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Effects of Different Pretreatments on the Bond Strength of a Dual-Cure Resin Core Material to Various Fiber-Reinforced Composite Posts

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Abstract - *The aim of this study was to evaluate the effects of different pretreatments on the bond strength of a dual-cure resin core to 3 types of fiber posts. Bond strength was measured using a push-out design. One-sided t-Test of Hypothesis with unknown variance was performed (p-values < 5%). Sandblasting abrasion with 50 µ alumina particles at a specific distance, pressure and time was the only surface treatment in DT Light Post® and Transluma Post™ that increased the bond strength to dual cure resin composite cores. FRC Postec Plus® post did not shown an increase in bond strength in any group.*

KEY WORDS: post, fiber post, surface treatment, push-out bond strength, composite resin core material

INTRODUCTION

Twenty years ago, fiber-reinforced composite (FRC) root canal posts were introduced as an alternative to more conventional materials[1,2], and their use increased in the last decade. The major advantage of FRC posts is their similar elastic modulus to dentin, producing a stress field similar to that of natural teeth, whereas metal posts exhibit high stress concentrations at the post-dentin interface[3-7].

Clinical studies have also demonstrated high success rates without the occurrence of root fractures[8]. Otherwise, laboratory and clinical research has found that debonding is a common cause of failure encountered between fiber posts and resin cores or root dentin and resin cement interfaces as a result of inadequate bond strength between their interfaces[8-10]. The retention between the core and prefabricated post materials is critical to post and core longevity[11]; in order to improve the bond strength between the post and the resin core, many surface pretreatment procedures for posts have been investigated, including silanization, sandblasting with 50µm Al_2O_3, hydrofluoric acid and hydrogen peroxide immersion.

Recently published data provide evidence that silane coating of the post surface increases post core bond strength[12,13]. Some uncertainty still remains with respect to the mechanisms that are actually responsible for this enhancing effect[14]. Previous studies reported that a mild form of airborne-particle abrasion (50µm alumina particles, 2.5 bars, 5 seconds, 30 mm) resulted in a statistically significant increase in retention of glass-fiber posts without producing visible changes in the post form that occurred with a stronger regimen (50µm alumina particles, 2.5 bars, 10 seconds, 15mm) [15,16]. Etching with hydrofluoric acid is intended to create a roughening of the surface that allows for micromechanical interlocking of resin to the restoration. Hydrofluoric acid, in combination with a silane-coupling agent, is often used to enhance the bond strength between composite resin and feldspathic ceramics. Because silica and quartz fibers are comparable in chemical structure with ceramic materials, hydrofluoric acid was recently proposed for etching fiber posts[17,18].

Pre-treatment with hydrogen peroxide was found to be effective for enhancing the retention between epoxy resin-based and methacrylate-based resin fiber post systems and core materials[17,19,20]. Hydrogen peroxide is able to dissolve the epoxy resin matrix, breaking epoxy resin bonds and exposing the surface of fibers to silanization[19,20]. This method was found to be effective for enhancing the retention between epoxy resin-based and methacrylate-based resin fiber post systems and core materials[17,20]. However, the relationship between the bond strength of resin core materials and surface roughness of fiber posts is lacking, as is the combination of sandblasting and additional pretreatments with this system.

Several materials have been used for core build-ups that differ in their mechanical properties, viscosities and setting reactions[21]. In a microscopic study[22], flowable composites achieved structural homogeneity and continuity with the post surface, which was superior to hybrid composites. However, the latter materials are expected to provide higher mechanical properties than the lightly filled flowable composites. Also, several composite resins specifically formulated for abutment build-up are currently available in the market.

Therefore, the aim of the present study was to investigate the effects of different pretreatments on the bond strength of dual-cure resin core materials to various fiber-reinforced composite posts. The null hypothesis was that there were no differences on interfacial push-out bond strength between fiber posts subjected to different surface treatments and dual-cured core build-up composite.

