Effect of silanization on bond strengths of fiber posts to various resin cements

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Objectives: To investigate the effects of pretreatment (silanization) on bond strengths between 3 different types of fiber posts and 4 resin cements, respectively. **Method and Materials:** Prefabricated quartz-fiber (Unicore Post, Ultradent) and prefabricated glass-fiber (FRC Postec, Ivoclar Vivadent) posts with a cross-linked polymer matrix and individually formed glass-fiber posts with an interpenetrating polymer network (IPN Post, Stick Tech) ($n = 160$ each) were inserted into resin composite disks (2 mm thick) using the following resin cements and silane solutions: Panavia F/Porcelain Bond Activator (Kuraray), PermaFlo DC/Silane (Ultradent), Variolink II/Monobond S (Ivoclar Vivadent) and RelyX Unicem/Espe Sil (3M Espe). Nonsilanated posts served as controls. The push-out bond strengths were determined before ($n = 10$) and after ($n = 10$) thermocycling (2,000 cycles, 5°C to 55°C, dwelling time 30 seconds). **Results:** Bond strengths (mean [SD]) were significantly affected by the resin cement ($P < .001$), the pretreatment ($P < .001$), and the type of post ($P < .001$), but not by thermocycling ($P = .955$, 4-way ANOVA). The IPN post demonstrated significantly higher bond strengths compared to the other posts ($P < .05$; Tukey *B*). Silanization significantly increased bond strengths (15.2 [5.2] MPa) compared to those of the control groups (13.9 [4.9] MPa). **Conclusion:** The type of fiber post revealed a significant influence on bond strengths, whereas the effects of silanization appeared to be clinically negligible. *(Quintessence Int 2007;38:121–128)*

Key words: fiber post, push-out bond strengths, silane, thermocycling

Clinical failures involving endodontically treated teeth reconstructed with posts are mainly attributed to cementation flaws of posts or root fractures, which are the most serious type of failure.[1,2] It has been suggested that the rigidity of a root canal post should be equal or close to that of dentin to distribute occlusal forces along the length of the root to prevent root fractures.[3] Fiber-reinforced composite (FRC) posts were introduced in 1990 and have demonstrated favorable physical properties, eg, a modulus of elasticity close to that of dentin.[4]

Commonly used prefabricated FRC posts exhibit a highly cross-linked polymer matrix between the fibers, and because of the high conversion rate they should be more or less nonreactive; therefore, bonding between prefabricated FRC posts and adhesive luting agents can be hampered.[5] A recently developed FRC material consists of continuous unidirectional glass fibers and a multiphase polymer matrix. This polymer matrix reveals a semi-interpenetrating polymer network (IPN) with both linear polymer phases, polymethylmethacrylate (PMMA), and cross-linked poly-

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Table 1 **Composition and ingredients of all materials used in the present investigation**

Luting agent	Bonding agent	Silane solution	Manufacturer	Composition of resin composites	Composition of primers	Effective silane	Solution of silane	Light curing
Panavia F	Ed Primer	Porcelain Bond Activator	Kuraray, Okayama, Japan	Barium glass powder, sodium fluoride, dimethacrylate, MDP, silica, benzoyl peroxide, amine, sodium aromatic sulfinate	10-MDP, HEMA, N-methacryl 5-aminosalicylic, sodium benzene sulfinate, N, N'diethanol p-toluidine, water	3-trimethoxysilylpropylmethacrylate (3-MPS)	Bisphenol-a-polyethoxydimethacrylate	40 s
PermaFlo DC	PermaFlo DC Primers A&B	Silane	Ultradent, Salt Lake City, Utah	Bis-GMA, benzoyl peroxide, tertiary amine	§§	MPS	Isopropanol 92%	40 s
Variolink II	Excite DSC	Monobond S	Ivoclar Vivadent, Schaan, Liechtenstein	Bis-GMA, urethane dimethacrylate, triethyleneglycol dimethacrylate, ytterbium trifluoride barium glass, silica	HEMA, bis-GMA, glycerine dimethacrylate, phosphoric acid acrylate, highly dispersed silica, ethanol	MPS	Ethanol 52%, distilled water 47%	40 s
RelyX Unicem		Espe Sil	3M Espe, Seefeld, Germany	Silica, glass, calcium hydroxide, methacrylated phosphoric ester, dimethacrylate, acetate	No primer available	Amino-silane	Ethanol > 90%	40 s

