Role of Chlorhexidine on Long-term Bond Strength of Self-adhesive Composite Cements to Intraradicular Dentin

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Purpose: To examine the effect of CHX pre-treatment on long-term bond strength of fiber posts luted with self-adhesive resin cements.

Materials and Methods: Seventy-two single-rooted teeth were selected for root canal treatment and post space preparation. The tested self-adhesive cement/post combinations were (N = 36): 1. RelyX Fiber-Posts luted with RelyX Unicem; 2. Rebilda Posts luted with Bifix SE Cement. For both self-adhesive cements, half of the specimens (experimental groups) were luted after the application of a solution of 2% CHX, while no CHX application was performed for the remaining specimens (control groups). Luted specimens were cut and used for push-out bond strength evaluation immediately, and after storage in artificial saliva for 6 months or 1 year. Additional specimens were processed for quantitative interfacial nanoleakage analysis.

Results: ANOVA showed that the variable times of storage had a significant influence on the results ($p < 0.05$), while no influence of the luting procedure (cements with or without CHX) on the final outcome ($p > 0.05$) was found. Tukey's pairwise post-hoc test showed that the radicular bond strength decreased with time of storage. In particular, a significant difference was found between T0 and T1y, but not between T0 and T6m. In contrast, in terms of pretreatment, no significant reduction in push-out bond strength was observed, irrespective of the aging time.

Conclusion: CHX pretreatment did not prevent bond strength degradation of fiber posts luted with self-adhesive cements.

Keywords: chlorhexidine, dentin bonding agents, fiber posts, nanoleakage analysis, push out, self-adhesive cements.

J Adhes Dent 2017; 19: 341–348.
doi: 10.3290/j.jad.a38896

Submitted for publication: 27.10.16; accepted for publication: 09.07.17

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Fiber-reinforced posts in combination with bonding materials are routinely employed to restore endodontically treated teeth.[10] Because the elastic moduli of fiber posts and radicular dentin are similar, they have been advocated for creating tooth-restoration units with improved resistance to dislodgement.[15] Quartz-fiber posts also fulfill contemporary esthetic dentistry demands for metal-free restorations.[37]

As the success of post-supported restorations relies on dentinal adhesion, the choice of appropriate bonding agents and resin-based materials for luting fiber posts to root canal dentin is a challenge that may affect the longevity of these restorations.[18] Several factors can adversely affect bonding to root canals, including the method of root canal instrumentation,[2,39] the type of adhesive, the mode of composite polymerization, and the presence of a difficult-to-bond intraradicular dentin substrate after the use of sodium hypochlorite as an oxidizing intracanal irrigant.[4,16,37]

Fiber posts may be cemented using dual-curing resin-based cements in combination with etch-and-rinse or self-etch adhesives, or using the recently formulated self-adhesive cements (ie, all-in-one resin-based materials) that are purported to achieve simultaneous bonding between the intraradicular dentin and the post.[26] The etch-and-rinse strategy requires a wet dentin substrate for optimal bonding,[34] although wetness within the root canal is difficult to control. The self-etch approach does not require rinsing, thereby overcoming the problem of substrate moisture control and simplifying the clinical procedures, since phosphoric acid etching is not required.[17,45] The self-adhesive cements allow simultaneous bonding between the fiber post and intraradicular dentin, further simplifying clinical procedures and reducing technique sensitivity.[1,33]

Bond longevity and stability of the adhesive/dentin interface are adversely affected by physical and chemical factors.[6,8] Even when the coronal seal is effective, degradation of the composite-dentin interface may occur through activation of endogenous dentin matrix metalloproteinases (MMPs) that are present both in the coronal and radicular dentin.[27,28,31,36,42] Previous studies have shown that these enzymes contribute to the degradation of suboptimally impregnated collagen fibrils within the hybrid layer, and expedite the degradation of the adhesive interface over time.[6,11]

Chlorhexidine (CHX), a non-toxic MMP inhibitor, has been proposed for the preservation of bond stability over time used in combination with the dentin adhesives.[5,12,19,25,31,43] Moreover, CHX also has been used as an irrigant in post endodontic restorations because of its antibacterial activity, substantivity, and biocompatibility.[13,21] However, CHX ability to preserve the bond strength created by self-adhesive cements to intraradicular dentin has not been fully clarified.

