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ORIGINAL ARTICLE

The effects of calcium hydroxide removal methods on bond strength of Epiphany SE with two irrigation protocols

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Abstract

Objective: The purpose of this study was to compare the effects of five calcium hydroxide removal methods on the bond strength of Epiphany SE sealer after canal irrigation with NaOCl+EDTA or NaOCl+MTAD. ***Materials and methods:*** The 120 roots were instrumented by using the ProTaper rotary system under irrigation with 2.5% sodium hypochlorite and randomly divided into two major groups according to the final irrigation: 1.3% NaOCl + MTAD and 5% NaOCl + 17% EDTA. For controls, 10 roots from each of the final irrigations with NaOCl + MTAD and NaOCl + EDTA (20 roots) were not filled with $Ca(OH)_2$. The intra-canal paste, $Ca(OH)_2$ was applied to each of the 100 remaining roots and stored at 37°C for 7 days. Each group was sub-divided into five sub-groups (n = 10) according to the removal technique for the intra-canal dressing: Group-1: recapitulation with size 30 K file + 3 ml of saline solution, Group-2: recapitulation with size 30 K file + 3 ml of 5% NaOCl, Group-3: using 5% NaOCl + 17% EDTA, Group-4: using 5%NaOCl + 15% citric acid, and Group-5: using 1.3% NaOCl + 5 ml MTAD. The root canals were filled with Resilon and Epiphany SE sealer. The bond strength was measured. ***Results:*** The mean bond strength of Epiphany SE to root dentine irrigated with NaOCl + MTAD was lower than that of NaOCl + EDTA. The highest bond strengths were 3.31 ± 0.057 and 2.60 ± 0.054 in the NaOCl + citric acid group when $Ca(OH)_2$ was applied to roots treated with NaOCl + EDTA and NaOCl + MTAD, respectively ($p < 0.05$). ***Conclusion:*** For root canals treated with NaOCl + EDTA or NaOCl + MTAD, the use of NaOCl + chelating agent for $Ca(OH)_2$ removal does not adversely affect the bond strength of Epiphany SE to dentin.

Key Words: *calcium hydroxide, removal methods, bond strength, irrigation*

Introduction

The success of endodontic treatment depends on correct shaping, cleaning and obturation to eliminate or prevent microbial contamination of the root canal system. Irrigation is used for the removal of tissue remnants and dentin debris during the mechanical instrumentation of the root canal [1]. There are a variety of irrigation agents available. Sodium hypochlorite (NaOCl) is the most universally used irrigating solution. Recently, a new developed endodontic irrigant containing a mixture 3% doxycycline, 4.25% citric acid and detergent (Tween 80) became available as Bio pure MTAD (Dentsply Tulsa Dental, Tulsa, OK). The recently revised MTAD protocol for clinical use is an initial irrigation for 20 min with 1.3% NaOCl, followed by a 5 min final rinse with MTAD [2].

Despite careful mechanical instrumentation and the use of antimicrobial irrigating agents, infections of the pulp space are difficult to eradicate consistently in one clinical treatment session [3]. Therefore, the use of an effective intra-canal medicament has been suggested by several studies as an important procedure for bacterial reduction [1,3]. Calcium hydroxide [$Ca(OH)_2$] has been widely used for endodontic treatment because of its antibacterial and biological properties [3]. However, the removal of root canal medicament completely before obturation provides better penetration of root canal sealer to dentin tubules [4]. Both the leakage of the obturation is decreased and also the bonding is increased [5]. One of the desirable properties of the root canal filling material is its ability to adhere to root canal dentin. High quality adhesion eliminates any space for the multiplication of micro-organisms between the filling material and the root canal wall [5].

There are many root canal sealers used in clinical practice to hermetically obturate root canals. Resilon

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core material and Epiphany SE (Pentron Clinical Technologies, Wallingford, CT) are the popular obturation materials introduced as an alternative to gutta-percha and traditional root canal sealers. Epiphany SE is a self-etching dualcure resin-based sealer that eliminates the traditional priming step in bonding the core material to the dentin tubules [5].

It was reported in several studies [4,6–8] that irrigating solutions and intra-canal medications can affect the bond strength [4] and adhesion [6] of endodontic sealers to dentin. Therefore, all interappointment dressings have to be removed from the root canal prior to filling [7] to avoid the negative interference between medicament and material [8]. Several studies [9–12] have been performed to assess the efficacy of different techniques on the removal of intra-canal medicaments from the root canal [9]. The authors found no reports of studies of the bond strength after $Ca(OH)_2$ dressing to the root dentin irrigated with NaOCl + MTAD. There are also no reported studies on the efficacy of techniques for removing $Ca(OH)_2$ on bond strength with NaOCl + MTAD.

