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Effects of two different chelation solutions and activation protocols on the push-out bond strength of a bioceramic sealer

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Abstract

Aim: This study evaluated the effects of glycolic acid (GA) and ethylenediaminetetraacetic acid (EDTA) chelation solutions on the push-out bond strength of AH Plus Bioceramic Sealer when activated using passive ultrasonic irrigation (PUI) and XP-endo Finisher (XPF) activation protocols.

Methods: A total of 48 similarly sized extracted mandibular premolars were randomly assigned to four groups (n = 12). All canals were prepared using Reciproc (25.08) instruments. Irrigation was performed using 5 ml of 5% sodium hypochlorite (NaOCl) between instruments, totaling 20 ml of NaOCl per canal. The teeth were divided into four groups for the final irrigation: Group 1: GA+PUI; Group 2: GA+XPF; Group 3: 17% EDTA+PUI; and Group 4: 17% EDTA +XPF. NaOCl was activated for 1 minute in all groups, followed by chelator activation for 30 seconds with the designated activation system. Canals were filled using the single-cone technique and AH Plus Bioceramic Sealer. The samples were incubated for one week. A single horizontal middle dentin section of 2 ± 0.1 mm thickness was prepared from each root. Push-out bond strength was tested using a universal testing machine with a loading rate of 0.5 mm/min. The data were analyzed using the Kruskal-Wallis H and Mann-Whitney U tests, with a $p < 0.05$ significance level.

Results: There were no statistically significant differences among the GA+PUI, GA+XPF, EDTA+PUI, and EDTA+XPF four groups ($p > 0.05$).

Conclusion: Both EDTA and GA showed similar bond strength effectiveness when activated by either irrigation activation system.

Keywords: Passive ultrasonic irrigation, XP-endo Finisher, push-out bond strength, EDTA, glycolic acid

Introduction

Eliminating pathogenic microorganisms via chemical and mechanical preparation facilitates filling root canals three-dimensionally, ensuring endodontic treatment success (1). Sealing root canals with a chemically inert, biodegradable material is fundamental to success. Preventing microleakage and achieving a hermetic seal requires the root canal sealer to adhere appropriately to both the radicular dentin and gutta-percha. Failure at either interface (either the gutta-percha-sealer or dentin-sealer) can result in microleakage and affect adhesion (2). Adhesion is the ability of the root canal sealer to bond to the radicular dentin wall and gutta-percha, while cohesion is the ability of the sealer and master cone to bond to the canal wall, forming a monoblock structure (3). Enhancing adhesive bonding by obtaining a clean dentin surface is essential for long-term success. One way to increase bond strength is to remove the smear layer (4).

Ethylenediaminetetraacetic acid (EDTA) forms soluble chelate complexes by binding calcium (Ca^{+2}) ions with its diamine components (5, 6). After instrumentation, a 1-minute application of 17% EDTA during root canal treatment is recommended to effectively remove the smear layer's inorganic components and dentin debris (7). Despite its self-limiting chelation mechanism, prolonged use of high concentrations of EDTA can cause dentin erosion (8). A study has shown that glycolic acid (GA), a fruit acid, can remove the smear layer with lower toxicity than EDTA (9). Glycolic acid (GA) has been supported as an alternative irrigant in root canal treatment, effective at neutral or acidic pH, and has been shown to cause less erosion on dentin's mechanical properties than EDTA (10).

The British Endodontic Society recommends manual agitation, ultrasonic activation, negative pressure irrigation, and heat activation for sodium hypochlorite (NaOCl), EDTA, and chlorhexidine (11). Passive ultrasonic activation (PUI) can disrupt endodontic biofilm, facilitating better penetration of irrigants along endodontic dentin walls. Various studies have shown that ultrasonic activation effectively removes the smear layer in apical and isthmus regions (12).