Thus, the aim of the present study was to examine the effect of 2% CHX pretreatment on the stability of composite-dentin bonds created by self-adhesive cements used for luting fiber posts. The null hypotheses tested were that pre-treatment of post spaces with 2% CHX has no effect on either 1) the bond strength of self-adhesive composite cements to intraradicular dentin over time or 2) the interfacial nanoleakage expression along the bonded interface after aging.

MATERIALS AND METHODS

Seventy-two human incisors extracted for periodontal reasons were selected for the study. Informed consent was obtained, according to the protocol approved by the University of Trieste, Italy. The inclusion criteria were the absence of radicular caries or fractures, no previous root canal treatment, post or crown, and a minimum root length of 13 ± 3 mm. The teeth were stored at 4°C in 1% chloramine-T solution and used within 1 month after extraction. Adhering soft tissue and debris were cleaned off and the crowns were removed using a low-speed diamond saw under water cooling (Micromet, Remet; Bologna, Italy). Cleaning and shaping was performed with a crown-down technique using FlexMaster rotary instruments on an E-Master handpiece (Dentsply DeTrey; Konstanz, Germany). Irrigation was performed with 5% NaOCl and 17% EDTA during cleaning and shaping of the root canal. The working length was established 1 mm coronal to the anatomical root apex. The cleaned and shaped root canals were obturated with gutta-percha and an epoxy resin-based sealer (AH Plus; Dentsply), using continuous-wave warm vertical compaction. Backfilling was performed using System B (Elements Obturation Unit, SybronEndo; Orange, CA USA).

The specimens were randomly divided into four groups (n = 18). Each group was treated with one of the following self-adhesive cement/post combinations:

- Group 1: An 8-mm post space was created using calibrated burs (3M ESPE), then irrigated with 0.2 ml of 2% CHX digluconate (Bisco; Schaumburg, IL, USA) for 1 min. The post space was dried with adsorbent paper points, followed by application of RelyX Unicem (3M ESPE; St Paul, MN, USA) to lute a RelyX FiberPost (3M ESPE; size: yellow; length: 20 mm; coronal diameter: 1.30 mm; apical diameter: 0.70 mm), in accordance with the manufacturer's instructions.
- Group 2: An 8-mm post space was created using calibrated burs (3M ESPE) and then no CHX pre-treatment was employed. RelyX Unicem was applied to the post space to lute a RelyX FiberPost. The latter had the same dimensions as those used in Group 1.
- Group 3: An 8-mm post space was created using calibrated burs (Voco; Cuxhaven, Germany) and then each post pace was irrigated with 0.2 ml of 2% CHX digluconate (Bisco) for 1 min. The post space was dried with adsorbent paper points, followed by application of Bifix SE Voco (Voco) to lute a Rebilda Post (Voco; size: green; length: 19 mm; coronal diameter: 1.20 mm; apical diameter: 0.65 mm), in accordance with the manufacturer's instructions.
- Group 4: An 8-mm post space was created using calibrated burs (Voco) and then no CHX pre-treatment was employed. Bifix SE Voco was applied to the post space to lute a Rebilda Post. The latter had the same dimensions as those used in Group 3.

Specimen Preparation for Push-out Bond Strength Evaluation

Fifteen specimens from each group were designated for micro-push-out bond strength evaluation in accordance with Mazzoni et al.[26] After 24 h, the root portions corresponding to the bonded fiber posts were sectioned transversely into five to six 1-mm-thick serial slices using a low-speed saw under water cooling (Micromet, Remet). The apical surfaces of the slices were marked with a dot using permanent black ink, then the slices were categorized as coronal (2 slices), middle (2 slices), and apical (1 or 2 slices), and randomly assigned to three different storage times: 24 h (T0), 6 months (T6m) or 1 year (T1y). Slices were incubated in artificial saliva at 37°C, and the storage medium of T6m and T1y specimens was changed every week.

