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ORIGINAL ARTICLE

Effect of three radicular dentine treatments and two luting cements on the regional bond strength of quartz fibre posts

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Abstract The purpose was to investigate by push-out tests and scanning electron microscopy (SEM)/energy-dispersive spectroscopy (EDS) the effect, after first acid etching the post space walls, of three radicular dentine treatments on the regional bond strength of quartz fibre posts placed using two heavily filled resin luting cements. The crowns of 39 extracted maxillary central incisors were sectioned transversely 2 mm coronal to the labial cement-enamel junction and the roots endodontically treated. After standardized post space preparations and etching 15 s with 32% phosphoric acid, 36 roots were randomly divided into six equal groups. Quartz fibre posts (D.T. LIGHT-POST) were placed using three radicular dentine treatments (0.9% sodium chloride (NaCl) for 60 s, 10% sodium hypochlorite (NaOCl) for 60 s, 17% ethylenediaminetetraacetic acid (EDTA) for 60 s followed by 5.25% NaOCl for 60 s) and two resin composite luting cements (ONE-STEP PLUS/DUO-LINK; ONE-STEP PLUS/LuxaCore Dual). Transverse segments (S1–S7), 1.00 mm (SD=0.05 mm) thick, were sectioned from the coronal 8 mm of each root. Push-out bond strength tests were performed on coronal, middle and apical post space segments (S2, S4, S6) at a crosshead speed of 0.5 mm/min. Data were recorded and analyzed using a two-way mixed ANOVA design (a=0.05). Three segments (S1, S5, S7) from roots in each group were examined using SEM/EDS. After post space preparation, acid etching and using each of the three radicular dentine treatments, the three remaining roots were sectioned longitudinally for SEM observation of the post space walls. At all root segment sites, the mean bond strengths from using 0.9% NaCl were significantly lower than for the other two radicular dentine treatments ($P \leq 0.02$), and DUO-LINK cement had significantly higher mean bond strengths than LuxaCore Dual cement ($P \leq 0.01$). There was a significant linear trend for reduced bond strengths from coronal to apical post space segments ($P < 0.001$), which was supported by the SEM/EDS observations of dentine tubule appearance and resin tag formation. Acid etching followed by either 10% NaOCl or 17% EDTA and 5.25% NaOCl dentine treatments of the post spaces provided good adhesion and resin luting cement tag infiltration of dentinal tubules in the coronal and middle segments in particular.

Keywords Fibre post · Push-out test · Bond strength · Luting cement

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Introduction

Reconstructing endodontically treated teeth having excessive loss of coronal structure by using prefabricated fibre post-and-core systems is a widely accepted treatment option [1, 2]. The advantages of fibre post-and-core retained restorations have been demonstrated in laboratory [3–5] and clinical studies [1, 6, 7]. Important characteristics of fibre posts (dowels) include a modulus of elasticity more similar to that of dentine [8] and an ability to be bonded to dentine using an adhesive technique [9, 10].

Posts provide retention for the core and artificial crown and distribute occlusal stresses to the root. Because debonding is the most frequent cause of clinical failure of adhesively luted fibre posts [6, 11], a high bond strength between the fibre post and the post space walls is essential. The effective bonding of fibre post systems may be influenced by various factors such as the type of fibre post [12], the condition of the radicular dentine and the density and orientation of dentine tubules along the post space walls [9, 13] and the adhesive luting technique and materials used comprising resin-based dentine adhesives and cements [14–16].

The retention of fibre posts depends on adequate bond strengths both between the post and resin luting cement and between the resin luting cement and post space dentine. Debonding along the resin luting cement–dentine interface has been identified as the most frequent clinical failure mode of adhesively luted fibre posts [10, 11, 16, 17]. A comparison of bond strengths of the resin luting cement–dentine interface with those of the resin luting cement–fibre post interface reported significantly lower bond strengths for the former [18].

Surface treatments of radicular dentine with different agents may cause alterations in the chemical and structural composition of human dentine which, in turn, may change its permeability and solubility characteristics [19]. These alterations have the potential to affect significantly the bonding of adhesive materials to the treated dentine surfaces [20]. Therefore, the surface treatment of radicular dentine requires further investigation to determine appropriate conditions for achieving optimal adhesion at the resin luting cement–dentine interface.

Following endodontic instrumentation, an effective method to remove organic and inorganic thick smear layer remnants from root canal walls and potentially allow better dentinal tubule penetration is to irrigate the root canal with ethylenediaminetetraacetic acid (EDTA) and sodium hypochlorite (NaOCl) [21, 22]. However, several studies have reported conflicting effects of EDTA and/or NaOCl endodontic irrigants on the bond strengths of resinous materials to radicular dentine. Some studies reported negative findings [23, 24], whereas others indicated positive results [25–28].