The XP-endo Finisher file system (XPF; FKG Dentaire SA, La Chaux-de-Fonds, Switzerland) is highly flexible and made from Ni-Ti Max-Wire alloy, designed to improve canal disinfection after root canal shaping. With an ISO #25 diameter and zero taper, this file transforms from a straight form in the martensitic phase (M phase) below 20°C to a spoon-like shape in the austenitic phase (A phase) at body temperature, expanding apically (13). This adaptability allows XPF to reach canal areas that would be inaccessible using standard irrigation protocols, enhancing irrigation efficiency (14).

To the best of our knowledge, no study has evaluated the bond strength of a bioceramic sealer activated by chelators using similar experimental protocols with XP-endo Finisher and PUI. This study aims to investigate the effects of the final irrigation agents EDTA and GA on the bond strength of AH Plus Bioceramic sealer when activated using XP-endo Finisher and PUI. The null hypothesis is that the type of activation does not significantly affect the bond strength of the chelators.

Materials and Methods

This study received ethical approval from the Local Ethics Committee of the Faculty of Dentistry at Dicle University, and the protocol number was 2024-15.

Sample size calculation

Based on a similar study, the sample size was calculated using G*Power 3.1 software (Heinrich Heine University, Düsseldorf, Germany). The calculation indicated that at least six sections per group were needed with 95% confidence (1-α), 96.4% test power (1-β), and f = 0.972 (15). To ensure robustness, the study was conducted on 48 teeth.

Sample selection

Forty-eight mature, single-rooted, single-canal mandibular premolars extracted for orthodontic or periodontal reasons, without cracks or fractures, were used. Soft and hard tissue residues were removed with periodontal curettes, and the teeth were stored in saline solution at room temperature.

Sample preparation and grouping

The access cavities were prepared by removing the pulp chamber roof with a diamond pear-shaped red size 2 bur (Komet, Lemgo, Germany) and shaped with an Endo-Z bur (Dentsply Maillefer, Tulsa, USA). An ISO 10 K-type file (VDW, Munich, Germany) was used to measure the working length after the root canals were pathfinded using an ISO 06 C-Pilot file. The file was then inserted into the root canal and gently guided in the apical direction until its tip appeared at the physiological foramen limits under magnification (7.5×). After access cavity preparation, the apical patency and canal count were confirmed using a #15 K-file (Dentsply Maillefer, Ballaigues, Switzerland). Teeth were decoronated to standardize to a length of 15 mm. Working lengths were determined to be 1 mm short of the major apical foramen. Teeth were embedded in silicone molds and cold acrylic (Imicryl SC; Imicryl Dental Materials, Inc, Konya, Türkiye) to form blocks.

All irrigation procedures were conducted at 37°C to simulate intraoral temperature using a water bath (JSR Research Inc., Korea). Pre-flaring was performed with #15 and #20 K-files, followed by shaping with Reciproc R25 (25.08) (VDW, Munich, Germany) and endodontic motor (X-Smart Plus, Dentsply Sirona, Ballaigues, Switzerland) per the manufacturer's instructions.

Irrigation was performed using a 31-gauge side-vented TruNatomy irrigation needle (Dentsply Maillefer, Ballaigues, Switzerland) with a total of 20 ml 5% NaOCl per canal in four 5 ml increments before final irrigation. Forty-eight mandibular premolar teeth were randomly assigned to four groups (n = 12) (Fig. 1).

Group 1: Rinsed with 5 ml 5% NaOCl and activated with PUI for 1 minute (Eighteeth Ultra X, Eighteeth, Jiangsu, China) ultrasonic device set to power mode 2 with 20.02 tip), followed by 5 ml distilled water (DW), then 5 ml 17% GA activated with PUI for 30 seconds, and final rinse with 5 ml DW.

Group 2: Rinsed with 5 ml 5% NaOCl and activated with PUI for 1 minute, followed by 5 ml DW, then 5 ml 17% GA activated with XPF for 30 seconds, and final rinse with 5 ml DW.

Group 3: Rinsed with 5 ml 5% NaOCl and activated with PUI for 1 minute, followed by 5 ml DW, then 5 ml 17% EDTA activated with PUI for 30 seconds, and final rinse with 5 ml DW.

