The Effect of Different Final Irrigant Activation Techniques on the Bond Strength of an Epoxy Resin–based Endodontic Sealer: A Preliminary Study

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Abstract

Introduction: The aim of this study was to evaluate whether or not different final irrigation activation techniques affect the bond strength of an epoxy resin–based endodontic sealer (AH Plus; Dentsply DeTrey, Konstanz, Germany) to the root canal walls of different root thirds. **Methods:** Eighty single-rooted human mandibular premolars were prepared by using the ProTaper system (Dentsply Maillefer, Ballaigues, Switzerland) to size F4, and a final irrigation regimen using 3% sodium hypochlorite and 17% EDTA was performed. The specimens were randomly divided into 4 groups ($n = 20$) according to the final irrigation activation technique used as follows: no activation (control), manual dynamic activation (MDA), CanalBrush (Coltene Whaledent, Altststten, Switzerland) activation, and ultrasonic activation. Five specimens from each group were prepared for scanning electron microscopic observation to assess the smear layer removal after the final irrigation procedures. All remaining roots were then obturated with gutta-percha and AH Plus sealer. A push-out test was used to measure the bond strength between the root canal dentin and AH Plus sealer. The data obtained from the push-out test were analyzed using 2-way analysis of variance and Tukey post hoc tests. **Results:** The bond strength values mostly decreased in the coronoapical direction ($P < .001$). In the coronal and middle thirds, ultrasonic activiation showed a higher bond strength than other groups ($P < .05$). In the apical third, MDA displayed the highest bond strength to root dentin ($P < .05$). The majority of specimens exhibited cohesive failures. **Conclusions:** The bond strength of AH Plus sealer to root canal dentin may improve with ultrasonic activation in the coronal and middle thirds and MDA in the apical third. *(J Endod 2014;40:862–866)*

Key Words

AH Plus sealer, bond strength, irrigation activation

The main goal of root canal treatment is the efficient disinfection of the root canal system and the prevention of reinfection (1). A combination of mechanical preparation with an effective irrigating regimen, the use of intracanal medicaments between appointments, and obturation of the root canal system are tools that can be used to attain this goal (2, 3). After chemomechanical preparation, a smear layer (an amorphous and irregular layer) 1- to 2-μm-thick is formed on the root canal walls. It consists of inorganic dentin debris and organic substances containing fragments of odontoblastic processes, microorganisms, their byproducts, and necrotic pulp tissues (4). The smear layer can harbor remnants of necrotic pulp tissues and bacterial biofilms. Residual biofilms can serve as a potential source of persistent infection and treatment failure (5). In addition, the smear layer could inhibit penetration of the root canal irrigation solutions and medicaments into dentinal tubules (6). Moreover, it has been indicated that removal of the smear layer may increase the bond strength of filling material to canal walls (7). However, Saleh et al (8) stated that the penetration of the endodontic sealers into the dentinal tubules when the smear layer was removed was not associated with a higher bond strength.

The irrigation of the root canal is an essential procedure in the endodontic treatment for the removal of the smear layer. Currently, the alternate use of sodium hypochlorite (NaOCl) and EDTA irrigants is recommended to remove both the inorganic and organic components of the smear layer (9). It was stated that the effectiveness of irrigants is associated with their direct contact with the entire canal wall. However, this might not be achieved with conventional needle irrigation because of the complex nature of root canal anatomy (10). Different irrigation activation techniques have been proposed to improve the efficacy of irrigation solutions within the root canal system. These techniques include activation with gutta-percha cones, lasers, brushes, negative pressure irrigation technique, and sonic and ultrasonic devices (11).

Because dentin surface treatment with different irrigation regimens causes alteration in the chemical and structural composition of human dentin, the permeability and solubility characteristics of dentin may change (12, 13) and hence affect the adhesion of filling materials to dentin surfaces (14). There are many studies regarding the effect of different final irrigation regimens on the bond strength of canal filling materials. According to the results of the studies, authors claim that final irrigating protocols impact the adhesion of sealers to root dentin (15–18).

Although the effect of final irrigation without activation on the bond strength of root canal sealers was evaluated, there is, to our knowledge, no study examining the bond strength of endodontic sealers to root canal dentin after the activation of final irrigants. Therefore, the purpose of this study was to evaluate the effect of different final irrigation

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activation techniques on the bond strength of an epoxy resin–based sealer (AH Plus; Dentsply DeTrey, Konstanz, Germany). The null hypothesis was that various final irrigation activation techniques may change the bond strength of AH Plus sealer in different root thirds.

