

# Evaluation of bond strength between glass fiber and resin composite using different protocols for dental splinting

Amaral R. Fabrício, Queiroz C. José Renato<sup>1</sup>, Leite P. P. Fabíola<sup>2</sup>, Reskalla N. J. F. Helcio<sup>2</sup>, Rodrigo Furtado de Carvalho<sup>3</sup>, Özcan Mutlu<sup>4</sup>

Department of Dental Materials and Prosthodontics, Araraquara Dental School, UNESP University, Estadual Paulista, <sup>1</sup>Department of Physics, Aeronautical Institute of Technology, São José dos Campos, <sup>2</sup>Department of Restorative Dentistry, Federal University of Juiz de Fora, Faculty of Dentistry, <sup>3</sup>Department of Dental Materials and Prosthodontics, São José dos Campos Dental School, UNESP University, Estadual Paulista, Brazil, <sup>4</sup>Department of Dentistry and Oral Hygiene, Head of Dental Materials Unit, Center for Dental and Oral Medicine, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, University of Zürich, Rämistrasse, Zürich, Switzerland

**Address for correspondence:**  
Dr. Rodrigo Furtado de Carvalho,  
São Mateus, 270/601,  
36025-000, Bairro São Mateus, Juiz de  
Fora, MG, Brazil.  
E-mail: rf-carvalho@hotmail.com

## ABSTRACT

**Context:** Many different polymeric materials to chair-side application on pre-impregnated glass fibers (PGF) are available and different protocols are used in clinical procedure. **Aims:** This study evaluated protocols used for dental splinting on adhesion between PGF and resin. **Settings and Design:** 42 pair of nano composite resin blocks with  $(6 \times 6 \times 8) \text{ mm}^3$  were assigned into seven groups ( $n=6$ ) and bonded according to the protocol: Gar) adhesive, resin; Ggr) glass fiber, resin; Ggar) glass fiber, adhesive, resin; Gfgar) flowable resin, glass fiber, adhesive, resin; Ggafr) glass fiber, adhesive, flowable resin, resin; Ggfar) glass fiber, flowable resin, adhesive, resin; Gfgr) flowable resin, glass fiber, resin. **Materials and Methods:** Micro sticks obtained from each group were submitted to the micro tensile bond strength test. **Statistical Analysis:** The data were statistically evaluated using ANOVA and Tukey's test (5%). **Results:** The protocol had a significant effect on the bond strength results ( $P=0.00$ ). Gar and Ggar resulted in the highest bond strength with no statistical difference. **Conclusions:** The use of adhesive agent showed to be efficient to promote initial adhesion between fiber and nano composite resin.

## Key words

Adhesion, bond strength, fiber, resin composite, resin flow

## INTRODUCTION

Direct chair-side splint is a simple and reversible clinical conduct with minimal or no tooth preparation in dentistry,<sup>[1,2]</sup> which promotes tooth stabilization after periodontal or orthodontic treatment to prevent progressive increase tooth mobility, space maintenance, protection of post traumatic teeth, prevents passive or active tooth movement and improving function with esthetic and comfort, mainly in patients with compromised dentitions.<sup>[1,3,4]</sup> Retainers are often made of either stainless steel wires or fiber-reinforced composites (FRC) of diverse types.<sup>[5]</sup>

The failures rates when stainless steel wire in the bonded orthodontic post-treatment stabilization splint is used are high.<sup>[5]</sup> Possible air voids in the manually further impregnated FRC that can cause stress concentration points that could result in a weakening of the polymer matrix of splint promote pre-impregnated FRC as a good option to use in splinting treatment. Defects in the interphase resin/material reinforcement interfere in transmission force between fiber and matrix. Furthermore, voids of poorly impregnated fibers become an inclusion body in the splinting. The oxygen could inhibit resin matrix polymerization, decrease load-bearing capacity of the FRC and increase water absorption that cause negative effect in mechanical properties.<sup>[4,6-8]</sup>