Therefore, the aim of this study was to compare the effects of five $Ca(OH)_2$ removal methods on the bond strength of Epiphany SE sealer after canal irrigation with NaOCl + EDTA or NaOCl + MTAD.

Materials and methods

One hundred and twenty extracted human single-rooted teeth were collected and used in this study. The teeth were thoroughly cleaned by removing hard deposits by curetting and the soft deposits by soaking in 5.25% sodium hypochlorite (NaOCl) (Wizard, Rehber Kimya San. ve Tic., Istanbul, Turkey) for 30 min. The teeth were then stored in distilled water at 4°C until used in this study. This study was approved by the Research Ethics Committee of Ondokuz Mayis University.

The crowns were removed at the cemento–enamel junction using a water-cooled diamond disc (Edenta AG, CH-9434, Switzerland) mounted in a low-speed handpiece and the pulp tissue was removed with a K-file (Dentsply/Maillefer, Ballaigues, Switzerland) under irrigation with 2.5% NaOCl (Wizard, Rehber Kimya San. ve Tic.). The working length was determined by introducing a size 15 K-file into the canal until it reached the apical foramen and withdrawing it 1 mm.

The root canals were instrumented by using the ProTaper rotary system (Dentsply, Maillefer, Ballaigues, Switzerland) according to the manufacturer's instructions (Pro-Taper Universal Guidelines; Dentsply, Maillefer, 2008), including S1, S2, F1, F2 and F3 files and irrigation with 2 mL of 2.5% NaOCl between instrumentations. Apical patency was verified with a size 15 K-file.

The 120 roots were then randomly divided into two major groups ($n = 60$) according to the final irrigation: 2 mL 1.3% NaOCl (Wizard, Rehber Kimya San. ve Tic.) and 5 ml MTAD (Biopure, Dentsply Tulsa Dental, Tulsa, OK) and 2 mL 5% NaOCl and 3 mL 17% EDTA (Wizard, Rehber Kimya San. ve Tic.). All irrigation solutions were delivered 2 mm short of the working length through a 27G needle (Endo-Eze Irrigator tip, Ultradent Products, Inc., UT, South Jordan). The root canals were then dried with paper points. For controls, 10 roots from each of the final irrigations with NaOCl + MTAD and NaOCl + EDTA (20 roots) were not filled with Ca $(OH)_2$. The intra-canal paste, $Ca(OH)_2$ (Metapaste, Meta Biomed Co Ltd, Korea) was applied with its injector to each of 100 remaining roots until the canal was completely filled. The radiographs were taken to confirm the completeness of $Ca(OH)_2$ filled inside the root canals. The coronal openings were sealed with a cotton pellet and a temporary filling material (Cavit; ESPE America, Norristown, PA). The samples were then stored at 100% humidity at 37°C for 7 days.

Each group was further sub-divided into five subgroups ($n = 10$) according to the removal technique for the intra-canal dressing:

- Group 1: recapitulation with size 30 K file + 3 mL of saline solution;
- Group 2: recapitulation with size 30 K file + 3 mL of 5% NaOCl;
- Group 3: using 3 mL of 5% NaOCl + 3 mL of 17% EDTA for 1 min;
- Group 4: using 3 mL of 5% NaOCl + 3 mL of 15% citric acid (Köksal, Köksal Eczane ve Medikal Ürünler Tic., Samsun, Turkey); and
- Group 5: using 3 mL of 1.3% NaOCl + 5 mL of MTAD for 5 min.

A final rinse with 3 mL distilled water were performed in all groups before filling the root canals. All specimens were dried with absorbent paper points (Suredent Corp., Gyeonggi-do, Korea) and the root canals were filled using a lateral condensation technique with Resilon/Epiphany SE sealer (Pentron Clinical Technologies, Wallingford, CT). The excess material was removed with moist gauze and the sealer was light-cured (Lunar, Benlioglu, Turkey) with a halogen light-curing unit for 40 s at the coronal access of the root, according to the manufacturer's instructions. All samples were then stored at 37°C and 100% humidity for 48 h to allow the sealer cements to set completely.