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MATERIALS AND METHOD

Ninety FRC posts were divided into three groups: Thirty glass fiber posts of ISO 120 Transluma Post™ (Lot 0800007844 and 0800005159, Bisco™), 30 quartz fiber posts of n°3 DT Light Post® (Lot 0800007828 and 0700006082, Bisco), and 30 glass fiber posts of n°3 FRC Postec Plus® (Lot L06687 and L28072, Ivoclar-Vivadent®). Each group was subdivided into six subgroups (*n=5*) according to different surface treatments.

Subgroup specimen preparation

Subgroup 0 (CONTROL): Each post was cleaned with 70 % alcohol for 60 s and then dried with oil-free air for 30s. No surface treatment.

Subgroup 1: Each post was individually immersed in a plastic container with 10% hydrofluoric acid (HA) porcelain etchant (Lot 017055A, Dentsply) for 15 seconds. After soaking in hydrofluoric acid, each post was removed, rinsed with air spray with distilled and deionized water for 30 seconds and dried with oil-free air for 30s. The samples were stored in hermetically sealed flasks containing silica gel for humidity control. A single dose of hydrofluoric acid was applied to each post, avoiding the reuse of the acid gel.

Subgroup 2: Each post was individually sandblasted (SB) using an intraoral sandblasting device (Microetcher™ II, Danville) for 5 seconds, at a distance of 30 mm and with a pressure of 2.5 bars. An apparatus, developed in a previous study[23], standardized the distance between the micro-etching tip of the Microetcher™ II and the surface of the FRC posts. After sandblasting, each post was rinsed with air spray and distilled and deionized water for 30 seconds and dried with oil-free air for 30s. The samples were stored in hermetically sealed flasks containing silica gel for humidity control.

Subgroup 3: Each post was individually immersed in a plastic container with a 24% hydrogen peroxide (HP) water solution obtained from diluted hydrogen peroxide P.A. 30% (Lot 0801838, Vetec Química Fina LTDA) for 10 min at room temperature. After the application of hydrogen peroxide, each post was rinsed with air spray using distilled and deionized water for 30 seconds and dried with oil-free air for 30s. The samples were stored in hermetically sealed flasks containing silica gel for humidity control. A single dose of 24% hydrogen peroxide water solution was applied to each post, avoiding the reuse of the solution.

Subgroup 4 (SB-HA): was submitted to sandblasting (as with Group 2), followed by immersion in 10% hydrofluoric acid porcelain etchant (as with Group 1).

Subgroup 5 (SB-HP): was submitted to sandblasting (as with Group 2), followed by immersion in 24% hydrogen peroxide (as with Group 3).

A silane-coupling agent (Monobond-S Lot K30207, Ivoclar Vivadent®) was applied in a single layer using a brush on the post surface, left to air dry for 60s at room temperature and then dried with a gentle stream of air in all subgroups. Subsequently, a single layer of the bonding agent AllBond 3® (Lot 0900009229 - Bisco™) was applied with a brush on the post surface, dried with a gentle stream of air to remove possible excesses and cured for 10s with a halogen light according to the manufacturer's recommendations (Optilux 500 – Curing Light; Demetron, Kerr) using an 8mm Curved Turbo Light Guide (8mm x 70mm, part number 21020). The light output was verified at greater than 800 mW/cm^2 throughout the study with a built-in curing radiometer and fiber posts were directly irradiated from the upper side of major diameter portion.

