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Effects of different irrigation agents on the bond strength of fiber posts to dentin structure

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Purpose: This study aims to investigate the push-out bond strength of fiber-reinforced posts following passive ultrasonic activation of the post space using EDTA, MTAD, Dual Rinse, and SmearOFF as final irrigation solutions, and to classify the associated modes of bond failure.

Methods: A total of 60 human mandibular premolar teeth were instrumented and obturated using a bioceramic-based root canal sealer. Post spaces were prepared using a fiber post drill set, maintaining 5 mm of the apical canal filling remained, and the samples were randomly divided into four groups (EDTA; MTAD; Dual Rinse; SmearOFF) (n=15). Intermittent passive ultrasonic activation protocols were applied to the final rinsing solutions. Fiber-reinforced posts were cemented into the post spaces using the self-adhesive resin cement Panavia SA Cement Universal.

Results: No statistically significant differences were found among the different final irrigation protocols applied. Although there was no statistical difference in the main effect, the main effect strength was highest in the EDTA group and lowest in the Dual Rinse group. Adhesive failure was the most frequently observed failure mode, while cohesive failures were observed at the lowest level.

Conclusion: EDTA, Dual Rinse, MTAD, and SmearOFF solutions have demonstrated comparable effects for post-space irrigation.

Keywords: Dual rinse; EDTA; SmearOFF; MTAD; push-out.

Introduction

Restoring endodontically treated teeth that have lost a significant portion of their coronal structure often necessitates the use of intra-radicular posts. Among the critical considerations during post-space preparation is the formation of a smear layer, which may hinder the bonding performance between resin-based cements and radicular dentin (1,2). To address this issue, clinicians commonly utilize sodium hypochlorite (NaOCl) in conjunction with chelating agents that facilitate the removal of inorganic remnants. One of the most frequently used agents for this purpose is ethylenediaminetetraacetic acid (EDTA), known for its effectiveness in eliminating the inorganic component of the smear layer. Nevertheless, prolonged exposure to EDTA may cause undesirable dentin erosion, especially when used beyond recommended durations (3).

MTAD, an irrigant composed of doxycycline, citric acid, and a detergent (polysorbate), has shown promise in effectively removing the smear layer, especially in the apical third of root canals. Compared to EDTA, MTAD has been

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associated with reduced dentin erosion in the coronal and middle thirds. However, due to its lack of organic tissue dissolution capability, MTAD is typically used in conjunction with NaOCl (4,5). Previous in vivo studies suggest that sequential irrigation with 1.3% NaOCl followed by MTAD produces postoperative outcomes comparable to those achieved using 5.25% NaOCl and 17% EDTA (6). The widely recommended clinical approach involves a 20-minute rinse with NaOCl followed by a 5-minute application of MTAD (7).

Dual Rinse HEDP represents a newer generation of irrigants, offering a simplified regimen by combining etidronic acid with NaOCl (8). This formulation maintains the tissue-dissolving and antimicrobial capabilities of NaOCl for up to one hour and prevents smear layer formation during mechanical instrumentation (9). One of the key advantages of Dual Rinse is its efficiency; by eliminating the need to alternate between separate chelators and disinfectants, it reduces procedural time and complexity.

SmearOFF is a one-step irrigation solution composed of 18% EDTA and a small concentration of chlorhexidine (CHX) (<1%), formulated to remove the smear layer and provide disinfection simultaneously (10). It has a near-neutral pH of approximately 9 (11), and unlike conventional CHX solutions, has been shown not to form precipitates when used after NaOCl, eliminating the need for an intermediate rinse with saline or distilled water (10,12,13).

Panavia SA Cement Universal, the self-adhesive resin cement used in this study, integrates the application ease of conventional cements with the mechanical strength and esthetic benefits of resin cements (14). Its dual-cure nature enables reliable polymerization even in deeper regions where light penetration is limited. This one-step material requires no separate surface treatment, reducing technique sensitivity and minimizing the risk of post-operative sensitivity.

This study explores the influence of different final irrigation protocols—EDTA, MTAD, Dual Rinse, and SmearOFF—on the interfacial strength of glass fiber posts, as assessed through push-out analysis. Each solution was activated using passive ultrasonic activation (PUA) to improve debridement efficacy. The research also aims to classify the predominant failure modes observed during bond strength testing, offering additional insights into the adhesive interaction between dentin, fiber posts, and resin cements.