(10-MDP) 10-methacryloyloxydecyl dihydrogen phosphate; (HEMA) hydroxyethyl methacrylate; (MPS) methoxysilylpropylmethacrylate; (bis-GMA) bisphenol glycidyl methacrylate

§§Data not provided by manufacturer

mer phases. Monomers of adhesive resins can penetrate into the linear polymer phase and form an interdiffusion bonding by polymerization.[6] In a recent investigation, higher flexural properties could be demonstrated for the new FRC material with the IPN structure compared to commercially prefabricated FRC posts.[7] Furthermore, IPN posts revealed better interfacial adhesion to resin cements compared to prefabricated FRC posts.[8]

For FRC post production, glass or quartz fibers are industrially coated with a silane to improve adhesion to the resin matrix, to protect the fibers from damage during handling, to modify the catalytic and wettability properties of the fiber surface, and to increase the chemical resistance of the fiber matrix interface especially to water.[9] To improve the bond strengths between adhesive luting cements and FRC posts, silanization of the post surface has been suggested but revealed contradictory effects.[10-14] Up to now it remains unclear whether the type of fiber post influences the effects of silanization on bond strengths of resin cements to FRC posts.

The aim of the present investigation was to evaluate the influence of silanization of 3 different types of FRC posts on bond strengths to various resin cements before and after thermocycling. The formulated null hypothesis was that the bond strengths to FRC posts were not affected by the type of post, the pretreatment (silanization), the resin cement, or thermocycling.

METHOD AND MATERIALS

Three different types of FRC posts (n = 480) were selected for the present investigation: prefabricated quartz-fiber posts (Unicore

Posts Size 3, Ultradent), prefabricated glass-fiber posts (FRC Postec Size 3), and individually formed glass-fiber posts with an IPN matrix (IPN Post Ever Stick, Stick Tech). The aim of this study was to investigate the bond strengths of the mentioned fiber posts to 4 different resin cements with and without silanization before and after thermocycling. The following materials were used: Panavia F/Porcelain Bond Activator (Kuraray), PermaFlo DC/Silan (Ultradent), Variolink II/Monobond S (Ivoclar Vivadent), and RelyX Unicem/Espe Sil (3M Espe) (Table 1).

The IPN posts were individually formed using a transparent plastic mold with a round cutout (diameter 1.5 mm) and light cured for 60 seconds (1,200 mW/cm², Astralis 10, Ivoclar Vivadent). Subsequently, the cure adhesive of the IPN posts (Stick Resin, Stick Tech) was applied onto the surface of the post and, according to the manufacturer's instructions, light cured for 20 seconds (1,200 mW/cm², Astralis 10).

The prefabricated posts were cleaned with alcohol, and all posts were inserted into 2-mm-thick resin composite disks (Adamant, Ivoclar Vivadent) using the materials mentioned above. These disks were fabricated for the present investigation (diameter 5 mm), with a central hole being congruent to the coronal parallel part of each post. The disks were placed into plastic molds that were also produced for this study. The plastic molds were provided with post space preparations congruent to the 3 different post types used; for the top of the post the molds revealed cutouts according to the size of the composite disks. To facilitate sample preparation, the plastic molds could be separated and fixed with screws (Fig 1). For insertion of the posts into the composite disks, the resin cements were applied onto the surface of the posts and into the orifice of the composite disks. Subsequently, the posts were inserted into the post space preparations of the respective plastic molds (Fig 2). Using this design, a central position of the post perpendicular to the composite disk was guaranteed. Light curing was performed in each group for 40 seconds (1,200 mW/cm², Astralis 10). The light source was positioned on top of the post.

