

Surface conditioning of glass fiber-reinforced epoxy resin: which protocol achieves bond strength and surface characteristics comparable to human dentin?

Condicionalmento da superfície de resina epóxi reforçada com fibra de vidro: qual protocolo proporciona resistência de união e características de superfície comparáveis à dentina humana?

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ABSTRACT

Objective: The aim of this study was to evaluate which surface conditioning protocol enables glass fiber-reinforced epoxy resin (GFRER), used as a dentin analogue, to achieve microshear bond strength (μ SBS), surface roughness, and contact angle values comparable to those of human dentin. **Material and Methods:** Human dentin specimens were used as the reference group (DENT), while GFRER slices were assigned to five surface-conditioning protocols: no conditioning (CTR), 5% hydrofluoric acid (HF5), 10% hydrofluoric acid (HF10), 37% phosphoric acid (H_3PO_4), or air abrasion with 110 μ m aluminum oxide particles (AA). Thirteen substrate specimens were included in each group ($n = 13$). Five resin cement cylinders were prepared on each specimen, which were then subjected to 12,000 thermal cycles. The bond strength value for each specimen was calculated as the mean of the five cylinders. Data were analyzed by one-way ANOVA and Tukey's post hoc test ($\alpha = 0.05$). Failure pattern, surface roughness, and contact angle were also evaluated. **Results:** No GFRER group differed from DENT in μ SBS (9.2 ± 3.1 MPa; $p > 0.05$). Among GFRER groups, HF5 (12.3 ± 2.0 MPa) showed higher μ SBS than CTR (8.2 ± 3.6 MPa) and HF10 (8.6 ± 3.3 MPa) ($p < 0.05$). Adhesive failures predominated. CTR and H_3PO_4 were the only GFRER conditions showing both roughness and contact angle statistically similar to DENT. Surface topography was modified by all conditioning protocols. **Conclusion:** GFRER produced μ SBS comparable to human dentin regardless of the conditioning protocol. CTR and H_3PO_4 produced surface-related outcomes most similar to DENT.

KEYWORDS

Dental bonding; Dentin; Epoxy resins; Shear strength; Wettability.

RESUMO

Objetivo: O objetivo deste estudo foi avaliar qual protocolo de condicionamento de superfície permite que a resina epóxi reforçada com fibra de vidro (GFRER), utilizada como análogo de dentina, apresente valores de resistência de união ao microcisalhamento (μ SBS), rugosidade superficial e ângulo de contato comparáveis aos da dentina humana. **Material e Métodos:** Espécimes de dentina humana foram utilizados como grupo de referência (DENT), enquanto fatias de GFRER foram distribuídas em cinco protocolos de condicionamento superficial: sem condicionamento (CTR), ácido fluorídrico a 5% (HF5), ácido fluorídrico a 10% (HF10), ácido fosfórico a 37% (H_3PO_4) ou jateamento com partículas de óxido de alumínio de 110 μ m (AA). Treze espécimes de substrato foram incluídos em cada grupo ($n = 13$). Cinco cilindros de cimento resinoso foram confeccionados sobre cada espécime, que posteriormente foram submetidos a 12.000 ciclos térmicos. O valor de resistência de união de cada espécime foi calculado como a média dos cinco cilindros. Os dados foram analisados por ANOVA 1 fator e teste post hoc de Tukey ($\alpha = 0,05$). O padrão de falha, rugosidade superficial e ângulo de contato também foram avaliados. **Resultados:** Nenhum grupo GFRER

diferiu do DENT em μ SBS ($9,2 \pm 3,1$ MPa; $p > 0,05$). Entre os grupos GFRER, HF5 ($12,3 \pm 2,0$ MPa) apresentou maior μ SBS que CTR ($8,2 \pm 3,6$ MPa) e HF10 ($8,6 \pm 3,3$ MPa) ($p < 0,05$). Falhas adesivas predominaram. CTR e H_3PO_4 foram as únicas condições de GFRER que apresentaram rugosidade e ângulo de contato estatisticamente semelhantes ao DENT. A topografia superficial foi modificada por todos os protocolos de condicionamento. **Conclusão:** A GFRER apresentou μ SBS comparável à dentina humana independentemente do protocolo de condicionamento. CTR e H_3PO_4 produziram resultados relacionados à superfície mais semelhantes ao DENT.

PALAVRAS-CHAVE

Adesão dentária; Dentina; Resinas epóxi; Resistência ao cisalhamento; Molhabilidade.

