Cementation in adhesive dentistry: the weakest link

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Citation for published version (APA):
CHAPTER 3

Influence of surface pretreatment of fiber posts on cement delamination

Abstract

Objectives: To evaluate the influence of post surface pretreatment on the delamination strength of different cements from a prefabricated FRC post tested in a three-point bending test.

Methods: Three cements were tested; RelyX Unicem, DC Core Automix, and Panavia F 2.0. Per cement, 40 posts (D.T. Light Post Illusion size 3) were divided into four groups; no pretreatment (control), sandblasting, silanization, and sandblasting followed by silanization. A cement layer was applied to the posts using a standardized poly-oxy-methacrylate mold. The specimens were subjected to a three-point bending test recording the initial and catastrophic failure loads. Two-way ANOVA and Tukey post-hoc tests were used to analyze the differences between the variables.

Results: At the initial failure load, all specimens demonstrated delamination of the cement layer, therefore initial failure load was defined as delamination strength. With RelyX Unicem, none of the pretreatments showed significant differences. When using Panavia F 2.0, silanization (735 (51) MPa) resulted in higher initial failure values than sandblasting (600 (118) MPa). When DC Core Automix was used, silanization (732 (144) MPa) produced significantly higher initial failure values than the no pretreatment group (518 (115) MPa) and the combined sandblasting and silanization group (560 (223) MPa). Two failure types were observed; cohesive and adhesive failure. In the silanization groups, more cohesive failures were observed for all cements tested.

Significance: Especially when non self-adhesive cements are used, silanization of fiber posts has a beneficial effect on cement delamination strength and failure type.
Introduction

In recent years, there has been a clear shift from metal alloy posts to the use of fiber posts. These posts offer a number of mechanical and clinical advantages. Their elastic modulus is closer to that of dentin compared to metal posts [1], which reduces the risk of root fracture. Indeed, clinically, root fracture is less apparent in endodontic teeth restored with glass fiber posts than in teeth restored with metallic posts [2]. A major problem with all types of posts is reliable cementation. This applies to glass fiber posts especially, because they are supposed to be cemented adhesively. An observation that illustrates this problem is the fact that debonding is the most common mode of failure of the fiber-reinforced composite post restoration [3, 4]. Endodontic failure [5, 6] and secondary caries [7] are also a common mode of failure, which can be a direct effect of microleakage due to failure of the adhesive bond. There are many factors that affect the retention of post systems, for example post design, type of luting agent, cementation procedure, preparation, shape, and condition of the post space among others [8]. Another factor affecting the success of the total adhesive system is the adhesion between cement and post. This bond has to withstand stresses induced by the normal masticatory function, which can eventually result in debonding of the post-core restoration.

Different pretreatment methods have been proposed to improve the retention of fiber posts. Silanization is often used to improve adhesion of cementation to a fiber post. This has the benefit of being a fast chair-side procedure. Several studies show that the microtensile bond strengths between post and cement can be improved by silanizing the post [9, 10]. However, there is no consensus on this subject. One study using a push-out bond strength test set-up, reported that the use of a silane coupling agent in combination with sandblasting did not increase the bond strengths when resin cements were used, although it was commented that this could be due to the limited adherence of the luting cement to the root canal walls [11]. Another study, by the same research group, reported that the use of a silane coupling agent alone did not increase the bond strengths when resin cements were used [12]. Another method of potentially creating extra retention is sandblasting. Sandblasting results in roughening of the post surface, leading to an increase in micromechanical retention [13]. Although some studies show that sandblasting the surface of fiber-reinforced composite posts significantly improved the retention of posts adhesively luted with a dual-cured resin
cement [14], the effectiveness of sandblasting is still debatable [13, 15]. The main problem with this method is the lack of selectivity. The matrix and fibers are both affected by the sandblasting, which could lead to damage of the post structure [13].

Retention of posts has been widely investigated, but these studies usually involve extracted teeth in order to provide clinically relevant results. The post retention is often measured by means of (micro) push-out strength measurements [13] or by microtensile bond strength testing [13]. Although push-out and microtensile bond strength tests relate to how posts behave in the clinical situation, only the strength of the weakest part of the restoration is determined. Failure can occur either on the cement-dentin interface, or on the cement-post interface, or cohesively within the cement, post or dentin. Moreover, the failure is not likely to take place at the cement-post interface, knowing that the bond between post and cement is usually stronger than the bond between cement and dentin. Therefore, these tests are not the best way to evaluate the bond strength between post and cement.

