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In vitro evaluation of the shear bond strength between fiber posts and methacrylate or silorane based composite resins

Pedro José Andrade da Silva^{1*}, Roberta Tarkany Basting Hoffing¹, Flávia L. B. do Amaral¹, Cecília P. Turssi¹, Carlos Eduardo Sabrosa Borges da Silva² and Fabiana Mantovani Gomes França¹

*Correspondence:

pjora@ig.com.br

¹ São Leopoldo Mandic Institute and Dental Research Center, Rua José Rocha Junqueira, 13, Ponte Preta, Campinas, SP 13045-755, Brazil

Full list of author information is available at the end of the article

Abstract:

Fiber posts (FP) are commonly used for extensive coronal reconstructions, often being associated with composite resins (CR) in order to minimize the space between the post and the intraradicular dentin. This study evaluated the shear bond strength of three different CR to a FP surface, with and without FP pretreatment with adhesive. Sixty #3 FP (Exacto Translúcido) were divided into six experimental groups ($n = 10$), according to the surface treatment used: G1—Filtek™ Z250; G2—Filtek™ P90; G3—Filtek™ bulk fill restorative; G4—Scotchbond™ Universal adhesive + Filtek™ Z250; G5—Silorane adhesive + Filtek™ P90 and G6—Scotchbond™ Universal adhesive + Filtek™ Bulkfill restorative. Two 2.0 mm thick specimens were obtained from each fiber post unit and submitted to a push-out test in a universal testing machine with a 5 mm/min cross-head speed and 5 Kilo-newton load cell. Mean and standard deviation values for bond strength (MPa) were: G1: 5.54 ± 1.78 ; G2: 1.69 ± 1.02 ; G3: 5.31 ± 1.58 ; G4: 7.39 ± 2.05 ; G5: 6.07 ± 1.88 ; G6: 5.50 ± 3.03 . The results were analyzed through two-way analysis of variance (ANOVA) and statistical significant differences were determined by Tukey test ($p = 0.05$). When no adhesive was used, the bond strength was statistically significantly higher for G1 and G3, which were not statistically significantly different between them. Adhesive application showed statistically significantly higher bond strength only for G5. Without the FP pretreatment with adhesive, the bond strength was statistically significantly higher for the methacrylate-based resins and the FP pretreatment with adhesive increased the bond strength only for the silorane-based resin and changed the prevalence of failure mode type to cohesive in all composite resin study groups. The use of silorane-based composite resins to customize posts require the use of adhesive as FP surface pretreatment. The use of adhesive as pretreatment of simulated customized FP, regardless of the CR used, appear to improve the mechanical behavior of the FP-CR unit.

Keywords: Post and core technique, Composite resins, Silorane resins, Reinforced fiber post, Esthetic core

Background

FP are used to aid the retention of restorative materials in endodontically treated teeth with severe destruction [1, 2]. In some clinical situations, the space between the FP and the walls of the root canal may be too wide due to the root canal anatomy and/or treatment [1, 3, 4]. As the thickness of the composite resin cement directly influences the FP adaptation and consequently the bond strength [3, 4], thinner cement layers are desirable for better mechanical properties [5, 6]. In addition, the greater the space between FP and the root canal wall, the greater chance for catastrophic root fractures [7] due to eventual post loosening. There are two techniques proposed in the literature to minimize this space, the use of accessory fiber posts [8, 9] and customization of the fiber post with CR [10–12]. The purpose of both techniques is to reduce the effect of shrinkage stress associated with CR cements, obtaining a more stable adhesive interface [10–13].

CR can be used to fill in this space as well as the core build up material [1, 14–16]. The vast majority of methacrylate-based CR has polymerization shrinkage between 2 and 5 % [17], whereas silorane-based CR exhibits polymerization shrinkage below 1 % [18, 19], which would make the adaptation of the post to the root canal better.

New CR materials have been developed to be used in thicker increments either as an intermediate layer in composite resin restorations or as the final restorative material. A photoactive group was added to these to control the kinetic of the polymerization and also a polymerization modulator in the center of the dimethacrylate urethane monomer, increasing the monomer size compared to regular restorative CR. This modification reduces the polymerization shrinkage compared to the flowable and packable methacrylate-based CR [20, 21]. This CR is used to simplify the incremental technique and reduce clinical hours, with up to 4 mm increment thickness [22, 23]. The depth polymerization capability and lower polymerization shrinkage of this group of composite resins could aid in obtaining the most root-wall-fit personalization of the fiber post within the customization post technique, guarantying the thinner cement layer possible (Table 1).