Composite core preparation

To make the core build-ups, a special apparatus to centralize the posts in relation to the composite build- ups was constructed. This device consisted of a cylindrical brass base and a system of five white polytetrafluoroethylene matrixes that fits on top of the base. A channel for post-placement was prepared in the center of the three cylindrical base of polytetrafluoroethylene matrixes to a depth, allowing 2 mm of the experimental posts to extend above the base into the other parts of the matrix. Each post system had a cylindrical specific matrix designed. Then, the upper part of the apparatus, two removable patterns with two standardized moulds, was placed on the center of the base device. The mould formed core build-ups with a diameter of 5mm. This selected diameter correspond to the maximum mesiodistal widths of natural anterior maxillary and mandibular teeth measured at the level of the CEJ after crown preparation[24,25]. Experimental specimens were made by placing a post in the base and filling the matrix with composite, resulting in a total core height of 2 mm[26]. The moulds in the polytetrafluoroethylene pattern ensured standardized shapes of the composite core build-ups and equal distribution of the core material around the posts. The experimental design is shown in Figure 1.

The composite cores were built up using a dual-cured core build-up composite (Biscore™ shade Natural Ref. A-1730NB Lot 0900007845 - Bisco™). The base and catalyst pastes of the composite were mixed in a 1:1 ratio and applied in bulk from a Centrix syringe (Centrix System Lot 09070720 and 07040458 – DFL Industria e Comercio S.A.). Gross excess composite was removed from the top of the polytetrafluoroethylene matrixes and cured for 20 s according to the manufacturer's recommendation using a halogen light-curing unit (Optilux 500 – Curing Light; Demetron, Kerr). The light output was verified at greater than 600 mW/cm^2 throughout the study with a curing radiometer (Demetron/Kerr, Danbury, CT). The tip of the polymerization unit was placed in contact with the composite that was directly irradiated from the upper side of the mould. After removing the cured post-core build-up cylinders from the mould of the patterns, a further irradiation of 20s was carried out from all sides in order to ensure complete polymerization of the dual curing composite core build-up material. The same investigator performed all of the procedures. The experimental design of composite core is shown in Figure 2.

The upper surface of each specimen (approximately 2.1-mm-thick) were polished with 600-grit silicon carbide paper in a polishing machine under water cooling (WOCO SF 20). The thickness of the slices was continuously checked using a digital caliper (Model 500-144B, Mitutoyo Sul Americana Ltda) until they were reduced to 2.0 ± 0.05mm in all diameters.

Figure 1. *Cylindrical brass base and the white polytetrafluoroethylene system.*

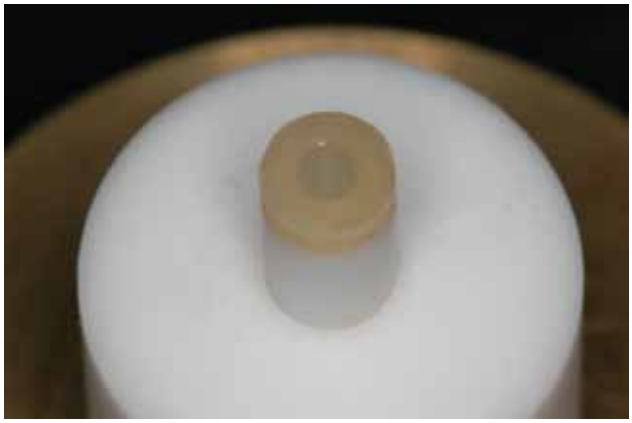

Figure 2. *The moulds in the polytetrafluoroethylene pattern formed core build-ups with a diameter of 5 mm.*