The micro-push-out bond strength test was performed on these slabs using a universal testing machine (Sun 500

Table 1 Composition and application mode of the self-adhesive cements tested in the study

Luting agent	Composition of the luting agent	Filler (wt%)	Fiber Post	Adhesive luting procedure
RelyX Unicem (3M ESPE)	Methacrylated phosphoric esters Dimethacrylates Acetate Substituted pyrimidine Peroxy compound Sodium persulfate Initiator Stabilizer	Glass powder, fumed silica, calcium hydroxide (72 wt%)	RelyX Fiber	Mix the capsule; apply RelyX Unicem cement directly into the post space through a disposable application tip attached to the capsule; place the post; leave the cement to auto-cure for 5 min and then light cure for 40 s.
Bifix SE (VOCO)	Bis-GMA UDMA Gly-DMA Phosphate monomers Initiators Stabilizers	Glass (70 wt%)	Rebilda Post	Apply directly Bifix-SE into the post space a disposable application mixing tip; place the post; light cure for 40 s.

Galdabini; Milano, Italy) connected to a load cell, operating at a crosshead speed of 1 mm/min. The apical surface displaying the ink dot was placed facing the punch tip, ensuring that load forces were applied from an apical to coronal direction. Bond failure was manifested by the dislodgment of the fiber post from the root section. Push-out bond strength data were converted to MPa by dividing the load in Newton by the bonded surface area (S_L) calculated at the dentin adhesive in mm^2; S_L was calculated as the lateral surface area of a truncated cone using the formula: $S_L = (R+r) \times [((h^2 + (R-r])^2]^{0.5}$, where R is the coronal post radius, r the apical post radius, and h the thickness of the slice. The widest and narrowest diameters of the post and the thickness of the slice were individually measured using a digital caliper with 0.01-mm accuracy.

All fractured specimens were analyzed with a stereomicroscope at 60X (Stemi 2000-C, Carl Zeiss; Jena, Germany) to determine whether the failure mode was adhesive between luting cement and dentin (AD), adhesive between luting cement and post (AP), cohesive within the luting cement (CC), or mixed (M). The maximum failure load was recorded in Newton (N) and converted into MPa in accordance with Mazzoni et al.[26]

As values were normally distributed according to the Kolmogorov-Smirnov test, the data were analyzed with three-way ANOVA to examine the effects of the luting cement, chlorhexidine application, and storage time, as well as the interaction of these factors on push-out bond strength. Post-hoc pairwise comparisons were performed using Tukey's test. The chi-squared test was used to analyze differences in the failure modes. For all tests, statistical significance was pre-set at $\alpha = 0.05$.

Interfacial Nanoleakage Analysis

For each group, three teeth (1 per aging group) were used for the evaluation of interfacial nanoleakage expression in accordance with Mazzoni et al.[26] Specimens were cut into 1-mm-thick slabs as previously described and not further demineralized, then the unembedded sections were covered with nail varnish, leaving 1 mm of root dentin exposed along the luting cement-dentin interface. The specimens were immersed in 50 wt% ammoniacal silver nitrate solution for 24 h, followed by immersion in a photo-developing solution in accordance with Tay et al.[41] Sections were bonded on glass slides, ground to a thickness of approximately 40 µm under running water with 600-, 800-, 1200-, and 2400-grit silicon carbide (SiC) paper (LS2; Remet), and observed under normal transmitted light using a light microscope (Nikon Eclipse, Nikon; Tokyo, Japan). The amount of silver tracer deposited along the interface (ie, the degree of interfacial nanoleakage) was scored by two observers in accordance with Saboia et al.[35] In brief, interfacial nanoleakage was scored on a scale of 0 to 4 based on the percentage of adhesive surface showing silver nitrate deposition: 0: no nanoleakage; 1: < 25% with nanoleakage; 2: 25%–50% with nanoleakage: 3: 50%–75% with nanoleakage; 4: > 75% with nanoleakage. Intra-examiner reliability was assessed using the kappa (κ) test. Because the data were not normally distributed, statistical differences among nanoleakage group scores were analyzed with Kruskall-Wallis ANOVA. Pairwise comparisons between group means were made using the Mann-Whitney U-test (level of significance: $p < 0.05$). The level of significance was adjusted according to Bonferroni's correction.