Several studies have evaluated the shear bond strength between customized FP and dentin [24–27]. However the fracture patterns are usually about failures in adhesion between post and resin material, either composite or resin cement [3, 28]. In that matter, different surface treatments have been described, such as silanization and application of an adhesive system [3, 4, 28–32], to ensure the bond between the resin-based materials and FP. However, for the customized-post technique there are not enough studies to determine if the adhesive use in the preparation of FP surface is actually required [33, 34].

Therefore, the current study evaluated the bond strength of silorane-based, a bulkfill methacrylate-based and a conventional methacrylate-based CR to a fiber post, with and without the use of adhesive systems.

The null hypothesis was that there was no difference on the shear bond strength between FP and tested CR, regardless of adhesive application.

Methods

Specimen preparation

FP were divided into 06 (six) groups, cardinally numbered, from 1 to 6, with 10 (ten) posts each, according to the surface treatment and CR selected, as shown in Table 2. The sample size was based on literature [35, 36].

Table 1 Materials, manufacturer, composition and application

Material/manufacturer	Composition	Application
Filtek™ Z250, A2 Shade, 3 M/ESPE	75–85 % silanized ceramic; 1–10 % BISEMA6; 1–10 % UDMA; 1–10 % BIS-GMA; <5 % TEGDMA; <5 % aluminum oxide; <0.5 % benzotriazole; <0.2 % EDMAB	2.5 mm maximum increment, photo-activated for 80 s
Filtek™ P90, A2 Shade, 3 M/ESPE	5–15 % 3,4-epoxi-ciclohexil-ethyl-cyclopolimethyl-siloxane; 5–15 % bis-3,4-epoxi-ciclohexil-ethyl-fenil-methyl-silane; 50–70 % silanized quartz; 10–20 % Yttrium fluoride; camphorquinone	2.5 mm maximum increment, photo-activated for 80 s
Filtek™ Bulk Fill Restorative, A2 Shade, 3 M/ESPE	60–70 % silanized ceramic; 10–20 % aromatic di methacrylate urethane; 1–10 % Ytterbium fluoride; 1–10 % UDMA; 1–10 % silanized silica; <5 % DDDMA; <5 % water; <5 % silanized zirconium; <1 % modified methacrylate monomer; <0.5 % EDMAB; <0.5 % benzotriazole	2.5 mm maximum increment, photo-activated for 80 s
Scotchbond™ Universal, 3 M/ESPE	15–25 % BIS-GMA; 15–25 % HEMA; 5–15 % decamethylene di methacrylate; 10–15 % ethanol; 10–15 % water; 5–15 % silanized silica; 1–10 % propenoic acid, 2-methyl, decanediole and phosphoric anidryde byproducts; 1–5 % itaconic and acrylic co-polymer acids; <2 % dimethyl amine benzoate; <2 % camphorquinone; <2 % dimethyl amine ethyl methacrylate; <2 % methyl ethyl ketone	Application of a thin layer for 15 s, Air drying for 5 s, Light-curing for 20 s
Filtek™ P90 SA, 3 M/ESPE	Primer: 15–25 % HEMA; 15–25 % BIS-GMA; 10–15 % water; 10–15 % ethanol; 5–15 % phosphoric acid methacryl-oxi-hexil-esters; 8–12 % silanized silica; 5–10 % 1,6-hexanediol di methacrylate; <5 % itaconic and acrylic acid co-polymer; <5 % (dimethyl amine) ethyl methacrylate; <3 % DL-camphorquinone; <3 % phosphine oxide; Bond: 70–80 % replaced di methacrylate; 5–10 % silanized silica; 5–10 % TEGDMA; <5 % phosphoric acid methacryl-oxi-hexil-esters; <3 % DL-camphorquinone; 1,6 hexanediol di methacrylate	Homogenous bond application, Light-curing for 20 s
Exacto Translúcido #03, Angelus®	80 % glass fiber, 20 % epoxy resin	Cleansing with ethanol 70 % for 15 s
Silano Angelus®	X-R-Si (OR) 3n, X: organic-functional group bonds to composite resin, R: methylene group, OR: hydrolysable group bonds with porcelains, composite resins and glass fiber posts, Si: Silicium. n: 0–3	Thin layer application 60 s waiting time Smooth air drying for 5 s

