

## ORIGINAL RESEARCH

# Bond Strength of Fiber-reinforced Posts to Deproteinized Root Canal Dentin

<sup>1</sup>Adilson Yoshio Furuse, <sup>2</sup>Leonardo Fernandes Cunha, <sup>3</sup>Samantha Pugsley Baratto, <sup>4</sup>Denise Piotto Leonardi  
<sup>5</sup>Gisele Aihara Haragushiku, <sup>6</sup>Carla Castiglia Gonzaga

## ABSTRACT

**Aim:** This study evaluated the push-out bond strength of cemented fiber posts after deproteinization of root canal dentin walls with NaOCl. The effect of the application of an antioxidant solution (sodium ascorbate) was also evaluated.

**Materials and methods:** A three-step etch-and-rinse (Scotchbond - 3M Espe) and a one-step self-etching adhesive (Xeno III - Dentsply Caulk) were evaluated. Thirty bovine incisor roots were divided into 3 groups:

- Irrigation with physiologic solution (control).
- Deproteinization with 10 minutes irrigation of 5% NaOCl.
- Deproteinization with NaOCl followed by 10 minutes irrigation with 10% ascorbic acid. Fiber posts were cemented with a dual-cured cement (Rely X ARC - 3M ESPE). The push-out bond strength was evaluated after 24 hours of storage in distilled water. The data were analyzed with three-way ANOVA, one-way ANOVA and Tukey's test ( $\alpha = 0.05$ ).

**Results:** There were significant differences between groups ( $p < 0.05$ ). The bond strength of Scotchbond was not influenced by the deproteinization. Xeno III showed a decrease in bond strength when deproteinized with 5% NaOCl ( $p < 0.05$ ). For Xeno III, the subsequent irrigation with ascorbic acid was able to reverse the effect of the deproteinization. Considering the radicular thirds, the bond strength varied in the sequence—apical < middle < coronal.

**Conclusion:** Only the all-in-one adhesive was influenced by the deproteinization. Considering the respective control groups, both systems showed similar bond strength results.

**Clinical significance:** The decreased bond strength of the self-etching adhesive following deproteinization seems to be related to the oxidant effect of the NaOCl solution and the subsequent irrigation with ascorbic acid was able to reverse the effect of the deproteinization.

**Keywords:** Dental adhesives, Fiber posts, Root canal surface treatment, NaOCl, Ascorbic acid.

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## INTRODUCTION

Fiber-reinforced posts have been widely used to restore endodontically treated teeth that present little remaining structure. The choice of material is attributed to the good mechanical behavior of this type of post. As fiber posts have an elastic modulus similar to that of dentin, less stress from occlusion is generated at the root and root fractures; when they occur, they are less severe than when metal posts are employed.<sup>1</sup> It is well known that to obtain a satisfactory clinical behavior, these posts must be bonded to the dental structures.

In the context of bonding anything to the root canal walls, the procedure itself is critical due to the heterogeneity of the dentin substrate with its highorganic content. Thus, many doubts arise during the cementation of fiber posts, especially when the several aspects involved in using resin-based cements are considered. The retention of fiber-reinforced posts is adhesive-dependent and may vary depending on whether an etch-and-rinse or a self-etching adhesive is employed.<sup>2</sup> These two major classes of adhesives used in dentistry are basically based on how the dentin substrate is treated or not with phosphoric acid, and whether the smear layer is removed or not along with the hydroxyapatite nanocrystals surrounding the collagen fibrils. The approach of exposing the collagen network and substituting the mineral content by a polymer is known as hybridization. This idea of hybridization or formation of a hybrid layer means that the collagen network of the dentin plays an important role on the adhesion process and that the longevity of the bonding depends on the impregnation of this network by adhesive resin monomers. Other aspects such as the surface treatment of the post, type of cement, post space irrigation protocol and regional differences at the radicular thirds of the root are also important clinical aspects that generate many doubts.<sup>3</sup>

<sup>1,4-6</sup>Professor, <sup>2</sup>Assistant Professor, <sup>3,5</sup>Private Practice