Group 4: Rinsed with 5 ml 5% NaOCl and activated with PUI for 1 minute, followed by 5 ml DW, then 5 ml 17% EDTA activated with XPF for 30 seconds, and final rinse with 5 ml DW.

The root canals were filled using the single-cone technique with AH Plus Bioceramic sealer. Access cavities were sealed with the temporary filling material (3 M ESPE, St. Paul, MN, USA). Samples were incubated at 37°C with 100% humidity for one week.

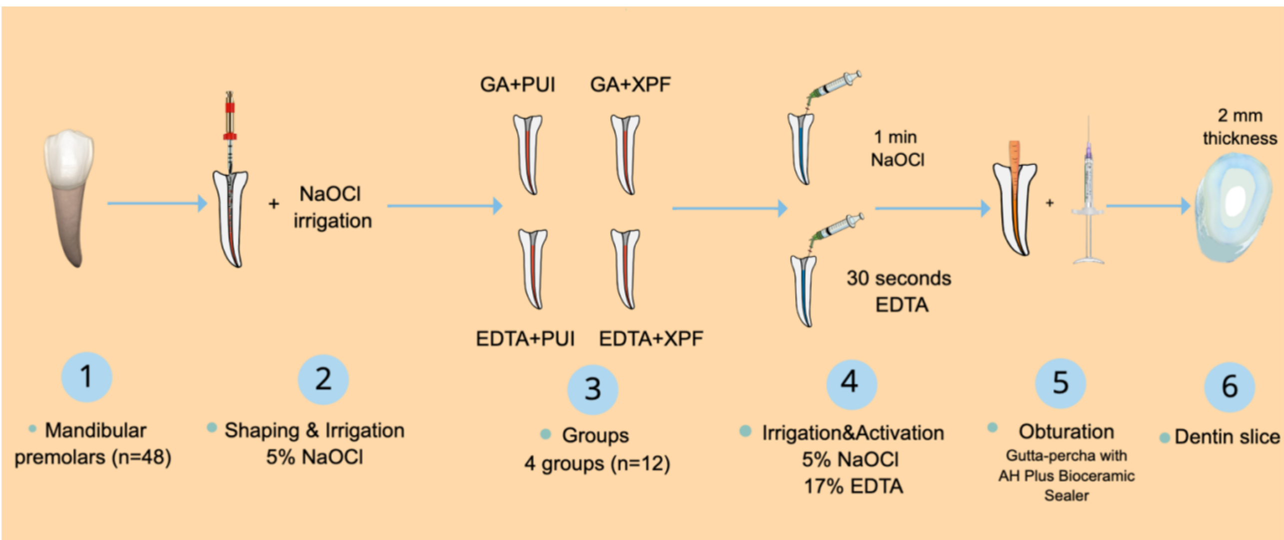

Figure 1. Schematic representation of the step-by-step procedure and dentine slice acquisition.

Preparation for push-out testing

Teeth embedded in acrylic were sectioned horizontally at 5 mm and 7 mm levels from the root apex using a precision cutting device (Isomet 1000, Buehler, Lake Bluff, IL, USA) with a 0.3 mm diamond blade (Metkon, Microcut precision cutter, Bursa, Turkey). Section thickness was measured with a digital caliper (Mitutoyo Corp., Tokyo, Japan) to obtain 2±0.1 mm slices.

Push-out test

The samples were subjected to a push-out test using a universal testing machine (Instron, Canton, MA, USA) with a piston speed of 0.5 mm/min. The test tip was positioned to contact only the repair material. Vertical force was applied to the gutta-percha. The maximum force at the point of material dislocation was recorded in Newtons. This force value was then divided by the interfacial area of the cement and dentin to convert it to megapascals (MPa). The values were recorded according to the groups.

Statistical analysis

Analyses were performed by using SPSS 25.0 software. (IBM Inc., Armonk, New York, USA).

The Shapiro-Wilk test revealed that the data did not follow a normal distribution. The Kruskal-Wallis H test was used to determine statistically significant differences among the groups. Mann-Whitney U test was employed for pairwise comparisons. The alpha level was set at 0.05.