To pre-impregnated glass fibers (PGF), the inorganic material is covered with a silane coupling agent and then pulled through convoluted paths around supports with a bath of light-and/or heat-curable monomer systems of polymers.<sup>[6]</sup> PGF acquires adequate flexure modulus and flexure strength,<sup>[2]</sup> adequate handling characteristics, improving adhesion because of the semi-IPN (interpenetrating polymer network) structure

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of the polymer matrix.<sup>[5,9]</sup> In addition, when the PGF are correctly processed induce no cytotoxicity.<sup>[10]</sup>

However, the perfect adhesion is required to transfer load from the matrix to the PGF. Without adhesion, the principle of fiber reinforced systems would not work, i.e., the strong fiber carries the load while the matrix distributes it and transfers from one fiber to the other.<sup>[11]</sup>

Many different polymeric materials to chair side application on PGF are available, and different protocols are used in clinical procedure.<sup>[1-3,5,7]</sup> An excessively strong interface between PGF and polymeric matrix leads to a rigid composite while in the case of weak adhesion, the above mentioned principle does not work; thus the strength of adhesion must be set to an ideal value.<sup>[11]</sup>

In order to determine the protocol used for dental splinting on adhesion between PGF and resin, this study evaluated the micro tensile bond strength between a PGF and different polymeric materials used in resin/fiber contact (RFC) (resin composite, resin flow and resin bond). The hypothesis is that resin bond agent improves bond strength on PGF among other materials.

## MATERIALS AND METHODS

The brand names, material types, manufacturers and batch numbers of the products used in the current study are presented in Table 1.

Eighty-four blocks (4.5 × 8 × 7) mm of nano composite (concept advanced, Vigodent, Rio de Janeiro, RJ, Brazil) were confectioned (incremental technique: 1.5 mm of thickness from each increment) using an addition silicone putty mold (Elite HD, Zhermack, Badia Polesine, Italy) and photo-cured (XL 3000, 3M ESPE, St. Paul, MN, USA). The intensity of the light was verified to be no lower than 500 mW/cm<sup>2</sup> using a radiometer before starting light polymerization in each group. The adhesion surface of each block was leveled and polished in a machine using silicon carbide papers in sequence (600, 800, and 1200 grit) under water cooling.

**Table 1: Brand name, material type and manufacturer of materials used in the current study**

Brand name	Material type	Manufacture	Batch number
Concept advanced	Nano particulated resin composite	Vigodent, Rio de Janeiro, Brazil	221-09
Grand Tec	Pre-impregnated fiber glass	VOCO, Cuxhaven, Germany	03528
Grandio	Flowable resin	VOCO, Cuxhaven, Germany	0842295
Single bond	Adhesive bond agent	3M ESPE, St Paul, USA	8RY

All blocks were cleaned in sonic bath during 5 min using distilled water, dried with air-spray (free oil contaminants) and randomly divided among the groups. To each resin block pair were bonded with materials applied on the adhesive surface following six strategies used for FRC construction and divided into seven groups: Gar) adhesive + resin composite; Ggr) glass fiber + resin composite; Ggar) glass fiber + adhesive + resin composite; Gfgar) flowable resin + glass fiber + adhesive + resin composite; Ggafr) glass fiber + adhesive + flowable resin + resin composite; Ggfar) glass fiber + flowable resin + adhesive + resin composite; Gfgr) flowable resin + glass fiber + resin composite. Each material was used according manufactures recommendations.

### Specimen preparation for the microtensile bond strength test

Resin-resin blocks were sectioned using a diamond disc at low-speed, under water cooling, in a sectioning machine (LabCut 1010, Extec, Enfield, CT, USA). The first and the last section were discarded in case of the possibility of excess or absence of material at the interface that might alter the results, using only the central specimens. It was obtained 6 microsticks from each block whit a bonded area measuring approximately 1.0 ± 0.1 mm<sup>2</sup> adhesive surface areas and 10 mm length. The micro sticks obtained from each resin block were kept in distilled water at 37°C for 48 h.