<sup>1</sup>Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo Bauru, São Paulo, Brazil

<sup>2-6</sup>Graduate Program in Dentistry, Positivo University, Curitiba Parana, Brazil

**Corresponding Author:** Adilson Yoshio Furuse, Professor Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo CEP 17012-901, Bauru, São Paulo, Brazil, Phone: +55 14 3235-8253, e-mail: furuse@usp.br

Besides the variety of adhesive systems and cements, some materials commonly employed during the endodontic therapy and their components may compromise the retention of fiber posts.<sup>3,4</sup> During the endodontic treatment, irrigation with antimicrobial solution such as sodium hypochlorite (NaOCl) is an important step in achieving a favorable outcome. However, if NaOCl is used to irrigate root canals before the cementation of fiber-reinforced posts, the bond strength may be jeopardized.<sup>5</sup> This potential for NaOCl solutions to decrease the bond strength of fiber-reinforced posts cemented to dentin may be attributed to their oxidant effects.

On the other hand, the treatment with NaOCl has been used as a dentin-deproteinization method<sup>6</sup> to improve adhesive wettability.<sup>7</sup> The role played by the organic matrix of the dentin on the bonding process should be evaluated because the enzymatic degradation of the collagen fibrils within the hybrid layer by the collagenolytic activity of specific matrix metalloproteinases may occur overtime and for this reason is a concern.<sup>8,9</sup> It has been hypothesized that the removal of the collagen layer by the action of NaOCl solutions would allow better resin penetration into the dentin<sup>10</sup> in addition to preventing nanoleakage.<sup>11</sup> Moreover, the bonding performance on NaOCl-deproteinized dentin differs between adhesive systems<sup>6,12</sup> and needs further study. In fact, Vargas et al (1997) and Munksgaard (2002) found higher bond strength values for some adhesives.<sup>10,13</sup>

Antioxidant agents such as ascorbic acid and sodium ascorbate have been evaluated as possible methods of reversing the negative effects of oxidants on the bond strength of resin-based materials to dentin.<sup>5</sup> Additionally, oxidant materials have shown to decrease the fracture resistance of endodontically treated teeth and sodium ascorbate seems to reverse this negative effect.<sup>14</sup> However, more studies are necessary to confirm these results before employing antioxidants on a daily basis. Thus, the purposes of the present study were to evaluate:

1. The push-out bond strength of cemented fiber posts after deproteinization by the irrigation of postspaces with 5% sodium hypochlorite.
2. The effect of subsequent irrigation with 10% sodium ascorbate.
3. The bond strength of fiber posts cemented with either a three-step etch-and-rinse or a one-step self-etching adhesive. The working hypotheses evaluated were that the push-out shear bond strength of fiber-reinforced posts cemented on root canal dentin would vary depending on the adhesive system employed; and that the deproteinization of postspaces with NaOCl and subsequent irrigation with an antioxidant solution such as ascorbic acid would significantly influence the shear bond strength.

## MATERIALS AND METHODS

Thirty freshly extracted bovine incisors with anatomically similar roots and fully developed apices were selected and cleaned. All roots were cut to a uniform length of 17 mm and then obturated with gutta-percha and calcium hydroxide sealer (Sealer 26, Dentsply, Petrópolis, RJ, Brazil). The root canal openings were sealed with glass ionomer cement (Vid-rion R, White SS, São Paulo, SP, Brazil) after obturation and samples were stored at 37°C in distilled water for 1 week. Afterward, the postspaces were prepared to a depth of 13 mm with 3 Largo burs provided by the postmanufacturer (Exacto, Angelus, Londrina, PR, Brazil). The roots were rinsed with water to remove remaining debris, blot-dried with paper points, and then divided into three groups (n = 10):

- G1: Roots were irrigated with physiological saline solution for 10 minutes (control).
- G2: Roots were deproteinized by irrigation with 5.25 v/v% NaOCl (Farmadoctor, Curitiba, PR, Brazil) for 10 minutes.
- G3: Roots were deproteinized by irrigation with 5.25v/v% NaOCl for 10 minutes, rinsed with water and rinsed with freshly prepared 10v/v% ascorbic acid for 10 minutes.