Resin-based core materials are bonded directly to fibre posts, often using a total-etch bonding system, without the intermediate use of a resin luting cement. It has not been extensively investigated whether a similar bonding efficacy to radicular dentine could be obtained when using the same total-etch bonding system with a heavily filled adhesive resin as luting cement.

Based on these considerations, the purpose of the present in vitro study is to investigate by regional push-out tests and scanning electron microscopy (SEM)/energy-dispersive spectroscopy (EDS) the effect, after first acid etching the post space walls, of three radicular dentine treatments on the bond strength of quartz fibre posts placed using two heavily filled resin composites as luting cements. The null hypothesis proposed is that there are no significant differences in mean regional bond strengths among the radicular dentine treatments and between the luting cements.

Materials and methods

Specimen preparation

Thirty-nine human sound maxillary central incisors, extracted for periodontal reasons, with fully developed apices and straight root canals were selected for this study. Suitable, cleaned incisors were examined stereoscopically (SZM45-B2, Nanking Shun Yu Optical Instrument Co. Ltd, People's Republic of China) at magnification ×10 for possible cracks or caries, and radiographs were taken to discard those teeth with irregularly shaped canals. All teeth were stored in 0.9% saline solution at 4°C for no longer than 2 weeks.

The natural crowns were sectioned with a water-cooled, low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) perpendicular to the long axis 2 mm coronal to the labial cementoenamel junction. Using a standardized technique, the root canals were then endodontically prepared before being obturated with a root canal sealer (AH 26, Dentsply International, Inc., York, PA, USA) and gutta percha points (Dentsply International, Inc.) using cold lateral compaction. The endodontically treated roots were stored in 0.9% saline solution at 37°C for 1 week to allow the sealer to set.

Post spaces were prepared uniformly to a depth of 10 mm with #3 D.T. Pre-Shaping Drills (Bisco, Inc., Schaumberg, IL, USA) leaving 3 mm of intact gutta percha as the apical seal. The absence of remnants of gutta percha on the walls of the post spaces was confirmed radiographically. The walls of the prepared post-holes were etched for 15 s using 32% phosphoric acid semi-gel (UNI-ETCH, Bisco, Inc., Schaumberg, IL, USA) applied with disposable micro-brushes, rinsed thoroughly with distilled water and completely dried with paper points. Thirty-six prepared roots were then assigned to six equal groups (A1, A2, B1, B2, C1, C2) according to a table of random numbers. The root lengths and the mesial–distal and labial–palatal diameters of each root at the root face were measured to 0.02 mm with a vernier caliper (Vernier Caliper Model 93218-0654, Harbin Measuring and Cutting Tool Group Co. Ltd., Harbin, People's Republic of China). There were no significant differences in these dimensions among the six groups (Table 1).

Quartz fibre-reinforced double-tapered posts having a 1.2 mm apical and a 2.2 mm coronal diameter (#3 D.T. LIGHT-POST, Bisco, Inc.) were placed after using three radicular dentine treatments (groups A1, A2: rinsed with 0.9% sodium chloride (NaCl) for 60 s and dried lightly; groups B1, B2: rinsed with 10% sodium hypochlorite (NaOCl) for 60 s and dried completely; groups C1, C2: rinsed with 17% EDTA for 60 s followed by 5.25% NaOCl for 60 s and dried completely, using paper points). The fibre posts were cemented using a lightly filled resin-based adhesive (ONE-STEP PLUS, Bisco, Inc.) and two heavily filled (~70 wt.%) dual-cure resin composite cements (groups A1, B1, C1: DUO-LINK (Bisco, Inc.); groups A2, B2, C2: LuxaCore Dual (DMG, Hamburg, Germany)). The materials were used according to the manufacturers' instructions discussed below.

Two thin layers of ONE-STEP PLUS resin-based adhesive were applied to the dentine walls of the post-holes using disposable micro-brushes. Excess bonding agent was removed carefully with paper points and the canal walls gently air-dried for 10 s, before the adhesive was light cured (Variable Intensity Polymerizer Jr, Bisco, Inc.) from the canal opening for 20 s at 500 mW/cm^2. The fibre posts were also coated with a thin layer of the light cured adhesive.

The two dual-syringe dispensed cements, DUO-LINK and LuxaCore Dual, were then each injected into the post spaces of their respective groups and the fibre posts inserted to full depth. Excess cement was immediately removed. The tip of the light unit was placed directly on the coronal end of each fibre post and the cement light cured for 40 s. All treated roots were stored in 0.9% saline solution at 37°C for 1 week.

A paralleling jig was used to ensure vertical alignment of each root while being embedded in a cylinder of clear self-cured acrylic resin (Shanghai Dental Materials Manufacturing Co., Shanghai, People's Republic of China). Using a water-cooled Isomet (Buehler Ltd) low-speed diamond saw, each root (n=36) was sectioned perpendicular to the long axis into 1.00 mm (SD=0.05 mm) segments labelled S1–S7 from coronal to apical (Fig. 1).