RESULTS

Push-out Bond Strength

Push-out bond strength for the different groups of self-adhesive composite cements is reported in Table 2. Three-way ANOVA showed that the different storage durations had a significant influence on the results ($p < 0.05$), while the other variables – luting cement and pretreatment with CHX – did not influence the final outcome ($p > 0.05$). Tukey's pairwise post-hoc test showed that the radicular bond strength decreased with increasing storage duration; spe-

Table 2 Means and standard deviations of push-out bond strength (in MPa) for each self-adhesive cement and aging interval (0 and 6 months, 1 year) [number of sections tested for each group]

Cements	CHX	Push-out bond strength (n = 5 teeth/group/storage time)		
		T0	T6m	T1y
RelyX Unicem	2% CHX	8.0 ± 4.1^{aA} [29]	8.4 ± 3.2^{aA} [29]	5.5 ± 2.6^{bA} [27]
RelyX Unicem	not used	8.4 ± 4.1^{aA} [26]	8.2 ± 4.2^{aA} [27]	5.4 ± 2.4^{bA} [30]
Bifix SE	2% CHX	6.9 ± 3.5^{aA} [26]	5.5 ± 2.6^{bB} [26]	5.2 ± 1.9^{bA} [29]
Bifix SE	not used	7.2 ± 3.3^{aA} [27]	5.7 ± 2.6^{bB} [29]	5.8 ± 2.3^{bA} [29]

Same superscript lowercase letters in rows indicate no difference between storage time ($p > 0.05$), same superscript uppercase letters in columns indicate no difference between luting procedures ($p > 0.05$).

Table 3 Mode of failure for each self-adhesive cement and aging interval (0 and 6 months, 1 year)*

Cement	CHX	Aging time	(% failure mode)			
			AD	AP	CC	M
RelyX Unicem	2% CHX	T0	62.5	8.3	12.5	16.6
		T6m	50.0	15.0	25.0	10.0
		T1y	47.3	15.7	5.2	31.5
RelyX Unicem	not used	T0	58.8	5.8	5.8	29.5
		T6m	55.5	5.5	33.3	5.5
		T1y	55.5	7.4	14.8	22.2
Bifix SE	2% CHX	T0	64.0	25.0	8.0	8.0
		T6m	64.2	10.7	14.2	10.7
		T1y	72.0	8.0	4.0	16.0
Bifix SE	not used	T0	64.0	25.0	8.0	8.0
		T6m	64.2	10.7	14.2	10.7
		T1y	72.0	8.0	4	16.0

*Percentages of slices demonstrating adhesive failure between luting agent and dentin (AD), adhesive failure between luting agent and post (AP), cohesive failure within the luting agent (CC), or mixed failure (M).

cifically, there is a significant difference between T0 and T1y, but not between T0 and T6m.

Failure mode analysis revealed that the predominant failure mode was adhesive between luting cement and intraradicular dentin (Table 3).