Two subgroups were studied (n = 5) according to the adhesive used:

1. A three-step etch-and-rinse adhesive (Scotchbond Multi-Purpose Plus (SBMP), 3M ESPE, St. Paul, MN, USA).
2. A one-step self-etching adhesive (Xeno III – Dentsply/Caulk, Milford, DE, USA). All posts were cemented with the same dual-cured cement (RelyX ARC, 3M ESPE, St. Paul, MN, USA). Both adhesive systems were applied following the manufacturers' instructions and were continuously light-cured with a halogen light source at approximately 500 mW/cm<sup>2</sup>, as measured by the incorporated radiometer (Elipar Trilight, 3M ESPE, Seefeld, Bavaria, Germany). For the etch-and-rinse subgroup, the irrigation process occurred after the acid etching. All adhesives used are presented in Table 1.

Nontransilluminating tapered posts with a coronal diameter of 2.0 mm and an apical diameter of 1.1 mm were used (Exacto 3, Angelus, Londrina, PR, Brazil). All posts were coated with cement and slowly seated by finger pressure. Specimens were stored in 37°C water for 24 hours. All specimens were prepared with controlled air humidity (55 ± 5%) and temperature (23 ± 1°C).

After storage, each root was cut horizontally with a slow-speed, water-cooled diamond saw (IsoMet 2000, Buehler, Lake Bluff, IL, EUA) to produce one slice of 1 mm in thickness for each root region (apical, middle and cervical). Slices of the fiber post-restored roots were loaded in the apical-coronal direction until postsegment extrusion

**Table 1:** Adhesives, composition and manufacturer's instructions

Adhesive/Manufacturer	Composition	Instructions for use
Scotchbond - 3M/Espe, St. Paul, MN, USA	Primer: Aqueous solution of HEMA and polyalkenoic acid copolymer; Bonding: Bis-GMA, HEMA and initiation system	Phosphoric acid-etching for 15 seconds; rinse with water; dry gently for 2 seconds; apply primer and dry gently for 5 seconds; apply adhesive to enamel; light-cure for 10 seconds
Xeno III - Dentsply/Caulk, Milford, DE, USA	Liquid A: 2-hydroxyethyl methacrylate (HEMA), purified water, Ethanol Urethane dimethacrylate resin, Butylated hydroxy toluene (BHT), Highly dispersed silicon dioxide Liquid B: Phosphoric acid modified polymethacrylate resin, Mono fluoro phosphazene-modified methacrylate resin, Urethane dimethacrylate resin, BHT, Camphorquinone, Ethyl-4-dimethylaminobenzoate	Liquid A: Shake bottle two to three times prior to use. Dispense one drop of liquid A into the mixing well. Liquid B: Dispense one drop of Liquid B into Liquid A in the mixing well. Mix liquid for approximately 5 seconds with the applicator tip supplied. With the applicator provided, apply generous amounts to thoroughly wet all cavity surfaces. Leave undisturbed for at least 20 seconds. Uniformly spread the adhesive using a gentle stream of oil-free air for at least 2 seconds until there is a uniform thickness and no further flow of the adhesive to ensure proper removal of solvent. Avoid thinning the adhesive layer by excessive air-drying. Light-cure for at least 10 seconds

in a universal testing machine (EMIC, São José dos Pinhais, PR, Brazil) occurred at a crosshead speed of 0.5 mm/min. The bond strength in MPa was calculated by dividing the load at failure (in N) by the area of the bonded interface. The area of the bonded interface was calculated as follows:

