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Reliability of Fiber Post Bonding to Root Canal Dentin After Simulated Clinical Function *In Vitro*

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Clinical Relevance

The present data generated *in vitro* suggests that retention of fiber posts may be reduced after clinical function and that the adhesive interface inside the root canal undergoes certain degradation processes. Therefore, endodontically treated teeth restored using fiber posts may benefit from additional reinforcement via coronal restoration using adequate ferrules and/or adhesive techniques.

SUMMARY

The aim of this study was to investigate the effect of thermomechanical loading (TML) on the bond strength of fiber posts luted with three different resin cements.

Sixty-six extracted human anterior teeth were endodontically treated and restored with fiber posts (RelyX Fiber Posts, 3M ESPE) using three commercially available resin cements and three corresponding core build-up materials (n=22 each): Panavia F 2.0/Clearfil DC Core Automix (Kuraray), Variolink II/Multicore Flow (Ivoclar Vivadent), and RelyX Unicem/Filtek Z250 (3M ESPE). Twelve specimens of each group received all-ceramic crowns and were subjected to TML. The other 10 specimens were stored in saline solution for 24 hours. The roots were sectioned and bond strength was measured using a push-out test. Adhesive interfaces of two specimens of each group subjected to TML were analyzed using field emission scanning electron microscopy (FES-EM).

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Bond strengths of fiber posts were significantly affected by the type of resin cement (*p*<0.0005) and TML (*p*<0.0005; two-way analysis of variance). TML significantly reduced bond strengths for all materials ((6.0 (6.2) MPa)) compared with initial bond strengths ((14.9 (10.4) MPa)). RelyX Unicem resulted in significantly higher bond strengths before ((18.3 (10.3) MPa)) and after TML ((9.8 (7.5) MPa)) compared with the other materials (*p*<0.0005; Tukey HSD). Using FESEM, Variolink II and Panavia F demonstrated a hybrid layer partly detached from the underlying resin cement, whereas no hybrid layer was observed for RelyX Unicem.

The decrease in bond strength after TML suggests that retention of fiber posts may be reduced after clinical function. Therefore, endodontically treated teeth that are restored using fiber posts may benefit from additional reinforcement via coronal restorations using adequate ferrules and/or adhesive techniques.

INTRODUCTION

The restoration of endodontically treated teeth using adhesively luted fiber-reinforced composite (FRC) posts is routinely performed in dental practice. Although clinical studies revealed promising results for using fiber posts,[1,2] bonding to root canal dentin is still a challenge because of limited access and visibility, reduced number of dentinal tubules in the apical third of the root, and deposition of cementum and secondary dentin.[3] In addition, the configuration factor, or C-factor, inside the root canal has been shown to be extremely high,[4] and the polymerization of light- or dual-curing resin cements might be hampered due to the varying degree of the light-transmitting ability of translucent fiber posts.[5] The large variety of products on the market for luting fiber posts, along with the intrinsic difficulties of bonding inside the root canal, complicate the selection of a luting strategy that may provide long-lasting bonding to root dentin. Consequently, it is important to investigate the stability of the adhesive interface of fiber posts inside the root canal to gain information about the long-term performance of adhesively luted FRC posts.

Self-adhesive resin cements are easy to handle and provide a time-saving procedure as no etching and bonding steps are required. *In vitro* investigations on the bonding behavior of the self-adhesive resin cement RelyX Unicem (3M ESPE, Seefeld, Germany) have resulted in contradictory findings.[6–11] Another approach is the use of a separate self-etching or an etch-and-rinse adhesive prior to inserting the resin cement inside the root canal. It has been speculated that the use of phosphoric acid inside the root canal might be advantageous with respect to dissolving the thick smear layer.[12] On the other hand, a universal occurrence of interfacial gaps inside the root canal along the hybrid layer surface of etch-and-rinse adhesives was observed using scanning electron microscopy (SEM).[13]