Push-out bond tests and failure modes

Push-out bond tests were performed on three different post space segments of each root (S2, S4, S6) using a universal load testing machine (MTS Synergie 100, MTS, Eden Prairie, MN, USA) at a crosshead speed of 0.5 mm/min. The maximum push-out force (N) for bond failure was recorded and the bond strength (MPa) calculated by dividing N by the area of the bonded interface:

$$\mathrm{MPa} = N/2\,\pi rh,$$

where π=3.14, r is the post radius (millimetres) and h is the thickness of the post segment (millimetres). Using the vernier caliper (Harbin Measuring and Cutting Tool Group Co. Ltd), the thicknesses of the segments and the radii of the fibre posts were measured to 0.02 mm before testing. After performing the push-out bond strength tests, the failed specimens were examined stereoscopically (Nanking Shun Yu Optical Instrument Co. Ltd) at magnification ×40 to determine the bond failure modes.

SEM specimen preparation

The remaining three post space prepared roots were allocated to each of the three dentine treatments (groups A, B, C) mentioned previously and then sectioned longitudinally for examination at magnification ×3,000 to examine morphological changes at the coronal, middle and apical post space regions of the radicular dentine surfaces.

Table 1 Mean (standard deviation) measurements of randomly assigned maxillary central incisor roots in each group

Radicular dentine treatment group (N=6)	Luting cement	Dimensions (mm)		
		Labial–palatal root face width	Mesial–distal root face width	Root length
A1—0.9% NaCl	DUO-LINK	6.29 (0.39)	6.28 (0.24)	13.37 (0.86)
A2—0.9% NaCl	LuxaCore	6.25 (0.37)	6.19 (0.20)	13.37 (0.87)
B1—10% NaOCl	DUO-LINK	6.33 (0.64)	6.16 (0.38)	13.09 (0.51)
B2—10% NaOCl	LuxaCore	6.32 (0.20)	6.11 (0.40)	13.35 (1.23)
C1—17% EDTA+5.25% NaOCl	DUO-LINK	6.32 (0.58)	6.33 (0.20)	13.36 (1.02)
C2—17% EDTA+5.25% NaOCl	LuxaCore	6.43 (0.35)	6.18 (0.36)	13.37 (0.98)
P value (one-way ANOVA)		0.99	0.92	0.99

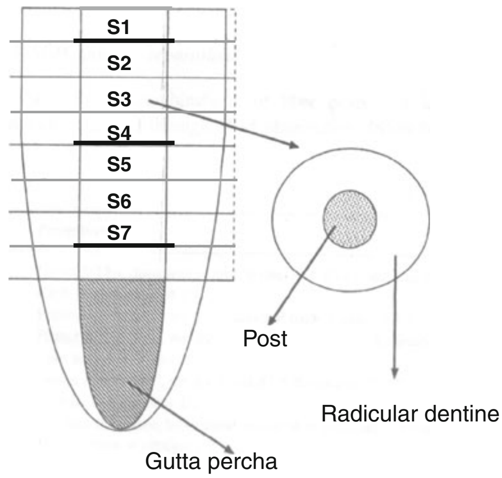

Fig. 1 Diagrammatic representation of the coronal, middle and apical post space segments S1–S7. Push-out tests used segments S2, S4 and S6. SEM histological observations and energy-dispersive spectroscopic measurements used segments S1, S5 and S7

Coronal, middle and apical post space segments (S1, S5, S7) were also examined in selected roots from each of the six fibre post-treated groups (Fig. 1). The flat surface dentine–cement interfaces of the segments were etched with 32% phosphoric acid semi-gel (UNI-ETCH, Bisco, Inc.) for 30 s, deproteinated with 5% NaOCl for 5 min, rinsed with distilled water for 1 min, gradually dried with increasing concentrations of ethanol and sputter-coated with gold before being examined by SEM (JSM-5610LV/NORAN-VANTAGE, JEOL Ltd., Tokyo, Japan) and by energy-dispersive spectroscopic microanalysis for the presence of barium present in the two luting cements.

Statistical analysis

A statistical software package (SAS 9.1.3, SAS Institute Inc., Cary, NC, USA) was used for the tests. The push-out bond strengths at the three radicular dentine post space segment sites were analyzed using a two-way mixed ANOVA design, and the cement infiltrations into root dentine walls at similar post space segment sites were analyzed using one-way ANOVA, with Tukey post hoc tests as required. The probability level was set at α=0.05 for statistical significance.