INTRODUCTION

The replacement and improvement of materials in the dental market are constant. The dental industry employs *in vitro* studies to evaluate quality and performance of these new materials. Although *in vitro* studies have inherent limitations, they play an essential role in the preclinical evaluation of dental materials by allowing the screening of material performance and supporting the design of future clinical studies. Extracted human teeth are considered the preferred substrate because they enable testing under conditions that more closely resemble the clinical situation [1]. However, the use of human dental substrate has some disadvantages, such as lack of substrate standardization [2,3], in addition to ethical aspects that make obtaining samples more difficult [4]. Furthermore, human dental tissues have become harder to find with the improvement in the population's oral health.

Human dentin is a mineralized tissue that occupies the largest portion of human teeth in weight and volume. One of the most distinct characteristics of this tissue is the network of tubules (approximately $0.5\text{--}1.5\ \mu\text{m}$ in diameter) that radiate from the pulp cavity toward the dentinoenamel junction [5]. Microscopic evaluations show that there are substantial spatial variations in the number, diameter, and orientation of the tubule lumens in the tooth crown [5]. These characteristics vary with the distance from the pulp, the physiology and the traumatic history of the tooth [6,7]. The search for alternative materials capable of replacing and mimicking the dentinal substrate is crucial when considering all of these structural, morphological and physiological variations distinctly presented by each dental element used for *in vitro* tests. Epoxy resin-based substrates have been used as dentin analogues due to their mechanical properties similar to dentin and their ability to provide standardized conditions in laboratory studies [8,9].

Kelly et al. [10] validated the use of an epoxy resin material filled with glass fibers, finding sufficiently similar results to dentin regarding its elastic modulus (18.6 GPa) and adhesion (to moist dentin, when the epoxy resin was treated with 8% HF acid etching followed by silane application). They also observed a similar failure pattern of the restorative set to those observed in cases with clinical failure [10]. Recent *in-vitro* studies have also used epoxy resin dies as standardized supporting substrates when evaluating the mechanical behavior of indirect restorations [8,9].

Although previous studies support the use of GFRER as a standardized dentin analogue, its bonding performance may depend on the surface-conditioning protocol applied before adhesive procedures. Different strategies have been reported in the literature, including hydrofluoric acid etching at variable concentrations, such as 10% [3,11-13], 8% [10], and 5% [14-17], as well as different acid action times [10,18,19], concomitant with the use of silane [11,14], adhesives [3,10,19] and primers [12,17], etching with 37% phosphoric acid [20,21], or even alumina-particle air abrasion [2,18,19], have been reported in several studies published in the literature. However, the effect of these different surface treatments on the epoxy resin material is not known, nor which presents the most similar performance to that presented by human dentin.

It is known that hydrofluoric acid etching is used to selectively remove the material's surface portion, exposing the vitreous matrix and creating surface microroughness in vitreous ceramics [12,22,23]. In evaluating the effect of different conditioning times with 10% hydrofluoric acid on a similar GFRER material, Merlo et al. [13] noted that the increase in acid exposure can affect the integrity of the glass fibers, causing peripheral degradation. To the best of our knowledge, it is not known whether different acids and concentrations also cause this effect. Blasting is another type of

surface treatment that is used in friable materials such as acid-resistant ceramics and indirect resin composites, in which the airborne abrasion increases the surface roughness and the bonding area, which alters the wettability. However, particle impact can cause surface defects and stress concentrations, which can interfere with the restoration performance [24-27].

Thus, based on the aforementioned information, the aim of this study was to evaluate which surface conditioning protocol enables glass fiber-reinforced epoxy resin (GFRER), used as a dentin analogue, to achieve microshear bond strength (μ SBS), surface roughness, and contact angle values comparable to those of human dentin. The null hypotheses were that: (1) surface conditioning protocols would not affect the microshear bond strength (μ SBS), surface roughness, or contact angle of glass fiber-reinforced epoxy resin (GFRER); and (2) there would be no differences in μ SBS, surface roughness, or contact angle between GFRER and human dentin, regardless of the surface conditioning protocol used.

METHODOLOGY

The description of the materials used in the present study, commercial names, manufacturer and composition are listed in Table I.

The present *in vitro* study was approved by the Research Ethics Committee of the Federal University of Santa Maria (CAAE: 44655321.1.00005346). Human molar teeth used in this study were donated by the Human Teeth Bank of the Federal University of Santa Maria. The sample size was determined from the study by Kelly et al. [10] through a sample calculation considering a statistical power of 80%, mean standard deviation of 2.2 and a detectable difference of 2.6 MPa. The sample size was defined as 13 slices of substrate for each group ($N = 13$), with 5 cylinders of resin cement cemented per slice. The bond strength value for each specimen was calculated as the mean of the five cylinders.

This study consists of 6 experimental groups according to Table II.