Little is known about the long-term bonding behavior of various luting agents based on different bonding strategies inside the root canal.[14–16] One study indicated an increase of debonding at the tooth restoration margins of various post and core restorations due to mechanical loading[17]; these findings were corroborated by another investigation that revealed a significant reduction of bond integrity after three months of water storage inside the root canal.[18] Conversely, no effects of mechanical cycling on bond strength of fiber posts to root dentin could be detected, although this study revealed effects of mechanical cycling on bond strength of zirconia posts, indicating that post rigidity might influence the effects of mechanical loading.[15] Consequently, the aim of the present study was to investigate the effect of thermomechanical loading (TML) on bond strength of fiber posts luted with a self-adhesive resin cement, a one-step self-etching adhesive, and a two-step etch-and-rinse system. The null hypothesis to be tested was that bond strengths of fiber posts are not affected by TML or type of luting cement system.

METHODS AND MATERIALS

Specimen Preparation

The crowns of 66 extracted human upper central anterior teeth were sectioned at the proximal cementoenamel junction using a diamond blade under constant water cooling. Root canal preparation was performed at a working length of −1 mm from the apical foramen using FlexMaster rotary instruments (VDW, Munich, Germany) with a crown-down technique. Apical enlargement was performed to size 0.02/50, and the teeth were filled by means of cold lateral condensation using gutta-percha points (VDW) and AH Plus (Dentsply DeTrey, Konstanz, Germany) as a sealer and stored in water for 24 hours.

The specimens were randomly divided into three groups of 22 teeth each. The root canals were enlarged with a slow-speed drill provided by the manufacturer of the selected post system (RelyX Fiber Post Size 2, 3M ESPE). The depth of the post

Table 1: *Composition of Materials Used*

Luting Agent (Lot No.)	Bonding Agent (Lot No.)	Core Build-up Material/ Adhesive (Lot No.)	Manu-facturer	Composition of Composite Resins	Composition of Primers	Composition of Core Build-up/ Adhesive	Irrigation After Post Space Prepar-ation	Application of Resin Cement
Panavia F 2.0 (41173)	Ed Primer (41173)	Clearfil DC Core Automix (039AA) Ed Primer (41173)	Kuraray, Osaka, Japan	Barium glass powder, sodium fluoride, dimethacrylate, 10-MDP, silica, benzoyl peroxide, amine, sodium aromatic sulfinate	10-MDP, HEMA, N-methacryl 5-aminosalcylic, sodium benzene sulfinate, N,N′diethanol p-toluidine, water	Silanated glass and silica, Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, dl-camphor-quinone, benzoylperoxide	CHX 0.2%	Cement onto the post surface and into the orifice of the canal
Variolink II (base: K09191, catalyst: K0511)	Excite DSC (H9851)	Multicore Flow (I22087) AdheSE (K0319)	Ivoclar Vivadent, Schaan, Liechtenstein	Bis-GMA, UDMA, TEGDMA, ytterbium trifluoride, barium glass, silica	HEMA, Bis-GMA, dimethacrylate, phosphonic acid acrylate, silica, ethanol, catalysts, stabilizers	**Multicore:** dimethacrylate, barium glass, fillers, Ba-Al-fluorosilicate glass, silicon dioxide, ytterbium trifluoride, catalysts, stabilizer, pigments **AdheSE:** phosphonic acid acrylate, Bis-acrylamide, water, initiators, stabilizer, dimetha-crylates, HEMA, silicon dioxide, initiators, stabilizer	CHX 0.2%	Cement onto the post surface and into the orifice of the canal
RelyX Unicem Aplicap (290958)		Filtek Z250 (7XN) Adper Prompt L Pop (292918)	3M ESPE, Seefeld, Germany	Glass powder, calcium hydroxide, methacrylated phosphoric ester, dimethacrylate, initiators	No primer	**Filtek Z250:** zirconia and silica fillers, Bis-GMA, UDMA, Bis-EMA **Adper Prompt LPop:** Methacrylic phosphates, Bis-GMA, photoinitiator camphor-quinone, water, HEMA, polyalkenoic acid polymer	NaOCl 1%, H_2O	Cement was applied into the canal using the provided elongation tip