Results

Push-out bond tests and failure modes

The mean bond strengths (MPa) achieved on extrusion of the fibre posts from the three post space segment sites in each treatment group are shown in Table 2 and illustrated in Fig. 2. There were statistically significant main effects of radicular dentine treatments and luting cements ($P<0.0001$), without significant interaction ($P=0.24$). At all post space segment sites, the mean bond strengths from using 0.9% NaCl were significantly lower than for the other two radicular dentine treatments ($P\leq0.02$), which had rather similar bond strengths ($P\geq0.22$). DUO-LINK cement had significantly higher bond strengths than LuxaCore Dual cement at all post space segment sites ($P\leq0.01$). Irrespective of dentine treatment and cement, there was a significant linear trend for reduced bond strengths from coronal to apical segment sites ($P<0.001$).

The distribution of the push-out test failure modes is shown in Table 3. Adhesive failures accounted for most (68.5%) of the 108 observations. Adhesive failures between dentine and luting cements were the most common (35.2%), followed by adhesive failures between fibre posts and luting cements (29.6%), cohesive failures in dentine (18.5%), cohesive failures in fibre posts (13.0%) and mixed adhesive failures (3.7%). Cohesive failures in luting cements alone were not observed. Group A had the highest adhesive and the lowest cohesive failures, but there was no significant difference between groups B and C in the distribution of adhesive and cohesive failures (Fisher's exact test, $P=0.81$).

SEM observations

Surface morphology In the three longitudinally sectioned post space prepared roots, the morphological changes in the radicular dentine surfaces following the three treatments (groups A, B, C) were distinctly different at the coronal, middle and apical regions (Fig. 3). In general, remnants of the smear layer and residual plugs in dentinal tubules from post space preparation increased towards the apical root region. When compared with group A, reduced smear layer remnants and residual plugs were associated with enlarged tubule orifices in groups B and C.

Adhesive interfaces The coronal, middle and apical post space segments (S1, S5, S7) showed distinctly different appearances for both the DUO-LINK and the LuxaCore Dual cement specimens (Fig. 4). In general, the numbers and the lengths of resin-infiltrated dentinal tubules decreased from the coronal to the apical root regions in all three dentine treatment groups. The numbers and the lengths of resin-infiltrated dentinal tubules were also generally reduced more in group A than in groups B and C for both luting cements.

Energy-dispersive spectroscopic microanalysis The mean Ba (weight percent) findings from energy-dispersive spectroscopic microanalysis of the dentine–cement interfaces in each treatment group are shown in Table 4. LuxaCore Dual treated with 17% EDTA followed by 5.25% NaOCl (group C2) showed a significantly higher mean Ba (weight percent) than any of the other five groups ($P<0.0001$).

Table 2 Mean (standard deviation) bond strengths (MPa) achieved on extrusion of the posts from each root segment site in each group

Radicular dentine treatment group (N=6×3 segments)	Luting cement	Root segment site		
		S2 (coronal)	S4 (middle)	S6 (apical)
A1—0.9% NaCl	DUO-LINK	9.77 (2.01)	6.97 (2.17)	2.15 (2.00)
A2—0.9% NaCl	LuxaCore	4.41 (2.20)	2.32 (1.30)	1.04 (0.85)
B1—10% NaOCl	DUO-LINK	13.50 (2.24)	10.12 (1.98)	5.39 (1.99)
B2—10% NaOCl	LuxaCore	8.98 (2.12)	4.60 (1.44)	2.82 (1.30)
C1—17% EDTA+5.25% NaOCl	DUO-LINK	11.54 (1.95)	8.58 (1.89)	4.09 (1.66)
C2—17% EDTA+5.25% NaOCl	LuxaCore	10.15 (1.98)	4.29 (1.70)	2.98 (1.10)
P value (one-way ANOVA)		<0.0001[a]	<0.0001[a]	0.0008[a]

[a] Significantly different

Discussion

Limited adhesion between resin luting cement and post space dentine was reported as being responsible for the ineffectiveness of post retention observed in vitro [10, 16, 18, 29]. Therefore, it is important to investigate various methods for enhancing adhesion between radicular dentine and resin luting cements, as improved bond strengths will lead to better clinical retention of fibre post-and-core retained artificial crowns [30].

Push-out bond strength and failure modes

There is a lack of agreement in literature on the thickness (h) of the push-out test specimen that should be used. Various publications have employed 1.0–2.5 mm thicknesses [5, 15, 16, 28], with 1.0-mm-thick segments being used in the present study. The influence of specimen thickness on the test results does not appear to have been specifically investigated.

Adequate long-term adhesion of fibre posts to radicular dentine might be difficult to achieve because of numerous adverse factors. There are technical difficulties in adequately controlling moisture [31] and placing/removing various adhesive and other dental materials at/from the bottom of long, narrow post spaces [9, 13, 18, 32]. Extremely high polymerization stresses are generated by resin luting cements in the confined post spaces because of unfavourable cavity configuration factors that restrict the flow of the cements during their setting [16, 18, 33].