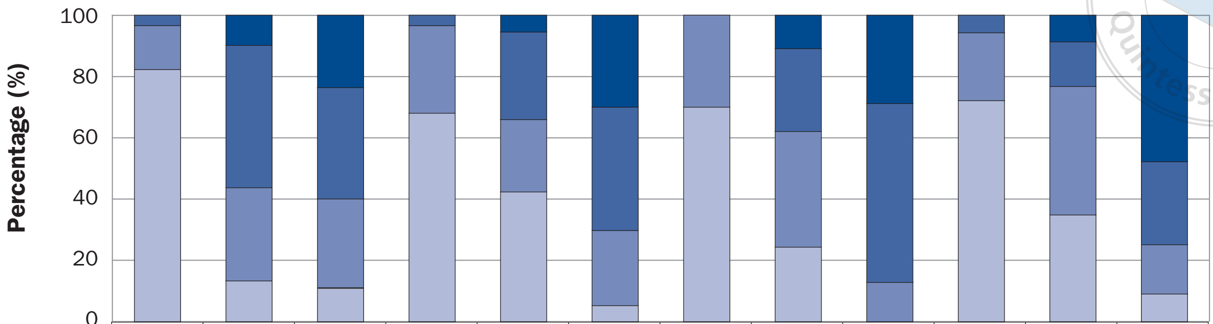
Interfacial Nanoleakage

Nanoleakage expression is reported in Fig 1. At T0, interfacial nanoleakage in specimens pre-treated with CHX (Figs 2a and 4a) was not significantly different from the control group specimens (Figs 3a and 5a) for both self-adhesive cements (Fig 1; $p > 0.05$). Storage in artificial saliva for 6 months (T6m) and 1 year (T1y) significantly increased interfacial nanoleakage expression compared to T0, in both the experimental (Figs 2b, 2c, 4b, 5c) and the control groups (Figs 3b, 3c, 5b, 5c; Fig 1; $p < 0.05$).

DISCUSSION

No difference in push-out bond strength or interfacial nanoleakage expression was observed between specimens pre-treated with CHX and the control specimens, irrespective of the type of self-adhesive resin cement employed and aging time. These results do not justify rejection of the two null hypotheses tested, that pre-treatment of post-spaces with 2% CHX has no effect on the push-out bond strength of self-adhesive resin cements to intraradicular dentin over time, and that pre-treatment of post-spaces with 2% CHX has no effect on the interfacial nanoleakage expression along the bonded interface after aging.

Dentin bonding procedures expose and activate MMPs in both the coronal and radicular dentin, particularly when etch-and-rinse adhesive systems are used.[27,28,30,40] Chlorhexi-



	RelyX Unicem + 2% CHX T0 (N = 39)	RelyX Unicem + 2% CHX T6m (N = 38)	RelyX Unicem + 2% CHX T1y (N = 38)	RelyX Unicem T0 (N = 39)	RelyX Unicem T6m (N = 39)	RelyX Unicem T1y (N = 37)	Bifix SE + 2% CHX T0 (N = 38)	Bifix SE + 2% CHX T6m (N = 39)	Bifix SE + 2% CHX T12 (N = 39)	Bifix SE T0 (N = 39)	Bifix SE T6m (N = 40)	Bifix SE T1y (N = 39)
> 75	0	9	26	0	5	29	0	10	28	0	8	47
50≤75	3	47	37	3	29	41	0	28	59	5	15	28
25≤50	15	30	29	29	24	25	30	38	13	23	42	16
< 25	82	14	11	68	42	5	70	24	0	72	35	9
0	0	0	0	0	0	0	0	0	0	0	0	0

Fig 1 Interfacial nanoleakage expression was evaluated in all groups, and the percentage of sections and nanoleakage scores are listed. Interfacial nanoleakage was scored based on the percentage of the adhesive surface showing silver nitrate deposition. 0: no nanoleakage; 1: <25% with nanoleakage, 2: 25% to ≤ 50% with nanoleakage; 3: 50% to ≤ 75% with nanoleakage; 4: > 75% with nanoleakage. Increasing interfacial nanoleakage expressed by deepening shades of blue. N = number of analyzed sections.

