Effect of Adhesive Resin Cements and Post Surface Silanization on the Bond Strengths of Adhesively Inserted Fiber Posts

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Abstract
This study evaluated the tensile bond strengths and the effect of silanization of fiber posts inserted with different adhesive systems. Sixty DT Light Posts (size 1) were used. Thirty posts were pretreated with silane. The posts were cemented into form-congruent artificial root canals (12 mm) of bovine dentine. Six groups were formed: G1, Prime&Bond NT/Calibra; G2, Monobond-S+Prime&Bond NT/Calibra; G3, ED Primer/Panavia 21ex; G4, Monobond-S+ED Primer/Panavia 21ex; G5, RelyX Unicem; and G6, Monobond-S+RelyX Unicem. The mean (standard deviation) tensile bond strengths (megapascals) were 7.69 (0.85) for G1, 7.15 (1.01) for G2, 6.73 (0.85) for G3, 6.78 (0.97) for G4, 4.79 (0.58) for G5, and 4.74 (0.88) for G6. G1 achieved significantly higher bond strengths than G3 and G5; G3 had significantly higher values than G5 (P < .05; Scheffé procedure). Silanization had no significant effect (P > .05, one-way analysis of variance). Tensile bond strengths were significantly influenced by the type of resin cement. Silanization of fiber post surfaces seems to have no clinical relevance. (J Endod 2007;33:840–843)

Key Words
Adhesive resin system, bond strength, fiber posts, post surface conditioning, silanization

Techniques and recommendations for the restoration of endodontically treated teeth have changed from the use of rigid prefabricated metal posts to composite resins and fiber-reinforced composite (FRC) posts (1–5). Different resin cement systems have been evaluated with respect to the insertion of FRC posts (6, 7). In addition, various pretreatments of post surfaces have been described to enhance the interface bond between the FRC posts and composite resins (8, 9). The role of silane to improve the bonding to FRC posts is discussed controversially (10).

Previous studies used human teeth or artificial root canal models for microtensile and push-out bond strength measurements (2, 7, 11, 12). The frequent occurrence of premature failures of specimens and the high variability of values are regarded as a disadvantage of the microtensile method (11). The push-out test is highly dependent on the position of the punch pin and only reflects the shear strength of thin slices of the whole specimen (7, 13). In human teeth, bonding to the root canal might be influenced by the variability of intraradicular dentine (14). To avoid the disadvantages of inhomogeneity of human root canal dentine, the present investigation was conducted by using exclusively 3-year-old bovine dentine. The aims of the present study were to investigate the retention of FRC posts, inserted with 3 different adhesive resin cements (a total-etch, a self-etch bonding system, as well as a self-adhesive resin cement) into artificial root canals of bovine dentine, and to determine the effect of previous silanization of the bonding surface by using a pull-out test. The null hypothesis was that the tensile bond strengths of FRC posts are not significantly affected by the type of resin cement and the pretreatment with a silane coupling agent.

Materials and Methods
Sixty 3-year-old (lower permanent) bovine incisors were extracted immediately after slaughter and stored in saline. The teeth were sectioned, and remaining blocks of root dentine were obtained. The dentine portions were fixed upright in aluminum molds with standardized alkali-coupling molds for embedding into resin blocks (Technovit; Heraeus Kulzer, Wehrheim, Germany) (Fig. 1A). Subsequently, standardized post spaces were prepared into the dentine blocks to a depth of 12 mm (1.5-mm distance to the pulp chamber) with a low-speed drilling machine (Gillardon, Hamburg, Germany), with the manufacturer's corresponding post drill system. The artificial root canals were rinsed with 1% sodium hypochlorite solution and dried.

Sixty fiber posts (DT Light Post; VDW, Munich, Germany) size 1 (length, 20.0 mm) were used. The DT Light Post is composed of unidirectional quartz fibers (60% volume) embedded in an epoxy resin matrix and has different tapers in the apical (2%) and the medium third (6%). Thirty posts were treated with a silane coupling agent (3-methacryloxypropyl trimethoxysilane, 1% in weight; Monobond-S; Ivoclar Vivadent, Schaan, Liechtenstein) for 60 seconds with a disposable brush and dried. The FRC posts were inserted by using Prime&Bond NT/Calibra (Dentsply, Konstanz, Germany), ED Primer/Panavia 21ex (Kuraray, Okayama, Japan), and RelyX Unicem (3M ESPE, Seefeld, Germany) according to the respective manufacturers' instructions.

Six experimental groups (n = 10) were formed: Group 1, Prime&Bond NT/Calibra; Group 2, Monobond-S + Prime&Bond NT/Calibra; Group 3, ED Primer/Pana-
and catalyst paste were mixed and applied onto the post surfaces. The posts were inserted, and the cement was left to cure.

RelyX Unicem is a dual-curing and self-etching resin cement that does not require any pretreatment of dentin. After capsule trituration the cement was applied onto the posts' surfaces. Insertion and light-curing (Optilux 401) through the post followed for 60 seconds.

Seven days after cementation and storage in humid atmosphere, the specimens were mounted in a universal testing machine (Instron Typ 4204; Instron Co, Canton, MA). The crosshead speed was 1.0 mm per minute. Bond strengths were expressed in megapascals (MPa). The total bonding surface at an insertion depth of 12 mm was 44.8 mm².

After testing, the bonding surfaces were analyzed with a light microscope ( Axioskop 40; Carl Zeiss, Jena, Germany) at 36× magnification, followed by evaluation of representative specimens with scanning electron microscopy (LEO 435 VP, LEO Elektronenmikroskope; Carl Zeiss, Oberkochen, Germany). The percentage of the bonding surface covered with resin cement after testing was determined approximately by 2 evaluators under a light microscope ( Axioskop 40), laying a transparent scaled foil onto the posts. Each post was evaluated from 2 sides (rotated 180 degrees) (Fig. 1B).