Abbreviations: 10-MDP, 10-methacryloyloxydecyl dihydrogenphosphate; BIS-EMA, ethoxylated bisphenol A glycol dimethacrylate; Bis-GMA, bisphenol A diglycidyl methacrylate; CHX, chlorhexidine digluconate; HEMA, 2-hydroxyethyl methacrylate; NaOCl, sodium hypochlorite; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

space preparation was 8 mm. Irrigation was performed after post space preparation according to the manufacturer's recommendations (Table 1).

RelyX Fiber Posts (3M ESPE) were tried in and inserted with one of the following three different resin cements according to the manufacturer's instructions: 1) Panavia F 2.0 (Kuraray, Osaka, Japan), 2) Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein), and 3) RelyX Unicem (3M ESPE). The posts were inserted into the canal, excess was removed, and light curing was performed using an LED curing unit (1200 mW/cm^2; Elipar Freelight 2, 3M ESPE) according to the manufacturer's recommendations. Light intensity of the light-curing unit was checked prior to use (LED radiometer, Demetron, Kerr, Orange, CA, USA). The core build-ups were conducted using the corresponding core build-up materials listed in Table 1. Twelve teeth of each group received preparations for all-ceramic crowns, including a circumferential 1.2-mm shoulder and a ferrule of 2 mm. With the help of silicone impressions of the original crowns, 36 lithium-disilicate ceramic crowns were fabricated (IPS e.max Press, Ivoclar Vivadent) and adhesively luted using the same materials used for post luting. Light curing was performed for 40 seconds from each surface (Elipar Freelight 2, 3M ESPE). After seven days of storage in distilled water at 37°C, the specimens were subjected to thermomechanical fatigue including 5000 thermal cycles (5°C/55°C, two minutes each cycle) and 1.2×10^6 mastication cycles at an angle of 135°. A force of 50 N was applied 3 mm below the incisal edge on the palatal surface of the crown (EGO chewing simulator, EGO Kältetechnik GmbH, Regensburg, Germany). After thermomechanical fatigue, the specimens were stored again for 21 days in distilled water at 37°C. The other 10 teeth of each group were stored in 100% humidity without crowns for 24 hours to allow for complete polymerization.

Push-Out Testing

The roots were sectioned perpendicular to the long axis of the root into four slices (thickness of 1 mm) using a band saw (Exakt Apparatebau, Norderstedt, Germany), and for 10 specimens per group, micro push-out testing was performed (Universal testing machine, Zwick, Roell, Ulm, Germany) at a crosshead speed of 0.5 mm/min. With regard to the tapered design of the post, three different sizes of punch pins as well as three different openings were used for the push-out testing.[6] The maximum stress was calculated from the recorded peak load divided by the computed surface. To calculate the exact bonding surface, the tapered design of the posts with regard to the respective part of the post was considered. Therefore, each specimen was measured with a micrometer screw (Mitutoyo Messgeräte GmbH, Neuss, Germany), and the bonding surface was calculated using the formula of a conical frustrum: $\pi(R_1 + R_2)\sqrt{(R_1 +^{11} R_2)^{22} 2 + h^2}$. After the push-out test, each specimen was observed using a stereomicroscope (DV 4, Zeiss, Jena, Germany) at 40× magnification to determine the failure mode. The specimens were divided into four groups according to the failure modes: 1) adhesive failures between dentin and cement, 2) adhesive failures between post and cement, 3) mixed failures, and 4) cohesive failures inside the post.