$$A = \{[\pi(R+r)][h^2 + (R-r)^2]0.5\}$$

where,

A is the area of the bonded interface,

$\pi$  is the constant 3.14,

R is the radius of the postsegment (mm) at the cervical region,

r is the radius of the postsegment (mm) at the apical region,

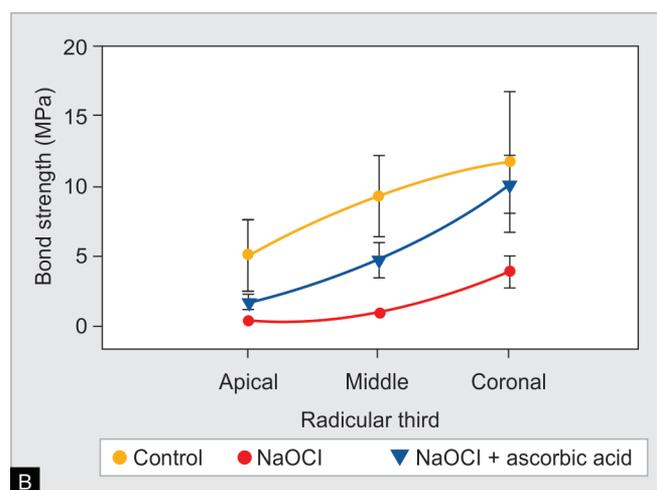
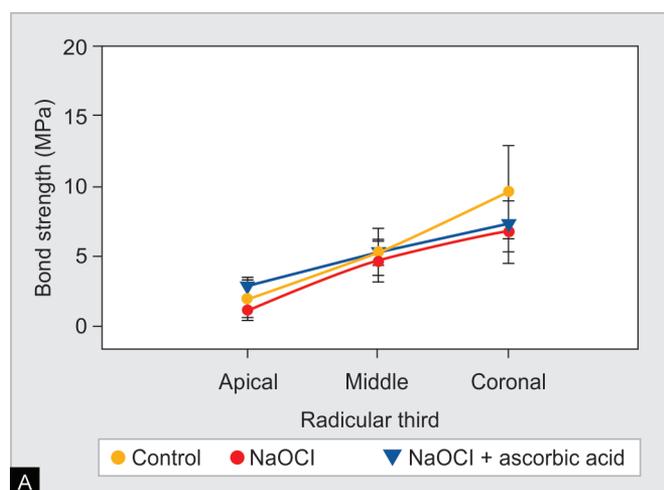
h is the thickness of the postsegment (mm).

The data were statistically analyzed using three-way ANOVA, considering the factors adhesive, irrigation protocol and radicular thirds. Multiple comparisons were made using Tukey's test ( $\alpha = 0.05$ ). For the sake of comparing each condition individually, regardless of the adhesive, irrigation protocol or radicular third, a one-way ANOVA and a Tukey's test ( $\alpha = 0.05$ ) were also applied. The failure modes were

analyzed by visual inspection with an 18 $\times$  magnification under a stereomicroscope and were classified as mainly adhesive at the cement-dentin interface, mainly adhesive at the cement-post interface, mainly cohesive at the dentin or mainly cohesive at the cement.

## RESULTS

The mean values (in MPa) and standard deviations are shown in Table 2 and Figures 1A and B. The three-way ANOVA showed significant differences between groups ( $p < 0.05$ ). Independent of the adhesive system or the irrigation protocol evaluated, the apical region showed lower push-out bond strength values ( $p < 0.05$ ). The bond strength of SBMP was not influenced by the irrigation protocol. Only Xeno III showed a significant decrease in bond strength when deproteinized with 5% NaOCl ( $p < 0.05$ ). For Xeno III, the subsequent irrigation with ascorbic acid was able to reverse the effect of the deproteinization with NaOCl. Considering the radicular third, the bond strength varied in the following sequence—apical < middle < coronal. The results from the multiple comparisons obtained by the one-way ANOVA are



**Figs 1A and B:** Mean bond strength values (MPa) according to the irrigation protocol and radicular thirds: (A) Scotchbond and (B) Xeno III

**Table 2:** Mean bond strength (MPa) and standard deviation values according to adhesives, irrigation protocol and radicular thirds