Table 1. *Materials used in the study*

Material	*Composition*	*Manufacturer*
FRC Postec Plus®	Dimethacrylates approx. 21 % Ytterbium fluoride approx. 9 % Glass fibres approx. 70 % Catalysts and stabilizers < 0.5 %	Ivoclar-Vivadent AG, Schaan, Liechtenstein
Transluma Post™	60% glass fibers 40% epoxy resin	Bisco™ Incorporated, Illinois, USA
DT Light Post®	60% quartz fibers 40% epoxy resin	Bisco™ Incorporated, Illinois, USA
Hydrofluoric acid porcelain etcher	10% Hydrofluoric acid, water, thickening and stain	Dentsply Indústria e Comercio LTDA, RJ, BRA
Microetcher™ II	Aluminum oxide (50µ) sandblaster	Danville Engineering, CA, USA
24% Hydrogen peroxide water solution	Diluted hydrogen peroxide P.A. 30%	Vetec Química Fina LTDA, RJ, BRA
Monobond S	1% wt 3-methacryloxypropyltrimethoxysilane (3-MPS), ethanol/water-based solvent	Ivoclar-Vivadent AG, Schaan, Liechtenstein
All Bond 3®	Ethanol (Part A) > 90% NTG-GMA Salt (Part A) > 1% Bis-GMA (Part B) > 40% HEMA (Part B) > 20% BPDM (Part B) > 5%	Bisco™ Incorporated, Illinois, USA
Biscore™ shade Natural Ref. A-1730NB	BASE:. Bis-GMA > 15% Glass filler < 65% Urethane Dimethacrylate < 9% Fused silica < 26% CATALYST:.Bis-GMA < 28% Triethyleneglycol Dimethacrylate <11% Glass Filler < 55% Fused silica < 21%	Bisco™ Incorporated, Illinois, USA

Table 2. *Subgroups and their respective surface treatments*

Subgroups	*Surface treatments*
Subgroup 0 (Control)	Cleaned with 70 % alcohol for 60 s and dried with oil-free air for 30s. No surface treatment.
Subgroup 1 (HA)	10% Hydrofluoric acid porcelain etchant, 15s
Subgroup 2 (SB)	Sandblasting Al_2O_3 50µ, d* = 30 mm, 5s
Subgroup 3 (HP)	24% Hydrogen peroxide, 10 min
Subgroup 4 (SB-HA)	Sandblasting Al_2O_3 50µ, d* = 30 mm, 5s + 10% Hydrofluoric acid porcelain etchant, 15s
Subgroup 5 (SB-HP)	Sandblasting Al_2O_3 50µ, d* = 30 mm, 5s + 24% Hydrogen peroxide, 10 min

*d = distance between micro-etching tip and the surface of the fiber post

The specimens were stored in distilled and deionized water for 24 hours at 37ºC in a humidor (Model Q-317M233 900W Quimis Aparelhos Cientificos Ltda) (100% relative humidity), before testing. The materials used in this study are presented in Table 1. Subgroup descriptions and their respective surface treatments are shown in Table 2.

Push-out test

After storage, each specimen was placed into a hole of a supporting table; as a result, the specimens were centered and a load was applied using a push-out pin (figure 3). Diameter sizes of the opening of the supporting table and of the push-out pin were 2.5mm and 1.2 mm, respectively, to enable the pushed-out post to pass through the hole of the supporting table. The diameter of the hole was larger than that of each post type. The posts were loaded vertically to their surface using the matching set until the post segments dislodged from the core build-up. The punch was aligned so that it only contacted the post upon loading. The interfacial push-out bond strength was measured in a universal testing machine (model Autograph AG-IS MO, Shimadzu do Brasil Comércio Ltda), with a load cell type SLBL-5kN at a crosshead speed of 0.5mm/min. The interfacial strength (MPa) was calculated as the quotient of the maximum force required to dislodge the post and the bonding area: $\sigma = F/\pi hd$ [where F was the load (in N), and h and d were the height and the diameter of the post segment, respectively (in mm)].

In order to point out which pretreatment significantly enhanced the bond strengths, after performing the push-out testing, each subgroup interfacial-strength mean was independently compared with the control group mean. Therefore, a one-sided t-Test of Hypothesis with unknown variance[27] was developed. The effect of the combined treatments was then tested. To do that, the SB-HA (subgroup 4) and SB-HP (subgroup 5) subgroups were submitted to the same statistical test, having the sandblasting (SB) treatment (subgroup 2) as the control group. Under this scheme, the t-test points out whether a sandblasting combined treatment enhances the bond strength obtained by a pure sandblasting treatment.