Specimen preparation

Human teeth

Non-carious human molar teeth were cleaned (periodontal curette MacCall 11/12 – Golgran, São Caetano do Sul, Brazil) and the occlusal

surfaces were ground using a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, United States) with #400 grit silicon carbide sand paper to remove the tooth's enamel until dentin exposure and surface standardization. During specimen preparation and before the bonding procedure, the exposed dentin surfaces were kept moist with gauze soaked in saline solution at room temperature to avoid dentin dehydration. This procedure followed the general recommendation of ISO/TS 11405:2015 that tooth surfaces should be kept wet during preparation, since dentin is especially sensitive to dehydration [28].

Glass fiber - reinforced epoxy resin (GFRER)

Glass fiber-reinforced epoxy resin material (Epóxi Pultrudado F, Protec) in a round-rod format (1000 mm $\times$ 10 mm) was cut into 2.7 mm -thick discs in a cutting machine (Isomet 1000, Buehler) with a diamond disc under constant refrigeration.

Embedding

Each DENT or GFRER slice was embedded in a cylindrical polyvinyl chloride (PVC) mold using a self-cured acrylic resin (VIPI Flash, Pirassununga, Brazil). For this, the specimens (dentin and GFRER) were fixed on the glass plate, wrapped in a sheet of wax number 7 (Lysanda, São Paulo, Brazil) with 2 mm height, leaving the top and bottom surfaces free of wax to preserve its bonding surface above the PVC level. Then, the PVC was placed over the specimen, the acrylic resin was poured into the mold and waited until its final polymerization. All specimens were polished (#400 and #600) [29] in a polishing machine (EcoMet/AutoMet 250, Buehler) to remove any wax or acrylic resin residue from the bonding surface. The specimens were cleaned in an ultrasonic bath with distilled water for 5 minutes (1440 D, 50/60 Hz, Odontobras, Ribeirão Preto, Brazil).

Surface treatment

All surface-conditioning procedures for DENT and GFRER groups were performed by a single trained operator.

Human teeth (DENT)

The dentin surface was conditioned with 37% phosphoric acid (Acid Gel, Villevie) for 15 seconds, washed with air-water-spray for 30 seconds, and lightly dried with gentle oil-free air-spray.

Table I - Description of materials, trade mark, manufacturer and composition

Material	Trademark / manufacturer	Composition
Fiber glass-reinforced epoxy resin – Round-rod	Epóxi Pultrudado F (Protec, São Paulo, Brazil)	Fiber glass wire and epoxy resin
37% phosphoric acid	Acid Gel (Villevie, Joinville, Brazil)	37% phosphoric acid
10% hydrofluoric acid	Condac Porcelana 10%, (FGM, Joinville, Brazil)	10% hydrofluoric acid
5% hydrofluoric acid	Condac Porcelana 5%, (FGM)	5% hydrofluoric acid
110 µm alumina particles	Oxido de Alumínio (Polidental, Cotia, Brazil)	alumina oxide particles (110 µm)
Primer coupling agent	Monobond N (Ivoclar, Schaan, Liechtenstein)	methacrylate, phosphoric acid methacrylate and sulphide methacrylate
Dual-curing dental adhesive	Excite F DSC (Ivoclar)	HEMA, dimethacrylate, phosphonic acidacrylate, highly dispersed silicone dioxide, initiators, stabilizers, potassium fluoride, alcohol solution
Resin cement	Variolink N (Ivoclar)	Catalyst: Barium glass filler and mixed oxide, dimethacrylates, ytterbium trifluoride, initiators and stabilizers, and pigments. Base: Barium glass filler and mixed oxide, dimethacrylates, ytterbium trifluoride, initiators and stabilizers, and pigments

HEMA = hidroxyethylmethacrylate.

Table II - Study design

Substrate	Surface Treatment	Groups
Human dentin	37% Phosphoric Acid, 15 seconds	DENT
	Without surface conditioning	CTR
Glass Fiber- Reinforced Epoxy resin - Round Rod format	5% Hydrofluoric Acid, 30 seconds	HF5
	10% Hydrofluoric Acid, 60 seconds	HF10
	37% Phosphoric Acid, 30 seconds	H ₃ PO ₄
	110 µm alumina particles air-abrasion	AA

Afterwards, the Excite F DSC adhesive (Ivoclar) was actively applied for 10 seconds, and air-spray was applied to uniform the layer and light-cured with a LED device (Radii Cal, SDI; Bayswater, Australia) with an intensity of 1200 mW/cm² for 10 seconds.

Glass fiber-reinforced epoxy resin material (GFRER)

For all GFRER groups, after the respective surface treatment and cleaning procedure, the silane bonding agent (Monobond N, Ivoclar) was actively applied for 15 s with a microbrush, left undisturbed for 45 s, and dried with an air jet.

Control (CTR)

No surface conditioning was performed. Only the silane bonding agent was applied as described above.