Following acid etching of dentine, a resin-based bond is produced when resin monomers infiltrate the exposed collagen and dentinal tubules in the demineralized dentine, producing a hybrid layer with resin tags [34]. However, because of incomplete resin infiltration, hybrid layer gap formation and also nanoleakage have been observed [35], which can lead to deterioration of the adhesion between resin monomers and dentine [36]. The collagen layer has been found not to contribute significantly to the interfacial bond strength of resin to dentine [25, 30], and adhesion with some bonding resins may be increased if the previously demineralized collagen layer is first dissolved and removed by a nonspecific proteolytic agent such as NaOCl [26, 37].

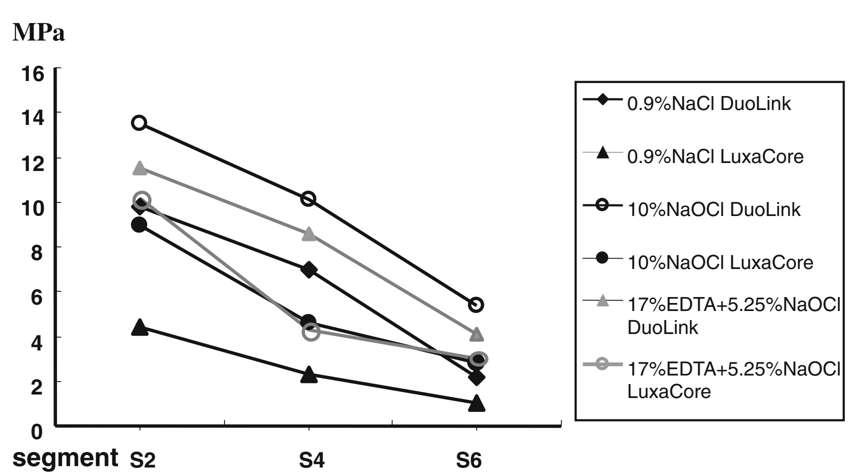

Fig. 2 Effects of the radicular dentine treatment, luting cement and post space segment site on the push-out bond strengths (MPa)

Table 3 Distribution of the failure mode numbers from the push-out tests

Failure modes (N=108)	Treatment group (N=6×3 segments)						
	A1	A2	B1	B2	C1	C2	Total
Adhesive between dentine and luting cement	14	8	2	2	6	6	38
Adhesive between post and luting cement	4	6	6	6	6	4	32
Cohesive in dentine		4	2	6	6	2	20
Cohesive in fibre post			6	2		6	14
Mixed adhesive between dentine, cement and post			2	2			4
Chi-square test=10.65, (df=1, 2)[a]	P=0.005[b]						

[a] Merged cells: adhesive vs cohesive failures for the three (A, B, C) dentine treatment methods