RESULTS

The subgroups data for each group are shown in Tables 3-5, where the subgroups are identified by their acronyms labels instead of by their numbers. For the sake of completeness, the failure loads (in N) and the associated interfacial strengths (in MPa) are presented, but the whole statistical analysis presented in this current work were applied on the interfacial strength data only.

Firstly, within the analyzed pretreatments, the sandblast treatment is necessary to significantly enhance the bond strength. This conclusion can be found in Table 6, where the p-values represent the statistical significance of the equivalence between the control and the other subgroups. Thus, subgroups exhibiting p-values lower than 5%, shown in bold and italic format, are said to be significantly different from the control group in terms of bond strength; one can fairly affirm that such treatments are capable of enhancing the related groups (post types) bond strength.

Also in Table 6, the second group, the FRC Postec Plus® post type, was shown to be insensible to any of the pretreatments.

Finally, concerning the effectiveness in improving the bond strength by combining sandblasting with another pretreatment, Table 7 provides evidence that such attempts are worthless. No significant difference between the SB group and the combined groups (SB-HA and SB-HP) were found (p-values are greater than 5%).

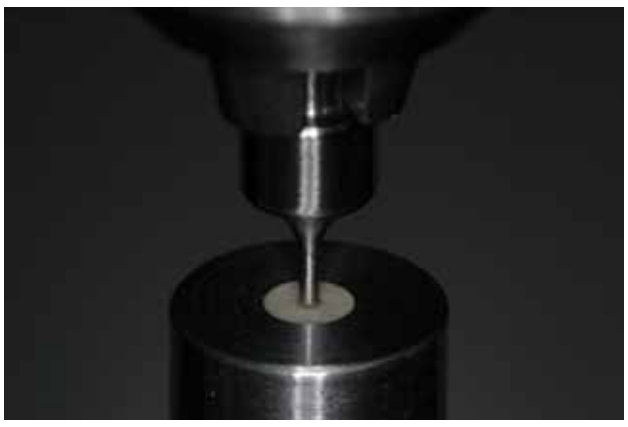

Figure 3. *Push-out pin design.*

DISCUSSION

Group and subgroup pretreatments were used according to a non-destructive method in a previous study[23] that proved sandblasting abrasion with alumina 50µ at a specific distance, pressure and time (30 mm at 2.5 bars for 5 seconds) as the only surface treatment that modified the surface topography of glass and quartz fiber posts, providing a significant increase in roughness. This corroborated the importance of this method and its standard use.

In a previous study[28], fiber posts as used alone revealed a large variation in coefficients of thermal expansion between reinforcing fibers and matrix polymers. The decrease in bond strengths and flexural properties of fiber posts and core materials after long-term water storage and thermocycling (TC) may be caused by plasticization of the polymer matrix by water due to water sorption and swelling, depending on the residual stresses inside the specimens according with other studies[26,28,29]. In the present study, samples were not submitted to TC.

Several studies suggest using silane-coupling agents in coating applications to promote adhesion between inorganic surfaces and polymeric molecules. Silanization results in hybrid organic-inorganic compounds, and should promote adhesion between organic and inorganic matrices due to an intrinsic dual reactivity[30].

Some authors reported an enhancing effect on bond strengths to core materials after the application of silane solutions[19,31]. Chemical adhesion after silane coupling of FRC post surfaces can therefore only be established to exposed fibers or filler particles of the post. Similarly, others studies confirmed the benefit of silane to enhance the bond strength of a dual-cure resin core material to translucent fiber posts[31-33].