Materials and Methods

Tooth Selection and Preparation

The sample size for the bond strength test was 60 at 93% power and a significance level of 0.05 using data (effect size = 0.608) obtained from a pilot study. An additional 20 teeth were added to the sample for evaluation with a scanning electron microscope after the final irrigation activation procedures. Thus, 80 extracted single-rooted human mandibular premolars with a similar root length from the cementoenamel junction to the root apex were selected for this study and were stored at 4°C in a saline solution. Preoperative mesiodistal and buccolingual radiographs were taken to verify the presence of a single canal. Criteria for tooth selection included a completely formed apex and the absence of previous root filling, resorption, or calcifications. The length of the teeth was standardized to 21 mm by trimming the crowns of teeth with silicon carbide abrasive paper. The teeth were completely not decoronated, and the crowns served as a reservoir for the irrigation solution. Endodontic access cavities were prepared using diamond burs (Diatech; Coltene Whaledent, Altststten, Switzerland) with a high-speed handpiece under water cooling. A #10 K-file (Dentsply Maillefer, Ballaigues, Switzerland) was inserted into each canal until its tip was just visible at the apical foramen and the length was measured. The working length (WL) was established by subtracting 1 mm from this measurement. To prevent the escape of irrigants from the root apex by simulating a clinical situation, the apex was sealed with melted wax (Modelling Wax; Dentsply DeTrey, Weybridge, UK). The root canals were prepared by using the ProTaper rotary instruments (Dentsply Maillefer) to a size 40, 0.06 taper (F4) to the WL. Between the use of each instrument, the canals were irrigated with 2 mL 3% NaOCl solution using a syringe and 29-G needle (NaviTip; Ultradent, South Jordan, UT).

After completion of the chemomechanical preparation, specimens were randomly divided into 1 control group and 3 experimental groups (n = 20). The description of the treatment of each group follows.

No-activation Group (Control) (n = 20). A final irrigation was performed with 5 mL 3% NaOCl followed by 5 mL 17% EDTA using a syringe and a 29-G needle (NaviTip) placed 1 mm short of the WL. No additional activation of irrigants was performed.

Manual Dynamic Activation Group (n = 20). The canals were flooded with 5 mL 3% NaOCl followed by 5 mL 17% EDTA, and each irrigant was activated manually to the WL by using a size F4 (Dentsply Maillefer) gutta-percha cone. The frequency of activation used was 100 push-pull strokes per minute. Four gutta-percha cones per root were used.

CanalBrush Group (n = 20). Activation of 5 mL 3% NaOCl and 5 mL 17% EDTA was performed using a CanalBrush (CB) with a tip diameter of 0.25 mm (Coltene Whaledent) in a handpiece set at 600 rpm. The brush was used with a gentle up-and-down motion at 1 mm from the WL. One CB per root was used.

Ultrasonic Activation Group (n = 20). In this group, 5 mL 3% NaOCl and 5 mL 17% EDTA were each passively activated using an ultrasonic device (EMS, Le Sentier, Switzerland). A smooth ultrasonic file (size 15, .02 taper) (ESI Instrument, EMS) was placed into the canal to 1 mm short of the WL without touching the walls, enabling it to vibrate freely. The ultrasonic file was activated at a power setting of 6. One ultrasonic tip was used for 5 root canals.

The activation time for each irrigant was 1 minute. The total working time for NaOCl and EDTA was 2 minutes for all groups. Finally, the specimens were irrigated with 5 mL distilled water to prevent further irrigant action. The total irrigation volume for final irrigation procedures was 15 mL for all groups.

Scanning Electron Microscopy Examination after Final Irrigation Activation Procedures

Five specimens from each group were left for scanning electron microscopic (Leo-440; Leo Electron Microscopy, Cambridge, England) observation of the smear layer removal after final activation procedures. Grooves were prepared with a water-cooled diamond bur on the buccal and lingual surfaces of the teeth, and the teeth were split along their long axis in a buccolingual direction using a hammer and chisel. For scanning electron microscopic analysis, the samples were dehydrated and coated with gold-palladium particles (20 nm). Three photomicrographs were taken from the coronal, middle, and apical thirds of the root canals at 2000× magnification to evaluate the cleanliness of the canal walls. The open dentinal tubules were counted on each photomicrograph using Adobe Photoshop software (Adobe Systems, San Jose, CA).