SEM Analysis

The slices of two sectioned specimens per group that were subjected to TML were fixated in 2.5% glutaraldehyde at pH 7.4 for 12 hours at 4°C,[19] dehydrated in ascending concentrations of ethanol, and polished. Subsequently, the interfaces were treated with 6 N hydrochloric acid for 30 seconds followed by a 10-minute immersion in 2.5% sodium hypochlorite and coated with Au-Pd (DV 502A Vacuum Evaporator), and secondary images were obtained at 5 kV using a field emission scanning electron microscope (FESEM; S-4700, Hitachi High Technologies America Inc, Pleasanton, CA, USA).

Statistical Analysis

Statistical analysis was performed using SPSS version 16.0 software (SPSS, Chicago, IL, USA). The alpha (Type I) error level was set to 0.05. To achieve normality in the study groups, bond strengths were subjected to a suitable Box-Cox transformation before analysis. Then the effects of resin cement and TML on bond strength were analyzed using two-way analysis of variance (ANOVA) and Tukey HSD post hoc test. The effects of the materials on the failure modes were investigated using Pearson's chi-square test.

RESULTS

Bond strengths of fiber posts were significantly affected by the type of resin cement ($p<0.0005$) and TML ($p<0.0005$; two-way ANOVA). TML significantly reduced bond strengths for all materials ((6.0 (6.2) MPa)) compared with initial bond strength values ((14.9 (10.4) MPa)). RelyX Unicem revealed significantly higher bond strengths before ((18.3 (10.3) MPa)) and after TML ((9.8 (7.5) MPa)) compared with Panavia F before ((13.2 (9.5) MPa))

Table 2: *Analyses of the Failure Modes (in Percentages) in Each Group*[a]

Resin Cement	Thermomechanical Loading	Failure Mode, %					*p* Value
		I Adhesive Cement Dentin	II Adhesive Post Cement	III Mixed	IV Cohesive Post	Not to Assess	
RelyX Unicem	Initial	20	78	2	0	0	$p<0.0005$
	TML	58.5	19.5	19.5	0	2.5	
Panavia F 2.0	Initial	28	68	4	0	0	$p=0.005$
	TML	43.9	36.6	19.5	0	0	
Variolink II	Initial	72	20	8	0	0	p=0.148
	TML	65.1	25,6	9.3	0	0	

[a] *$p<0.05$ indicates significant effects of thermomechanical loading (TML) on the failure modes (Pearson).*

and after TML ((3.5 (2.8) MPa)). Rely X Unicem also resulted in significantly higher bond strengths than Variolink II before ((13.2 (10.6) MPa)) and after TML ((4.8 (5.5) MPa)); ($p<0.0005$; Tukey HSD).

Analyses of failure modes with respect to the resin cement and TML are presented in Table 2. TML significantly affected the failure modes of the materials RelyX Unicem ($p<0.0005$) and Panavia F 2.0 ($p=0.005$) but not the failure modes of Variolink II ($p=0.148$; Pearson's chi-square test).

Representative images for Variolink revealed a distinctive hybrid layer and numerous resin tags (Figure 1a). Higher magnification revealed a detached hybrid layer from the underlying resin cement (Figure 1b). For the self-etching primer, ED Primer/Panavia F 2.0, a narrow hybrid layer was observed (Figure 1c,d); however, parts of the hybrid layer were also detached from the underlying resin cement. The self-adhesive resin cement RelyX Unicem showed no hybrid layer formation under the magnification used in this study, and scarce penetration into dentinal tubules could be observed (Figure 1e,f).

DISCUSSION

The null hypothesis of the present study had to be rejected since the resin cement and TML significantly affected bond strengths of fiber posts inside the root canal.

The resin cements of the present study were selected according to different bonding strategies and were all dual-cure resin cements. Light polymerization of these cements leads to a higher conversion rate,[20] and this might affect their physical-mechanical properties.[21] However, a decrease in light intensity from the coronal to the apical aspect of translucent fiber posts has been reported, and this could affect the curing efficacy of resin composites in the depth of the root canal.[5] Consequently, the bond strengths of the luting agents used in the present study could have been affected by a possible insufficient degree of conversion of the luting agents. However, this assumption could not be supported by the results of the failure mode analysis, for which a low percentage of cohesive cement failures was observed. Consequently, this aspect should be evaluated in further studies.