### µTBS test

Each micro sticks were attached with cyanoacrylate gel (Super Bonder Gel, Loctite Ltd., São Paulo, SP, Brazil), keeping the adhesive zone free, to the rods of a device adapted for this test. Micro sticks were positioned parallel to the long axis of the device in order to reduce the bending stresses. The device was fixated in the universal testing machine (EMIC DL-1000, Santo José dos Pinhais, PR, Brazil), as parallel as possible in relation to application of the tensile load, and testing was performed at a cross-head speed of 1 mm/min.

The bond strength was calculated according to the formula  $R=F/A$ , where “R” is the strength (MPa), “F” is the load required for rupture of the micro sticks and “A” is the interface area of the specimens (mm<sup>2</sup>).

### Fracture analysis

The fractured surfaces of the microsticks were analyzed in an optical microscope at ×60 (Mitutoyo, Measuring Microscope MFA, Kawasaki, Japan), and the scanning electron microscope (LEO 435VPi, LEO-Zeiss, Tokyo, Japan) at ×35 to ×100 magnification in SE mode to characterize the failure mode.

The fractures surfaces were classified according to the following scores: (a) adhesive failure along the interfacial region between the PGF and composites, (b) mixed

failure (adhesive failure between the resin and PGF together with cohesive failure of the resins); (c) cohesive failure along in resin composite or flowable resin.

**Statistical analysis**

Statistical analysis (Minitab 16, Minitab Inc., Pensilvânia, USA) to bond strength results was performed using one-way ANOVA and multiple comparisons with Tukey's adjustment test. P values less than 0.05 were considered to be statistically significant in all tests.

**RESULTS**

One-way ANOVA [Table 2] revealed that the  $\mu$ TBS values were significantly affected by splinting strategy. The results of Tukey's multiple comparison tests demonstrated that unfilled resin (resin bond agent and resin flow) presented significantly higher results than the other groups with material reinforcement [Table 3].

The fracture analysis performed in the micro sticks submitted to  $\mu$ TBS test [Figures 1 and 2] revealed that the pattern of failure was predominantly adhesive along the interface between the PGF and resin matrix for all groups; mixed failure also was observed in the groups that receiving unfilled resin on PGF; cohesive fracture was observed in the Gar (control group) with cohesive fracture of the composite resin.

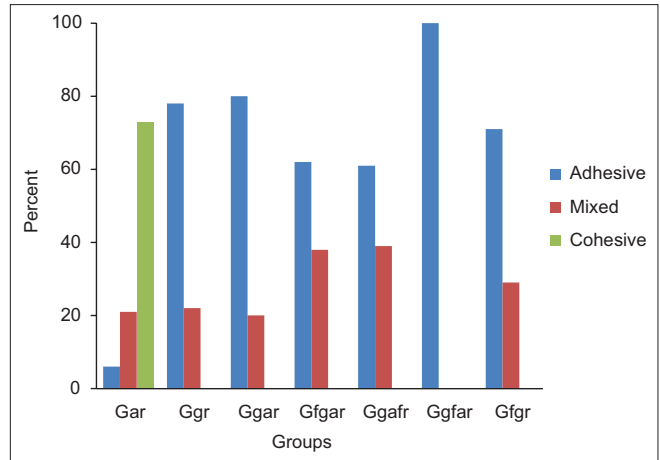


Figure 1: Fracture analysis in specimens submitted to microtensile bond strength (%)

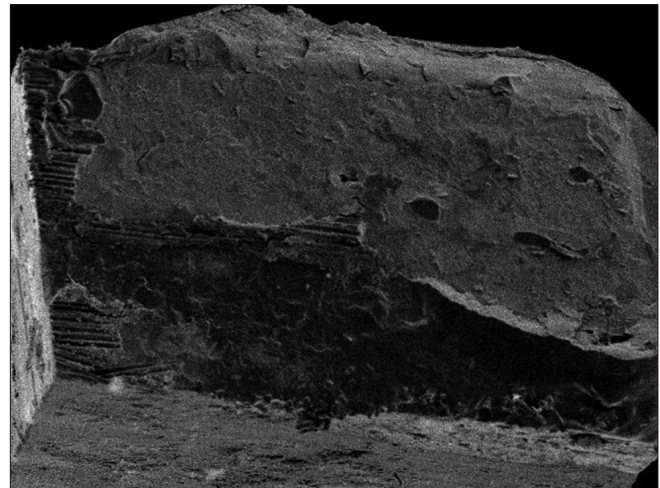


Figure 2a: Scanning electron microscope images of fracture surface of microsticks: Adhesive surface presented air bubble in mixed failure to specimen of Ggr