BISEMA6 bisphenol a polyethylene glycol diether dimethacrylate; *UDMA* diurethane dimethacrylate; *BISGMA* bisphenol a diglycidyl ether dimethacrylate; *TEGDMA* triethylene glycol dimethacrylate; *EDMAB* ethyl 4-dimethyl aminobenzoate; *HEMA* 2-hydroxyethyl methacrylate

Following manufacturer's instructions all FP were cleansed with a gauge embedded in ethanol 70 % (Ciclofarma) for 10 s and treated with silane (Angelus®). Following manufacturer recommendations, all FP were previously treated with silane (Angelus®). In groups G4, G5 and G6, the FP was treated with the correspondent adhesive (Table 2), with a thin layer application, as recommended by the manufacturer, followed by air drying for 05 s in order to evaporate the solvent and homogenize the adhesive thickness and light curing with a light emitting diode (LED) (Elipar™ Paradigm™; 3 M/ESPE, Seefeld, Germany) for 20 s.

Once treated, the FP was individually placed into a transparent plastic matrix (Fig. 1) specially designed to keep the posts in an up-right position, keeping them centralized and to standardize the CR filling procedure. The matrix was then, incrementally filled,

Table 2 Experimental groups

Group	N	Surface treatment
1	10	Filtek™ Z250 filling
2	10	Filtek™ P90 filling
3	10	Filtek™ Bulk Fill Restorative filling
4	10	Scotchbond™ Universal adhesive application; Filtek Z250 filling
5	10	Filtek™ P90 silorane adhesive application; Filtek™ P90 filling
6	10	Scotchbond™ Universal adhesive application; Filtek™ bulk fill restorative filling

leaving approximately 6.0 mm of the posts extremity free at the top and 1.0 mm at the bottom end and 2.0 mm of composite resin around the post. Each increment was inserted around the FP (Fig. 2) and condensed with a Teflon instrument (Fig. 3) and light cured 04 (four) times for 20 s each, with an 1200 mW/cm² irradiance LED (Elipar™ Paradigm™; 3 M/ESPE). All CR used were of A2 shade.

The posts involved with CR were removed from the plastic matrix and stored in distilled water for 24 h in an oven (ECB 3; Odontobrás, Ribeirão Preto, SP, Brazil) at 37 °C. The FP-CR samples were then perpendicularly glued to an acrylic plate with wax (Asfer,

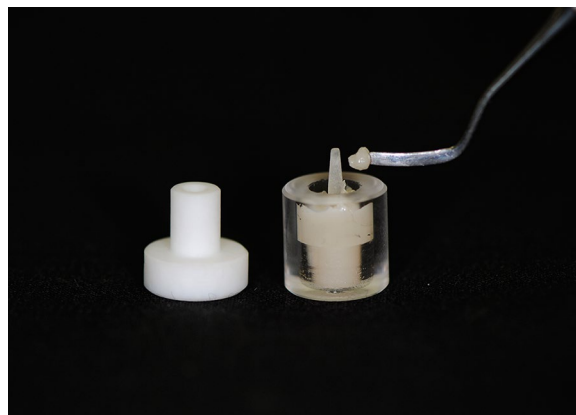
**Fig. 1** FP-CR unit builder matrix**Fig. 2** CR increment filling



Fig. 3 CR condensing

São Caetano do Sul, SP, Brazil) and positioned in a precision cutter machine (IsoMet™ 1000; Buehler, Bluff, IL, USA). With a wafering diamond blade (11-4244 15HC; Buehler), three parallel cuts were made perpendicularly to the posts long axis (Fig. 4), obtaining two 2.0 mm-thick testing specimens from each post (Fig. 5), resulting in 20 test samples for each experimental group. For each FP-CR sample, an average value for the bond strength was calculated from both test samples derived from it.

Push-out test

A stainless steel base was positioned on a universal testing machine (EZ test EZ-LX; Shimadzu Corporation, Suzhou, Sū, China) to perform the push-out test. This base had a 12.8 mm diameter-wide table designed to receive a second stainless steel base that was prepared to receive the test samples. This second base had a 2.5 mm diameter-wide orifice in the center and was fabricated to allow the post to drop into it during testing. All samples were loaded with the bottom part of the post facing up.