The thin push-out test is considered as a valid method to analyze the bond strengths of fiber posts to root canal dentin.[22] Nevertheless, the exposure of the fiber post to the dislodging forces during the push-out test cannot be compared with functional forces during clinical service.[10] Moreover, the sectioning process, especially of the teeth subjected to TML, may induce artifacts that could influence the test results. In addition, remnants of root canal obturation materials left inside the root canal and accessory canals[23] could affect bond strengths of

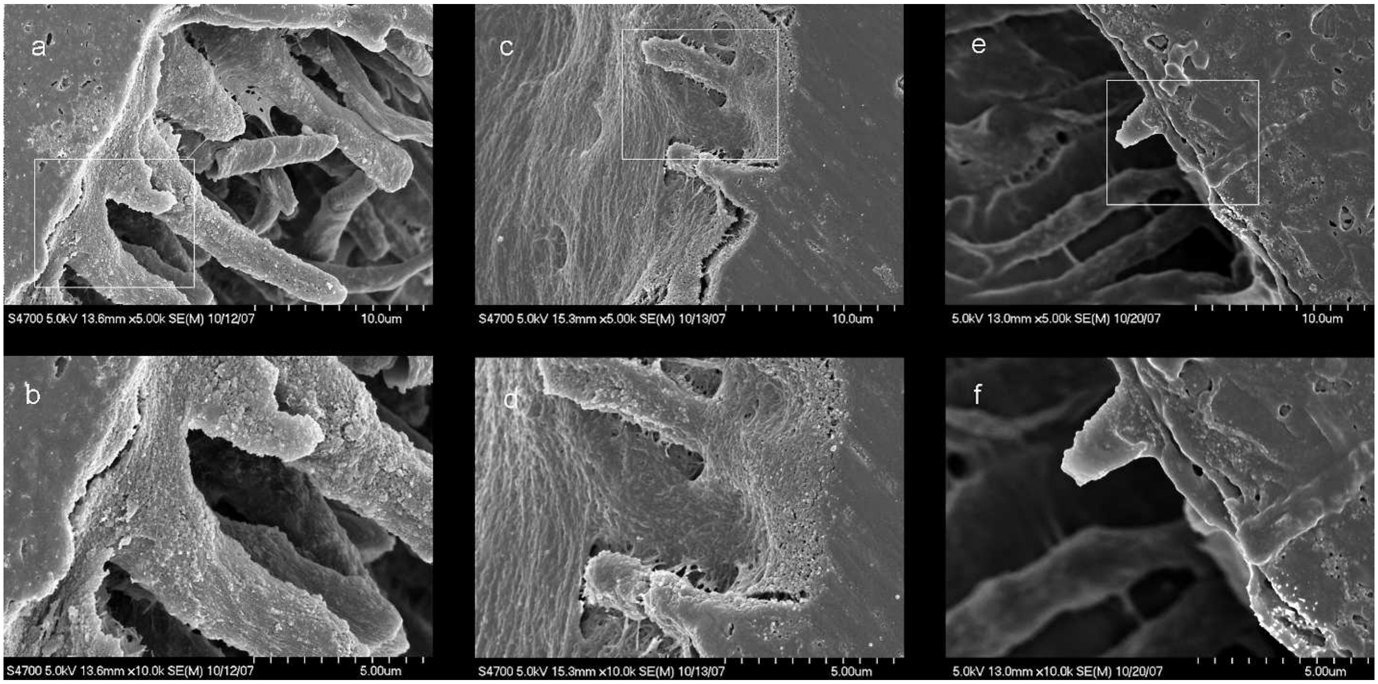

Figure 1. *(a): Representative images for Variolink revealed a distinctive hybrid layer and numerous resin tags. (b): Higher magnification depicted a detached hybrid layer from the underlying resin cement. (c, d): For the self-etching primer, ED Primer/Panavia F 2.0, a narrow hybrid layer could be detected; parts of the hybrid layer were also detached from the underlying resin cement. (e, f): The self-adhesive resin cement RelyX Unicem showed no hybrid layer formation with the magnification used in this study, and scarce penetration into dentinal tubules could be observed.*

fiber posts inside the canal. This would explain, at least partially, the high standard deviation that was observed in some groups.