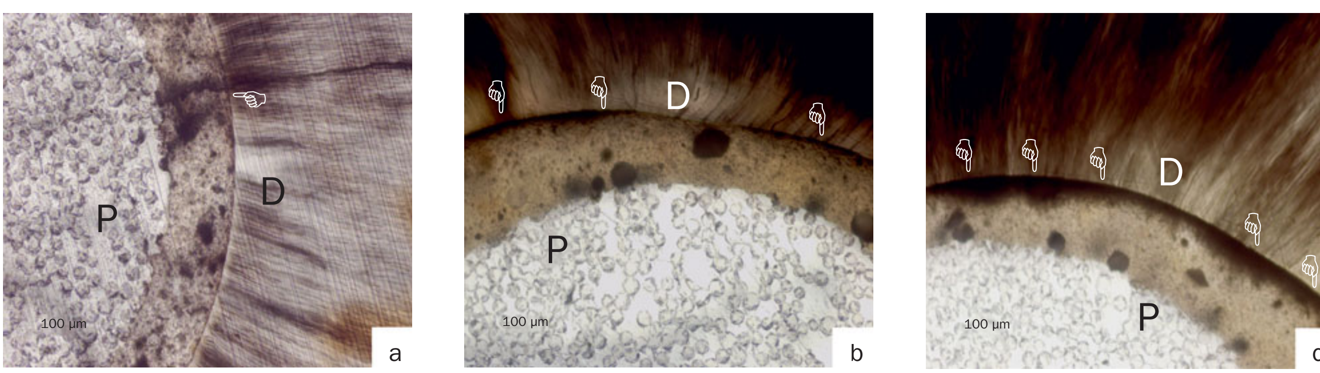


Fig 2 Light micrographs showing the adhesive interface created by RelyX Unicem+2%CHX pretreatment at T0 (a), T6m (b) or T1y (c) of storage in artificial saliva at 37°C. D = dentin; P = fiber post; pointers = silver nitrate deposits. Increasing deposits of silver were found after aging compared to T0 despite CHX pretreatment. Scale bar: 100 µm.

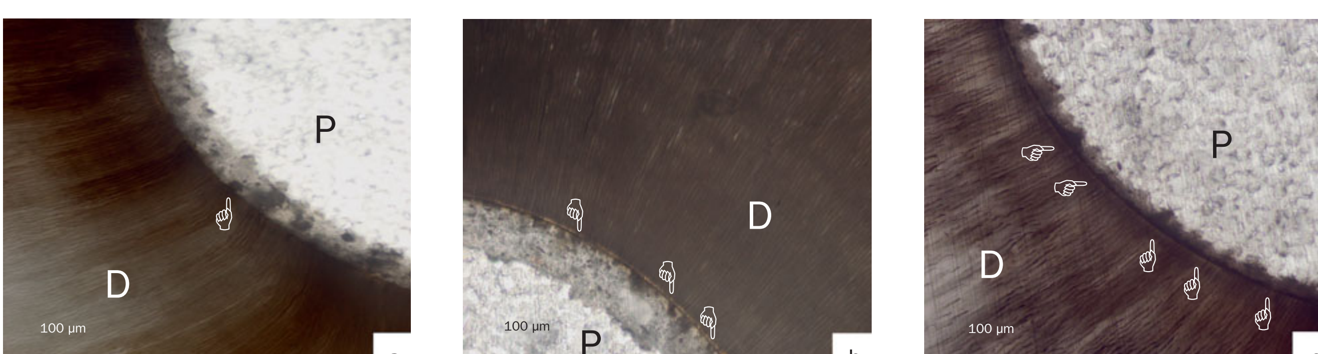


Fig 3 Light micrographs showing the adhesive interface created by RelyX Unicem at T0 (a), T6m (b) or T1y (c) of storage in artificial saliva at 37°C. D = dentin; P = fiber post; pointers = silver nitrate deposits. Nanoleakage significantly increased after aging. Scale bar: 100 µm.

