Effect of Final Irrigation Protocol on Push-Out Bond Strength of Bioceramic and Resin-Based Root Canal Sealers: An In-Vitro Study

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Abstract
Background: The success of endodontic treatment depends on effective obturation and the adhesion between root canal sealers and dentin. Final irrigation protocols influence dentin surface characteristics and bonding.
Aim: To evaluate and compare push-out bond strength of bioceramic and resin-based sealers following different final irrigation protocols.
Materials and Methods: Twenty-four extracted single-rooted premolars were standardized to 14 mm and instrumented up to size 25/.06. Samples were divided into four groups (n=6): Group I – EDTA + Resin, Group II – EDTA + Bioceramic, Group III – EDTA + HEDP + Resin, Group IV – EDTA + HEDP + Bioceramic. Push-out bond strength was tested using a universal testing machine. Data were analyzed using one-way ANOVA and Tukey post hoc test ($p<0.05$).
Results: Bioceramic sealers showed significantly higher bond strength than resin-based sealers. EDTA + HEDP significantly enhanced bond strength.
Conclusion: Final irrigation protocol plays a crucial role in sealer adhesion. HEDP improves bonding, particularly with bioceramic sealers.

Keywords: Bioceramic sealer, HEDP, EDTA, Push-out bond strength, Endodontics

1. Introduction

Three-dimensional obturation is critical for preventing reinfection of the root canal system and ensuring long-term treatment success. The adhesion between root canal sealers and dentin contributes significantly to resistance against dislodgement forces and microleakage [1]. Push-out bond strength testing is widely used to evaluate the adhesive properties of endodontic sealers due to its reliability and reproducibility [2].

The smear layer formed during instrumentation can impede sealer penetration and adaptation [3]. EDTA is commonly used to remove this layer; however, prolonged exposure can result in excessive dentin demineralization and collagen matrix degradation [4]. These changes may compromise bonding, especially for resin-based sealers that rely on hybrid layer formation [9].

Etidronic acid (HEDP) is a weaker chelating agent that can be used alongside sodium hypochlorite, allowing continuous chelation while preserving dentin integrity [5,6].

Bioceramic sealers are calcium silicate–based materials that chemically bond to dentin by forming hydroxyapatite [7]. They exhibit bioactivity, dimensional stability, and slight expansion upon setting [8]. In contrast, resin-based sealers rely on micromechanical retention [9].

This study aimed to evaluate the effect of final irrigation protocols on the push-out bond strength of bioceramic and resin-based sealers.

2. Materials and Methods

2.1 Study Design

This in vitro experimental study was conducted to evaluate the effect of different final irrigation protocols on the push-out bond strength of bioceramic and resin-based root canal sealers.

2.2 Sample Selection and Storage

Twenty-four freshly extracted human single-rooted premolars were selected for the study. Teeth were extracted for orthodontic or periodontal reasons.

Inclusion criteria:

- Single straight root canals
- Fully formed apices
- No caries, cracks, fractures, or resorption

Exclusion criteria:

- Previously endodontically treated teeth
- Curved canals
- Calcified canals

Following extraction, teeth were cleaned of soft tissue debris and calculus using ultrasonic scalers and stored in 0.1% thymol solution at room temperature until use to prevent dehydration and microbial growth.

2.3 Standardization of Samples

The crowns were removed using a diamond disc under continuous water cooling to obtain a standardized root length of 14 mm.

Working length was determined by inserting a size #10 K-file into the canal until it was visible at the apical foramen and then subtracting 1 mm from this length.

2.4 Root Canal Preparation

Biomechanical preparation was performed using rotary nickel-titanium instruments up to size 25/.06 taper.

Irrigation during instrumentation was carried out using 3% sodium hypochlorite (NaOCl) delivered with a side-vented irrigation needle placed 1 mm short of the working length. Each canal received 2 mL of NaOCl between successive instruments.

After completion of instrumentation, canals were flushed with distilled water to remove residual irrigants.

2.5 Final Irrigation Protocol

The samples were randomly divided into four experimental groups (n = 6):

- Group I: 17% EDTA for 1 minute + Resin-based sealer
- Group II: HEDP (9%) + Resin-based sealer
- Group III: 17% EDTA for 1 minute + Bioceramic sealer
- Group IV: HEDP (9%) + Bioceramic sealer

For EDTA groups, canals were irrigated with 5 mL of 17% EDTA for 1 minute, followed by a final rinse with distilled water.
For HEDP groups, a solution of 9% etidronic acid (HEDP) was used in combination with sodium hypochlorite to allow continuous chelation throughout the irrigation phase.
All canals were finally dried using sterile paper points prior to obturation.

2.6 Obturation Procedure

Obturation was performed using the single-cone technique corresponding to the master apical file size (25/.06 gutta-percha cones).

