of the fiber, and a tensile  
74 force (F) is applied on the fiber. If the length of the fiber is greater than  $L_c$ , the fiber will break.  
75 Otherwise, the fiber slips from within the matrix without breaking. By pull-out test it is also possible  
76 to study the influence of fiber-matrix bond strength ( $\tau$ ), shear stress ( $\mu$ ) and shrinkage of the matrix  
77 by the pressure on the fiber ( $P_0$ ) on the mechanical properties of the composite [34, 37].

78 Therefore, research work on fiber pull-out from a matrix is essential to understand the stress  
79 transfer on different types of composites. [34]. This work seeks to verify the interaction between a  
80 geopolymer slag-based matrix and synthetic glass and carbon fibers, comparing the fiber-matrix bond  
81 strength between the fiber and the matrix obtained by pull-out test. It aims to examine the cracking  
82 pattern of the fibers embedded in the specimens to discover the critical embedded length ( $L_c$ ) and  
83 analyze relative displacements occurring at the fiber-matrix interface.

## 84 2. Materials and methods

85 This research used 100% ground blast furnace slag (GBFS), provided by ECOCEM from France,  
86 as the basic precursor material for the fabrication of the geopolymer paste specimens. The chemical  
87 composition for GBFS can be found in Table 1. Blast furnace slag is produced by drying and grinding  
88 granulated blast furnace slag. The degree of depolymerisation (DP) for the GBFS was found 1.44,  
89 which is an indicator of slag activity, that is considered good within a range from 1.3 to 1.5 [50].  $D_{50}$   
90 represents the average particle size in the production and application of powder materials, which can  
91 affect the durability of the geopolymer, since a large surface area leads to a higher polymerization  
92 rate and a difference in the number of voids [51]. For the GBFS used,  $D_{50} = 11.8 \mu\text{m}$ .

93 Table 1 - Chemical composition (%) of precursors

Precursor	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	Na <sub>2</sub> O	SO <sub>3</sub>	TiO <sub>2</sub>	MnO
GBFS	43.2	37.2	10.5	0.6	7	0.6	0.1	0.5	0.3

94

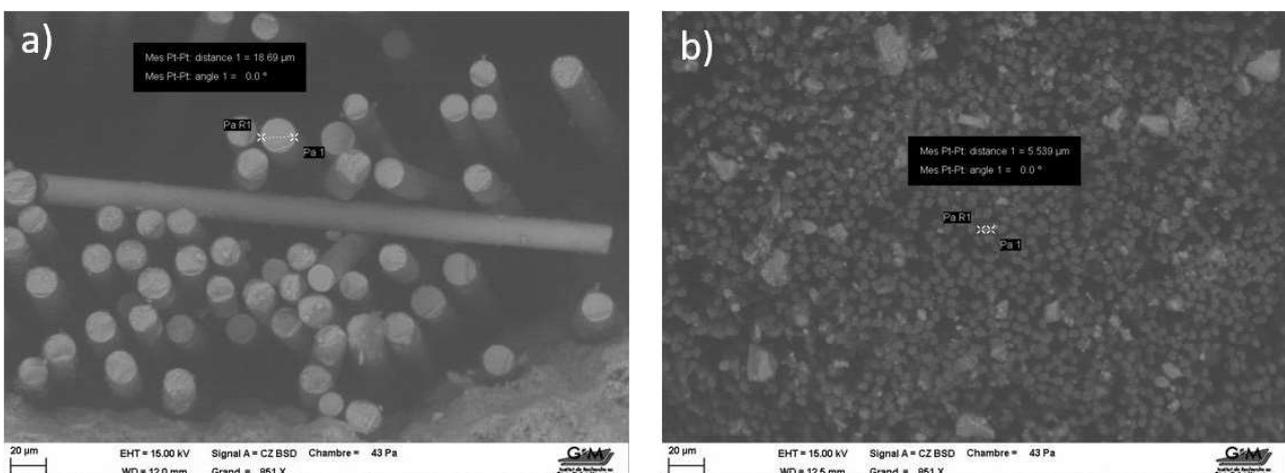
95 The alkaline activator solution employed in the mixtures, in its proper proportions, were prepared  
96 by mixing the sodium hydroxide (NaOH) solution with the sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) solution.