The aim of the present study was to simulate and accelerate aging *in vitro* in a clinically relevant manner to assess the long-term behavior of adhesively luted fiber posts. Consequently, the specimens subjected to TML were restored using all-ceramic crowns to ensure a clinically relevant loading to mimic the clinical situation as closely as possible. In the control group, the initial push-out bond strength was measured after 24 hours. This would not allow enough time for the fabrication of crowns in this group. However, there might be a possibility of either mechanical disruption during preparation or stresses from crown cementation affecting the cement-post interface. These possible effects cannot be detected in the design used for this study.

A significant decrease was observed in bond strength after TML of all samples irrespective of the resin cement used. Changes in the bonded interface *in vivo* may be caused by flexure of the restored tooth under occlusal stresses. Because of water sorption at the resin-dentin interface, fluid movement may occur at the junction of adhesive resin, hybrid layer, and dentin during loading of the restoration. This could result in mechanical and chemical degradation of the cured resin.[24] The degree of degradation has been shown to be related to the chemical composition of the monomers, the degree of conversion, and the degree of cross-linking in the polymerized matrix.[25] Moreover, degradation of dentin-adhesive interfaces might be caused by discrepancies between the depth of acid demineralization and that of hydrophilic monomer infiltration. This may result in unprotected collagen fibrils at the bottom of the hybrid layer that can be hydrolyzed by the free water entrapped around the collagen fibrils.[26] It has also been demonstrated that the bottom of the hybrid layer formed by self-etching adhesives contains unprotected collagen fibrils.[27] The activity of bacteria-produced collagenases, as well as the activation of host-derived matrix metalloproteinases, has been shown to contribute to the degradation of the adhesive interface.[28,29] This could compromise the long-term bonding behavior of all investigated systems after TML.

A recent study also revealed a significant decrease in bond strength after thermocycling (40,000 cycles, representing approximately four years of functional service[30]) for the self-adhesive resin cement RelyX Unicem and the self-etch adhesive system Ed Primer/Panavia F 2.0, but not for the etch-and-rinse strategy XP Bond/CoreXFlow.[31] The initiator-catalyst system in the self-cure activator of XP Bond might promote adhesion of compatible dual-curable resin-based luting agents to the adhesive layer and accelerate their polymerization.[32] However, RelyX Unicem also revealed significantly higher bond strength values after thermocycling compared with Panavia F 2.0 as observed in the present study.[31]

The results of the present study corroborate those of a previous investigation that demonstrated distinctive nanoleakage patterns for the etch-and-rinse system Excite DSC/Variolink II and the self-etch adhesive Ed Primer/Panavia F 2.0 up to 0.8 mm inside the root canal after TML, whereas the self-adhesive resin cement RelyX Unicem was able to prevent distinctive leakage at this penetration depth.[33] In that study, none of the luting systems was able to hermetically seal the root canal if leakage occurred around the margins of the coronal restoration, which correlates well with the observed decrease in bond strengths for all materials in the present study. Conversely, Bottino and others reported no effect on the push-out bond strength of adhesively luted FRC posts after 2,000,000 cycles of mechanical loading compared with an unloaded control group.[15] This loading protocol was conducted without thermocycling, which is known to challenge the adhesive interface both chemically (hot water accelerates hydrolysis and elution of the interface components) and mechanically (repetitive contraction and expansion stresses), besides enhancing the effect of temperature and water-mediated aging phenomena.[34]