In this study, the null hypothesis (H_0) stated that the final irrigation protocol would have no statistically significant effect on the push-out bond strength of fiber posts to root canal dentin.

Materials and Methods

The present study, approved by the Local Ethics Committee of Dicle University Faculty of Dentistry (No: 2023/19, Date: 26/04/2023) and conducted according to the Declaration of Helsinki. All experimental procedures were conducted in the research laboratories of the Faculties of Dentistry at Dicle University and Selçuk University (Konya, Türkiye). A total of 60 human mandibular premolars extracted for periodontal reasons were used. Only teeth with single straight canals, mature apices, and no visible cracks or caries were included. Teeth with root fractures, previous endodontic treatment, resorption, or canal calcification were excluded. All procedures in this study were performed by a single operator. The sample size was determined by a priori power analysis using G Power software (version 3.1, University of Düsseldorf, Germany). Based on an alpha level of 0.05, a power of 0.80, and a medium effect size, the analysis indicated a requirement of 45 observations per solution group (15 samples × 3 root regions), totaling a minimum of 180 observations.

The working length for each specimen was determined using a #15 K-file (Dentsply Maillefer, Ballaigues, Switzerland) placed 1 mm short of the major apical foramen. No magnification system (such as dental loupes or an operating microscope) was used during working length determination or any other procedural step. Root canal instrumentation was performed with T-Endo MUST NiTi rotary files (Dentac, Istanbul, Türkiye), utilizing M25 (25/06) and M40 (40/04) file sequences in combination with an Ai-motor endomotor (Woodpecker, Guilin, China). During canal shaping, 2 mL of 5% NaOCl (Microvem, Istanbul, Türkiye) was delivered following each file change using a 27-gauge open-ended syringe (Ultradent, South Jordan, UT, USA). The total NaOCl volume per canal amounted to 10 mL. Following chemomechanical instrumentation, 2 mL of 17% EDTA (Promida Ltd., Eskişehir, Türkiye), was applied to each canal for one minute. Subsequently, a final rinse was performed using 3 mL of 5% NaOCl. To facilitate irrigant activation, the final NaOCl rinse was subjected to passive ultrasonic agitation using an ultrasonic tip (Irrisafe, #20–21; Satelec, Merignac-Cedex, France) positioned 1 mm short of the established working length. Activation was carried out for 30 seconds, at a frequency of 30 kHz and medium power setting, as recommended by the manufacturer, after which the canals were rinsed with distilled water to eliminate any residual irrigant.

The canals were then dried using sterile absorbent paper points (President Dental, Munich, Germany). Obturation was performed with the single-cone technique using gutta-percha cones (Dentsply Maillefer, Ballaigues, Switzerland) and a bioceramic-based root canal sealer (AH Plus Bioceramic Sealer; Dentsply Sirona, Erlangen, Germany). Coronal orifices were sealed with a temporary restorative material (Cavit; 3M ESPE, Seefeld, Germany), and the teeth were stored in distilled water at 37°C for one week to ensure complete setting of the sealer prior to post-space preparation.

Post gaps measuring 9 mm in depth were created using a fiber post drill system, while ensuring that 5 mm of apical filling material remained intact. Following preparation, the 60 specimens were randomly assigned to four experimental groups, each comprising 15 samples.

- Dual Rinse (Medcem GmbH, Weinfelden, Switzerland) group: 10 mL Dual Rinse, 1 min PUA
- EDTA group: 5 mL 5% NaOCl + 5 mL 17% EDTA, 1 min PUA
- MTAD (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA) group: 5 mL 1.3% NaOCl + 5 mL MTAD, 1 min PUA
- SmearOFF (Vista Dental Products, Racine, WI, USA) group: 5 mL 5% NaOCl + 5 mL SmearOFF, 1 min PUA

The PUA protocol involved intermittent application, with 2.5 mL of solution followed by 30 seconds of activation, repeated twice.

After drying with absorbent paper points (President Dental, Munich, Germany), fiber posts (Exacto No: 2, Angelus, Londrina, PR) were cemented using Panavia SA Cement Universal (Kuraray, Okayama, Japan). The resin cement was applied using an intra-canal tip, excess cement was removed, and polymerization was performed using an LED curing unit (Bluephase 20i, Ivoclar Vivadent, Liechtenstein) at 1200 mW/cm^2 for 20 seconds.