Currently, there is no test that reflects the clinical situation exactly. In order to gain a better understanding of bond strengths of composite cements to the post surface, it is beneficial to address this problem from different perspectives. In this light, the adhesion of cements to the post surface has been investigated using a new testing methodology developed by the authors which involves a three-point bending test. The purpose of this study was to evaluate the influence of different cements and post surface pre-treatment methods on the delamination strength of the cement from the post surface.

**Materials and methods**

In a poly-oxy-methacrylate (POM) block, standardized post spaces were prepared. An initial hole was drilled in the block using a 1 mm parallel carbide bur in the lathe cut machine (MF 70, Proxxon, Germany), and then the D.T. Light Post special burs (RTD, St. Egreve, France) were used to finish the post space preparation to the desired size and depth, which was a post space corresponding with a number three sized post to full preparation depth. In this way, a non-bonding mold was created as can be seen in Figure 3.1.
Influence of surface pretreatment of fiber posts on cement delamination

Figure 3.1  Fabrication of the test specimens. (A) Preparing the post space preparations in a polyoxy-methacrylate block. (B) POM mold with preparation of ten post spaces. (C) The fiber posts are fitted in the post spaces of the mold. (D) Cement is applied in the post spaces, posts are placed and samples are removed after setting of the cement.

Using this mold, a thin, uniform and in dimensions clinically relevant cement layer could be applied to the fiber posts. Per cement, 40 fiber posts (DT light Post Illusion size 3, RTD, St. Egrève, France), were divided into four groups and pretreated in different manners. The first group (control) was cleaned with ethanol 80% and dried with compressed air, no post surface pretreatments were carried out. In the second group, the posts were sandblasted (Danville Engineering) perpendicular to their long axis for 2 s with 50 μm aluminum oxide particles with a pressure of 4000 HPa (4 Bar) at a distance of 5 cm and cleaned with ethanol. In the third group, the posts were silanized by mixing Photo Bond liquid A and B and Porcelain Bond activator in equal portions. This mixture was applied to the post using a microbrush and polymerized with an Astralis 5 polymerization light (Vivadent, Germany) for 10 s. In the fourth group, sandblasting was followed by silanization.
Table 3.1  Materials used in this study.

<table>
<thead>
<tr>
<th>Product</th>
<th>Manufacturer</th>
<th>Batch number</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clearfil DC Core Automix Paste</td>
<td>Kuraray</td>
<td>00035A</td>
<td>Catalyst paste: Bis-GMA, TEGDMA, silanated colloidal silica, barium glass, D,L-camphorquinone, benzoyl peroxide Universal paste: Bis-GMA, TEGDMA, silanated colloidal silica, barium glass, N,N-diethanol p-toluidine</td>
</tr>
<tr>
<td>Clearfil Photo Bond Bonding agent Universal</td>
<td>Kuraray</td>
<td>00502C</td>
<td>N,N-di-ethanol-p-toluidine, sodium benzene sulfinate, ethanol</td>
</tr>
<tr>
<td>Clearfil Photo Bond Bonding Agent Catalyst</td>
<td>Kuraray</td>
<td>00403</td>
<td>MDP, Bis-GMA, HEMA, hydrophobic alifatic dimethacrylate, camphorquinone, benzoyl peroxide</td>
</tr>
<tr>
<td>Clearfil Porcelain Bond Activator</td>
<td>Kuraray</td>
<td>00207A</td>
<td>Hydrophobic dimethacrylate,  γ-methacryloxy propyltrimethoxy silane (γ-MPS)</td>
</tr>
<tr>
<td>Panavia F 2.0 Paste A</td>
<td>Kuraray</td>
<td>00148A</td>
<td>Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, sodium aromatic sulfinate (TPBSS), N, N-diethanol-p-toluidine, surface treated (functionalized) sodium fluoride, silanated barium glass</td>
</tr>
<tr>
<td>Panavia F 2.0 Paste B</td>
<td>Kuraray</td>
<td>00148A</td>
<td>MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, silanated silica, photoinitiator, dibenzoylperoxide</td>
</tr>
<tr>
<td>D.T. Light-Post Illusion</td>
<td>RTD</td>
<td></td>
<td>Radiopaque translucent quartzfiber post</td>
</tr>
</tbody>
</table>

Kuraray Medical Inc., Okoyama, Japan; 3M ESPE, Seefeld, Germany; RTD, St. Egrève France. Bis-GMA: bisphenol A diglycidylmethacrylate; TEGDMA: triethylene glycol dimethacrylate; MDP: 10-methacryloxydecyl dihydrogenic phosphate; HEMA: 2-hydroxyethyl methacrylate.