Adhesive	Radicular third	Irrigation	Mean ( $\pm$ SD)	
Xeno III	Apical	Control	1.98 (1.35) <sup>AB</sup>	
		NaOCl	1.14 (0.71) <sup>AB</sup>	
		NaOCl + ascorbic acid	2.86 (0.61) <sup>ABC</sup>	
	Middle	Control	5.30 (1.69) <sup>BCDE</sup>	
		NaOCl	4.65 (1.44) <sup>ABCD</sup>	
		NaOCl + ascorbic acid	5.26 (0.93) <sup>BCDE</sup>	
	Coronal	Control	9.59 (3.29) <sup>EFG</sup>	
		NaOCl	6.73 (2.24) <sup>CDEF</sup>	
		NaOCl + ascorbic acid	7.35 (2.04) <sup>CDEFG</sup>	
	Xeno III	Apical	Control	5.04 (2.53) <sup>BCDE</sup>
			NaOCl	0.33 (0.10) <sup>A</sup>
			NaOCl + ascorbic acid	1.70 (0.54) <sup>AB</sup>
Middle		Control	9.27 (2.86) <sup>DEFG</sup>	
		NaOCl	0.91 (0.28) <sup>AB</sup>	
		NaOCl + ascorbic acid	4.68 (1.24) <sup>ABCD</sup>	
Coronal		Control	11.69 (5.02) <sup>G</sup>	
		NaOCl	3.87 (1.09) <sup>ABC</sup>	
		NaOCl + ascorbic acid	10.09 (2.1) <sup>FG</sup>	

The same superscript letter indicates no statistically significant difference ( $p < 0.05$ ); SD: standard deviation

displayed as superscripted letters in Table 2. Considering the control and NaOCl + ascorbic acid groups, both adhesives showed similar behavior at each radicular third. All failures occurred primarily at the cement-dentin interface.

## DISCUSSION

The present study was designed to evaluate the influence of the superficial deproteinization with NaOCl on the push-out bond strength of fiber posts cemented to root canals with a resin-based cement. Since, dentin adhesive systems started being used in dentistry, the idea of obtaining a good and stable adhesion to the dentin has focused on impregnating collagen fibrils with resin monomers. However, the role played by the collagen fibrils is an important issue to be evaluated, because there are concerns related to its degradation overtime.<sup>8,9</sup>

The NaOCl has been used as a dentin deproteinizing agent.<sup>6</sup> When NaOCl solutions contacts the organic matter, there is a saponification reaction between fatty acids and the hypochlorous acid, liberating water and the salt of carboxylic acid by the reaction with amino acids. There is a release of chlorine that combined with the protein amino group, forms chloramines. Hypochlorous acid ( $\text{HOCl}^-$ ) and hypochlorite ions ( $\text{OCl}^-$ ) lead to amino acid degradation and hydrolysis.<sup>15</sup>

Two other aspects evaluated were the regional differences normally observed when fiber-reinforced posts are adhesively cemented on root canals and the possibility of reversing the effect of NaOCl irrigation with a reducing agent. The deproteinization with NaOCl irrigation followed

by irrigation with ascorbic acid has shown good shear bond strength results when compared to irrigation with a physiologic solution for both a dual-cured resin-based cement and a self-adhesive one.<sup>5</sup> Ascorbic acid is a well-known antioxidant and is considered safe for use, as it is unlikely that any adverse biologic effect will occur, especially during the irrigation of root canals where the use of proper field isolation is mandatory to avoid contamination.

Some studies have been conducted to evaluate the role played by the deproteinization promoted by the application of NaOCl solutions prior to the use of dentin adhesive systems.<sup>6,12</sup> This concern is based on the fact that debonding is a common reason for the failure of fiber posts adhesively cemented to root canals and that it is a process normally related to the degradation of exposed collagen fibrils not impregnated by the adhesive. These discrepancies between the demineralization and infiltration of monomers would be, in theory, low or nonexistent when one-step self-etching adhesives are employed.<sup>16</sup> Although the use of these simplified systems tends to reduce the operative time by reducing the number of steps during application, minimizing the risk of making errors, the technique is still sensitive.<sup>17</sup>

For the etch-and-rinse subgroup, the irrigation process occurred after the acid-etching. This procedure was conducted to increase the removal of collagen by the deproteinizing action of the NaOCl solution. Similar protocols have been used in other studies.<sup>10,11</sup> It is known, however, that the deproteinization is not homogeneous and depends on the application time, concentration of the solution, volume of solution and temperature.<sup>18,19</sup> In the present study, a long period of irrigation with 5% NaOCl was employed to remove as much collagen as possible from the surface of the root canal walls. It is expected that the higher the concentration, the higher the deproteinizing potential of the solution. A 10% NaOCl solution was not employed because this is not the most commonly used concentration.