Push-out bond strength assessment

Specimens were then fixed with an autopolymerizing acrylic resin. All specimens were sectioned horizontally into $2 \times 3.0 \pm 0.1$ mm thick slices from 3 mm above the apex to 9 mm above the apex in a precision

sectioning machine using a water-cooled diamond saw (Isomet, Buehler, Lake Bluff, IL). Each slice was marked on its apical side with an indelible marker to avoid any constriction interference due to root canal taper during push-out testing. The thickness of each specimen was measured and recorded by a digital caliper (series 500 Caliper; Mitutoyo America Corp., Aurora, IL) with an accuracy of 0.01 mm.

Push-out bond test was performed with a 0.5 mm diameter cylindrical plunger mounted on a Universal Testing Machine (Lloyd LR 30K; Lloyd Instruments Ltd, Fareham, UK) managed by personal computer software (Nexygen-Ondio Version 4.0; Lloyd Instruments Ltd) at a cross-head speed of 0.5 mm/min until the sealer dislodgement was judged to have occurred. The load at failure recorded in Newtons (N) was divided by the area of the bonded interface for each specimen to calculate the bond strength in megapascals (MPa), according to the following formula [13]: Push-out bond strength (MPa) = P/A, where P is the maximum load (N) and A is the adhesion area of the root canal filling (mm^2). $A = 2\ \pi r \times h$, where π is the constant 3.14; r is the root canal space radius; and h is the thickness of the slice in mm. The measurements were performed by one operator who was blinded as to which samples were matched to which materials.

Data were analysed using a 2-way repeated-measures analysis of variance and Tukey's multiple comparison tests as factors with $\alpha = 0.05$.

Results

The mean push-out bond strength values in megapascals and standard deviations are shown in Table I.

The mean bond strength of Epiphany SE to root dentine irrigated with NaOCl + MTAD was lower than that of NaOCl + EDTA. However, 2-way ANOVA showed that Ca(OH)$_2$ application into the root canal adversely affected bond strength, except for the NaOCl + citric acid and NaOCl + EDTA groups. The highest bond strengths were 3.31 ± 0.057 and 2.60 ± 0.054 in the NaOCl + citric acid group when Ca(OH)$_2$ was applied to roots treated with NaOCl + EDTA and NaOCl + MTAD, respectively ($p < 0.05$). In contrast, the lowest bond strengths were 2.38 ± 0.045 and 1.79 ± 0.053 for the saline with K file group with the NaOCl + EDTA and NaOCl + MTAD treatments, respectively ($p < 0.05$).

Discussion

Ca(OH)$_2$ is widely used in endodontics. It acts as a physical barrier in the canal and prevents root canal reinfection [14]. Optimum adhesion requires intimate contact between the adhesive material and the substrate to facilitate molecular attraction and allow either chemical adhesion or penetration for micromechanical surface interlocking [15,16].

The use of chemical substances and files in root canal treatment is extremely important to promote cleanliness of dentine walls in endodontic treatment [17]. Incomplete removal of Ca(OH)$_2$ from the root canal surfaces prevented sealer from penetrating into the dentinal tubules, resulting in a potential reduction of sealer adaptation [18]. Hence, complete removal of calcium hydroxide from the root canal before filling is recommended.

Ca(OH)$_2$ removal techniques are normally effective in the coronal portion of the root, whereas they may be deficient in the middle and apical thirds. Margelos et al. [19] and Scelza et al. [20] found Ca(OH)$_2$ remnants mainly in the apical region. Therefore, 3 mm cross-sections were taken from the middle and upper apical regions of the roots in the current study. Several studies tried to determine the best protocol to remove all calcium hydroxide medication before root canal filling to maximize the penetration of the root canal sealer to the dentin surface. It is commonly accepted that alternating use of 5.25% NaOCl and 15% citric acid, recapitulation with the master apical file (MAF) in combination with irrigation and 5.25% NaOCl and 17% EDTA are the efficient protocols for Ca(OH)$_2$ removal [10–12]. Therefore, these known, efficient

Table I. The mean bond strength values of the groups (MPa).

	Irrigation protocols		
	NaOCl + EDTA	NaOCl + MTAD	*p*-value
Without Ca(OH)2	3.25 ± 0.060^a	2.52 ± 0.048^A	<0.05
Ca(OH)2 removal technique			
NaOCl + K file	3.06 ± 0.054^b	2.32 ± 0.048^B	
Saline + K File	2.38 ± 0.045^c	1.79 ± 0.053^C	
NaOCl + MTAD	2.62 ± 0.061^d	2.24 ± 0.060^D	
NaOCl + EDTA	3.19 ± 0.058^a	2.45 ± 0.047^A	
NaOCl + Citric acid	3.31 ± 0.057^a	2.60 ± 0.054^A	

There is no significant difference between groups with the same superscript letter ($p < 0.05$).