The samples were embedded in cold acrylic resin (Meliodent Rapid Repair, Heraeus-Kulzer) using cylindrical plastic molds, ensuring that the coronal surface of the tooth was level with the mold surface. Using a Microcut device (Microcut 201; Metkon, Bursa, Türkiye), an initial 1 mm thick cervical section was removed. Then, three consecutive transverse sections of 2.0 ± 0.1 mm thickness were obtained from each root. Specimens were categorized into apical, middle, and cervical thirds, and mounted on a mechanical testing device (Lloyd LS series; Ametek Inc., Lloyd Instruments, Fareham, UK), with the apical end oriented upward.

To dislodge the fiber posts from the canals, a force was applied using custom push-out pins with diameters of:

- 0.8 mm for the cervical region,
- 0.5 mm for the middle region,
- 0.3 mm for the apical region.

The loading speed was set to 1 mm/min.

Fracture load values were measured in Newtons (N) using Nexygen software integrated with the Lloyd LRX testing system (Fareham, UK). The bond strength was then calculated by converting the maximum load values to Megapascals (MPa) using the following formula:

$$\text{Bond strength (MPa)} = \frac{\text{Maximum load (N)}}{\text{Bonding area of the fiber post}}$$

For each section, the bonding area of the fiber post (mm2) was calculated using the formula:

Bonding area=2πrh

r represents the radius of the fiber post,

h represents the thickness of the section (in mm),

π (pi) was taken as 3.14.

The specimens subjected to the push-out bond strength test were subsequently evaluated under a stereomicroscope (Axiocam MRC, Carl Zeiss, Germany) at 25× magnification to determine the mode of failure.

Statistical Analysis

Statistical analyses were performed using IBM SPSS Statistics, Version 23 (IBM Corp., Armonk, NY, USA) and JAMOVI, Version 2.3.28 (Sydney, Australia). The Shapiro-Wilk test was utilized to evaluate the normality of data distribution. Two-Way Robust ANOVA from the Walrus package was utilized to compare bond strength across different irrigation solutions and regions, with Bonferroni correction applied for multiple comparisons. Final data were presented as median values accompanied by the lowest and highest ranges. A significance level of $p<0.050$ was considered.

Results

A statistically significant interaction was observed between the irrigation solution and root region ($p<0.001$) (Table 1). The highest median bond strength was recorded in the coronal region with Dual Rinse (7.92 MPa), while the lowest was in the apical region with Dual Rinse (1.01 MPa). However, statistical analysis revealed no meaningful variation across the irrigation protocols or root canal regions ($p>0.050$).

The failure modes observed following the push-out test, categorized by irrigation solution, are presented as frequencies and percentages in Table 2.

Table 1. Descriptive statistics (median, minimum, and maximum values) of bond strength according to irrigation solution and region, along with multiple comparison results

Irrigation solution	Region			
	Apical	Middle	Coronal	Total
Dual Rinse	1.01 (0.65 – 1.42)^{A}	6.51 (1.48 – 17.79)^{AB}	7.92 (2.97 – 19.58)^{B}	4.43 (0.65 – 19.58)
EDTA	5.14 (0.1 – 23.81)^{AB}	3.05 (1.33 – 13.27)^{AB}	5.95 (1.43 – 47.92)^{AB}	5.14 (0.1 – 47.92)
MTAD	3.12 (0.13 – 26.77)^{AB}	6.54 (1.04 – 17.04)^{AB}	4.89 (1.34 – 28.17)^{AB}	4.89 (0.13 – 28.17)
SmearOFF	3.59 (0.78 – 28.01)^{AB}	3.9 (1.1 – 13.48)^{AB}	7.6 (1.33 – 19.7)^{AB}	5 (0.78 – 28.01)

$^{A\text{-}B}$: Interactions with the same letter indicate no significant difference; Median (Minimum - Maximum).

Table 2. Frequency and percentage distribution [n(%)] of adhesive, cohesive, and mixed failure types for each solution.

Failure types	EDTA	MTAD	SmearOFF	Dual Rinse
Adhesive	32 (7.1%)	28(62.2%)	21 (46.6%)	35 (77.7%)
Cohesive	3 (6.6%)	5 (11.1%)	8 (17.7%)	1 (2.2%)
Mixed	10(22.2%)	12(26.6%)	16 (35.5%)	9 (20%)

Discussion

The ease of root canal sealer removal during post-space preparation is critical for the proper cementation and retention of fiber posts. Effective decontamination of the dentin surface during canal preparation significantly contributes to improved bond strength. In a comparative study, Vilas et al. (15) evaluated the adhesion of fiber posts in canals filled with Endofill, EndoSequence BC, and AH Plus sealers. Their findings showed that AH Plus was more easily removed from the canal walls compared to the other sealers, resulting in higher bond strength values for fiber post cementation. The present study used AH Plus Bioceramic Sealer, an improved and advanced version of AH Plus. This research is novel because AH Plus Bioceramic Sealer is a contemporary material that has not yet been widely studied in cavities prepared for post-space.