[b] Significantly different

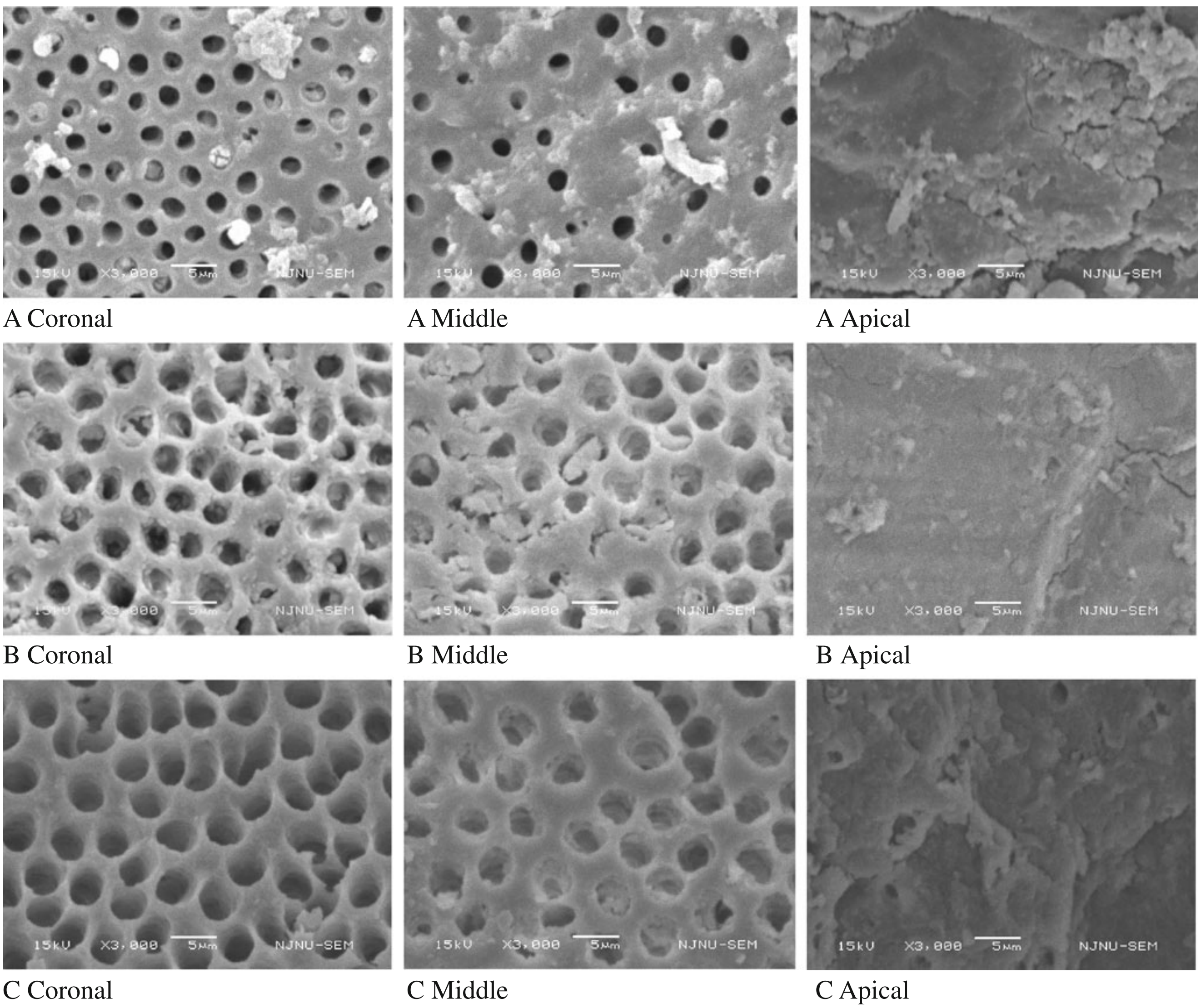

Fig. 3 SEM examples of the surface effects of the three dentine treatments at the coronal, middle and apical post space regions. *A* Group A—0.9% NaCl 60 s: Canal walls were generally smooth and regular. A thick amorphous smear layer of cutting debris covered the dentine in the apical region in particular, while many of the dentinal tubules in the middle region contained residual smear plugs. *B* Group B—10% NaOCl 60 s: Canal walls were generally rough and irregular. There was a reduced smear layer in the apical region and reduced numbers of dentinal tubules with residual smear plugs. The orifices of the tubules were enlarged from dissolution of the peritubular (intratubular) dentine. *C* Group C—17% EDTA 60 s followed by 5.25% NaOCl 60 s: Canal walls were generally smooth and regular. Very little smear layer and residual smear plugs in dentinal tubules were observed, allowing identification of some tubules in the apical region. The orifices of the dentinal tubules were significantly enlarged from dissolution of the peritubular and adjacent intertubular dentine. Original magnification, ×3,000

