Microtensile bond strength between a UDMA fiber post and different resin cements: Effect of pre-surface treatment

Renato Cassio ROPERTO¹, Thiago Soares PORTO¹, Lisa LANG¹, Sorin TEICH¹, Sean WEBER¹, Omar EL-MOWAFY² and Sizenando Toledo PORTO-NETO³

¹ Department of Comprehensive Care, School of Dental Medicine, Case Western Reserve University, 2124 Cornell Rd, Cleveland, 44106, USA
² Department of Clinical Science, Faculty of Dentistry, University of Toronto, 124 Edward Street, Ontario, M5G1G6, Canada
³ Department of Restorative Dentistry, Faculty of Dentistry, Sao Paulo State University, 1680 Humaita St, Araraquara, SP, 14801, Brazil
Corresponding author, Renato Cassio ROPERTO; E-mail: rcr67@case.edu

INTRODUCTION

First introduced in the 1990’s, glass reinforced fiber posts were a good alternative for restoring endodontically treated teeth¹-³. Their use became much more popular due to the evident similarity in modulus of elasticity to dentin; excellent aesthetic appearance; ability to bond directly to tooth structure, thereby avoiding a laboratory phase; easy removal if an endodontic retreatment is required; ability to transmit light into the root canal, and it eliminates the potential hazards of corrosion and allergic hypersensitivity⁴-⁵. Scientific studies have also shown that fiber posts can increase the core retention of the remaining radicular portion of tooth, by reinforcing the interface when the tooth is under functional loading⁶-⁹. For this reason, the choice of restorative adhesive technique is very important and has a direct impact on the longevity of the final restoration¹⁰,¹¹.

Glass-fiber posts are generally composed of reinforced glass fibers embedded in PMMA epoxy resin⁵,¹² or, in some cases, urethane dimethacrylate (UDMA)¹³, both of which have a similar flexural strength¹⁴. Some authors have reported a difficulty in creating a strong mechanical interlock between the luting agent materials and the PMMA epoxy resin utilized in most commercial fiber posts, without having some form of surface treatment¹⁵-²¹. Some examples of surface treatments are: particle sandblasting with Al₂O₃; chemical etching with ethyl alcohol or hydrogen peroxide; plasma etching; and other available treatments²⁸-²². Finally, a silane coupling treatment is utilized to establish a chemical bond between the treated post surface and the luting agent/core build-up material²³-²⁵.

During a fiber post cementation, failure of a resin cement and fiber post surface often occurs at the junction between both. This failure process requires better characterization. The objectives of the present study were to: firstly, evaluate the effect of commonly employed PMMA fiber post-surface treatments, on UDMA glass fiber posts. Secondly, determine if UDMA fiber posts have different adhesive behaviors when the various surface treatments are performed. The null hypotheses tested were: (1) there are no differences in topographic changes produced by all forms of surface treatments on the UDMA fiber post; (2) these treatments have no influence on the bond strength of the UDMA fiber post to the resin cements material.

MATERIALS AND METHODS

Sixty fiber posts (DT Logipost, Synca, Canada) with a maximum diameter of 1.5 and 20 mm long were used in this study. The posts were composed of longitudinally oriented fibers, coated with UDMA resin (urethane-dimethacrylate monomer-1,6-bis-[methacryloxyloxy-2-ethoxy carbony lamino]). The three different surface chemical treatments include: Group 1: no surface treatment and silane application only (G1); Group 2: immersion in ethyl alcohol (96°GL) for 10 min, followed by silane application (G2); and Group 3: immersion in...
24% H$_2$O$_2$ for 10 min, followed by silane application (G3). Each group was further divided into four subgroups based on the luting resin cement utilized: I-Unicem/3MESPE; II-Biscem/Bisco; III-Panavia SA/Kuraray and IV-DuoLink/Bisco.

The surface treatment for G1 consisted of one application of a single layer of silane (Silane Component, Bisco, Schaumburg, IL, USA) for 60 s followed by gentle air drying for an equal time. The surface treatment for G2 consisted of three successive steps: a) immersion of the post in a ethyl alcohol (96°GL) solution for 10 min; b) extensive rinsing with distilled water for 1 min; and c) application of a single layer of silane for 60 s followed by gentle air drying for an equal time. For G3, the treatment followed three successive steps, as well: a) immersion of the post in a 24% H$_2$O$_2$ solution for 10 min; b) extensive rinsing with tap water for 1 min; and c) application of a single layer of silane for 60 s followed by gentle air drying for an equal time.

Subsequently, a block build-up process was performed using four different resins: Unicem self-adhesive resin cement (3M ESPE, St. Paul, MN, USA), Biscem self-adhesive resin cement (Bisco), Panavia SA self-adhesive resin cement (Kuraray, Tokyo, Japan), and DuoLink conventional resin cement (Bisco). The composite resin cements (conventional and self-adhesive) were mixed and applied according to the manufacturer’s instructions.