Evidence suggests that a one-week waiting period after root canal obturation positively affects the bond strength of fiber posts and resin cement (16,17). Therefore, in our study, fiber post-placement was performed after a one-week waiting period to ensure the complete setting of the root canal sealer.

The resin cement used during fiber post application is intended to ensure seamless integration between the post and the tooth, creating a structure resistant to forces and forming an effective barrier against microleakage (18). Therefore, selecting an appropriate cement is crucial for achieving a successful bond. Self-adhesive resin cements are widely favored for their simplified application protocol, as they reduce technique sensitivity and eliminate the need for separate bonding agents or surface conditioning steps (19). However, during post-cementation, the progressive reduction in light penetration towards the apical region can result in insufficient photoactivation, leading to polymerization issues (20). To mitigate these drawbacks, our study utilized Panavia SA Cement Universal, a dual-cure self-adhesive resin cement that ensures reliable polymerization even in deeper regions.

The integrity of the interface among dentin, fiber post, and resin cement plays a pivotal role in determining the retention of fiber posts (21) Clearing the debris-rich layer that accumulates along canal walls during post instrumentation is widely regarded as a key step in promoting retention and optimizing the adhesive interface. If not adequately removed, the smear layer can occlude dentinal tubules, impair adhesion, and ultimately compromise the micromechanical interlocking between the post and the tooth structure (22,23). Consequently, meticulous cleaning of the root canal dentin surface is essential prior to fiber post cementation to ensure optimal bonding (23). The standard protocol for smear layer removal involves the application of 17% EDTA following NaOCl irrigation (24). Initially, NaOCl dissolves organic tissues within the root canal, while subsequent EDTA irrigation targets inorganic debris, effectively cleansing the canal walls and restoring the patency of dentinal tubules by eliminating smear layer residues (25).

The role of ultrasonic systems in improving the efficacy of irrigants during smear layer removal has been highlighted in multiple studies. Guo et al. (26) emphasized that ultrasonic activation serves as a beneficial adjunct, contributing significantly to enhanced canal cleanliness. Supporting this, Dioguardi et al. (27) demonstrated that passive ultrasonic activation (PUA) facilitates apical debridement by producing high-frequency acoustic turbulence and cavitation within the canal. Kuah et al. (28) further observed that a 60-second ultrasonic activation of 17% EDTA resulted in superior removal of smear remnants from the apical third. The performance of the EDTA group in the present study aligns with these previously reported findings.

Although no statistically significant difference was observed among regions within the EDTA group, the apical region demonstrated higher bond strength than the middle region. However, in their studies, Gu and Guo suggested that PUA has a similar effect to conventional irrigation techniques (22,26). The variability in outcomes with PUA may be attributed to differences in experimental setups and material choices. A control group without PUA was not included in the current study, as the primary aim was to compare the relative performance of different final irrigants under standardized activation conditions. The omission of an unactivated control group allows a focused evaluation of irrigant efficacy under clinically relevant agitation protocols.

Tay et al. (29) reported that MTAD exerts a more pronounced demineralizing effect on sound intraradicular dentin compared to EDTA, resulting in collagen matrices that are approximately 1.5 to 2 times thicker than those produced by EDTA. These enhanced matrices promote dentin hybridization with hydrophilic methacrylate-based cements without the need for phosphoric acid etching or using self-etch resin cement. While previous studies have indicated that root canals irrigated with MTAD and obturated with AH Plus achieve excellent coronal sealing (30), the thicker intraradicular collagen matrices generated by MTAD may compromise resin infiltration, leading to potential bonding deficiencies (31). Unlike the open leakage typically observed in the coronal region, incomplete resin infiltration within these matrices may result in nanometer-scale leakage (29). Additionally, Yurdagüven et al. (32) found that MTAD application significantly reduces the bonding strength of self-etch adhesives to coronal dentin. Consistent with these findings, our study demonstrated that the MTAD group presented lower bond strength in the coronal region compared to the middle region. This outcome is likely attributed to the intraradicular collagen matrices formed by MTAD, which may have hindered resin infiltration, particularly when using self-adhesive cement.