Table 3. *Subgroups data: Group 1 (DT Light Post)*

SUBGROUP:	*CONTROL*	*HA*	*SB*	*HP*	*SB-HA*	*SB-HP*
AVERAGE STRENTGH (N):	223.36	250.56	298.52	238.49	270.17	302.87
STD.DEV. STRENTGH (N):	26.56	40.95	10.68	12.40	25.13	26.29
AVERAGE INTERFACIAL STRENTGH (Mpa):	16.17	18.14	21.61	17.26	19.55	21.92
STD.DEV. INTERFACIAL STRENTGH (Mpa):	1.92	2.96	0.77	0.90	1.82	1.90

Table 4. *Subgroups data: Group 2 (FRC Postec Plus)*

SUBGROUP:	*CONTROL*	*HA*	*SB*	*HP*	*SB-HA*	*SB-HP*
AVERAGE STRENTGH (N):	301.57	269.60	314.58	316.06	319.32	313.88
STD.DEV. STRENTGH (N):	27.33	10.48	31.75	16.14	26.55	11.54
AVERAGE INTERFACIAL STRENTGH (Mpa):	24.01	21.46	25.05	25.16	25.42	24.99
STD.DEV. INTERFACIAL STRENTGH (Mpa):	2.18	0.83	2.53	1.29	2.11	0.92

Table 5. *Subgroups data: Group 3 (Transluma Post)*

SUBGROUP:	*CONTROL*	*HA*	*SB*	*HP*	*SB-HA*	*SB-HP*
AVERAGE STRENTGH (N):	263.87	279.58	332.33	249.37	304.17	316.36
STD.DEV. STRENTGH (N):	20.55	14.45	17.35	20.11	18.57	22.46
AVERAGE INTERFACIAL STRENTGH (Mpa):	28.01	29.68	35.28	26.47	32.29	33.58
STD.DEV. INTERFACIAL STRENTGH (Mpa):	2.18	1.53	1.84	2.14	1.97	2.38

Table 6. *Test of Hypothesis on the equality of the means: control versus others subgroups. Results shown in significance level (p-values)*

SUBGROUP:	*HA*	*SB*	*HP*	*SB-HA*	*SB-HP*
DT Light Post Group:	14.0%	0.5%	16.6%	1.8%	0.3%
FRC Postec Plus Group:	decrease*	25.9%	18.3%	17.3%	21.1%
Transluma Post Group:	11.0%	0.1%	decrease*	1.1%	0.6%

* The "decrease" label is displayed in the case where the difference between the control and another subgroup interfacial-strength means exhibits a negative value.

Table 7. *Test of Hypothesis on the equality of the means: sandblast versus others combined sandblast subgroups. Results shown in significance level (p-values)*

SUBGROUP:	*SB-HA*	*SB-HP*
DT Light Post:	decrease*	37.7%
FRC postec plus:	40.4%	decrease*
Transluma Post:	decrease*	decrease*

* The "decrease" label is displayed in the case where the difference between the control and another subgroup interfacial-strength means exhibits a negative value.

Different types of composite resins currently on the market could be used to build up a core onto a fiber post. Relatively stiff self-curing resins would have the advantage of providing a stable support to the crown. On the other hand, more elastic composites, such as flowable and light-activated materials, are easy to prepare with diamond burs for crown adaptation and tend to have easier handling and a better integration with the fiber post surface, leaving little room for bubbles/voids within the abutment, and for discontinuities along the core– post interface[34].

Another study did not demonstrate significantly different bond strength values between a flowable and a highly viscous composite resin core material, but it did find significant interactions between post type and core material. In this current study, it was suggested that a core material exhibiting a high monomer concentration (as with flowable materials) may interact better with some fiber post systems due to the higher amount of resin penetration. This was also in accordance with another study[35], which detected varying bond strength values of two core materials depending on the surface treatment.

As reported by other authors[19,36], the retention of FRC posts and composite resin core materials might depend on the chemical and micromechanical interactions occurring between the two, as well as on the viscosity of the core material (eg, the monomer concentration), and thus may differ between various types of posts and core materials.