Root Canal Obturation

Fifteen specimens remained in every group, and the canals were dried with paper points (Dentsply Maillefer). All canals were then obturated with an epoxy resin–based sealer, AH Plus, and F4 gutta-percha cones using the single-cone technique. Mesiodistal and buccolingual radiographs were taken to confirm complete filling. After root filling, the coronal 1 mm of the filling materials was removed from each specimen, and the space in each was filled with a temporary filling material (Cavit; 3M ESPE, Seefeld, Germany). Subsequently, all specimens were stored at 37°C in a 100% humidity for 2 weeks.

Push-out Testing. Each root was cut horizontally at a slow-speed using a water-cooled diamond saw (Isomet; Buehler, Lake Bluff, IL) at depths of 4, 7, and 10 mm to produce slices approximately 1-mm-thick from each root region (ie, apical, middle, and coronal). The thickness of each slice was measured using a digital calliper (Teknikel, Istanbul, Turkey) to an accuracy of 0.001 mm. Both the apical and coronal aspects of the specimens were then microscopically examined to confirm a circular canal shape (19). All slices were then scanned, and the diameters of filling materials measured using an electronic scale in software (Adobe Photoshop) to determine the diameters of plungers to be used for the push-out test. The push-out test was performed in a universal testing machine (Instron Corp, Canton, MA) by applying a continuous load to the apical side of each slice using 0.7-, 0.8-, and 0.9-mm diameter cylindrical plungers, respectively, matching the diameter of each canal third. The diameter of plungers was approximately 80% of the canal diameters. Loading was applied at a crosshead speed of 1 mm/min from the apical to the coronal direction until bond failure occured. The maximum load applied to filling material before failure was recorded in newtons and converted to megapascals (MPa) according to the following formula:

$$\text{Push} - \text{out bond strength (MPa)} = N/A$$

where N = maximum load (N) and A = adhesion area of root canal filling (mm^2).

The adhesion area of each section was calculated as: $(\pi r_1 + \pi r_2) \times L$, where $L = \sqrt{(r_1 - r_2)^2 + h^2}$, π is the constant 3.14, r_1 is the smaller radius, r_2 is the larger radius, and h is the thickness of the slice in millimeters.

Analysis of Failure Modes

The failure modes were examined under a stereomicroscope (BX60; Olympus, Tokyo, Japan) at 30× magnification. Each sample

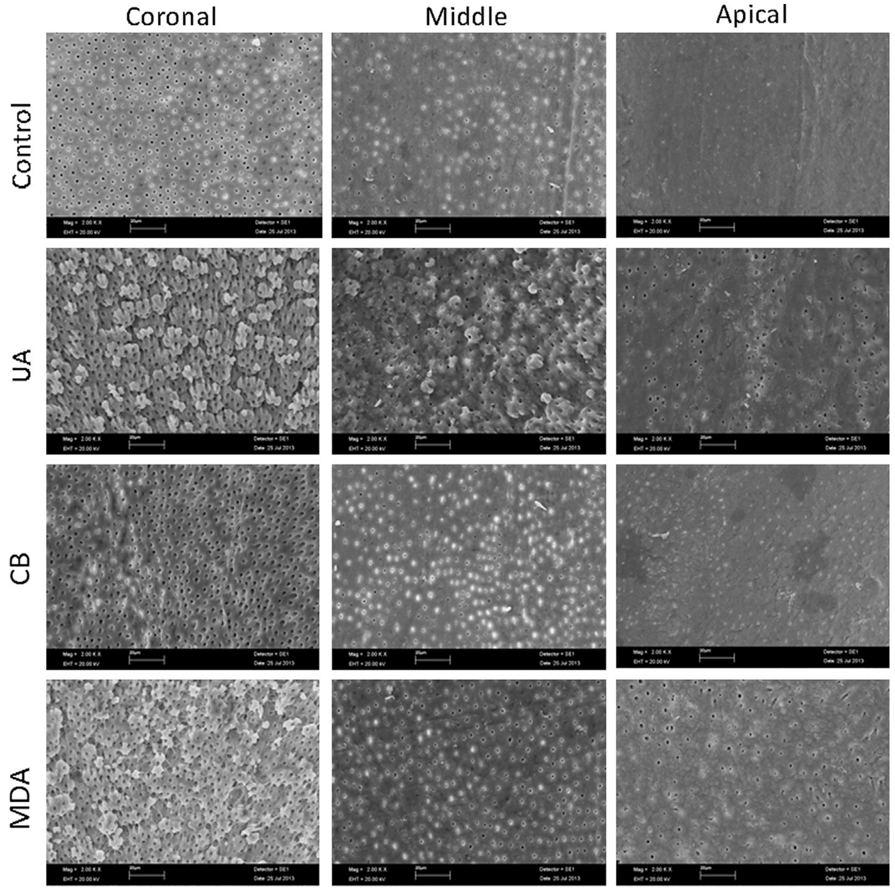

Figure 1. Representative scanning electron microscopic images showing selected samples from coronal, middle, and apical segments representing the different irrigant activation techniques (magnification ×2000).

was categorized into 1 of 3 failure modes: adhesive failure at the sealer dentin, cohesive failure within sealer, and mixed failure in both the sealer and dentin (20). Two specimens that were representative of the fracture modes from each group were further evaluated under a scanning electron microscope (Leo-440).