Erik et al. (11) reported that irrigation with SmearOFF significantly reduced post-bond strength. However, in our research, SmearOFF demonstrated the second-highest bond strength after EDTA. This difference may be attributed to the chlorhexidine (CHX) content in SmearOFF, which acts as a matrix metalloproteinase (MMP) inhibitor, preventing collagen degradation at the bonding interface. The suppression of collagen matrix degradation may have been a contributing factor to the improved bond strength documented in our findings (33).

According to Erik et al. (11), post-space irrigation using Dual Rinse resulted in apical and middle region bond strength values comparable to those attained with EDTA. Unlike some earlier reports, the present study showed that the coronal and middle thirds of the root canals exhibited the highest bond strength values. Although no statistically significant differences were identified among the final irrigation agents evaluated, a notable difference was detected between the coronal and apical segments in specimens treated with Dual Rinse. In light of these outcomes, the null hypothesis (H_0) was only partially supported.

Several factors may account for the progressive decline in bond strength observed from the coronal toward the apical region. Factors contributing to this decline include the restricted penetration of irrigation solutions into the apical third, largely attributed to insufficient solution volume, and the heightened anatomical complexity of the apical root canal system. In addition, the crown-down instrumentation technique leads to a higher accumulation of dentin debris within the apical third tubules, potentially compromising adhesion. The gradual reduction in both the quantity and surface area of dentinal tubules in the apical region also plays a significant role in the observed decline in bond strength (34–36).

Beyond tubular patency and dentin morphology, the choice of self-adhesive resin cement in our study also played a pivotal role in the outcomes. This observation is in accordance with the findings of Topçu et al. (37), who noted a lower bond strength in the apical region, primarily attributed to the superior accessibility of the coronal third for both adhesive application and light-curing energy. Moreover, prior studies have suggested that both hydrostatic conditions and the efficiency of adhesive light activation are impaired in the apical third, which is regarded as a key factor contributing to the reduced bond strength in this area (38). The bond failure modes were examined under 25× magnification using a stereomicroscope to assess the characteristics of interfacial disruption. Analysis revealed that adhesive failure was the predominant mode observed across all specimens.

The adhesive interface is commonly recognized as the most vulnerable area post-cementation, with adhesive failure between dentin and resin cement representing a critical issue in push-out bond strength evaluations (39). This study utilized Panavia SA Cement Universal, adhering strictly to the manufacturer's guidelines, without supplementary surface treatment. According to the manufacturer, Panavia SA is a one-step, user-friendly cement that provides reliable adhesion to diverse materials without requiring an additional primer. Nonetheless, the results of our study suggest that this cement did not attain optimal bonding efficacy. Fiber posts were cemented one-week post-root canal obturation in our investigation. Previous studies by Yuanli and Alsubait revealed that fiber posts cemented immediately following root canal treatment in teeth obturated with bioceramic sealers revealed enhanced bond strength (40,41). As a result, the one-week delay before post-cementation in our study may have negatively influenced the bonding performance, potentially leading to adhesive failure.

Despite the strengths of this study, several limitations should be acknowledged. The lack of standardization in NaOCl concentration among groups, use of a single resin cement type, and deviation from manufacturer-recommended MTAD application times may have introduced bias. Additionally, omitting distilled water or NaOCl rinsing after EDTA, as well as the unassessed thermal effects of passive ultrasonic activation on periodontal tissues, restrict the generalizability of these results. Further studies are needed to establish safe and effective PUA protocols under clinically relevant conditions.

Conclusion

This study highlights the substantial impact of post-space irrigation protocols on the bond strength of fiber posts to root canal dentin. Although all tested final irrigation solutions—EDTA, MTAD, Dual Rinse, and SmearOFF—yielded comparable overall bond strength outcomes when combined with passive ultrasonic activation, region-specific discrepancies were evident. Notably, the Dual Rinse group exhibited a statistically significant decrease in bond strength in the apical third compared to the coronal region. The predominance of adhesive failure modes underscores the susceptibility of the resin–dentin interface to variations in surface treatment. These findings suggest that, while the irrigation solutions appear similarly effective on a general level, their performance may vary across different regions of the root canal, particularly in the apical third, where reduced adhesion continues to pose clinical challenges. Therefore, further studies are necessary to clarify the physicochemical characteristics of these irrigation solutions and their interactions with dentinal substrates, especially in the anatomically complex apical areas of the root canal system.

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