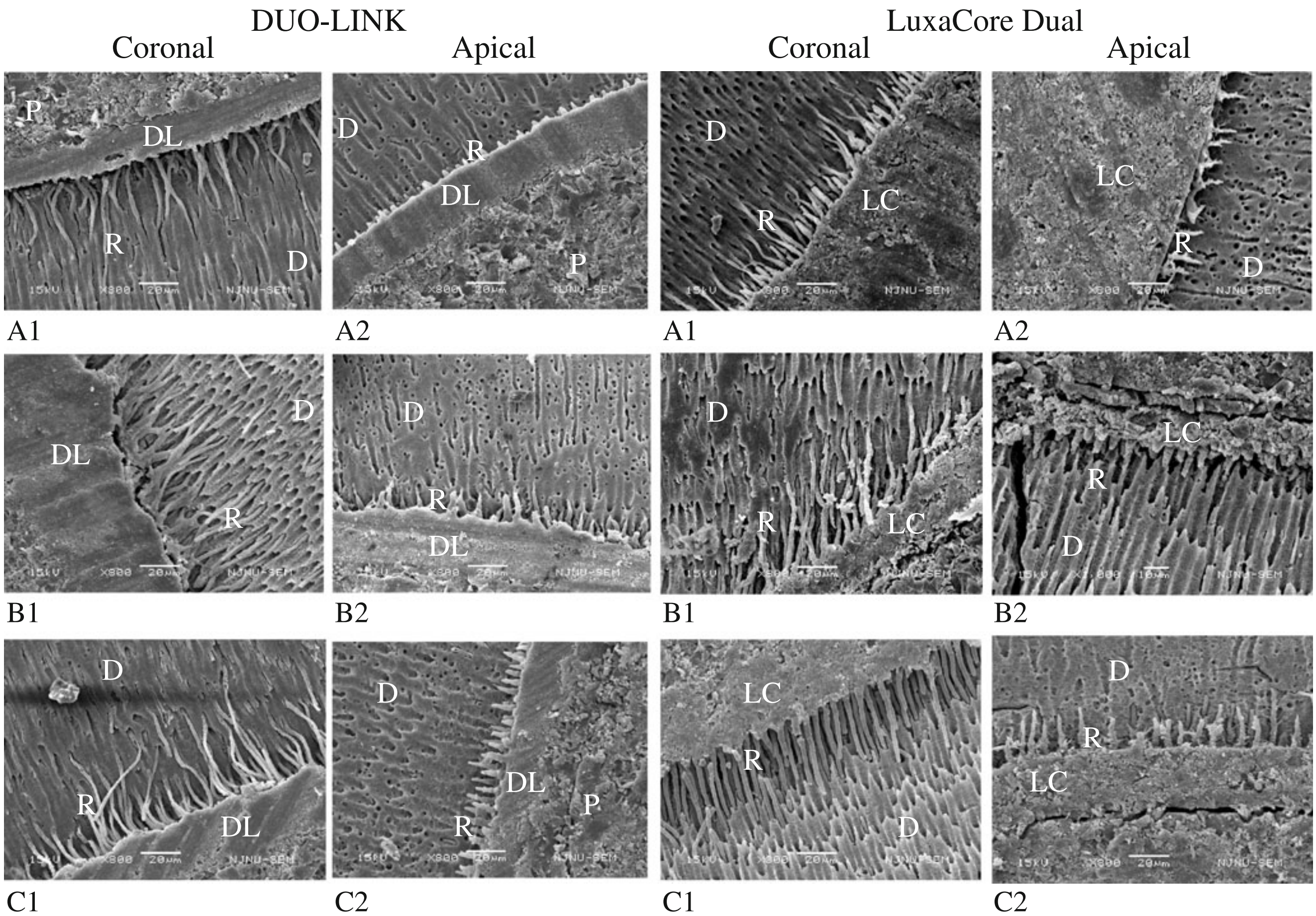

Fig. 4 SEM examples of the interfaces between the dentine and the bonded DUO-LINK and LuxaCore Dual cements in the coronal and apical post space segments (the resin tag appearances in the middle post space segments were intermediate between those of the two other segments). *D* dentine, *R* resin tags, *P* D.T. LIGHT-POST, *DL* DUO-LINK cement, *LC* LuxaCore Dual cement. *A1*, *A2* Group A—0.9% NaCl 60 s: Sparse irregular resin tags lacking lateral branches were observed, in the apical segments in particular. *B1*, *B2* Group B—10% NaOCl 60 s: More regular and longer resin tags with some lateral branches were observed in the coronal segments in particular. *C1*, *C2* Group C—17% EDTA 60 s followed by 5.25% NaOCl 60 s: Denser, regular and longer resin tags with some lateral branches were observed in the coronal segments in particular. Original magnification, ×800

In the present study, radicular dentine treatments with 10% NaOCl after dentine etching resulted in significant increases in push-out bond strengths compared with the NaCl controls (Table 2), irrespective of the post space segment site and resin luting cement used. In one study, the proteolytic action of NaOCl treatment after dentine etching removed the demineralized collagen layer to result in a porous irregular dentine substratum that provided more effective micromechanical retention [28]. It was considered that the proteolytic effect of NaOCl treatment after dentine etching avoided the possibility of incomplete infiltration of the adhesive resin adversely affecting the long-term durability of post retention [26].

Table 4 Energy-dispersive spectroscopic microanalysis of root segment dentine sites for the presence of barium from luting cement infiltration

Radicular dentine treatment group (N=1×3 segments)	Luting cement	Ba (wt.%; error)
A1—0.9% NaCl	DUO-LINK	0.90 (4.37) a
A2—0.9% NaCl	LuxaCore	3.59 (1.06) a
B1—10% NaOCl	DUO-LINK	2.43 (2.03) a
B2—10% NaOCl	LuxaCore	6.73 (1.67) a
C1—17% EDTA+5.25% NaOCl	DUO-LINK	2.64 (1.56) a
C2—17% EDTA+5.25% NaOCl	LuxaCore	16.60 (2.36)
P value (one-way ANOVA)		P<0.0001[b]

Means in each column with the same letter are not significantly different

[b] Significantly different

Groups treated with 17% EDTA followed by 5.25% NaOCl solutions after dentine etching also achieved significantly higher push-out bond strengths than those of the NaCl controls (Table 2). However, there is a divergence of opinion regarding the effectiveness of EDTA and NaOCl on the retention of endodontic posts. EDTA and NaOCl solutions will remove the thick surface smear layer on root canal walls and the smear plugs in dentinal tubules formed during post space preparation, to allow bonding resins to penetrate dentinal tubules and intertubular dentine. Such penetration should effectively seal the tubules and contribute to the bond strength of endodontic posts by creating more effective micromechanical retention [27]. But, in one study, EDTA did not significantly increase the retention of endodontic posts [39]. It was suggested that the use of EDTA and NaOCl solutions might be harmful and perhaps even weaken the root [40]. The findings from different studies appear to depend on the concentrations of the solutions used and the periods of application.