In the present study, the self-adhesive resin cement RelyX Unicem showed higher bond strengths compared with the other materials investigated. The results are in the same line as previous investigations.[6,7,35] Another study also found significantly higher push-out bond strengths of RelyX Unicem compared with Panavia F 2.0.[10] In contrast, other studies demonstrated either a similar bond strength for the self-adhesive resin cement compared with a self-etch approach or an etch-and rinse approach[11,31] or even lower bond strength values for the self-adhesive resin cement compared with a self-etch approach or an etch-and-rinse adhesive system.[8] In the last mentioned study, RelyX Unicem was used in the self-cure mode. The low degree of conversion of this cement in the self-cure mode[36] may have contributed to the lower bond strength values.

The bonding mechanism of RelyX Unicem differs from that of self-etching adhesives since no distinct demineralization and hybridization were observed upon Transmission Electron Microscopy morphological interface examination.[8] In addition, RelyX Unicem resulted in a shallow demineralization despite its low initial pH.[37] This is in agreement with the present FESEM analysis since no hybrid layer formation was observed under the magnification used and scarce penetration of the cement into dentinal tubules was observed (Figure 1e,f). The use of the self-adhesive resin cement RelyX Unicem with the RelyX Fiber Post could have contributed to the favorable bond strength of this cement in the present study since the manufacturer claims both chemical compatibility and strong micromechanical interlocking of this post-cement system. Despite the high bond strength of RelyX Unicem in the present study, the predominant failure mode of this cement was adhesive between post and cement before TML, indicating that the weak part of this interface was still between post and cement. For the luting agents RelyX Unicem and Panavia F 2.0, the predominant failure mode changed significantly after TML from adhesive between post and cement into adhesive between dentin and cement. This change corroborates the respective reduction in bond strengths and the potential degradation of the resin-dentin interface.

All materials were applied into the root canal according to the manufacturers' recommendations. RelyX Unicem was the only material that was inserted using a flexible root canal–shaped application tip, whereas all other materials were applied onto the post surface as well as into the orifice of the canal prior to post insertion. Although the direct application of resin cements with a syringe has been shown not to affect the bond strengths to root dentin of etch-and-rinse resin cements,[38] it could have contributed to the favorable results of RelyX Unicem, since the use of this application aid reduced the number of imperfections within the self-adhesive cement interface compared with the conventional application technique.[39]

The self-etching ED Primer of Panavia F 2.0 is a one-step adhesive. These adhesives have been described as behaving like semipermeable membranes after polymerization because of their higher concentration of hydrophilic monomers and the lack of the subsequent application of a more hydrophobic resin coating.[40] Water sorption by hydrophilic resin monomers within resin-dentin interfaces could contribute to their degradation over time[26] and affect the bond durability of one-step self-etching adhesives. Hydrophilicity and hydrolytic stability are antagonistic properties,[41] and this could explain the reduction of bond strength after TML for this cement despite the ability of functional monomers, ie, 10-methacryloxydecyl dihydrogen phosphate, present in ED Primer that may interact with hydroxyapatite to form calcium salts and contribute to a stable bond.[42]

The strong etching effect of phosphoric acid totally removes mineral crystals and exposes the collagen fibers.[43] The subsequently applied adhesive must infiltrate around these fibers. Porosities within an incompletely infiltrated hybrid layer, any discrepancy between the etching depth and the following resin infiltration depth, as well as technique sensitivity could have contributed to the low bond strength values of Excite DSC/Variolink II in the present study after TML. Nevertheless, this was the only etch-and-rinse cement evaluated in this study; thus, the current findings should not be generalized to all etch-and-rinse cements.

CONCLUSIONS

The decrease in bond strength after TML suggests that retention of fiber posts inside the root canal may be reduced after clinical function. Therefore, endodontically treated teeth that are restored using fiber posts may benefit from additional reinforcement via coronal restorations using adequate ferrules and/or adhesive techniques.

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