97 Sodium silicate (Na-Si) activator was prepared by mixing 10M sodium hydroxide (NaOH) with  
98 sodium silicate ( $\text{Na}_2\text{SiO}_3$ ), maintaining a  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio of 2.0. The produced Na-silicate  
99 activator contained 66.7% water with 0.7  $\text{Na}_2\text{O}/\text{SiO}_2$  molar ratio. NaOH was purchased from ALFA-  
100 AESAR in the form of pellets, white colored with 98% purity. Sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) was  
101 purchased from VWR in soluble form, of pH between 11-11.5 and density 1.35 g/cm at 20°C.

102 The precursor material ( $1,458.3 \text{ kg/m}^3$ ) was mixed for three minutes to have a more homogeneous  
103 mixture. Following, the activating solution ( $335.3 \text{ kg/m}^3$ ) mixed with the water ( $208.3 \text{ kg/m}^3$ ) was  
104 added to the dry mixture and blended for three minutes more. The percentage of water in the mixture  
105 was 12.4% and solid-to-liquid ratio 2.0.

106 The glass fiber used was commercial S2-glass which have high tensile strength of 3700 to 4300  
107 MPa [52], without alkaline oxides, containing 65%  $\text{SiO}_2$ , 10% MgO and 25%  $\text{Al}_2\text{O}_3$ . The diameter  
108 for a single fiber was measured, by Scanning Electronic Microscopy (SEM), using ZEISS EVO®4  
109 electron microscope equipped with a secondary electron sensor and a backscattered electrons sensor.  
110 After the pull-out test, prisms samples cores were sliced and polished into square shapes of  $2\text{cm} \times 2\text{cm}$   
111 by 0.5 cm thickness. Single glass fibers used measured around 17-20  $\mu\text{m}$  and single carbon fibers had  
112 a diameter of 5-8  $\mu\text{m}$ , as show in Figure 1. The fibers were not preconditioned as the aim was to study  
113 the interaction of the raw materials.

114 Figure 1 - Diameter of glass fiber (a) and carbon fiber (b) measured by SEM observations

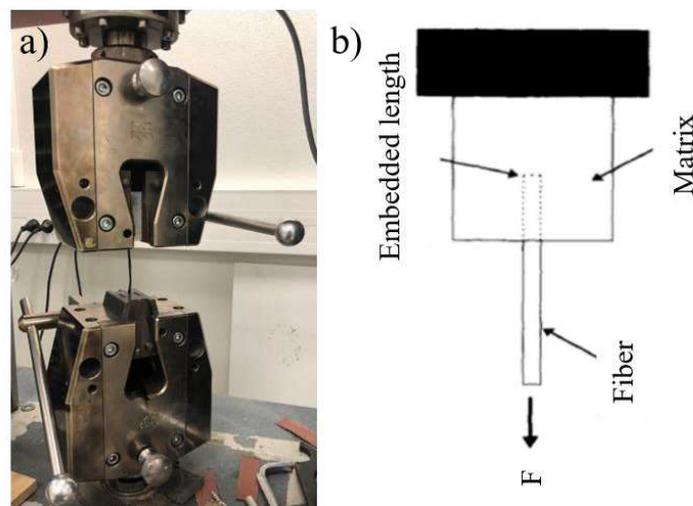


115

116 A total of 18 rectangular specimens were tested to find the critical embedded length and the  
117 maximum interfacial shear (bond) strength. Three essays were carried out for each type of fiber,  
118 carbon (CF) and glass (GF) at each embedded length of 10 mm, 20 mm and 30 mm [53]. The  
119 specimens were named according to the type of fiber used and the embedded length, e.g., for carbon  
120 fiber embedded 10 mm, specimen was named CF\_10mm. The geopolymer paste specimens all had  
121 five cm in height and cross section of approximately one cm<sup>2</sup> [54]. Each fiber strand, composed of  
122 multiple fibers, had approximately one millimeter in diameter, measured by SEM observations [55].  
123 The specimens were prepared at 20 °C and 50% humidity, demolded after 24 hours and kept in a  
124 plastic film until testing date. All the specimens were tested at seven days [54]. As observed in Alves  
125 [6], for this mixture, the GBFS had already been activated at this curing time.