When irrigated with physiologic solution, the push-out shear bond strength of fiber-reinforced posts cemented on root canal dentin did not vary depending on the adhesive system employed. This result suggests that both etch-and-rinse and self-etching systems may be used to cement fiber posts to root canals, which is in disagreement with other studies that evaluated the incompatibility between dual-cured resin-based cements and simplified dentin adhesive systems.<sup>20,21</sup>

The coronal region of the root showed higher bond strength values. Although a dual-curing cement was employed, the light-curing obviously influenced the results, which may explain why the incompatibility between the all-in-one adhesive and the dual-cured cement was not noteworthy. Other possible explanations for the regional differences may be related to the fact that the adhesion to the

apical and middle areas of the root may be more technique-sensitive than the coronal area and regional differences in the quantity, volume and orientation of the tubules at different levels of the root canal.<sup>22</sup>

Only Xeno III showed a statistically significant decrease in bond strength when treated with 5% NaOCl, although SBMP also showed a tendency toward decreasing bond strength. The irrigation of posts spaces with NaOCl and the subsequent irrigation with ascorbic acid were only effective in completely restoring the push-out bond strength when Xeno III was used. It is interesting to observe that for Xeno III, as the irrigation occurred in less-permeable dentin due to the presence of the smear layer, a decreased influence of the irrigation protocol would be expected, which was not confirmed.

The SBMP seems not to be influenced by irrigation with NaOCl for 10 minutes. Similar results for this adhesive system were observed by Vargas et al (1997), although these authors employed 2 minutes instead of 10 minutes of irrigation with NaOCl.<sup>10</sup> Other studies that evaluated the two-step etch-and-rinse version of this system, Singlebond, showed a significant decrease in bond strength when this adhesive was applied after irrigation with NaOCl.<sup>5,23</sup> In the case of SBMP, may be the third step of applying a hydrophobic layer of a high-pH adhesive (pH  $\approx$  5.6) after priming could decrease the negative effect of the surface oxidation.

The present study evaluated nontransilluminating tapered posts. It should be noted that the type of post could lead to different results and that further studies could be conducted to evaluate the role played by different types of fiber-reinforced posts as well as the long-term stability of the adhesion obtained after irrigation with various endodontic solutions. It is, however, interesting to observe that the retention of fiber posts may not be entirely affected by the postdesign, root surface roughness or resin cement-curing mode.<sup>24</sup>

The influence of the irrigation protocol employed previous to the cementation of fiber-reinforced posts seems to be adhesive-dependent. Although potentially affecting the formation of the hybrid layer, deproteinization with a NaOCl solution seems not to be more important than the oxidizing effect, as observed in the present study. Although, decreasing the bond strength of the self-etching adhesive, the effect of irrigation with NaOCl could be reversed by the antioxidizing action of ascorbic acid. Additionally, further *in vitro* and *in vivo* studies should be carried out to evaluate the effect of the studied deproteinization method on the longevity of resin-dentin bonds.

## CONCLUSION

The bond strength of the fiber-reinforced postevaluation varied only when the one-step self-etching adhesive was

employed. For this adhesive, the deproteinization with NaOCl significantly decreased the bond strength, while the subsequent irrigation with ascorbic acid increased the bond strength. Regional differences between the coronal, middle and apical areas of the root occurred for both adhesives, with higher values being observed in the coronal region.

## CLINICAL SIGNIFICANCE

The negative influence on bond strength after deproteinization is adhesive-dependant. The decrease on bond strength of the self-etching adhesive following deproteinization seems to be related to the oxidant effect of the NaOCl solution and the subsequent irrigation with ascorbic acid may reverse the effect of the deproteinization.

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