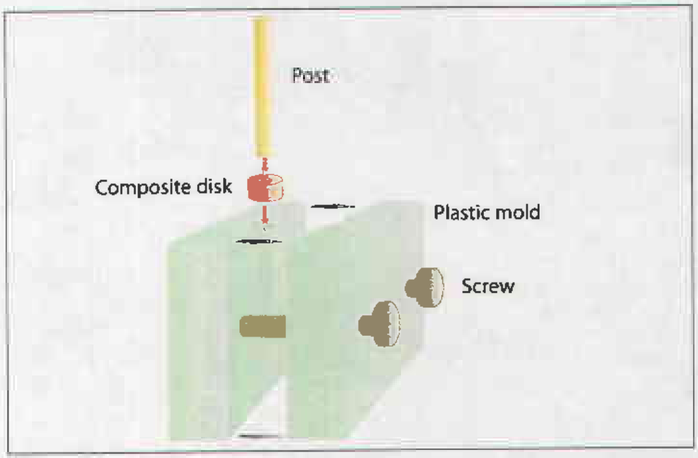

Fig 1 For post insertion, resin composite disks were prepared that could be fixed into precise-fitting cutouts at the coronal part of the post space preparation inside the plastic molds. Consequently, posts could be inserted into the composite disks, simulating a defined surface of the post space preparation inside a tooth.

In the pretreatment groups the silane solutions were applied onto the posts' surfaces and air dried at room temperature for 60 seconds. Subsequently, the adhesive system of the respective resin cement used was applied onto the posts' surfaces as well. With the controls only, the adhesive system of the respective resin cement was applied onto the cleaned posts' surfaces.

After light curing, the specimens were stored for 24 hours in saline solution at 37°C. Half of the specimens of each group were subjected to thermocycling (2,000 cycles, 5°C to 55°C, dwelling time 30 seconds), while push-out testing was performed immediately for the other half of the specimens to determine the initial bond strength values. For the push-out testing, the posts were placed into a hole of a supporting table; thus, the specimens were centered and the load was applied using a push-out pin (Fig 3). Diameter sizes of the opening of the supporting table and of the push-out pin were 2.5 and 1.2 mm, respectively. Push-out testing was conducted using a universal testing machine (Zwick, Roell) by loading each specimen at a crosshead speed of 0.5 mm/min until fracture. The maximum failure load was recorded in newtons and converted

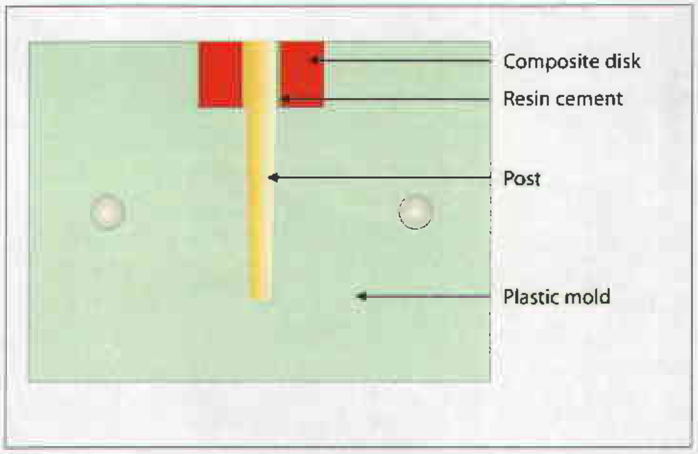

Fig 2 Longitudinal section through the plastic mold, revealing an inserted post, layer of resin cement, and composite disk.

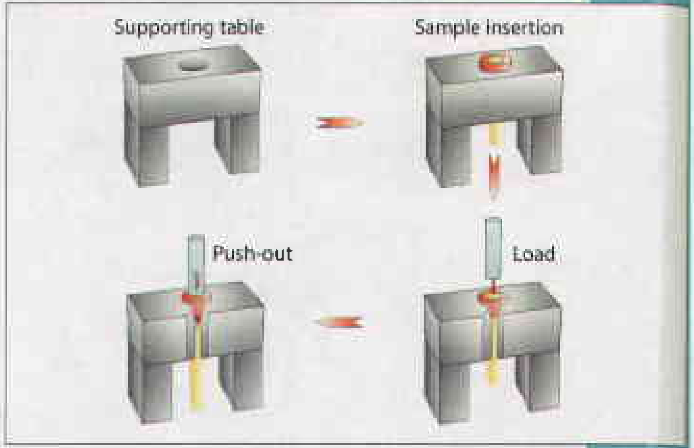

Fig 3 For push-out testing a supporting table with an opening of 2.5 mm was selected. After sample insertion, the load of the push-out pin (diameter 1.2 mm) was applied at a crosshead speed of 0.5 mm/min until fracture.