126 A displacement of 0.5 mm/min [56, 57] was applied to the fiber on a Swift/Roell Z050 machine  
127 (Figure 2a), adapted with a Restrained Top Constraint (RTC) grapple, with a load cell of 50kN,  
128 following the schematics presented on Figure 2b (adapted from Yue [37]). While the force was  
129 applied, the behavior at the fiber-matrix interface was verified through the graph generated. The test  
130 was finished once the fiber is totally pulled-out of the matrix. The data obtained from the fiber pull-  
131 out test determines the applied loads from the shear stress and the relative displacements occurring at  
132 the fiber-matrix interface. With these results, it is possible to compute the relative adhesion stress and  
133 the strength effectiveness conferred by the fibrous reinforcement [58].

134 Figure 2 – Experimental setup of pull-out test (a) and pull-out test schematics (b)



135

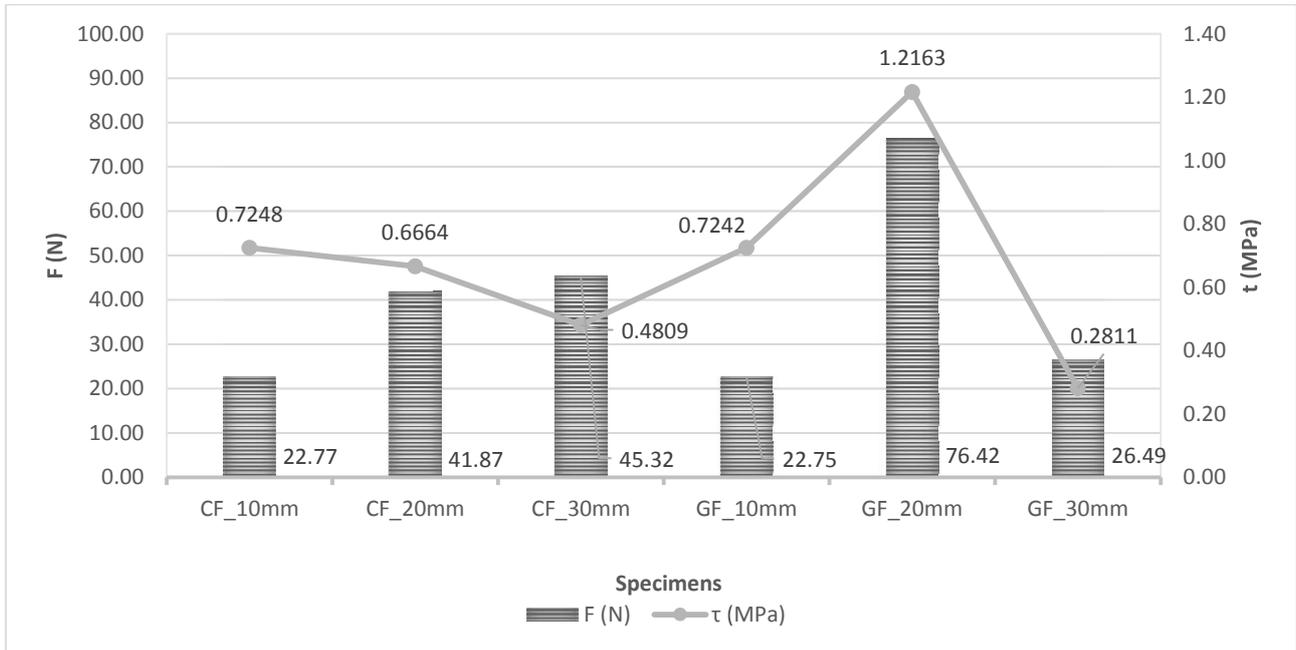
136 **3. Results and discussion**

137 Through the data analysis of the applied loads (F) and relative displacements ( $\delta$ ) that occur  
 138 between the fiber and the matrix, it is possible to define the values of adhesion stress ( $\tau$ ) [39].  
 139 Compiled results, according to the embedded length of the reinforcing fiber, are shown in Table 2  
 140 (standard deviation values are in parentheses) and Figure 3.