Each fiber post was positioned centrally in the cavity of a square shaped, silicon matrix of the following dimensions: 10 mm(L)×15 mm(W)×1.5 mm(H). The silicon matrix was then completely filled with composite resin cement. The excess composite resin cement was removed from the matrix and a plastic mylar strip was placed on top. Subsequently, a glass slide was placed on the matrix and light-curing was performed through the slide using a halogen light (Spectrum 800 Light Cure Unit, Dentsply DeTrey, Konstanz, Germany) with an output of 600 mW/cm$^2$ for 40 s. This resulted in a cured block of uniform thickness, with the post in the center and resin cement on both sides. Each block was then serially polished with sandpaper (400 to 100 µm grit) to obtain a uniform thickness of 1.5 mm.

The specimens were stored in distilled water for 24 h. Each block was then fixed to an acrylic block, with wax, and mounted on a slow-speed diamond saw (Isomet Buehler, Lake County, IL, USA) where it was sectioned into 5 bar-shaped sticks with the dimensions of approximately: 10 mm(L)×3.0 mm(W)×1.5 mm(H), (Fig. 1). Each bar-shape stick was attached with cyanoacrylate glue (Zapit, Dental Ventures of America, Melville, NY, USA) micro-sticks were also submitted for a failure analysis (Fig. 1).

The SPSS 23.0 software (IBM Software, Armonk, NY, USA) was used for statistical analysis. Means and standard deviations were calculated, and the data was analyzed by analysis of variance (ANOVA) using a factorial design in which post surface treatment technique and resin cement type acted as the independent variables. Multiple comparisons of means were performed by the post hoc statistical test.

Two additional posts from each surface treatment group were randomly selected for SEM longitudinally examination. Each post was mounted onto a metallic stub, platinum-sputtered (Polaron SL 515 machine, Watford, Herts, UK) and observed under a scanning electron microscope (Hitachi S2500, Tokyo, Japan) at different magnifications (35×, 500×, 1,000× and 2,000×).

**RESULTS**

The means and standard deviations of the bond strengths for each of the five experimental and control groups are shown in Table 1. The one-way ANOVA is shown in Table 2. Statistical analysis revealed no significant differences between post surface treatments ($p>0.05$). Statistical differences were found between the resin cements ($p<0.05$) as well as the interaction between surface treatment/resin cement ($p<0.05$). Regarding the resin cements utilized in this experiment, Tukey test revealed that the group I-Unicem resin cements were significantly higher ($p<0.05$) than resin cement groups II, III and IV. Resin cement group II showed the lowest values as compared to groups I and IV, however, no statistical difference was found when compared with resin cement group III. The resin cements DuoLink (Bisco) and Experimental (Kuraray), produced values with no statistical difference between each other ($p<0.05$). Concerning the interaction between resin cement and surface treatment, the statistical analysis revealed
that Group 3/resin cement IV showed comparatively higher bond strength values, however, not statistically different when compared with the other self-adhesive resin cement groups. For all groups, specimens failed predominantly at the interface resin cement-fiber post (adhesive failure).

**Table 1** Mean and standard deviation of the post-core bond strength calculated for all experimental groups

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Cement</th>
<th>Mean (SD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>Unicem</td>
<td>12.49 (1.20)</td>
</tr>
<tr>
<td></td>
<td>Biscem</td>
<td>8.14 (0.83)</td>
</tr>
<tr>
<td></td>
<td>Panavia SA</td>
<td>10.41 (0.70)</td>
</tr>
<tr>
<td></td>
<td>DuoLink</td>
<td>6.67 (0.89)</td>
</tr>
<tr>
<td>Alcohol</td>
<td>Unicem</td>
<td>10.37 (3.70)</td>
</tr>
<tr>
<td></td>
<td>Biscem</td>
<td>9.88 (1.12)</td>
</tr>
<tr>
<td></td>
<td>Panavia SA</td>
<td>10.26 (1.44)</td>
</tr>
<tr>
<td></td>
<td>DuoLink</td>
<td>9.78 (1.28)</td>
</tr>
<tr>
<td>H₂O₂</td>
<td>Unicem</td>
<td>12.92 (1.58)</td>
</tr>
<tr>
<td></td>
<td>Biscem</td>
<td>7.09 (0.97)</td>
</tr>
<tr>
<td></td>
<td>Panavia SA</td>
<td>8.25 (1.31)</td>
</tr>
<tr>
<td></td>
<td>DuoLink</td>
<td>13.90 (2.09)</td>
</tr>
</tbody>
</table>