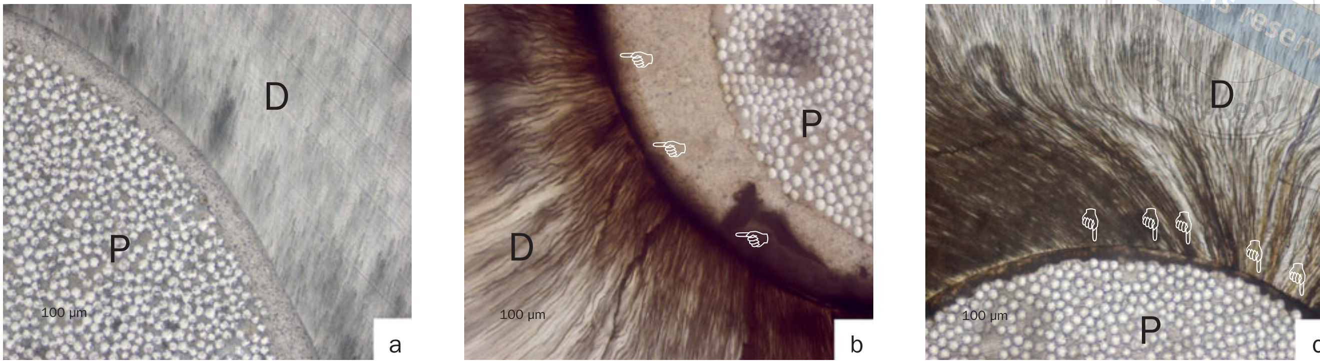


Fig 4 Light micrographs showing the adhesive interface created by Bifix SE+2%CHX pre-treatment at T0 (a), T6m (b) or T1y (c) of storage in artificial saliva at 37°C. D = dentin; P = fiber post; pointers = silver nitrate deposits. After 1 year of storage, interfacial nanoleakage expression significantly increased after aging. Scale bar: 100µm.

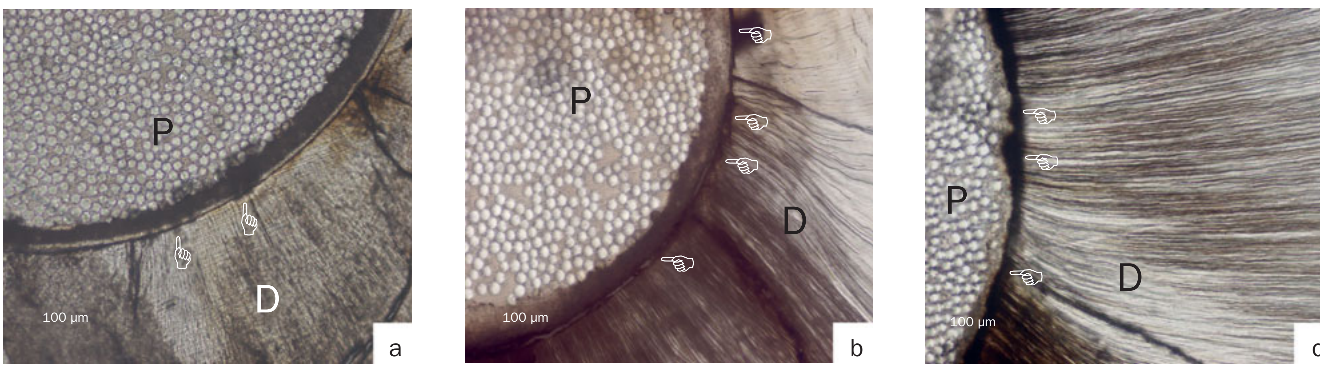


Fig 5 Light micrographs showing the adhesive interface created by Bifix SE at T0 (a), T6m (b) or T1y (c) of storage in artificial saliva at 37°C. D = dentin; P = fiber post; pointers = silver nitrate deposits. Similar to CHX pretreated specimens, nanoleakage significantly increased after aging. Scale bar: 100 µm.

dine has been reported to inhibit MMP activity within the hybrid layer, thereby contributing to the preservation of bond strength over time when applied on acid-etched dentin.[5,11,25]

In the present study, CHX was used in association with self-adhesive resin cements that do not require a preliminary etching step. Thus, CHX was applied directly on smear-layer–covered dentin surfaces. Current methods of cleaning and shaping root canals produce a smear layer of approximately 1 to 2 µm. In addition, part of the smear layer is packed into the dentinal tubules as smear plugs. The smear layer and smear plugs contain inorganic and organic debris that include fragments of odontoblastic processes, microorganisms and necrotic pulpal tissues.[3,24,32,44] Results obtained, however, are in contrast with those obtained for coronal dentin, where CHX is able to reduce degradation of the adhesive interface overtime.[7] In the current in vitro work, no differences were detected after one-year aging among the experimental and the control groups. To bond fiber posts on radicular dentin, a self-adhesive cement was employed. It is well known that this kind of cement is a mild self-etching material, which probably is unable to etch the entire depth of the smear layer,[29] thus not reaching and exposing the collagen. It could be speculated that the absence of differences among the groups after one year of storage was due to the fact that CHX did not remove the smear layer or change the smear layer thickness; it is unlikely that these cements can etch through thick smear layers into the underlying intact dentin. Even when thin smear layers are present, it is dubious whether these self-adhesive cements are capable of etching anything more than the most superficial part of the intact dentin beneath smear layers. Collagen fibrils within the intact dentin are protected by intrafibrillar apatite minerals. Thus, collagen molecules with partially- or fully-mineralized dentin are inaccessible to CHX or to collagen degradation pathways.[46] Our findings are in agreement with Lührs et al.[23] In that study, the authors examined the effect of CHX as a protease inhibitor on the microtensile bond strength of self-adhesive cements used for luting ceramics to dentin. That study reported significant bond strength reduction after aging, even when dentin was treated with CHX prior to the use of the self-adhesive cements.