141 Table 2 – Results for the different specimens of the pull-out test

Specimen	F (N)	$\delta$ (mm)	$\tau$ (MPa)
CF_10mm	22.77 (2.54)	3.04 (1.03)	0.7248 (0.1132)
CF_20mm	41.87 (4.03)	2.72 (0.28)	0.6664 (0.1745)
CF_30mm	45.32 (3.32)	1.39 (0.06)	0.4809 (0.1483)
GF_10mm	22.75 (3.87)	3.47 (0.17)	0.7242 (0.0863)
GF_20mm	76.42 (2.96)	2.82 (0.51)	1.2163 (0.1699)
GF_30mm	26.49 (5.12)	1.85 (0.70)	0.2811 (0.1311)

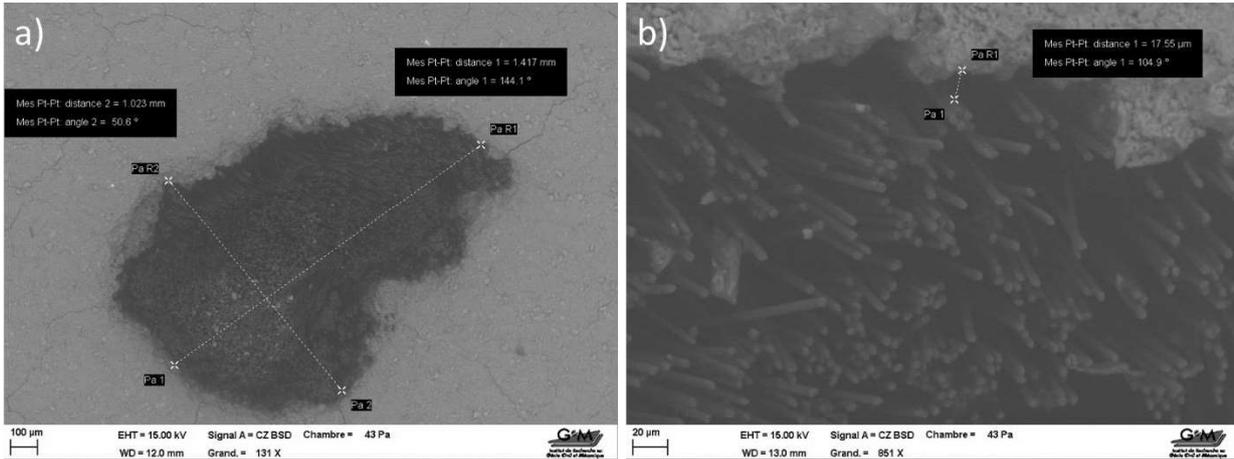
142 Figure 3 - Compiled values from the pull-out test  
 143



144  
 145 For specimens CF\_10mm and CF\_20mm the fiber was pulled out. Results show that the adhesion  
 146 stress was higher for specimens with 10 and 20 mm of embedded length of carbon fiber, with values  
 147 equal to 0.7248 and 0.6664 MPa, respectively. On specimen CF\_30mm, the fiber fractured after  
 148 reaching 45.32 N. For 30 mm length the lowest value for the adhesion stress was obtained reaching  
 149 0.4809 MPa. The values obtained show higher values of adhesion stress for composite CF\_10mm,