Table 2: Results of one-way analysis of variance for microtensile bond strength data (\*P<0.05)

Source	DF	SS	MS	F	P
Protocol	6	8992.4	1498.7	18.9	0.00
Error	245	19429.3	79.3		
Total	251	28421.7			

DF – Degree of freedom; SS – Sum of square; MS – Average square; F – Statistical value

Table 3: Mean and standard deviation of microtensile bond strength values (MP<sub>a</sub>) for different groups. Number of microsticks produced and percentage (%) of pre-test failures during sectioning and microstick preparation

Groups	Microsticks (%)	PTF (%)	Mean (SD)	Tukey's test*
Gar	36 (100)	3 (8.3)	22.4 (13.5)	a
Ggr	36 (100)	27 (75.0)	3.9 (4.3)	d
Ggar	36 (100)	1 (2.8)	17.2 (7.8)	ab
Gfgar	36 (100)	4 (11.1)	12.6 (7.0)	bc
Ggafr	36 (100)	18 (50.0)	6.4 (5.5)	d
Ggfar	36 (100)	20 (55.6)	7.8 (9.7)	c
Gfgr	36 (100)	15 (41.7)	12.2 (11.0)	bc

\*The same superscripted letters indicate no significant differences (Tukey's test, P<0.05); SD – Standard deviation; PTF – Pre-test failures; MP<sub>a</sub> – Mega pascal; Gar – Adhesive+resin; Ggr – Glass fiber, resin; Ggar – glass fiber, adhesive, resin; Gfgar – Flowable resin, glass fiber, adhesive, resin; Ggafr – Glass fiber, adhesive, flowable resin, resin; Ggfar – glass fiber, flowable resin, adhesive, resin; Gfgr – flowable resin, glass fiber, resin

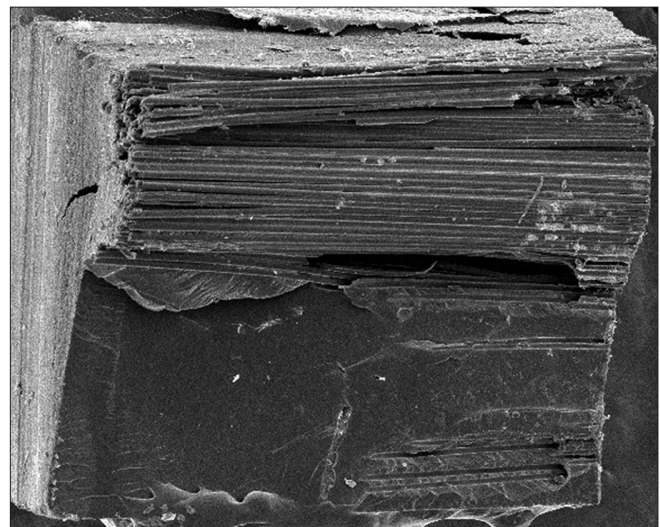


Figure 2b: Scanning electron microscope images of fracture surface of microsticks: Mixed failure to specimen of Ggar

## DISCUSSION

The results showed that resin bond agent applied on PGF in RFC improved bond strength in this multiphase resin system. The hypothesis was accepted.

Della Bona and van Noort related that  $\mu$ TBS test is a more appropriate method to evaluate the bond strength of adhesive interfaces.<sup>[12]</sup> It can be explained by the more uniform interfacial stresses distribution in this type of test than shear test. It should be emphasized that when the shear bond strength values with FRC materials are measured and reported, the anisotropic behavior of the FRC must be taken into consideration to avoid the false interpretation of the resultant bond strength data.<sup>[13]</sup> In order to minimize the influence of interfacial defects and eliminate non-uniform stress distribution at the adhesive interface, a tensile bond test with reduced testing area has been evaluated. Because of these aspects the  $\mu$ TBS was performed on this study.