5% hydrofluoric acid (HF5)

The GFRER material's surface was etched with 5% hydrofluoric acid (Condac Porcelain

5%, FGM) for 30 seconds and washed with air/water jet for 30 seconds [14-17]. Afterwards, the specimens were cleaned in an ultrasonic bath with distilled water for 5 min to remove possible precipitates and dried with an air jet for 15 s. Then, silane was applied as described above.

10% hydrofluoric acid (HF10)

The GFRER material's surface was etched with 10% hydrofluoric acid (10% Condac Porcelain, FGM) [3,11-13], for 60 seconds and washed with air/water jet for 30 seconds [3,11-13]. Then, ultrasonic cleaning, drying, and silane application were performed as described for HF5.

37% phosphoric acid (H₃PO₄)

The GFRER material's surface was etched with 37% phosphoric acid (Acid Gel 37%, Villevie) for 30 seconds and washed with air/water jet for 30 seconds [20,21]. Then, ultrasonic cleaning, drying, and silane application were performed as described for HF5.

Alumina particles abrasion (AA)

The GFRER material's surface was air-abraded with 110 μm alumina oxide particles (Polidental, Cotia, Brazil) for 20 seconds, with a pressure of 2 bar at a distance of 10 mm from the substrate [2,18,19]. After air abrasion, an oil-free air jet was applied to remove loose alumina particles from the surface, and silane was applied as described above.

Surface roughness analysis

The bonding surface roughness of all specimens was measured after each surface treatment. The measurements were performed using a contact stylus profilometer (SJ-410, Mitutoyo, Japan). The Ra (the arithmetic means of the absolute values of the peaks and valleys measured from a mean plane) and Rz (the mean distance between the five highest peaks and the five lowest surface valleys) were calculated by the mean of three measurements in each x and y axis of the specimen, considering the ISO:21920-2:2021 parameters [30].

Contact angle analysis

The contact angle was measured in all the specimens after each surface treatment (no silane application), via the sessile drop technique using a goniometer (Drop Shape analysis, model DSA 30S, Kruss GmbH, Hamburg, Germany) connected to a computer program (DSA3, V1 .0.3-08, Kruss). First, one drop of distilled water (11 μl) was dropped at the center of each specimen using a needle. The drop was then measured once every second for five seconds and a mean value was determined for each specimen.

Bonding procedure

After the respective surface treatment, 5 starch tubes (Isabela, São Caetano do Sul, Brazil) with 1.0 mm of height and 1.2 mm of internal diameter were placed on each treated specimen surface, and fixed with wax number 7 (Lysanda) [31]. A single trained operator performed the bonding procedures at room temperature (25 °C). The resin cement (Variolink N, Ivoclar) base and catalyst pastes were dispensed (1:1), manipulated according to the manufacturer's recommendations, and inserted with a resin spatula (Titanium resin filled n°.

6 – Indusbello Company, Londrina, Brazil) into the starch tubes. The resin cement excesses were carefully removed with a microbrush. The resin cement of each starch tube was then light-activated (1200 mW/cm² of intensity; Radii Cal, SDI) for 40 seconds. The specimens were stored in distilled water at 37 °C for 24 hours. Then, the starch tubes were carefully removed with a clinical probe (#5, Golgran) and the resin cement cylinders were individually assessed to guarantee that no defects were present at the interface. The resin cement cylinder was excluded if any irregularity was detected.

Aging procedure

After storage for 24 hours, all specimens underwent intermittent 12,000 thermal-cycles (Ethik Technology Limited – model 521-6D; Vargem Grande Paulista, São Paulo, Brazil) with the temperature ranging from 5 °C and 55 °C with 30 seconds of dwell time at each temperature and 2 seconds of transfer time.

Microshear bond strength test - μSBS

The PVC cylinders were placed in a jig attached to a universal testing machine (EMIC DL1000, São José dos Pinhais, Brazil) so that the cement cylinder was aligned with the center of the load cell, parallel to the adhesive interface. A stainless-steel wire ($\varnothing = 0.2 \text{ mm}$) was looped around the resin cement cylinder parallel and as close as possible to the cement-substrate interface. The shear load was applied (10 kgf load cell) at a rate of 1 mm/min until failure occurred, and the data were recorded in Newton (N). The bond strength (MPa) was calculated using the formula " $S = L/A$ ", where " S " is the strength (MPa), " L " is the load at which the specimen failed (N) and " A " is the area of the adhesive interface (1.13 mm²).

Failure analysis

All tested specimens were inspected in a stereomicroscope (Discovery V20, Carl-Zeiss, Gottingen, Germany) at 30 $\times$ magnification to verify the failure type. The failures were classified as adhesive (less than 50% of resin cement remained at the adhesive interface) or cohesive (more than 50% of resin cement remained in the adhesive interface). Representative images of each failure type were obtained at 30 $\times$ magnification in a stereomicroscope.