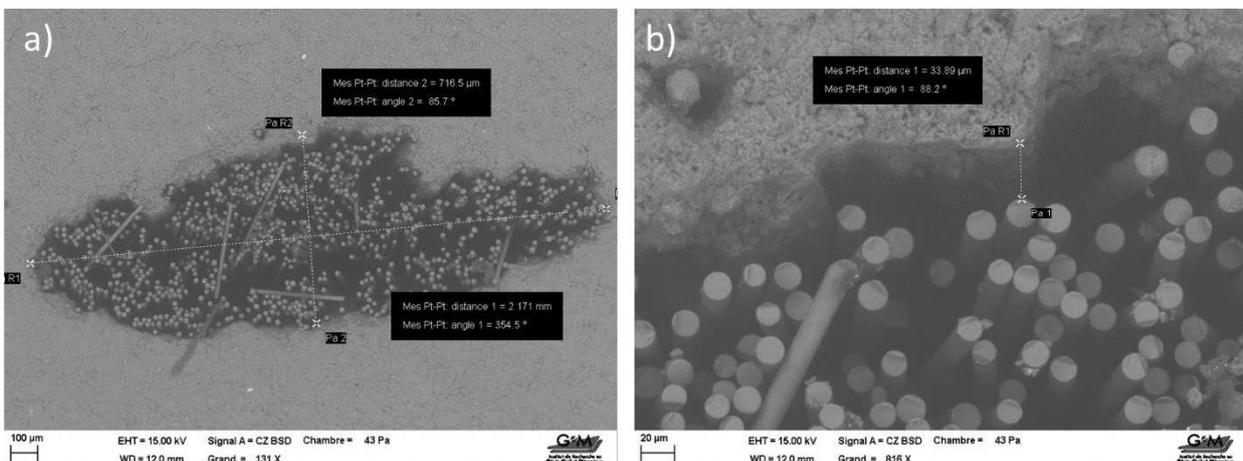
150 but higher force for CF\_30mm. Specimen CF\_20mm presented the best adherence due to the higher  
151 value of applied load, without fiber fracture. Figure 4 shows SEM observations of the cross section  
152 and interface for one of the carbon fiber specimens with embedded length of 20 mm.

153 Figure 4 - SEM observations for cross section of carbon fiber (a) and interface after pull-out (b)



154  
155 Glass fiber results showed same trend as that of carbon fiber. For specimens GF\_10mm and  
156 GF\_20mm the fiber was pulled out, with adhesion stress values of 0.7242 MPa and 1.2163 MPa,  
157 respectively. For 30 mm embedded length, a fracture on the fiber can be observed, at 26.49 N, due to  
158 the low value of 0.2811 MPa for adhesion stress. Thus, it is possible to affirm that the embedded  
159 length of 20 mm has the greatest capacity for transferring stresses between glass fiber and the matrix.  
160 Figure 5 shows SEM observations of the cross section and interface for one of the glass fiber  
161 specimens with embedded length of 20 mm.

162 Figure 5 - SEM observations for cross section of glass fiber (a) and interface after pull-out (b)

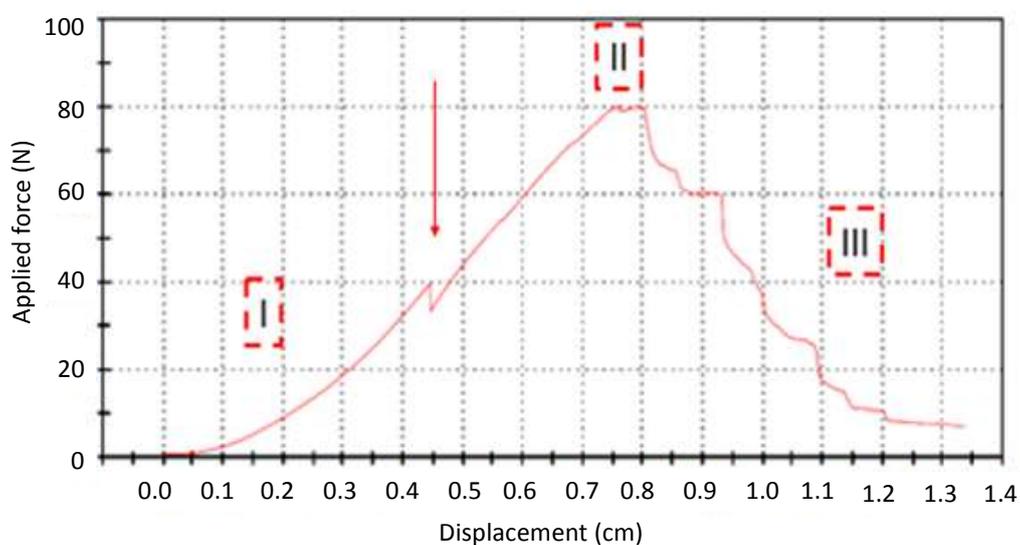


163

164 The critical length ( $L_c$ ) is a possible explanation for the fact that the embedded lengths of 20 mm  
165 had higher stress transfer capacity between the fiber and the matrix [59]. Lengths below  $L_c$  present a  
166 deficiency in transfer of loads and the fiber slides through the matrix. For lengths above  $L_c$ , a greater  
167 interaction between the fibers can be noted, causing failure, entanglement and decrease of the  
168 effective length. According to Feih et. al [35], sizing controls the interface for glass fiber composites,  
169 because it is responsible for the physical-chemical link between the fiber surface and the matrix  
170 system.