Statistical Analysis

The Kruskal-Wallis test was used to detect the effect of final irrigation activation techniques in the removal of the smear layer. Multiple comparisons were performed using the Mann-Whitney U test.

Two-way analysis of variance was used to detect the effect of the independent variables, final irrigant activation techniques (control, ultrasonic activation [UA], CB, and manual dynamic activation [MDA]) and root canal thirds (coronal, middle, and apical), and their interaction (final irrigant activation technique * root canal third) on push-out bond strength. The Tukey post hoc test was performed for multiple comparisons. The significance level was set at $P < .05$. All statistical analyses were performed using SPSS 16.0 software (SPSS Inc, Chicago, IL).

Results

The Removal of the Smear Layer

Figure 1 shows specimens examined using a scanning electron microscope at 2000× magnification. The Kruskal-Wallis statistic showed a significant difference among the groups with respect to the smear layer ($P < .05$). The Mann-Whitney statistic showed that the UA group had a higher number of open dentinal tubules in the coronal and middle thirds when compared with the other groups ($P < .05$). Also, the MDA group had a higher number of open dentinal tubules in the apical third than the other groups ($P < .05$).

Push-out Bond Strength

Mean bond strength values (MPa) after the push-out tests are shown in Figure 2. Two-way analysis of variance indicated that push-out bond strength values were significantly affected by both final irrigant activation techniques ($P < .05$) and root canal thirds ($P < .001$). There was a statistically significant interaction between final irrigant activation techniques and root canal thirds ($P < .05$). The coronal third had higher bond strength values than the middle third and the apical third ($P < .001$). The middle third had higher bond strength values than the apical third ($P < .001$).

In the coronal third, the bond strength in the UA group was higher than those in the control group ($P = .010$), CB ($P = .007$), and MDA groups ($P = .002$). In the middle third, the bond strength in the UA group was higher than those of the others ($P < .001$). In the apical third, the bond strength in the MDA group was higher than those in the control ($P < .001$), UA ($P = .002$), and CB groups ($P < .001$).

The modes of fracture are listed in Supplemental Table S1. (Supplemental Table S1 is available online at www.jendodon.com.) Cohesive failure was the most frequent type of failure in all groups except the apical region of the MDA group, which primarily exhibited the mixed failure mode.

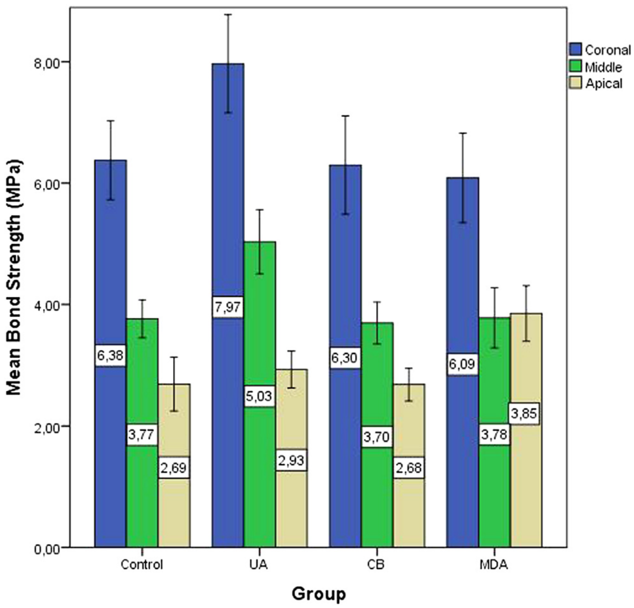

Figure 2. The push-out bond strength (MPa) of AH Plus sealer with respect to the test groups.