Topographic analysis

Two additional specimens of each group were produced, their surfaces were treated as mentioned above, dried in a laboratory desiccator and gold-sputtered to be analyzed in SEM (Secondary electrons, 20Kv; VEGA3, Tescan) to evaluate the materials' surface characteristics at 100× and 1000× magnification.

Data analysis

For roughness, contact angle, and μ SBS analyses, the substrate specimen was considered the experimental unit ($n = 13/\text{group}$). For μ SBS, five resin cement cylinders were bonded to each substrate specimen. Therefore, the μ SBS values obtained from the five cylinders bonded to the same specimen were averaged, generating one mean μ SBS value per specimen. Data normality and homogeneity of variances were verified using the Shapiro-Wilk and Levene's tests, respectively. Since the assumptions were met, roughness, contact angle, and μ SBS data were analyzed using one-way ANOVA followed by Tukey's post hoc test ($\alpha = 0.05$). The statistical analyses were performed using IBM SPSS Statistics software (IBM Corp., v.27; IBM, Armonk, NY, USA).

RESULTS

Considering the post-conditioning mean roughness analysis (Table III), the AA group exhibited the highest roughness values and was statistically similar to the HF10 group. In contrast, the DENT, H_3PO_4 , CTR, and HF5 groups showed comparable roughness values, with no significant differences among them.

Regarding the post-conditioning contact angle analysis (Figure 1), the CTR (49.3), DENT (50.5), H_3PO_4 (54.2) and AA (52.4) groups were statistically similar to each other and smaller than the HF5 (87.3) and HF10 groups (74.0).

There was no statistical difference ($p > 0.05$) between human dentin and GFRER treated with different surface treatments regarding the microshear bond strength (Table IV). Among the GFRER groups, HF5 showed higher μ SBS than CTR and HF10 (Table IV). Failure at the adhesive zone was the main type of failure in all the groups (Table IV and Figure 2). The AA group had the major number of pre-test (26%) and cohesive (10%) failures (Table IV).

The topographic images (Figure 3) show a dentin surface free from a smear layer with exposed dentinal tubules, configuring an ideal surface for adhesion. Considering the surface treatments proposed for the dentin-analogue material, the conditioning of the surface of the material caused surface alterations. Greater removal and degradation of the epoxy matrix and exposure of the glass fibers can be better observed in the HF10 group, while the degradation was lower in the HF5 group. The H_3PO_4 group showed surface smoothing, and the deposition of aluminum oxide particles made the surface rougher and more heterogeneous in the AA group.

DISCUSSION

The first null hypothesis was rejected, since surface conditioning affected the μ SBS to GFRER. HF5 resulted in higher bond strength than CTR and HF10. Additionally, the HF5 and HF10 groups showed the highest contact angle values, whereas the AA group exhibited the highest surface roughness values, which were statistically similar to those of the HF10 group. The second null hypothesis was partially accepted, since GFRER, regardless of the conditioning protocol, showed μ SBS comparable to human dentin; however, only CTR and H_3PO_4 presented both surface roughness and contact angle values statistically similar to dentin.

Although the validity of bond strength tests to predict the clinical performance of dental materials is debatable [32], evidence shows that clinical outcomes can, to some extent, be estimated based on laboratory results [33]. These studies can quickly and easily collect data on a specific parameter or property while holding other variables constant, and experimental groups can be simultaneously tested within the same test methodology [34]. Kelly et al. [10] used a GFRER material as a viable alternative substrate to substitute the human dentin for mechanical and adhesive tests. However, the different surface treatments applied to the material, mainly in mechanical tests, reported in the literature show methodological variability regarding the material preparation prior to luting procedures [11,16,17,20].

Chemical and physical surface modifications of restorative components are necessary to establish reliable adhesion. The topographic analysis (Figure 3) of the representative samples

Table III - Mean and standard deviation from roughness after the surface treatments