Bond strengths between core materials, resin luting agents and FRC posts can be measured using push-out tests. The push-out design can be regarded as a suitable method for evaluation of frictional resistance and post dislodgement[37], and, in this present study, the push-out design showed no premature failures and an acceptable variability of the data distribution. Furthermore, the dislocation resistance of adhesively bonded fiber posts seems to be largely derived from sliding friction in combination with chemical adhesion and micromechanical interlocking. Compared to another in vitro study where the adhesion of fiber posts to resin core materials was evaluated[26], the present investigation demonstrated compatible bond strength values. As polytetrafluoroethylene matrixes were used and stresses occurred after the resin core materials were cured around the FRC post, the push-out bond strength could be affected by the influence of storage temperature and time after polymerization. Adhesive interfaces usually reveal residual stresses according to the polymerization shrinkage of the components[38].

In this context, it is important to suggest that future studies should be done to test bond strengths of fiber posts sandblasted with alumina 50µ at a specific distance, pressure and time to luting agents, and root canal dentin, because the unity between post, luting agent, root canal dentin and resin composite core is a critical factor that should be complemented.

The selection of an adhesive system containing bis-GMA with hydroxyethyl methacrylate (HEMA) has been proven to be effective in penetrating into fiber post structure[39]. The preparation and polymerization of the posts performed shortly before building up the core structure could have contributed to the increased bond strengths due to continuing polymerization of these posts as related in another study[26].

The relationship between surface roughness of various fiber-reinforced composite posts with pretreatments on bond strength to dual-cure resin core material, if a previous investigation is compared against[23], were partially observed because, in the present study, the second group was shown to be insensible to any of the pretreatments. In a recent study[23], a topographical evaluation of different glass and quartz fiber posts surface treatments by a tridimensional surface roughness test demonstrated that the SB treatment was shown to be the only treatment to provide a significant roughness increase in all samples. In the present study, it was expected that, in all groups, the sandblast treatment would significantly enhance the bond strength to resin composite cores; however, in FRC Postec Plus® group, this did not happen. Different post types may present different behaviors if submitted to different pretreatments, and, most likely, their compositions are directly related to this difference and some variation in the resistance to surface conditioning may be present and should have future evaluation. Significant interactions between pretreatments and fiber post compositions may reflect contrary effects on bond strength to resin composite cores with different combinations.

CONCLUSION

Under the limitations of this current study, sandblasting abrasion with 50µ alumina particles at a specific distance, pressure and time was the only surface treatment for DT Light Post® and Transluma Post™ that increased bond strength to dual cure resin composite cores and is recommended in general dentistry. FRC Postec Plus® posts did not show an increase in bond strength in any group. Performing other pretreatments, such as hydrofluoric acid or hydrogen peroxide immersion, in addition to sandblasting appeared to be unnecessary.

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MANUFACTURES' DETAILS

- Bisco™ Incorporated, 1100W, Irving Park Rd, Schaumburg, Illinois 60193, USA Ivoclar Vivadent® AG, Bandererstrasse 2, FL-9494 Schaan, Liechtenstein
- Dentsply Industria e Comércio LTDA – Rua Alice Hervê, 86 Petrópolis RJ, BRA 25665-010
- Microetcher™ II - Danville Engineering, 1901 San Ramon Valley Blvd. San Ramon, CA 94583, USA
- Vetec Química Fina LTDA, R.Pastor Manoel Avelino de Souza 1021, Duque de Caxias, RJ, BRA 25250-000

- Kerr, 21 Commerce Drive, Danbury, CT 06810-4153, USA
- DFL Industria e Comercio S.A. Estrada do Guerenguê, 2059 22713-002 Rio de Janeiro- RJ, Brazil
- WOCO SF 20; Conrad
- Mitutoyo Sul Americana Ltda., Suzano, Brazil – Digital caliper
- Quimis Aparelhos Cientificos LTDA, Diadema, Sao Paulo, Brazil
- Shimadzu do Brasil Comércio Ltda. Divisão Analítica, Barra Funda, São Paulo, Brazil

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