A metallic arm, with an 1 mm diameter active tip attached to the load cell (5 Kilonewton), was aligned to the first metal base so its active tip would only touch the post

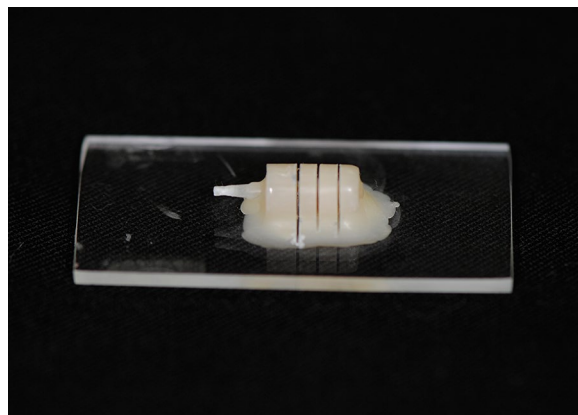
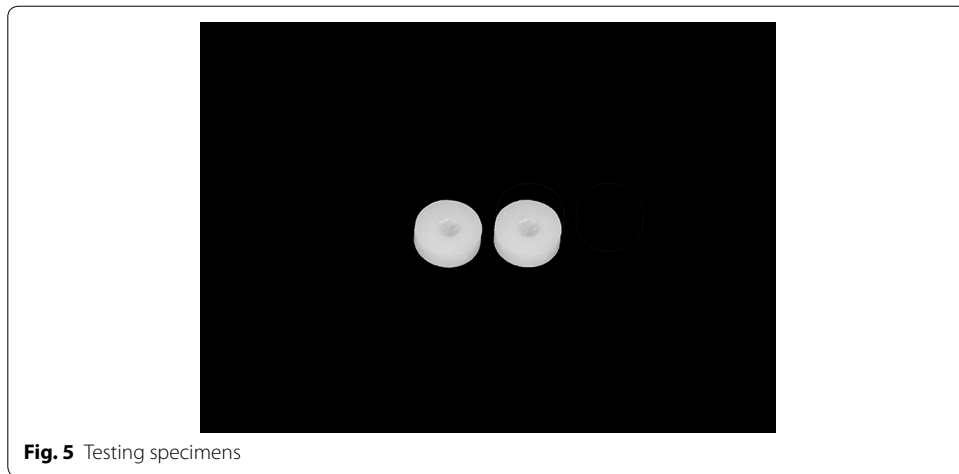


Fig. 4 FP-CR unit cuts



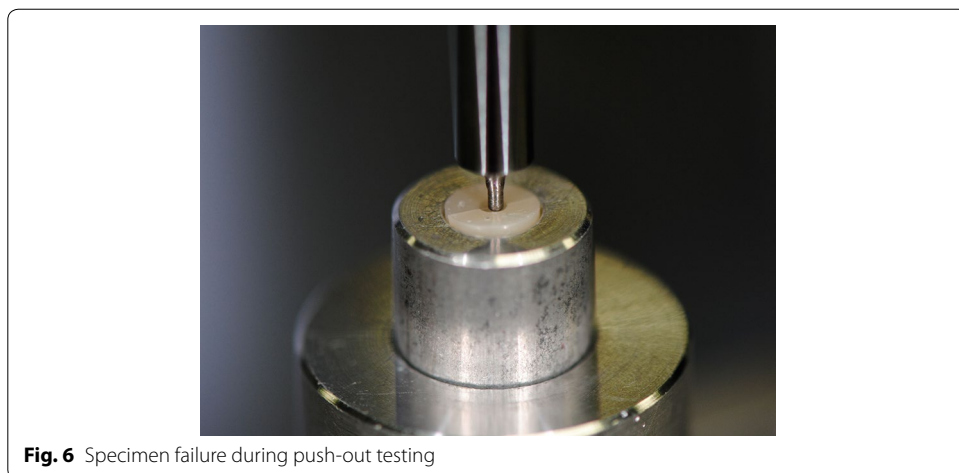
during testing. The push-out test was performed with a crosshead speed of 0.5 mm/min until automatic detection of failure (Fig. 6).

The shear bond strength values in Mega Pascal (MPa) were obtained by dividing the maximum force needed to dislodge the post, in Newton (N), by the adhesive interface area. The area was calculated through the diameters of the post and the thickness of the specimen. Therefore, the area = $\left\{ [\pi (R + r)] \cdot [h^2 (R - r)^2]^{0.5} \right\}$, where π is constant and equals 3.1416; R represents the radius of the top part of the post, r is the radius of the bottom part of the post and h represents the height of the post section, in mm.

The values were recorded and submitted to statistical analysis.