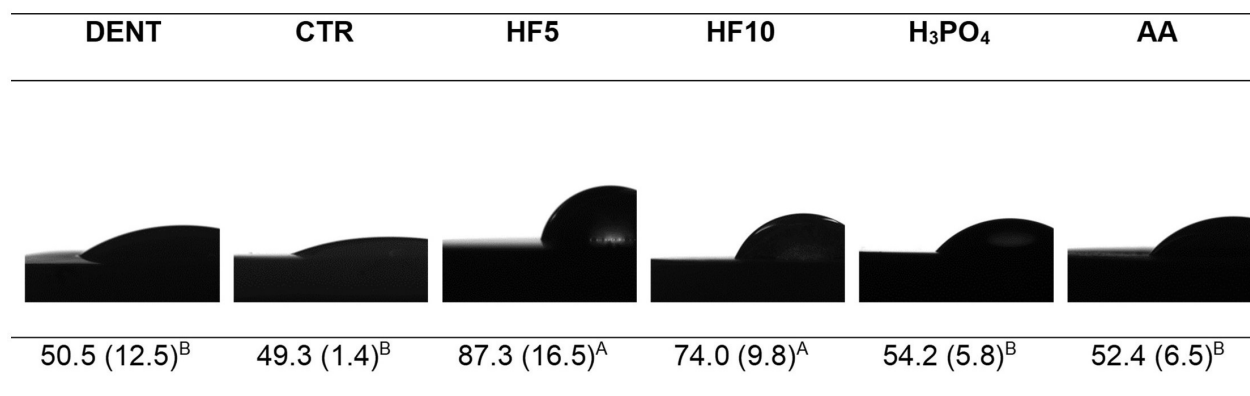
Substrate	Roughness	
	Ra	Rz
DENT	0.2 (0.7) ^{BC}	2.0 (0.6) ^{BC}
CTR	0.2 (0.1) ^C	1.4 (0.2) ^C
HF5	0.3 (0.1) ^{BC}	2.2 (0.2) ^B
HF10	0.5 (0.1) ^{AB}	3.1 (0.4) ^{AB}
H ₃ PO ₄	0.2 (0.1) ^C	1.4 (0.2) ^C
AA	2.0 (0.5) ^A	13.4 (1.3) ^A

Different uppercase letters in each column mean significant statistical difference based on Tukey post-hoc test ($\alpha = 0.05$) for roughness analysis (N = 13).

Table IV - Microshear bond strength (mean and standard deviation) in MPa, total of pre-test failures (PTF) and types of failure evaluated after the bond strength tests – predominantly adhesive failure or predominantly cohesive failure

Substrate	μ SBS – mean (SD)	Failure - n (%)		
		PTF	Cohesive	Adhesive
DENT	9.2 (3.1) ^{AB}	10 (15.3)	3 (4.6)	52 (80.0)
CTR	8.2 (3.6) ^B	3 (4.6)	1 (1.5)	61 (93.8)
HF5	12.3 (2) ^A	3 (4.6)	0 (0.0)	62 (95.3)
HF10	8.6 (3.3) ^B	9 (13.8)	3 (4.6)	53 (81.5)
H ₃ PO ₄	9.7 (3.9) ^{AB}	10 (15.3)	0 (0.0)	55 (84.6)
AA	8.8 (2.8) ^{AB}	17 (26.1)	7 (10.7)	41 (63.0)

Same uppercase letters indicate statistical similarity reported by Tukey post-hoc test ($\alpha = 0.05$) for microshear bond strength. Failures were calculated considering each resin cement cylinder bonded to each substrate specimen (n = 65/group).

**Figure 1** - Images of contact angle of the surfaces in each group post surface treatment: DENT (phosphoric acid 37%), CTR (control, without surface conditioning), HF5 (hydrofluoric acid 5%), HF10 (hydrofluoric acid 10%), H₃PO₄ (phosphoric acid 37%) and AA (110 μ m alumina particles air-abrasion).

of each group showed that the different proposed surface treatments have the ability to modify the GFRER material's surface as far as phosphoric acid modifies dentin. The dentin image showed a surface free from a smear layer and an unobstructed dentinal tubule, which is ideal for bonding agent application [6,35]. Considering the dentin-analogue material, the 10% hydrofluoric acid surface treatment caused greater removal of the epoxy resin matrix, exposing the glass fibers and causing their degradation, when compared

with the other surface treatments and with the control group (Figure 3). However, this greater exposure of glass fibers does not seem to have influenced the bond strength, since their values were statistically similar to the control group. Merlo et al. [13] evaluated the effect of different etching times of 10% hydrofluoric acid on the surface of a glass fiber-reinforced epoxy resin material, concluding that a shorter etching time (15 seconds) is sufficient to expose the glass fibers due to the selective removal of the resin matrix;