The post space molds were filled with RelyX Unicem, Panavia F 2.0 or DC Core Automix. The materials used in this study are shown in Table 3.1. The cements were mixed according to manufacturer’s instructions and applied in the mold using needle tubes (Accudose Needle Tubes, Centrix Inc., Shelton, CT, USA), to minimize
the amount of voids and air bubbles present within the cement, and to maintain a high level of homogeneity of the cements. After insertion of the cement in the mold, a (pretreated) fiber post was placed in each mold, and the cement was light-cured from coronally following manufacturer’s instructions using an Astralis 5 polymerization light. The posts were left in their molds for 24 h at 37 °C. After 24 h the posts with their attached cement layers were carefully removed from the POM mold and subjected to a three-point bending test.

![Diagram of three-point bending test set-up with dimensions.](image)

**Figure 3.2** Schematic representation of three-point bending test set-up with dimensions.

The three-point bending test was performed with a crosshead speed of 0.5 mm/min in a Hounsfield H109KM universal testing machine (Hounsfield, Redhill, UK). The loading angle was 90° and the distance between the supporting bars was 10 mm. A schematic representation of the test set-up with dimensions is given in Figure 3.2.

The diameter of the post at force application was carefully measured using digital sliding calipers with an accuracy of 0.01 mm. The posts were measured at the point of pressure appliance, which was clearly visible as a small white indentation in
the cement layer. To get a clear notion of the thickness of the post at that point, the calipers were placed in the middle of the indentation and the post was rotated 90°, measuring the largest possible diameter at the pressure point, thus erasing variety in diameter caused by compression of the cement or loss of cement caused by delamination as much as possible. The initial failure value (N) was recorded at the first dip in the stress-strain curve of the experiment. The catastrophic failure was recorded at fracture of the post. The flexural strength at initial and catastrophic failure was calculated using the following equation:

$$F_s = \frac{8Fl}{\pi d^3}$$

where $F_s$ (in MPa) is the calculated flexural strength at failure, $F$ is the load at failure (in N); $l$ is the distance between the points of support in the three-point bending test (in mm), in this case 10 mm; and $d$ is the diameter of the fiber post (in mm).

Two-way ANOVA (SigmaStat Version 3.0, SPSS Version 11.0, Inc., Chicago, USA) and Tukey post-hoc tests were used to analyze the differences between flexural strength, type of cement and type of pre-treatment. The significance level $\alpha$ was set at 0.05.

**Results**

All specimens demonstrated delamination of the cement layer from the post surface at the initial failure load. The initial failure strength was therefore defined as the delamination strength. Cement delamination was observed on the bottom part of the specimens.

The two-way analysis of variance showed a significant effect on cement ($F = 4.651; P = 0.011$) and method ($F = 30.253; P < 0.001$). There was also a significant interaction between cement and method ($F = 2.548; P = 0.013$). Comparing the pooled data of the three cements (see Table 3.2, Figure 3.3), RelyX Unicem proved to have significantly higher mean delamination strength values than DC Core Automix ($P = 0.008$). There were no further significant differences between the cements for delamination strength. The pooled data of the pretreatment methods (Table 3.2, Figure
show that silanization of the posts resulted in significantly higher delamination strengths than sandblasting of the posts ($P = 0.026$). There were no further overall differences between the pretreatment methods.

The cements and pretreatment method groups were also compared (Table 3.2, Figure 3.3). There were no statistically significant differences between pretreatment methods for RelyX Unicem. For Panavia F 2.0, silanization of the post resulted in higher delamination strengths than sandblasting ($P = 0.025$), there were no further significant differences. For DC Core Automix, silanization resulted in significantly higher delamination strengths than no pretreatment ($P = 0.001$) or sandblasting and silanization ($P = 0.021$). There was a tendency for silanization to have higher values than sandblasting, but this was not statistically significant ($P = 0.136$). Within the no pretreatment groups, both RelyX Unicem ($P = 0.006$) and Panavia F 2.0 ($P = 0.013$) had higher delamination strength values than DC Core Automix. There was, however, no statistically significant difference between the first two. Within the sandblasting and the silanization groups, there were no statistically significant differences in initial failure values between the different cements. Within the sandblasting plus silanization groups, however, RelyX Unicem had, again, higher delamination strength values compared to DC Core Automix ($P = 0.001$).

**Failure mode**

Two failure types were recorded; adhesive failure and cohesive failure. When adhesive failure occurred, the first dip in the stress-strain curve did not correspond with fracture of the post. The cement layer delaminated while the posts were still intact. When cohesive failure occurred, there was only one dip in the stress-strain curve. At this point, delamination of the cement layer occurred simultaneously with fracture of the post.
Table 3.3 summarizes the distribution of failure modes within the different groups. It also shows the mean values for the different groups calculated per failure type. It was generally observed that cohesive failures resulted in higher delamination strengths than adhesive failures.