171 The fiber material influences the efficiency of the reinforcement and of the displacement related  
172 to the applied load, due to chemical interactions [35] between the type of fiber and the matrix material.  
173 From the results, the fiber-matrix adhesion ( $\tau$ ) for the glass fiber system is better than for the carbon  
174 fiber system. This implies the glass fiber system has a matrix-to-fiber stress transfer more effective,  
175 which suggests higher tensile strength for the composite. Applied loads ( $F$ ) and relative displacements  
176 ( $\delta$ ) are higher for glass fiber, which suggests higher toughness for the glass fiber composite, as  
177 observed from the larger values of  $\tau$ . The applied load ( $F$ ) vs displacement ( $\delta$ ) curve for the specimen  
178 CF\_20mm, which presented bond strength and interaction in the fiber-matrix interface superior to the  
179 other specimens analyzed, is plotted in Figure 6.

180 Figure 6 – Pull-out test results for specimen GF\_20mm



181

182 When studying the results for the specimen that presented the best adherence (Figure 6), it is  
183 important to notice that the pull-out test can be divided into three phases [37, 58]. The initial  
184 debonding and sliding phase, found in region I of the graph, is characterized by being an elastic-linear  
185 section, where the load is constantly increased until reaching a nonlinear stretch. Region II is qualified  
186 as maximum fiber stress, where the extraction force reaches the maximum value ( $F_{\max}$ ), and  
187 decohesion becomes partial. After this phase, there is a constant drop in the load, corresponding to  
188 region III. This region is controlled by the friction resistance of the interface and consists of sliding  
189 and pull-out until the fiber is extracted and completely withdrawn [37].

#### 190 **4. Conclusions**

191 Understanding the interface properties helps in enhancing the structural properties of composite  
192 materials by improving the interfacial bonding. This work described a procedure to measure apparent  
193 interfacial shear strength from pull-out tests on fibrous reinforcements. The pull-out test provides  
194 results of fiber-matrix adhesion and interfacial properties that can characterize and assess fiber-  
195 reinforced composites. The interfacial parameters of applied loads ( $F$ ), relative displacements ( $\delta$ ) and  
196 adhesion stress ( $\tau$ ) can be determined from pull-out data.

197 Two types of fiber were studied, glass fiber and carbon fiber, in three different embedded lengths  
198 of 10 mm, 20 mm and 30 mm. This study is fundamental to determine which material can be  
199 considered more efficient and makes the matrix more resistant to tensile and deformation strength. In  
200 addition to the different behaviors noticed due to the type fibers used, the influence of the embedded  
201 length of the fiber was another factor analyzed. The embedded length affects the load transfers and,  
202 therefore, the reinforcement efficiency. It is possible, through these data, to infer which composite  
203 has a more effective matrix-to-fiber stress transfer and determine the fiber critical embedded length.

204 It was concluded that the fiber that presented the best adherence for GBFS-based matrix is glass  
205 fibers, with 20 mm embedded length. The value obtained for adhesion stress of glass fiber at 20 mm  
206 embedded length is 68% higher than for glass fiber at 10 mm embedded length. When comparing  
207 with the carbon fiber system that has the higher value of applied load without fiber fracture, the glass

208 fiber system value corresponding to fiber-matrix interface is 83% higher. Critical length was  
209 considered 20 mm. With embedded lengths superior to the critical length, the fiber failure can occur  
210 before interface debonding. Lengths bellow critical length also causes deficiency in transfer of loads  
211 because there is a minimum critical length of the fiber required for a valid shear pull-out.

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