into megapascals. The maximum stress was calculated from the recorded peak load divided by the computed surface. To calculate the exact bonding surface, the thickness (*h*) of each specimen was measured with a micrometer screw (Mitutoyo Messgeräte). The bonding surface was calculated using the formula of a cylinder ($2\pi \times r \times h$), since in all cases the parallel part of the tapered posts was inserted into the composite disks.

After push-out testing, each specimen was observed using a stereomicroscope (DV 4, Zeiss) at 40× magnification to determine the failure mode. This observation was performed by 2 independent operators, and the specimens were divided into 3 groups according to the failure mode: *(1)* adhesive failures between post and cement; *(2)* cohesive failures inside the cement; and *(3)* cohesive failures inside the post. In cases of mixed failures the decision was made according to the failure mode that occurred predominantly.

All statistical analyses were performed using commercially available software (SPSS for Windows 12.0, SPSS). The effect of resin cement, thermocycling, pretreatment, and type of post on bond strengths was analyzed using analysis of variance (ANOVA), and post hoc comparisons were performed using Tukey *B*. Analysis of failure modes was computed using chi-square testing. The level of significance for all statistical analyses was set at .05.

RESULTS

Bond strengths (mean [SD]) were significantly affected by the resin cement ($P < .001$), the pretreatment ($P < .001$), and the type of post ($P < .001$), but not by thermocycling ($P = .955$; 4-way ANOVA). Interactions were observed between type of post and resin cement ($P < .001$) and resin cement and pretreatment ($P < .001$).

Post hoc comparison revealed that the resin cement Variolink demonstrated significantly higher bond strengths (16.3 [4.5] MPa) than the other investigated materials Panavia (14.2 [4.7] MPa), RelyX Unicem (14.2 [4.1] MPa) and PermaFlo DC (13.6 [6.4] MPa) ($P < .05$, Tukey *B*). Concerning the post type, post hoc analysis showed significantly higher bond strengths for IPN Post (19.4 [4.6] MPa) compared to FRC Postec (11.9 [3.4] MPa) and Unicore posts (12.4 [3.1] MPa) ($P < .05$, Tukey *B*).

Without consideration of post type and resin cements, silane pretreatment revealed significantly higher bond strengths (15.3 [5.2] MPa) compared to the control groups (13.9 [4.9] MPa). The box-and-whisker plot (Fig 4) demonstrates the bond strengths between post types and resin cements of both controls and pretreatment groups. Silane pretreatment significantly increased the bond strengths of PermaFlo DC (14.9