Another in vitro work by Kul et al[21] showed no difference in push-out bond strength when CHX pretreatment was performed on root dentin, again confirming the results of the present study.

A recent study conducted by Di Hipólito et al[14] showed reduced bond strength when 2% CHX was used for dentin pretreatment. The combined use of scanning electron microscopy and energy-dispersive x-ray spectroscopy to examine 0.2% and 2.0% CHX-treated smear-layer–covered dentin substrates found precipitates and chlorine residues on the dentin surface after CHX application. Dentin surface precipitates could act as a physical barrier that jeopardized cement-dentin interaction. Likewise, chemical interference caused by the chlorine residues could hamper the effectiveness of composite cements.[14] A loss of bond strength in association with CHX use has also been detected by de Araújo et al,[13] underlining a reduction in MPa values after six months of aging, especially with CHX pretreatment.[13]

Although chemical bonding may have occurred between reactive monomers of the self-adhesive cements and the smear layer, the interface between weakly bonded smear layer and the underlying intact mineralized dentin remains the weakest link in the composite-dentin bond. This weak link resulted in further bond strength reduction over time when specimens were immersed in artificial saliva, regardless of CHX pretreatment, resulting in pronounced nanoleakage expression and loss of bond strength after aging. Both RelyX Unicem and Bifix SE exhibited adhesive failure as the predominant failure mode at all storage intervals, supporting the hypothesis that the weakest link may be the cement-dentin interface (Table 3).

Our results are in accordance with previous studies,[20,38] but are in conflict with the study by Lindblad et al,[22] in which the authors reported that CHX significantly improved the bond strength for RelyX Unicem. It must be emphasized that only short-term storage (3–7 days) was investigated in the Lindblad study, not the behavior over longer durations.[22]

Recent developments support the use of CHX blended into the adhesive/cement formulations. Despite the possible interferences with the degree of cure[9] when CHX is blended within the polymer, a recent study supports the incorporation of CHX into a self-etch primer.[47] In that study, it was demonstrated that improved bond strength over time could be achieved when CHX at high concentrations (up to 20%) was blended within the self-etching primer.

CONCLUSION

The use of 2% CHX to pretreat the root canal dentin before fiber post cementation using the tested self-adhesive composite cements does not improve the bond durability over time. The most likely reason is that the self-adhesive cements are not aggressive enough to etch beyond the smear layer, expose collagen fibrils or endogenous enzymes within the underlying intact dentin in order for CHX to exert its effect. As such, the decline in push-out bond strength and increase in nanoleakage expression of the tested self-adhesive cements over time observed in the present study could be caused by other factors, such as water sorption of the hydrophilic components of these cements, or the degradation of ester bonds of some of the resin monomer components, rather than activation of MMPs. Further in vitro investigations are necessary to better understand the aging behavior of self-adhesive cements in radicular dentin.

ACKNOWLEDGMENTS

The authors thank Mr. Aurelio Valmori for extensive laboratory assistance, and the manufacturers for kindly providing the materials. The study was partially supported by MIUR grants, Italy.

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Clinical relevance: The application of CHX pre-treatment does not prevent bond strength degradation of fiber posts luted with self-adhesive cements over time.