$Ca(OH)_2$ removal methods were employed for removal of $Ca(OH)_2$ in this study. However, no study was found on the efficacy of MTAD in $Ca(OH)_2$ removal.

Calt and Serper [21] showed that EDTA tends to have a corrosive effect on dentin when employed for more than 1 min. Hence, 17% EDTA was used as a final rinse for 1 min in this study. MTAD is recommended as a final rinse after initial irrigation with 1.3% NaOCl, according to manufacturer's instructions. Therefore, 1.3% NaOCl and MTAD were also used in this study.

With the correct matching of adhesive techniques to endodontic materials, the attachment of sealers to root dentine and obstruction properties improved. The Epiphany SE sealer chosen for the study was developed with that aim.

High bond strength of root canal sealers to radicular dentin is necessary for preserving the integrity of the seal in root canal filling. There are different mechanical methods of assessment of bond strength, such as shear bond strength [22], microtensile bond strength [4] and push-out tests [23] and different values of bond strength have been reported, even for the same material [23–27]. The push-out test is a reliable and efficient method of evaluating bond strength because it allows measurement of differences in bond strength at different root levels with adequate variability of the data distribution [24]. To avoid operator-dependent variability [25], the push-out procedures were performed by the same operator in the present study.

Although De-Deus et al. [26] found the bond strength of Epiphany SE and exposed root dentin treated with 2.5% NaOCl and 17% EDTA was 2.4 MPa, Wachlarowicz et al. [27] reported that NaOCl/EDTA and NaOCl/MTAD did not significantly improve Epiphany-dentin bond strength when compared with NaOCl used alone. In the present study, the mean bond strength of the NaOCl + EDTA and NaOCl + MTAD groups was 3.25 ± 0.060 and 2.52 ± 0.048 MPa, respectively, which were consistent with De-Deus et al.'s [26] study results. The lower bond strength values of the NaOCl + MTAD group compared to the NaOCl + EDTA group may be explained by the study of Tay et al. [28], who stated that a precipitate formed because of the oxidation of doxycyline by NaOCl. This degradation product had a high affinity for hydroxyl apatite [28]. However, only one study, that of Barbizam et al. [29], evaluated the bond strength of Epiphany sealer to dentin walls after placement of $Ca(OH)_2$ dressings and recapitulation with a K file and saline as the irrigant. Barbizam et al. [29] stated that the use of $Ca(OH)_2$ as an intracanal dressing material affected the adhesion of Epiphany to the root canal walls. The bond strength of the $Ca(OH)_2$ groups was 10.18 ± 1.9 MPa, while the strength of the non-$Ca(OH)_2$ groups was 13.82 ± 3.9 MPa. The cause of high values may be due to the use of Epiphany instead Epiphany SE and also the method of preparation of the dentin discs. Additionally, Shokouhinejad et al. [30] found the bond strength of Resilon/Epiphany SE after irrigation with 1.3% NaOCl + MTAD was lower than the present study result. This result might be attributed to the thickness of dentin samples.

To date, no other studies have evaluated the bond strength of Epiphany SE sealer to root dentin, with or without $Ca(OH)_2$ application. Although the use of $Ca(OH)_2$ medicaments decreased the bond strength of Epiphany SE sealer to root wall dentin in the present study, there was no reduction of bond strength of Epiphany SE when the $Ca(OH)_2$ was removed with NaOCl + chelation agent. The lowest bond strength was seen in the K-file and saline group because they are uneffective in removing $Ca(OH)_2$ [10]. The results for the removal techniques in the present study are consistent with those of Salgado et al. [10] that show that the inefficient removal of $Ca(OH)_2$ from dentin tubule orifices handicaps the penetration of Epiphany SE. In addition, the removal of $Ca(OH)_2$ with NaOCl + MTAD showed a slight decrease in bond strength. This may be explained by the presence of oxidation of doxycline by NaOCl in the dentin tubule orifices and/or the presence of $Ca(OH)_2$ particles in the dentin tubule orifices.

It is, therefore, concluded that for root canals treated with NaOCl + EDTA or NaOCl + MTAD, the use of NaOCl + chelating agent for $Ca(OH)_2$ removal does not adversely affect the bond strength of Epiphany SE to dentin. However, it is suggested that NaOCl + EDTA irrigation be used instead of NaOCl + MTAD irrigation for higher bond strength, if Epiphany SE is used as a sealer. Further clinical trials are necessary to confirm the results of *in vitro* studies.

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