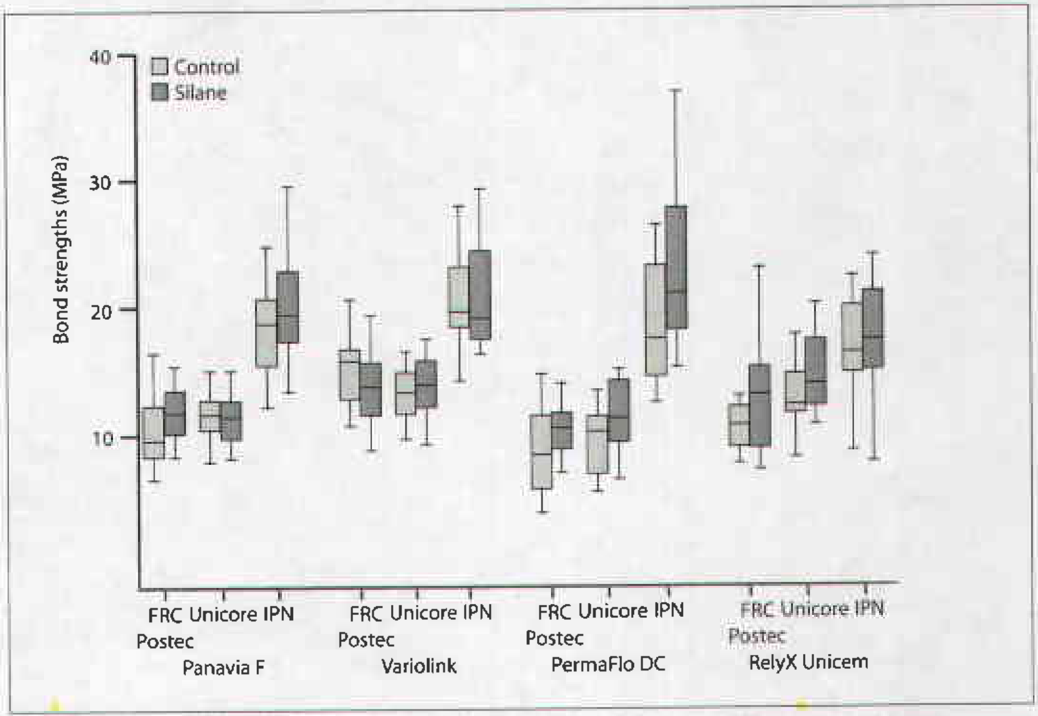

Fig 4 Box-and-whisker plot demonstrating the bond strengths of 3 types of fiber posts and 4 resin cements with and without silanization.

Table 2 **Failure modes of samples after push-out testing (absolute numbers and percentage relation)**

	Failure modes		
Material	**Adhesive post/cement**	**Cohesive inside cement**	**Cohesive inside post**
Panavia F	81 (67.5%)	31 (25.8%)	8 (6.7%)
Variolink	83 (69.2%)	29 (24.2%)	8 (6.6%)
PermaFlo DC	71 (59.2%)	38 (31.6%)	11 (9.2%)
RelyX Unicem	74 (61.7%)	38 (31.7%)	8 (6.6%)

[6.8] MPa) compared to the control groups (12.3 [5.8] MPa) (*P* = .025, *t* test) and RelyX Unicem (15.0 [4.2] MPa) compared to the controls (13.5 [3.9] MPa) (*P* = .042, *t* test); after Bonferroni correction with factor 4 none of these differences remained significant anymore.

Analyses of fracture mode are represented in Table 2. No significant differences between groups were observed (*P* > .05).

DISCUSSION

Bond strengths were significantly affected by the type of post, the resin cement, and the pretreatment, but not by thermocycling. Thus, the null hypothesis was partly rejected.

The present study investigated the bond strengths of various resin cements to different types of FRC posts using a push-out model. Push-out tests result in a shear stress at the interface between post and cement[15]; this is comparable to the stresses under clinical conditions. Furthermore, the push-out design used allowed polymerization stresses comparable to the clinical situation.[16] A previous study showed more reproducible bond strength measurements using a conical version of the push-out design compared to microtensile techniques.[17] However, designs evaluating microtensile bond strengths have been controversial because of premature failures that can occur during sample preparation, in particular in cases where bond strength is very low.[18]

The aim of the present investigation was to evaluate the bond strengths between posts and resin cements and the effect of silanization of FRC posts on bond strengths. Therefore, the posts were inserted into composite disks using plastic molds with artificially created post spaces. When posts are luted into extracted teeth, bonding to dentin might influence the bond strength values between posts and cements. It should be emphasized that this investigation focused only on bond strengths between posts and resin cements, even if the bond strengths to dentin seem to be interesting as well.

Resin cements used in the present investigation were selected based on different compositions of the cements as well as of the corresponding adhesive systems. The type of resin cement revealed a significant influence on bond strengths to FRC posts. Previous studies[11,19,20] also described a significant influence of the type of resin cement on bond strengths. The mentioned studies revealed increased bond strengths of resin cements containing functional phosphate monomers. Nevertheless, the observed differences in bond strengths in the present study were in a range of 2 to 3 MPa; therefore, the clinical relevance of these results can be considered of minor importance.