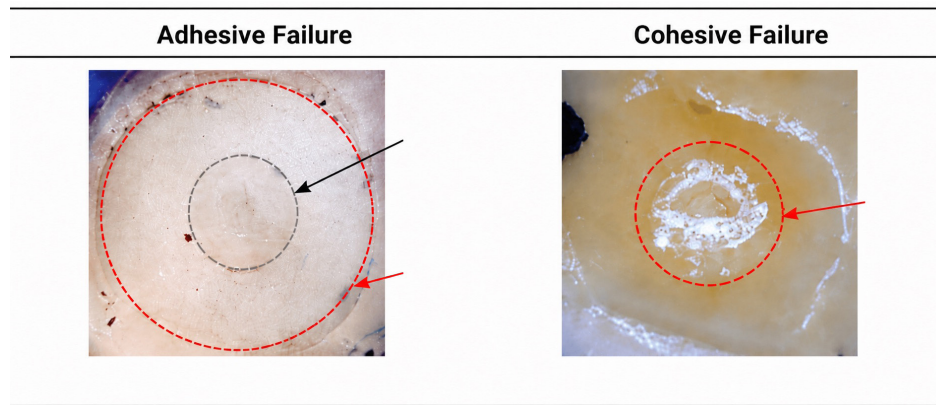


Figure 2 - Representative images of failure modes observed under a stereomicroscope at 30x magnification. In the adhesive failure (left), the red arrow indicates the outer boundary of the epoxy resin surface that was in contact with the starch matrix, whereas the black arrow indicates the surface that was in contact with the resin cement. The area within the inner dashed circle is free of resin cement residues. In the cohesive failure (right), resin cement residues are visible within the red circle.

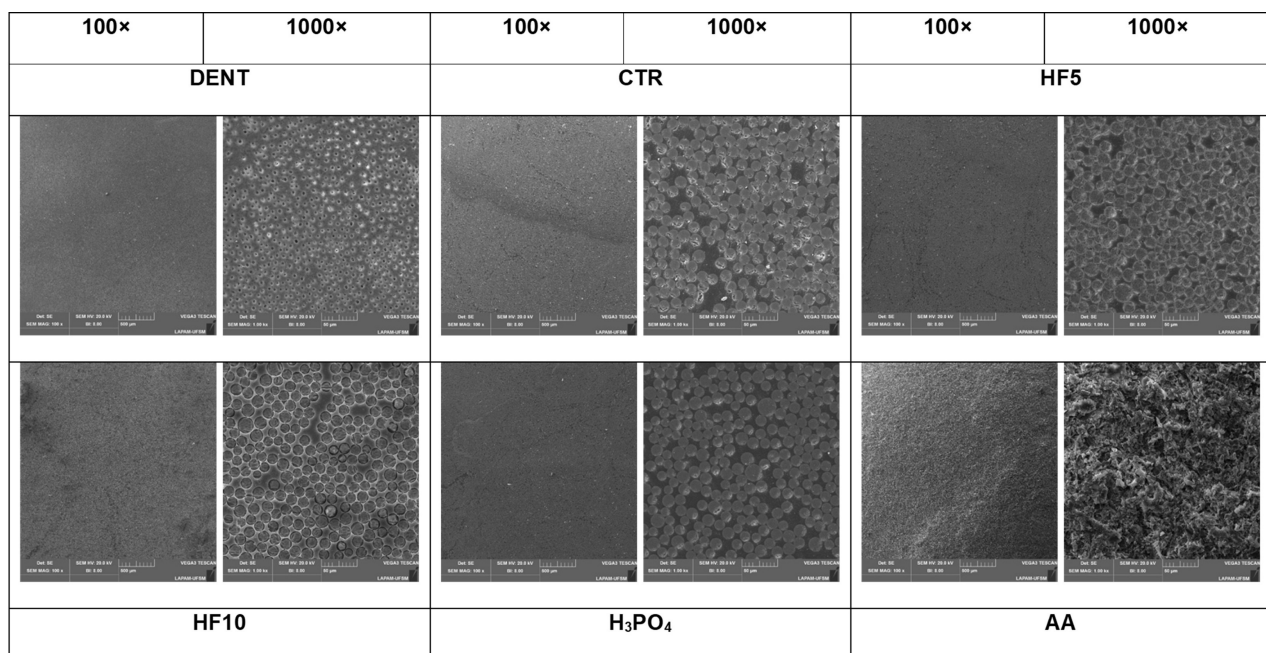


Figure 3 - Representative scanning electron microscopy (SEM) images of surface topography at 100x and 1000x magnification: treated dentin surface (DENT) and dentin-analogue material with respective surface treatments: silane application only (CTR), 5% hydrofluoric acid etching (HF5), 10% hydrofluoric acid etching (HF10), 37% phosphoric acid etching (H₃PO₄), and air abrasion with 110-μm aluminum oxide particles (AA).

furthermore, conditioning for longer (60 seconds) resulted in peripheral overtreatment of the fibers, compromising their structure and reliable adhesion between the substrate and the restorative material [13]. The 5% hydrofluoric acid etching showed less capacity for surface degradation when compared to 10% hydrofluoric acid etching (Figure 3), since it has a lower concentration and was used for a shorter time, which corroborates that the degradation of the glass fibers worsens the adhesive performance of the GFRER.