When using RelyX Unicem, any kind of pretreatment of the post resulted in more cohesive failure, this applied especially to pretreatments where silanization was used. In the no pretreatment group, there was no large difference observed in delamination strength between the specimens with cohesive and adhesive failure modes. When using silanization or both silanization and sandblasting, almost all specimens failed cohesively. The delamination strengths were higher than with no pretreatment or sandblasting alone.

With Panavia F 2.0, sandblasting alone did not result in more cohesive failure, but silanization and sandblasting plus silanization did. There were, however, no clear
differences in delamination strengths between failure types and groups. Silanization resulted in more cohesive failures compared to no pretreatment or sandblasting.

Silanization within the DC Core Automix groups results in a more favorable failure type (cohesive failure) than no pretreatment, sandblasting, and sandblasting plus silanization. There is little difference in delamination strength between failure modes within the silanization group. Within the other groups, the difference between the failure modes is much higher, in favor of the cohesive failure type. This implies that in the silanization groups, the delamination occurs at a later stage in the stress-strain curve, closer to the catastrophic failure of the post.
<table>
<thead>
<tr>
<th>Pretreatment method/cement</th>
<th>RelyX Unicem</th>
<th>Panavia F 2.0</th>
<th>DC Core Automix</th>
<th>Mean Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>No pretreatment (control)</td>
<td>691.1 (69.6)¹</td>
<td>677.5 (44.3)²</td>
<td>517.7 (114.7)ᵃᵇ¹,²</td>
<td>628.8 (112.3)</td>
</tr>
<tr>
<td>Sandblasting</td>
<td>675.5 (45.7)</td>
<td>567.4 (96.5)ᵃ</td>
<td>599.7 (118.2)</td>
<td>614.1 (99.9)ᵃ</td>
</tr>
<tr>
<td>Silanization</td>
<td>665.2 (237.8)</td>
<td>735.1 (51.1)ᵃ</td>
<td>731.5 (143.7)ᵃᵇ</td>
<td>710.6 (160.7)ᵃ</td>
</tr>
<tr>
<td>Sandblasting+Silanization</td>
<td>757.5 (201.6)¹</td>
<td>677.4 (47.5)</td>
<td>560.2 (222.6)ᵇ¹</td>
<td>665.0 (138.4)</td>
</tr>
</tbody>
</table>

Mean cement:  
743.1 (173.5)¹  
716.7 (136.2)  
667.1 (204.0)¹

ᵃᵇ: Statistically significant differences between pretreatment methods (within the same cement).  
¹,²: Statistically significant differences between cements (within the same pretreatment method).
Table 3.3  Distribution of failure type per study group with their mean delamination strength values (in MPa) and standard deviations in parentheses.

<table>
<thead>
<tr>
<th>Pretreatment</th>
<th>RelyX Unicem</th>
<th>Panavia F 2.0</th>
<th>DC Core Automix</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Failure type</td>
<td>Failure type</td>
<td>Failure type</td>
</tr>
<tr>
<td></td>
<td>n</td>
<td>n</td>
<td>n</td>
</tr>
<tr>
<td>Adhesive</td>
<td>691.0 (78.8)</td>
<td>722.7 (32.1)</td>
<td>517.7 (114.7)</td>
</tr>
<tr>
<td>Cohesive</td>
<td>691.4 (8.8)</td>
<td>658.2 (33.9)</td>
<td>736.1 (101.4)</td>
</tr>
<tr>
<td>Silanization</td>
<td>665.6 (39.2)</td>
<td>724.6 (211.4)</td>
<td>724.6 (93.3)</td>
</tr>
<tr>
<td>Sandblasting + Silanization</td>
<td>733.5 (105.8)</td>
<td>764.2 (20.6)</td>
<td>687.2 (52.6)</td>
</tr>
<tr>
<td>Sandblasting</td>
<td>667.2 (24.2)</td>
<td>724.6 (211.4)</td>
<td>740.4 (241.2)</td>
</tr>
<tr>
<td>No pretreatment (control)</td>
<td>713.9 (65.4)</td>
<td>610.8 (29.3)</td>
<td>824.3 (-)</td>
</tr>
<tr>
<td>Adhesive</td>
<td>658.2 (33.9)</td>
<td>610.8 (29.3)</td>
<td>824.3 (-)</td>
</tr>
<tr>
<td>Cohesive</td>
<td>665.6 (39.2)</td>
<td>656.5 (105.7)</td>
<td>824.3 (-)</td>
</tr>
</tbody>
</table>
Discussion