Results

Descriptive statistics for the groups are listed in Table 1 and visualized in Figure 2. No statistically significant differences were found among the GA+PUI, GA+XPF, EDTA+PUI, and EDTA+XPF groups ($p>0.05$).

Failure distribution is given in Table 2. While adhesive failure was seen in the GA+PUI and EDTA+XPF groups, it was not seen in the other groups.

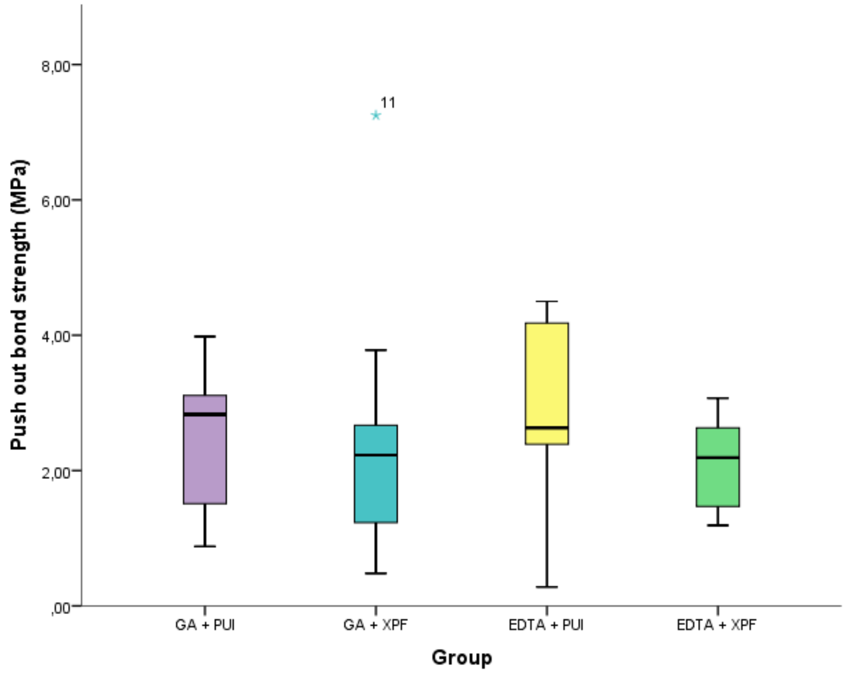

Figure 2. Visualization of median bond strength values using a box-plot graph

Table 1. Mean, standard deviation (SD), median, minimum (min), and maximum (max) values of push-out bond strength (MPa) for each group.

Group	Mean + SD	Median (Min - Max)
GA + PUI	2.47 ± 1.04	2.83 (0.88-3.98)
GA + XPF	2.49 ± 1.92	2.23 (0.48-7.25)
EDTA + PUI	2.84 ± 1.29	2.63 (0.28-4.5)
EDTA + XPF	2.06 ± 0.65	2.19 (1.19-3.07)

Abbreviations: GA: glycolic acid, **PUI:** passive ultrasonic activation, **EDTA:** ethylenediamine tetraacetic acid, **XPF:** XP-endo Finisher

Table 2. Distribution of failure types by groups

Group	N	Adhesive	Cohesive	Mixed (Adhesive + Cohesive)
GA+PUI	12	1	11	-
GA+XPF	12	-	9	3
EDTA+PUI	12	-	10	2
EDTA+XPF	12	2	7	3

Abbreviations: GA: glycolic acid, PUI: passive ultrasonic activation, **EDTA:** ethylenediamine tetraacetic acid, **XPF:** XP-endo Finisher

Discussion

This study tested the effects of two different chelating agents, activated using two different methods, on the bond strength of a bioceramic sealer. Given the lack of significant differences among the groups, the null hypothesis was accepted.

In endodontics, the push-out bond strength test evaluates the resistance of filling materials, the retention of posts, and the adhesion of sealers to dentin. It also provides good results for assessing intra-canal bonding strength (16). In a study testing the bioactive potential and cytocompatibility of the newly introduced AH Plus Bioceramic sealer, it was reported that this sealer exhibited significantly higher cytocompatibility and bioactive potential than AH Plus, while showing similar properties to Endosequence BC sealer. The researchers supported the use of this promising sealer in root canal treatment (17). Our study obtained results supporting its clinical use by testing it with different chelation solutions and activation methods.