Failure mode

After the push-out test, all specimens were observed under a stereoscopic loupe (Eikonai, São Paulo, SP, Brazil) with 40 \times magnification, to determine the failure mode, divided as: (1) adhesive failure between post and resin; (2) mixed failure and (3) cohesive failure within post and resin.



Statistical analysis

The results gathered for the shear bond strength are expressed by average and standard deviation and were submitted to two-way ANOVA testing and for average multiple comparisons Tukey post hoc test was employed. Significance level was set at 5 %.

Results

Two-way ANOVA, with test power of 57 %, showed there was interaction between the study factors (composite resin and adhesive application).

Tukey test revealed that in the absence of adhesive (groups G1, G2 and G3), the bond strength of the methacrylate-based CR (Filtek™ Z250 and Filtek™ Bulkfill restorative) to the FP was statistically significantly higher, but it did not differ among each other (Table 3).

The bond strength of the silorane-based CR (Filtek™ P90) specimens was not statistically significantly different from the others when the adhesive was applied. With such treatment, the shear bond strength for Filtek™ P90 increased significantly while for the other CR the adhesive application did not allow for better performance (Table 3).

For the failure mode, it was noticed that in G2 (Filtek™ P90) there was predominantly adhesive failures (55 %). When the adhesive for the same CR (G5—silorane adhesive + Filtek™ P90) was applied, the failures were cohesive in 90 % of the specimens. The same failure mode was most prevalent in all methacrylate-based CR groups when the adhesive was used (G4, 85 % and G6, 70 %). In G1 (Filtek™ Z250), mixed failures were predominant (60 %), while in G3 (Filtek™ Bulkfill restorative), there were adhesive (45 %) and mixed failures (40 %) in similar proportions, as noticeable in Graphic 1 (Fig. 7).

Table 3 Average and standard deviation values for shear bond strength of FP, according to technique

Composite Resin	Adhesive	
	Absent	Present
Methacrylate-based resin (Filtek™ Z250)	5.54 (1.78) Aa	7.39 (2.05) Aa
Silorane-based resin (Filtek™ P90)	1.69 (1.02) Bb	6.07 (1.88) Aa
Methacrylate bulk fill resin (Filtek™ Bulk Fill)	5.31 (1.58) Aa	5.50 (3.03) Aa

Statistical difference is indicated through different capital letters in the row and different small letters in the columns

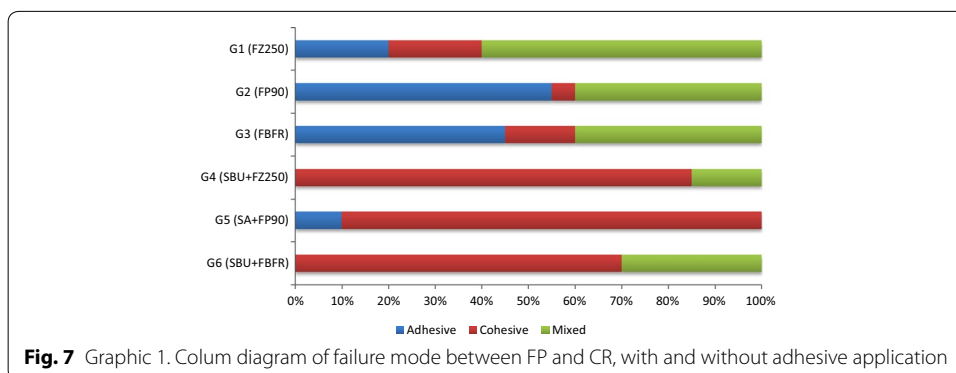


Fig. 7 Graphic 1. Colum diagram of failure mode between FP and CR, with and without adhesive application

Discussion

The results of this study rejected the null hypothesis, since the type of CR used and the FP pretreatment with adhesive affected the bond strength between the FP and the CR.

In this study the shear bond strength was tested through a push-out test, seeking similarity to the clinical situation, as the push-out test evaluate a failure that occurs parallel to the adhesive interface [30, 37, 38]. The 2.0 mm test sample thickness was selected in order to minimize the influence of the CR fracture resistance in the bond strength of the FP-CR sample tested [39–41].

The use of silane as surface pre-treatment for every group was based on the manufacturer's recommendation and in some literature reports that showed increase in the bond strength of silanized FP [1, 42, 43]. To simplify the methodology of this study, the studies of Rosatto et al. [44] and Novais [45] were considered regarding the use of pre-hydrolyzed silane and the absence of heat drying, respectively.