Discussion

In root canal obturation procedures, sealers are used to attain an impervious seal between the core materials and root canal walls. The high bond strength of a root canal sealer to intraradicular dentin through micromechanical retention or frictional resistance may be advantageous in maintaining the integrity of the sealer-dentin interface (21). Chemical adhesion between the dentin and sealer (with the exception of glass ionomer sealers) cannot be achieved. Therefore, it has been suggested that the mechanical interlocking of the sealer plug inside the dentinal tubules after smear layer removal may improve dislocation resistance of root filling materials (22). Dentin surface treatment with endodontic irrigants may affect the adhesion of sealers on root canal walls (23) because irrigants can alter the dentin surface composition (12). The null hypothesis was accepted based on the results of the present study.

In the present study, the bond strength primarily decreased in the coronal to apical direction. This result is comparable with results from several studies showing that the adhesion of root sealers generally decreased in the coronal to apical direction (24, 25). This can be explained by the decreasing tubule density from coronal to apical, which reduces sealer penetration into the smaller tubule diameter in the apical thirds (26, 27). The lack of access to the apical region of irrigation solutions and the consequent incomplete removal of the smear layer may decrease the penetration of sealer into dentinal tubules and may thereby affect adhesion in the apical region (28). In this sense, enhancing direct contact of the final irrigation solution with the entire canal wall can be helpful in improving the adhesion of sealer. Therefore, the present study focused on determining whether or not the bond strength of AH Plus sealer improves after different final irrigant activation techniques, especially in the apical region.

Recent studies have focused on the use of final irrigation activation techniques to provide maximum access of irrigation solutions to all areas of the root canal system and thereby optimize removal of the smear layer and to enhance sealer penetration. Moon et al (29) evaluated the effects of irrigant activation on sealer penetration, and their results showed that irrigant activation may improve sealer penetration into dentinal tubules. They also stated that sealer penetration into the tubules can be used as an indicator for smear layer removal and may improve retention of the filling material.

AH Plus sealer, which can be considered the gold standard material for testing endodontic sealers' resistance to dislodgment because it presents advantages in comparison with other materials usually used, was used in the present study (30, 31). The results of the present study showed that the bond strength of AH Plus sealer increased after the activation of final irrigants. In the coronal and middle thirds, UA improved the bond strength of the AH Plus sealer when compared with other groups, and MDA improved the bond strength of the sealer in the apical third. The CB group did not show an improvement in the adhesion of AH Plus sealer. Scanning electron microscopic images (Fig. 1) showed that the UA group had a higher number of open dentinal tubules in the coronal and middle thirds when compared with the other groups. Also, the MDA group had a higher number of open dentinal tubules in apical third than the other groups. Given these results, although a correlation between the bond strength and penetration of sealer has been unproven, the effective removal of the smear layer may improve the adhesion of AH Plus sealer with increased penetration of AH Plus sealer into dentinal tubules. It has been stated that good penetration, adaptation, and adhesion properties will have a positive effect on sealing because of the increased surface contact between the sealer and dentin (32). The adhesion provided by canal sealers eliminates the space that might otherwise allow fluid to infiltrate the sealer-dentine interface (31). This may also be one of the factors attributing to the long-term success of endodontically treated teeth.

The use of conventional needle irrigation without activation during final irrigation may cause gasses present in the apical region to form a vapor lock into which fluid penetration is difficult. Therefore, the various final irrigant activation techniques may be helpful in breaking the vapor lock effect and moving the solutions apically (33). In the current study, the MDA group had the highest bond strength values in the apical region. McGill et al (34) stated that the push-pull motion of a well-fitting gutta-percha point in the canal space might generate higher intracanal pressure changes during pushing movements, leading to more effective delivery of the irrigant to the "untouched" canal surfaces. Effective cleaning of the apical region can thereby be accomplished. The superior bond strength values of the MDA group in the apical region may be caused by increased sealer penetration depending on effective removal of the smear layer in the apical region.

In the present study, inspection of the root slices revealed that the fracture modes were mainly cohesive for all groups except the apical region of the MDA group as shown in Supplemental Table S1. (Supplemental Table S1 is available online at www.jendodon.com.) This is in agreement with the findings of Prado et al (35) who reported a cohesive failure pattern in the AH Plus sealer group. This result may be because of the high adhesion capacity of AH Plus sealer to the canal dentin (36).

Conclusions

Under the conditions of this study, it can be concluded that the bond strength of AH Plus sealer to root canal dentin may improve with UA in the coronal and middle thirds and MDA in the apical third. Further study is required to confirm this preliminary data and to investigate the bond strength of different root canal sealers after final irrigant activation protocols.

Acknowledgments

The authors deny any conflicts of interest related to this study.

Supplementary Material

Supplementary material associated with this article can be found in the online version at www.jendodon.com (http://dx.doi.org/10.1016/j.joen.2013.10.012).

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