A study investigating the effects of different acidic solutions (GA, phosphoric acid, citric acid, and EDTA) used as final irrigation agents on the push-out bond strength of resin- and bioceramic-based (AH Plus and Bioserra) root canal sealers found that GA groups exhibited significantly higher bond strength than EDTA groups. Additionally, both GA and EDTA solutions significantly increased the bond strength of the bioceramic sealer compared to the resin-based sealer. However, in that study, the EDTA solution was 10%, unlike our study, where both EDTA and GA solutions were used at 17% concentration (18). In our study, no differences were observed in bond strength between the EDTA (17%) and GA (17%) groups. This could be interpreted as the lower concentration of EDTA negatively affecting bond strength and the effectiveness being significantly influenced as the concentration of EDTA approached that of GA.

In a study by Souza et al., the effects of GA, EDTA, and QMix on smear layer and root dentin bond strength were evaluated with and without activation (PUI). GA increased bond strength more than EDTA, both alone and with activation (12). The authors attributed this increase to the small size and acidic pH of GA molecules facilitating effective penetration into the root dentin (9). Although our study did not show statistically significant differences, the highest median bond strength value (2.83 MPa) was obtained with GA solution in the PUI activation group. XPF did not improve bond strength as much as PUI in activating both solutions. This could be due to the cavitation effect produced by high-frequency vibrations in PUI, as suggested by the working principles of the two activation systems (19).

In a study using EDTA as the final irrigation agent, activation was performed with syringe needle irrigation, PUI, and XPF, and the bond strength of two different sealers was tested. The results showed no significant difference between PUI and XPF, while no significant difference was found between PUI and syringe needle irrigation with lower mean MPa values (20). The authors could not explain this but attributed it to the lack of cavitation or bubble effect in XPF. Similarly, in our study, the EDTA group showed lower median bond strength values with XPF compared to PUI.

Cohesive failure indicates the maximum adhesion strength of materials, while adhesive failure, which occurs due to weak bonding, is an undesirable failure type (21). The most common failure type in all groups was cohesive. Only one sample in the GA - PUI group showed adhesive failure, while two samples in the EDTA + XPF group showed adhesive failure. Therefore, the median bond strength values of these two groups are consistent with the failure types.

There are studies using XPF and PUI for EDTA activation, but no study has evaluated the bond strength of a bioceramic sealer activated by GA with XPF. The standardized irrigation protocol is a strength of our study.

However, like all in vitro studies, it has some limitations. Given that dentin tubule permeability affects sealer penetration, the lack of standardization in the age group of extracted teeth could have led to differences in tubule diameter, potentially affecting our results. Additionally, bond strength values examined only in the middle section do not represent the entire tooth.

Conclusion

When either EDTA or GA is used as the final irrigation agent, both PUI and XPF activation improve the bond strength of a bioceramic sealer to root canal walls to a similar extent. Further studies are needed to evaluate the bond strength of GA activation with XPF.

Disclosures

Acknowledgment: This study was verbally presented at the 10th International Endodontic Symposium, May 19-22, Antalya, Türkiye.

Ethical Approval: Ethics committee approval was received for this study from the Dicle University, Faculty of Dentistry, Local Ethics Committee, in accordance the World Medical Association Declaration of Helsinki, with the approval number: 2024-15.

Peer-review: Externally peer-reviewed.

Author Contributions: Concept – M.B.; Design – M.B., M.Y.Ç.; Supervision – M.Y.Ç., H.K.; Materials – M.B., M.Y.Ç., H.K.; Data collection &/or processing – H.K., G.R.T.A.; Analysis and/or interpretation – M.Y.Ç., H.K.; Literature search – M.B., H.K., G.R.T.A.; Writing – M.B.; Critical review – M.Y.Ç.

Conflict of Interest: There is no any conflict of interest.

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