According to the literature [2,36], phosphoric acid has been used as a cleaning agent for glass fiber-reinforced epoxy resin because it has no relevant corrosive action on this material [37,38].

In the present study, the SEM images suggest that H₃PO₄ seems to have only cleaned the surface when compared with the control group, without the marked fiber degradation observed after hydrofluoric acid etching, especially in the HF10 group (Figure 3). Therefore, the relevance of H₃PO₄ should not be interpreted as an adhesive-enhancing effect, since this treatment did not increase μ SBS compared with the other conditioning protocols. Instead, H₃PO₄ produced surface roughness and contact angle values statistically similar to those observed for dentin (Table III and Figure 1), indicating that this protocol may be useful when the aim is to maintain surface characteristics closer to the

reference substrate. In contrast, HF5 resulted in higher μ SBS than CTR and HF10, but it did not reproduce the surface-related outcomes of dentin, particularly regarding contact angle (Figure 1).

The positive results found in the CTR group may be associated with the chemical interaction between the epoxy resin matrix and the glass fibers exposed on the GFRER surface (Figure 3) [39]. Furthermore, silane is known to increase surface wettability, promoting chemical interaction between the substrate surface and the resin cement [39,40].

The representative images of the AA group show aluminum oxide particle deposition on the surface of the material, providing a rougher and more heterogeneous surface (Table III and Figure 3), and presenting the highest surface roughness values (Table III). However, this surface treatment was not suitable for the GFRER material, as this group presented the highest number of pre-test and cohesive failures (Table IV). This can be explained by the impact force of the aluminum oxide particles during air-abrasion, causing a deleterious effect on the integrity of the glass fibers and making the adhesive interface more susceptible to the effects of aging [41,42]. This high occurrence of pre-test and cohesive failures indicates that air abrasion may have compromised the stability of the adhesive interface, which should be considered when interpreting the μ SBS results of the AA group.

When μ SBS was considered alone, all GFRER groups showed values statistically similar to human dentin, regardless of the surface-conditioning protocol. However, when surface-related outcomes were also considered, only CTR and H_3PO_4 presented roughness and contact angle values statistically similar to dentin. These findings suggest that CTR and H_3PO_4 were the most suitable surface treatments for GFRER when used as a dentin substitute in laboratory studies, since they provided surface characteristics more comparable to human dentin.

As limitations of the present study, it can depict the comparison of the adhesive behavior between human dentin and only one commercial GFRER material, and the use of only one resin cement. It is not possible to mimic all the complexities found in an oral environment in a single *in vitro* study, such as constant changes in pH and temperature, eccentric movements, or parafunctions. However, these studies are

important to help predict the performance of different long-term materials [32,43], so the extrapolation of the data obtained must be done with caution.

Ultimately, the use of a substrate that can replace human dentin *in vitro* studies is essential for execution and viability from the methodological viewpoint and the obtained results, without compromising its quality [10,29]. Based on this, future studies should seek greater characterization of the material regarding different testing methodologies and evaluate the mechanical performance of the material, which may cover different commercial brands and influence from different bonding systems.

CONCLUSION

GFRER produced μ SBS values comparable to those of human dentin regardless of the surface-conditioning protocol used. However, when surface-related outcomes were also taken into account, only the CTR and H_3PO_4 groups showed not only comparable microshear bond strength values but also roughness and contact angle values statistically similar to those of dentin. Therefore, these protocols may be considered more suitable when the aim is to preserve GFRER surface characteristics closer to those of the reference dentin substrate. In contrast, air abrasion with 110 μ m aluminum oxide particles is not recommended as a surface-conditioning protocol for GFRER in bond strength tests because of the high frequency of pre-test and cohesive failures.

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Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Author's Contributions

FDN: Conceptualization, Data Curation, Formal Analysis, Investigation, Methodology, Software, Validation, Visualization, Writing – Original Draft Preparation, Writing – Review & Editing. **LSR:** Data Curation, Formal Analysis, Investigation, Methodology, Software, Validation, Visualization, Writing – Original Draft Preparation, Writing – Review & Editing. **GCA:** Writing – Original Draft Preparation, Writing – Review & Editing. **GKRP:** Investigation, Methodology, Project Administration, Resources, Validation, Writing – Review & Editing. **LFV:** Conceptualization, Investigation, Methodology, Project Administration, Resources, Validation, Visualization, Writing – Review & Editing. **MPR:** Conceptualization, Formal Analysis, Funding Acquisition, Investigation, Methodology, Project Administration, Resources, Validation, Visualization, Writing – Review & Editing.

Conflict of Interest

The authors declare no conflict of interest.

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Regulatory Statement

This study was conducted in accordance with the guidelines of the Research Ethics Committee of the Federal University of Santa Maria. The approval code for this study is: CAAE 44655321.1.00005346.

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