Based on recent studies that showed that acid etching does not increase the bond strength [46], but could damage the FP epoxy-matrix [47], this study did not use any etching as FP surface pre-treatment.

A previous study mapped the formation of multiple gaps through the FP-CR interface when no surface treatment was used [2]. On the other hand, Lastumaki [48] showed the positive influence of adhesive usage as surface treatment on the bond strength of FP. In accordance, each CR correspondent adhesive was used.

The present study showed that the type of CR influences the bond strength only when the adhesives were not used as surface treatment, having methacrylate-based resins (FZ250 and FBFR) achieved higher bond strength values ($p < 0.05$) when compared to the silorane-based resin (FP90). This could be related to the difference in polymerization shrinkage between silorane-based resins (lower than 1 %) and the other CR (2 and 5 %) [15]. The volumetric reduction that occurs because of cross bonding between the molecules in the CR organic matrix would be reduced in silorane-based CR due to the opening of a cationic ring previously to the formations of such chemical bonds, during the material polymerization [18, 19, 49, 50]. It is known that silorane-based CR has significantly lower polymerization shrinkage, although showcasing similar mechanical properties to the methacrylate-based composites [51]. The volumetric decrease (shrinkage) of methacrylate-based composite materials could be responsible for the increase in the push-out bond strength of those materials to the FP through the tightening of the contact between post and CR.

The increase of bond strength when adhesives were applied in this study, only showed statistic significant difference ($p < 0.05$) between the silorane-based CR groups, which could be explained by the fact that these CR seem to have lower adhesion power to substrate than methacrylate-based CR [52]. In addition, the silorane adhesive system has two phases: one self-etching hydrophilic primer and one hydrophobic resinous adhesive [53].

Despite the shear bond strength showing no statistic difference among the methacrylate-based CR groups, the change in failure mode was noticeable. For all CR groups, when adhesive was not applied, adhesive and mixed failures were predominant for the low-shrinkage CR G2 (Filtek™ P90) + G3 (Filtek™ Bulkfill Restorative) and G1 (Filtek™ Z250) group, respectively, supporting once more the theory that the polymerization

shrinkage could influence the bond strength due to the increase in the contact between the FP and CR surfaces, which was suggested by Goracci et al. [54].

Furthermore, in all groups that the specific adhesive was applied (G4, G5 and G6), cohesive failure was predominant, which could be supported by the reduction of gaps through the adhesive interface, responsible for initiating mechanical failures and increasing the number of stress points inside the FP-CR sample [32].

The clinical relevance of these findings resides on the fact that adhesive application as surface treatment for personalizing FP could allow for a more safely indication of the customized-post technique, since the failure patterns in this study showed that the FP-CR sample showcased a “one-body” mechanical behavior when the specific adhesive was used.

Further studies are necessary in order to evaluate the *in vitro* influence of aging in the adhesive interface resistance and whether the positive performance of the adhesive-treated fiber post, especially regarding the failure mode and mechanical behavior, is similar to the performance seen in this *in vitro* study.

Conclusions

Within the limitations of this study, it can be concluded that:

- The use of silorane-based composite resins to customize posts require the use of adhesive as FP surface pretreatment;
- The use of adhesive as pretreatment of simulated customized FP, regardless of the CR used, appear to improve the mechanical behavior of the FP-CR unit;
- The null-hypothesis was discarded, as:
 - the bond strength was statistically higher for the methacrylate-based resins without the FP pretreatment with adhesive;
 - the FP pretreatment with adhesive increased the bond strength only for the silorane-based resin and changed the prevalence of failure mode type to cohesive in all composite resin study groups.

Abbreviations

FP: glass fiber post(s); CR: composite resin(s); LED: light emitting diode; MPa: mega pascal; N: Newton; ANOVA: analysis of variance.

Authors' contributions

PAS carried out all *in vitro* assays and drafted the manuscript. RTB helped to draft and in revising the manuscript. FA participated in the design of the study. CT performed the statistical analysis. CES conceived of the study and participated in its design. FMF participated in the coordination and helped to draft the manuscript. All authors read and approved the final manuscript.

Author details

¹ São Leopoldo Mandic Institute and Dental Research Center, Rua José Rocha Junqueira, 13, Ponte Preta, Campinas, SP 13045-755, Brazil. ² University of the State of Rio de Janeiro, Boulevard 28 de Setembro, 157, Vila Isabel, Rio de Janeiro, RJ 20550-030, Brazil.

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Competing interests

The authors declare that they have no competing interests.

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