Two different silane coupling agents were selected for the present investigation: 3 single-phase preactivated solutions (3-methoxysilylpropylmethacrylate and aminosilane) and a 2-component system (Porcelain Bond Activator/Ed Primer, Kuraray). Hydrolysis of this 2-component system occurs by mixing the silane coupler (3-MPS) with the acidic monomer (10-methacryloyloxydecyl-dihydrogen phosphate) just before application. Preactivated silane solution may exhibit a higher rate of hydrolysis compared to 2-component systems. In the latter this could result in an incomplete reaction if the solvent is not completely evaporated, affecting the bond strengths.[21]

The effects of silanization on bond strengths between fiber posts and resin cements have been debated. Various studies reported no effect on bond strengths after silanization of FRC posts,[11,13,14] whereas 2 further studies revealed an increasing effect of silanization on bond strengths to quartz- and glass-fiber posts.[10,12] The present investigation revealed a significant overall influence of silanization on bond strengths to FRC posts. When comparing the data, the observed difference was 1 MPa, and the statistical significance in this case is mainly attributed to the high number of samples. Therefore, the observed influence of silanization on bond strengths to FRC posts in the present investigation seemed to be clinically negligible.

Silane solutions are hybrid organic-inorganic compounds that can establish adhesion between organic and inorganic matrices by means of an intrinsic dual reactivity.[9] Therefore, a chemical coupling at the FRC post–resin cement interface is only possible between the resin cement and exposed fibers or filler particles of the post. Because of the differences in chemistry, no bonding is expected to occur between the methacrylate-based resin of the cements and the epoxy resin matrix of prefabricated FRC posts.[22] A recently published study revealed promising results in conditioning prefabricated epoxy resin–based FRC posts with different solutions, eg, potassium permanganate and hydrogen peroxide followed by silanization.[23] In a further study of the same working group a removal of epoxy resin was described after 20-minute application of 10% hydrogen peroxide or 10-minute application of 24% hydrogen peroxide to a depth of 50 µm, leaving undamaged fibers for silanization.[22] In both studies conditioning of the surface of the FRC posts followed by silanization enhanced bond strengths to resin core buildup materials.

The type of post revealed a significant influence on bond strengths in the present study. The IPN post demonstrated significantly higher bond strength values with all investigated resin cements. This corresponds with another study that demonstrated higher bond strengths of an individually formed IPN post compared to an FRC post with a cross-linked polymer matrix cemented into root canals of human molars.[6] In contrast to the other investigated groups the IPN post used in that study revealed no adhesive failures between post and cement, demonstrating a better adhesion of the resin cements to this post.

The surface of an FRC post with a cross-linked polymer matrix is well polymerized, and only small reactivity is left for free-radical polymerization bonding. This might explain the lower bond strength values of the prefabricated glass- and quartz-fiber posts in the present investigation. The IPN post consists of linear and cross-linked polymer phases. The linear phase (PMMA) can be dissolved by bisphenol glycidyl methacrylate (bis-GMA)–based adhesive resins, and an interdiffusion bonding obviously might be established.[6] Thus, the higher bond strength values of the IPN post observed in the present study underscore the superior bonding capacities of this post type.

The failure mode registered in all tested groups was predominantly adhesive between post and cement. This demonstrated the relative weakness of the post-composite bond that appears to be less strong than the resin cement or post itself.

Thermocycling revealed no effect on bond strengths between fiber posts and luting agents; this result may be due to the number of cycles used in the present investigation, and it might be speculated that an increased number of cycles would have revealed pronounced differences. However, information from the literature on the number of cycles necessary is scarce, and more research is clearly warranted in this field.

CONCLUSION

Even if the effects of silanization and resin cement proved to be significant with regard to bond strengths to FRC posts, the clinical relevance of the observed differences can be considered of minor importance. The main influence on bond strengths can be attributed to the type of post. IPN posts seemed to be less susceptible to loss of retention due to the higher bond strength values to resin cements compared to prefabricated FRC posts. Concerning the mechanical properties and clinical performance of IPN posts, further in vitro and in vivo studies are necessary.

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