The type of failure was determined as adhesive between post and composite, as cohesive within the composite, or as a mixed type of fracture. The tensile bond strength values of the 6 groups (n = 10) were compared with the one-way analysis of variance (ANOVA) and the Scheffé procedure at a level of significance at P < .05 (SPSS 14.0 for Windows; SPSS Inc, Chicago, IL).

Results

The mean tensile bond strength values and the mean percentage of the resin cement—covered bonding surfaces with standard deviations are shown in Table 1. The one-way ANOVA showed that the pretreatment with silane did not result in any statistical difference in tensile bond strengths (P > .05). The Scheffé procedure revealed that Prime&Bond NT/Calibra (group 1) achieved significantly higher bond strengths than RelyX Unicem (group 5) and ED Primer/Panavia 21ex (group 3) (P < .05). The cementation of the posts with ED Primer/Panavia 21ex resulted in significantly higher bond strength values than the use of RelyX Unicem (Scheffé procedure; P < .05).

Light microscopic and scanning electron microscopy analysis after the tensile bond strength tests showed that only mixed fracture types occurred on the bonding surfaces (Fig. 1C).

The highest percentage of covered bonding surface was evaluated after the cementation with Prime&Bond NT/Calibra.

Discussion

Bovine dentine was used in this investigation because of the limited availability and the inhomogeneity of extracted human teeth. Bovine teeth are a suitable alternative substitute for human dentine and have recently been used for pullout strength testing of a fiber-

![Figure 1](image-url)
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glass-reinforced composite post system (15–18). The posts were inserted into the artificial root canals without a root canal filling to avoid the effects of root canal treatment on the dentine (19, 20). It can be assumed that the direction of the dentinal tubules differed in the physiologic setting compared with the model used in the present study, and this could have affected the results with respect to the bonding behavior of the 3 investigated adhesive systems. However, the present model was highly standardized, and it should be emphasized that bonding to dentine highly depends on the formation of a hybrid layer, whereas tubule orientation and tag formation seem to be of minor importance (21). Nevertheless, the intrarelational aspects of the present study should be focused with respect to the cementation materials and the post silanization.

The null hypothesis of the present study has to be rejected. Prime&Bond NT/Calibra with phosphoric acid etching demonstrated significantly higher tensile bond strengths compared with the 1-step self-etching ED Primer/Panavia 21ex and with the self-etching resin cement RelyX Unicem. It can be assumed that as a result of the preparation of the artificial root canals into the bovine dentine blocks, a smear layer was created (22). A previous study investigating the adhesion of fiber posts to dentine showed that the use of phosphoric acid completely dissolved the smear layer, whereas the weaker self-etching ED primer revealed only partial dissolution of the layer. Both systems were able to etch the underlying dentine. RelyX Unicem lacks in genuine hybridization of the intact bonding substrates (12). This is in accordance with the present results, which indicate that the tensile bond strength values depend on the etching capacity and creation of sufficiently hybridized dentine of the adhesive systems used. On the other hand, recently published studies demonstrated that the bond strength of fiber posts in root canals rather depend on frictional resistance instead of adhesion. This highlights the demand for future investigations in this field (23, 24).

The use of silane is the subject of controversial discussion. A microtensile bond strength study demonstrated that silane significantly improved the bond strengths between FRC posts and resin composites (25). Recent studies showed that in push-out tests the bond strengths of the resin cements to FRC posts were not significantly affected by silanization (7, 10). The present study revealed that silanization of the bonding surfaces did not significantly affect the tensile bond strength values. In contrast to recent studies that investigated small units of post and composite resin material or thin cross-sectional slices (7, 10, 25), the present results represent the total tensile bond strength values in relation to the complete bonding surface of the FRC posts. Regarding these results, it can be assumed that the effects of silanization have no clinical relevance.

A silane agent can only bridge resins and OH-covered inorganic superfluously exposed glass fibers. The highly cross-linked polymers of the matrix in FRC posts do not have any functional groups for chemical reaction with silane molecules (25). With respect to the fact that the DT Light Post is composed of 60% (vol) glass fibers, silane might improve the bonding at the interface between FRC posts and the resin-based cement, dependent on the parts of superficially exposed glass fibers. The light microscopic evaluation revealed differences in the parts of resin cement–covered surfaces of the debonded posts. These results were not analyzed statistically, because this part was an approximate evaluation. However, it was remarkable that the posts inserted with Prime&Bond NT/Calibra demonstrated the highest percentage of covered bonding surfaces and the highest tensile bond strengths. As a conclusion, the interfacial bond between the adhesive system and the dentine seems to be weaker than the bonding to the FRC post surface. These observations indicate no influence of silanization on the adhesion of the resin-based cements to the FRC post surfaces. The mean parts of resin cement–covered post surfaces were almost the same for the respective materials with or without previous silanization.

Different types of post surface conditioning have been proposed for FRC posts (6, 9, 25, 26). In the present study, pretreatment of the posts was recommended only for Calibra (application and light-curing of 1 layer of Prime&Bond NT). The monomers probably infiltrated the space between the glass fibers and the microporosities of the epony resin matrix. The polymerized bonding resins might stabilize micromechanical retention between polymers and FRC posts, thus resulting in a higher percentage of resin cement–covered bonding surfaces.

The self-etching RelyX Unicem system revealed the lowest percentages of covered surfaces. This system might create a layer of the acidic monomers on the post surface that probably reduce the micromechanical interlocking and are therefore responsible for the weaker interfacial bond of the resin cement to the FRC posts. The failure modes of the present study indicate that there was no chemical bond between the composite resin cements and the fiber posts.

References