In this study, significant differences between the investigated groups were found. The differences were relatively small, and the standard deviations rather high. There was a significant difference for the delamination strength between RelyX Unicem and DC Core Automix, and certain pretreatment methods seemed to perform better than others. Silanization performed better overall than sandblasting. However, the differences did not count in equal manner for all cements tested. No significant differences between pretreatment methods were found for RelyX Unicem, Panavia F 2.0 only showed differences between sandblasting and silanization. DC Core Automix showed the most significant differences between the different pretreatment methods. There were significant differences between silanization and no pretreatment and between silanization and sandblasting plus silanization.

Apart from the delamination strength, the failure type was also important. Cohesive failure meant that the delamination strength of the cement layer from the post was as high as the strength of the post itself, and therefore the strongest bond achievable in this test set-up. Adhesive failure implied a delamination of the cement layer while the post was still intact. RelyX Unicem and Panavia F 2.0 performed better overall in terms of failure mode compared to DC Core Automix. Silanization resulted in a larger portion of cohesive failure for all three cements tested, whereas sandblasting did not result in such an effect. The combination of sandblasting and silanization resulted in high portions of cohesive failure, but this effect was more likely due to the silanization than to the sandblasting of the post. In fact, with DC Core Automix it was observed that sandblasting followed by silanization resulted in more adhesive failures and lower delamination strengths, especially for the adhesive failures, than silanization alone.

Silanization is based on the formation of covalent -Si-O-Si- bonds between hydroxyl groups of glass materials and the alkoxy groups of a silane. In this study porcelain bond activator was used, which contains \( \gamma \)-MPS (\( \gamma \)-methacryloxy propyltrimethoxy silane) as the reactive monomer. In general, bond strength can also be improved by sandblasting, which creates micromechanical retention through roughening of the post surface. It has been suggested that sandblasting results in volume loss of the post, hereby compromising the structural integrity, and therefore strength, of the post [11].
However, D’Arcangelo et al. showed that although the surface structure was altered by sandblasting, the flexural strength and flexural modules of fiber posts were not compromised [16]. In this study the cement delamination was investigated and not catastrophic failure, e.g. fracture of the post. Comparing delamination strengths of the sandblasted post and the untreated post did not show any significant differences. There are indications that different test methodologies result in different conclusions regarding sandblasting. Studies regarding the shear component of the stresses, e.g. push-out tests [17], showed more favorable results regarding sandblasting compared to the micro-tensile tests [15].

The observed differences in delamination strength between the cements suggest that the chemical composition is of great importance in choosing a pretreatment method when cementing a fiber post. RelyX Unicem is, according to the manufacturer, a self-adhesive resin cement. In the case of the adhesion to a fiber post, the methacrylated phosphoric esters in the RelyX Unicem cement react with the fiber post material. A similar reaction occurs in Panavia F 2.0. The phosphate groups of the MDP (10-methacryloxydecyl dihydrogenic phosphate) are responsible for what could be called a ‘self-adhesive process’. In DC Core Automix, such an effect is not present. This means that there is no direct chemical bond between the cement and post material, beside eventual reactions with unreacted existing C = C bonds on the surface of the fiber post. Although sandblasting improves the micromechanical retention, the delamination strength did not improve significantly. However, silanization of the post resulted in a delamination strength similar to that of the other two cements. Apparently, silanization improves the chemical bond the other two cements already have, which is necessary for a high delamination strength.

For the clinical situation, the most interesting results are the situations where the delamination strength is higher than the no pretreatment delamination strength for a given cement. In this study, this only applies to DC Core Automix with silanization of the post. For all other cements investigated, pretreatment did not lead to better results than no pretreatment. One should realize that the other cements were ‘self-adhesive’ cements, while DC Core Automix is a ‘normal’ composite cement. From a clinical perspective one should be aware that silanization is important if a composite cement without ‘self-adhesive’ properties is used. Moreover, the silanized fiber posts showed the more favorable cohesive failure mode compared to the other pretreatment
groups for all cements tested. The clinical situation is, however, much more complex than the test set-up used in this study. Compared to the clinical situation, all in vitro studies are a compromise. Nevertheless, the results show the proof of principle that silanization improves the bond strength between a fiber post and resin composites without self-adhesive properties.

**Conclusion**

Within the limitations of this study, it can be concluded that especially when non self-adhesive cements are used, silanization of fiber posts has a beneficial effect on the cement delamination strength and failure mode.
References