Regarding the properties of heterogeneous materials, they are determined by the same four factors; the characteristics of the components, composition, structure and interfacial interactions. Nevertheless, interfacial interactions and interfaces play a key role in all multicomponent materials irrespectively of the number and type of their components or their actual structure. They are equally important in particulate filled polymer fiber reinforced advanced composites or nanocomposites. The difficulty of estimating the role of interfaces and interphases may arise from the fact that the type, mechanism and strength of interaction developing between the phases in multicomponent materials may vary in a very wide range as a function of component characteristics.<sup>[11]</sup>

In the first FRCs used in dentistry (hand-fabricated), inadequate impregnation of fibers with the matrix polymer with air bubbles trapped between the fibers in the matrix was founded.<sup>[1,4,6]</sup> Resin PGF, under controlled conditions, offer superior properties such as handling, flexure modulus and flexure strength.<sup>[2,4,9]</sup> Failures were mostly debonding of the resin matrix from the PGF [Figure 1], which could be considered as the weakest part of the RFC, showing the stable adhesion between inorganic and organic materials of PGFs.

Little information is available in literature about the bonding of PGF and the resin composite, the matrix frequently used in chair-side restorations. Many different protocols are presented in literature using different materials.<sup>[7,14,15]</sup> The use of different materials with various properties and different compositions could affect the bond strength and the failure types between PGF and resin matrix. When long-term splint is required, as in teeth with immature short roots, horizontal root fractures or alveolar bone fractures, or in periodontal treatments

and when the retention is necessary following orthodontic treatment,<sup>[16-18]</sup> the failure of splint could have influence on the periodontal healing and stability of tooth.

The present study showed the use of a bonding agent on PGF prior the resin composite application was important to improve bond strength between PGF and the matrix composite to Ggar, in accordance with others studies.<sup>[1,14]</sup> This group was not significantly different from the Gar (control group) [Table 3]. The adhesive system holding hydroxyethylmethacrylate (HEMA) was used because previous studies have shown that low molecular weight HEMA can effectively penetrate to the linear phases of the matrix and thus enhance the bonding by interdiffusion<sup>[14]</sup> suggested too by fracture analysis of Gar (control group) shows in Figure 1. In addition, the low viscosity properties permitted the flow of adhesive bonding agent on spaces between fibers, increasing the contact area between different phases. Air bubble was founded on the fracture surface in Ggr showing the difference in fracture pattern compared with Ggar [Figure 2]. Thus, the adhesive bonding agent influenced on the  $\mu$ TBS results and in the failure type. Strong interface has an effect on stress distribution and interfacial fracture behavior improving mechanical properties of RFC. In the microscopic observation, Ggar presented high amount of adhesive failure from the PGF surface [Figure 1] when compared among other groups. Although, was the group that presented lower rate of pre-test failure during the cut of sticks [Table 3].

The fabricant recommends that GrandTEC-glass fiber must always be covered with at least one layer of a (flowable) composite for all applications. In the present study, the use of flowable resin composite (Gfgr) showed statistical difference in the  $\mu$ TBS results when compared with the control group (Gar). Then, the use only flowable resin was not the most efficient strategy to improve bond strength between glass fiber and resin composite. Moreover, this strategy (Gfgr) showed a high rate of pre-test failure and large standard deviation to  $\mu$ TBS results [Table 3]. Previous studies reported that swelling of the composite substrate surface with different solvents and the use of low-viscosity intermediate monomer resins, such as adhesive bonding agent, influenced on the bond between two composites,<sup>[14]</sup> in accordance with our results.

The basic condition of the application of FRC is the perfect adhesion between the components, causing better stress distribution to fiber without compromise interface adhesion between PGF/resin matrix, increasing strength to the fracture of splitting. This study showed that the efficacy of reinforcement could depend of the use of an adhesive bonding agent on PGF.

News research will be necessary to study the effect of the aging on this interface. Subsequently, studies about

the influence of this approach on the bond strength of splint by RFC in the tooth and on the fracture pattern in this interface are important to apply this protocol in clinical studies.

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