**Table 2** Two-way ANOVA

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of squares</th>
<th>df</th>
<th>Mean square</th>
<th>F</th>
<th>Sig</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corrected Model</td>
<td>282.474 (a)</td>
<td>11</td>
<td>25.679</td>
<td>9.699</td>
<td>0.000</td>
</tr>
<tr>
<td>Intercept</td>
<td>6,021.319</td>
<td>1</td>
<td>6,021.319</td>
<td>2,274.230</td>
<td>0.000</td>
</tr>
<tr>
<td>Cement</td>
<td>97.832</td>
<td>3</td>
<td>32.611</td>
<td>12.317</td>
<td>0.000</td>
</tr>
<tr>
<td>Treatment</td>
<td>12.477</td>
<td>2</td>
<td>6.239</td>
<td>2.356</td>
<td>0.106</td>
</tr>
<tr>
<td>Cement * Treatment</td>
<td>172.165</td>
<td>6</td>
<td>28.694</td>
<td>10.838</td>
<td>0.000</td>
</tr>
<tr>
<td>Error</td>
<td>127.086</td>
<td>48</td>
<td>2.648</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Total</td>
<td>6,430.880</td>
<td>60</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Corrected Total</td>
<td>409.561</td>
<td>59</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
</tbody>
</table>

**SEM**

Two posts were randomly selected from each group for SEM examination of the superficial aspect of the post following surface pre-treatment. Pre-treatment with ethyl alcohol (96°GL) or 24% H₂O₂ for 10 min resulted in minimal modification of the fiber post surface when compared with the control group. All groups showed only a small roughening of the surface along the entire post length regardless of the surface treatment performed. The exposed glass fibers were not damaged or fractured by the oxidative action of H₂O₂ or the ethyl alcohol. Longitudinal views of both groups revealed very small surface dissolution of the UDMA resin matrix, exposing some additional surface area of glass fibers for micromechanical retention. The underlying UDMA resin remained intact and exhibited no signs of cracking or damage regardless the surface treatment performed (Figs. 2–4).

Fig. 2  SEM of the control group (no surface treatment) in different magnification (35×, 500×, 1,000× and 2,000×). Arrow showing the glass fiber and start showing the UDMA resin matrix.
DISCUSSION

Incompatibility between materials is still a common problem in dental practice. The adhesion of a glass fiber post and luting agents and/or core build-up materials should have enough strength to support the occlusal stress and mechanical load of the post and core/crown restoration. Achieving proper adhesion is arguably the most challenging clinical aspect of this type of restoration, and clinical failure is a common outcome. Methods to improve the adhesion between glass fiber posts and build-up materials have been investigated extensively in the literature. Some methods utilize additional in-lab equipment, such as plasma treatment and sandblasting with Al₂O₃, which can be time consuming and costly. More simple methods using solvent solutions, such as ethyl alcohol (96GL) and 24% H₂O₂, are also capable of improving the adhesion of resin materials to the post surface by partially dissolving the epoxy bonds on the PMMA epoxy resin commonly used in a variety of commercial fiber posts. These solutions are also capable of roughening the surface by exposing the glass fibers, thus creating an ideal interlocking pattern between the post surface and the resin cement.

However, the material tested in this study uses a different monomer covering the glass fibers that is characterized by a more rigid and heavily cross-linked polymer matrix. When compared to PMMA epoxy resin matrix, the UDMA monomer structure (urethane-dimethacrylate monomer-1,6-bis-[methacryloyloxy-2-ethoxycarbonylamino]) presents a more stiff molecular structure with hydroxyl groups aimed to provide good bonding adhesion and interaction with other resin based materials without any necessary pre-surface treatment. This could be translated to a faster and more reliable clinical procedure without the necessity of expending additional time preparing the fiber post surface. For both PMMA and UDMA fiber posts, coupling agents are suggested to be used before the cementation procedure itself. The action of the silane (usually indicated after the surface etching treatment) involves the formation of covalent bonds from the reaction of the organo-functional group with the resin matrix; and the hydrolyzed alkoxy group with the mineral substrate (glass or silica) of the composite material. Thus, the
chemical removal of the superficial layer of PMMA or UDMA resin exposes additional glass fibers, which increases the surface area available to contact the silane molecules. The increased number of chemical bonds between the silanized glass fibers and the methacrylate-based resin material significantly improves the interfacial bond strength.

The PMMA epoxy resin, which exhibits a high degree of conversion and highly cross-linked structures within the polymer substrate, differs from the UDMA resin base material, which is minimally affected by chemical agents that act by oxidizing the substrates and breaking bonds. Furthermore, UDMA resin has more chemical affinity to luting agent materials as compared to PMMA epoxy resin, thereby negating the need for surface treatment prior to cementation. This finding was also supported by the results attained in this study. The results of the SEM investigation illustrated that the pattern of surface dissolution for UDMA fiber posts was minimal when treated with ethyl alcohol or hydrogen peroxide.

CONCLUSION

In conclusion, the post surface pre-treatment with ethyl alcohol or hydrogen peroxide did not significantly increase the bond strength between silanized UDMA glass fiber posts and the resin cements. However, the bond strength was significantly affected by the type of resin cement used. Nevertheless, it would be interesting to conduct long-term clinical studies to investigate the survival rate of fiber posts utilizing a similar type of resin cement used. Nevertheless, it would be interesting to conduct long-term clinical studies to investigate the survival rate of fiber posts utilizing a similar type of surface treatment, but under clinical conditions.

REFERENCES