In the present study, groups bonded with DUO-LINK had significantly higher push-out bond strengths than groups bonded with LuxaCore Dual ($P \leq 0.01$). The finding might have been related to the physical properties of the cements and to the use of ONE-STEP PLUS adhesive with both luting cements and might have been different if LuxaCore Dual had been used with LuxaBond-Total Etch (DMG). Though both of these dual-cure adhesive systems are applied after separate dentine etching, as required by both manufacturers, further research would provide additional information.

The distribution of the push-out test failure modes showed that adhesive failure of the bonding interfaces accounted for most failures whereas cohesive failure in either dentine or fibre posts accounted for least failures and suggested that interfacial adhesion was the weakest between the dentine and luting cements (Table 3). Therefore, it is important to enhance the bond between radicular dentine and adhesive resins. Obvious differences in the distribution of failure modes were noted in the experimental groups compared with the 0.9% NaCl controls. Almost all of the failures in the controls were adhesive, usually at the dentine–luting cement interface, while failures in the experimental groups were both adhesive and cohesive with relatively fewer adhesive failures at the dentine–luting cement interface. These findings, with an increased ratio of cohesive failures after the experimental treatments [41], indicate that adhesion at the resin cement–dentine interface in particular had been enhanced.

SEM/EDS observations

Radicular dentine bond strengths depend on adhesive resin infiltration into demineralized intertubular dentine and resin tag formation (tubule penetration and intertubular anastomoses) and possible chemical interactions at the resin–dentine interface [26]. Hybrid layer formation in the intertubular dentine and in the first portion (1–3 μm) of the tubules [34] provides essential micromechanical retention of the resin monomers during their polymerization shrinkage [42].

SEM observations of the 0.9% NaCl control specimens before bonding showed some instrumented smear remnants on the dentine surfaces and plugs occluding the orifices of dentinal tubules (Fig. 3A). After bonding, sparse regular short-tapered resin tags lacking lateral branches were present, with tag lengths and numbers reducing greatly from coronal to apical post space regions (Fig. 4A1, A2). The presence of a retained smear layer after post space preparation has been shown to decrease significantly the bond strength of resin to dentine [38, 43].

SEM observations of the dentine surfaces treated with 10% NaOCl appeared completely different from the control specimens before bonding, with smear remnants and plugs removed, and the orifices of the tubules slightly enlarged (Fig. 3B). After bonding, dense, irregular resin tags with frequent lateral branches were observed, which also decreased in numbers and lengths from coronal to apical post space regions (Fig. 4B1, B2). NaOCl removes demineralized collagen, leading to increased diameters of the resin tags [20].

SEM observations of the dentine surfaces treated with 17% EDTA followed by 5.25% NaOCl before bonding showed the entire absence of a smear layer and plugs, with dentinal tubules visible even at the apical region of the post space (Fig. 3C). Orifices of the dentinal tubules were greatly flared due to removal of peritubular dentine. After bonding, the numbers, lengths and diameters of resin tags in the dentinal tubules and lateral branches were increased but again were reduced from coronal to apical post space regions (Fig. 4C1, C2). The energy-dispersive spectroscopic microanalysis of Ba (weight percent) also indicated an increased resin luting cement infiltration of dentine tubules following NaOCl and EDTA/NaOCl dentine treatments (Table 4). Although, with all three dentine treatments, the measurements for Ba were higher for LuxaCore Dual than for DUO-LINK, this trend was not reflected in their bond strengths, which were significantly lower for LuxaCore Dual (Table 2), and might have been related to the different physical properties of the cements and the adhesive used.

The apical region of the root canal and of the post space preparation provides a challenge for satisfactory dentine adhesion because of problems with adequate access and unfavourable dentine structure. However, problems with dentine adhesion, associated with fibre post retention in the apical region in particular, may be ameliorated by first acid etching the post space walls and then using either NaOCl or EDTA/NaOCl treatments before the resin-based adhesive materials employed in the present study are placed.

Conclusions

Etching of the post space dentine with 32% phosphoric acid followed by either 10% NaOCl or 17% EDTA and 5.25% NaOCl treatments provided significantly improved adhesive bond strengths and resin luting cement tag infiltration of dentinal tubules in the coronal and middle post space segment sites in particular, when compared to 0.9% NaCl treatment. ONE-STEP PLUS/DUO-LINK cement had significantly higher mean bond strengths than ONE-STEP PLUS/LuxaCore Dual cement at all post space segment sites. Therefore, the null hypothesis was not accepted.

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Conflict of interest The authors declare that they have no conflict of interest.

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