- Resin-based sealer used: Sealmax-R
- Bioceramic sealer used: SmartSeal

The sealer was introduced into the canal using a lentulo spiral to ensure uniform distribution along the canal walls. The master cone was then placed to full working length.
Excess gutta-percha was removed using a heated instrument, and vertical compaction was performed at the coronal level.
All specimens were stored at 37°C and 100% humidity for 7 days to allow complete setting of the sealer.

2.7 Sectioning of Samples (fig a-c)

After the setting period, each root was sectioned horizontally using a precision diamond saw under water cooling.
Three slices of 2 mm thickness were obtained from the middle third of each root to ensure standardization and minimize anatomical variations.
The thickness of each slice was verified using a digital caliper.

2.8 Push-Out Bond Strength Testing (fig d)

Each specimen was positioned on a custom-made metal jig in a universal testing machine.
A cylindrical plunger of appropriate diameter (slightly smaller than the canal diameter) was used to apply load in an apico-coronal direction to avoid constriction interference.
The load was applied at a crosshead speed of 1 mm/min until bond failure occurred. The maximum load at failure was recorded in Newtons (N).
Push-out bond strength was calculated using the formula:
Bond strength (MPa) = Load (N) / Adhesion area (mm^2)
The adhesion area was calculated based on the formula for the lateral surface area of a truncated cone.

2.10 Statistical Analysis

All data were tabulated and analyzed using statistical software .

- Normality of data was assessed using the Shapiro–Wilk test
- One-way ANOVA was used to compare mean bond strength among groups
- Tukey's post hoc test was applied for intergroup comparisons

A p-value of <0.05 was considered statistically significant.

Results

All specimens were successfully prepared and subjected to push-out bond strength testing. The mean bond strength values (MPa) and standard deviations for all groups are presented in Table 1 and illustrated in Figure 1.

3.1 Comparison of Sealer Types

Bioceramic sealer groups (Group III and Group IV) demonstrated higher mean push-out bond strength compared to resin-based sealer groups (Group I and Group II).

- Group III (EDTA + Bioceramic): 4.2 ± 0.4 MPa
- Group IV (HEDP + Bioceramic): 4.9 ± 0.45 MPa

In contrast:

- Group I (EDTA + Resin): 2.8 ± 0.3 MPa
- Group II (HEDP + Resin): 3.4 ± 0.35 MPa

The difference between bioceramic and resin-based sealers was found to be statistically significant ($p < 0.05$).

3.2 Effect of Final Irrigation Protocol

Within both sealer categories, the use of HEDP resulted in higher bond strength compared to EDTA alone.

- **Resin sealer:**
  - EDTA (Group I): 2.8 MPa
  - HEDP (Group II): 3.4 MPa
- **Bioceramic sealer:**
  - EDTA (Group III): 4.2 MPa
  - HEDP (Group IV): 4.9 MPa

This indicates that HEDP significantly enhances adhesion irrespective of sealer type ($p < 0.05$).

3.3 Intergroup Comparison

One-way ANOVA revealed a statistically significant difference among the four groups ($p < 0.05$).

Post hoc Tukey analysis showed:

- Group IV had significantly higher bond strength compared to all other groups
- Group III was significantly higher than Groups I and II
- Group II was significantly higher than Group I

3.4 Overall Ranking of Groups

Based on mean bond strength values:

Group IV (HEBP + Bioceramic) > Group III (EDTA + Bioceramic) > Group II (HEBP + Resin) > Group I (EDTA + Resin)

Bioceramic sealers showed significantly higher bond strength than resin-based sealers ($p<0.05$).

- EDTA + HEDP groups demonstrated significantly higher values than EDTA alone.
- Highest: Group IV (Bioceramic + EDTA + HEDP)
- Lowest: Group I (Resin + EDTA)

4. Discussion

The present study evaluated the influence of final irrigation protocols on the push-out bond strength of bioceramic and resin-based sealers. The findings clearly indicate that both the type of sealer and the irrigation protocol significantly affect bonding performance.

Bioceramic sealers demonstrated superior bond strength compared to resin-based sealers across all groups. This can be attributed to their unique chemical bonding mechanism. These materials release calcium ions, which react with phosphate ions in dentinal fluid to form hydroxyapatite, creating a chemical bond at the sealer–dentin interface [7,10]. This process results in the formation of a mineral infiltration zone, enhancing both micromechanical interlocking and chemical adhesion [11].

Additionally, the hydrophilic nature of bioceramic sealers allows better penetration into dentinal tubules, especially in the presence of residual moisture [12]. This contrasts with resin-based sealers, which are highly technique-sensitive and require optimal moisture control. Excessive demineralization caused by EDTA may collapse the collagen network, thereby impairing resin infiltration and hybrid layer formation [4,9].

The results of this study also demonstrated that the addition of HEDP significantly improved bond strength. HEDP is a milder chelating agent that enables continuous chelation without causing aggressive dentin erosion [5]. It preserves dentin microhardness and maintains structural integrity while still effectively removing the smear layer [6,13].

In contrast, prolonged use of EDTA alone has been shown to cause dentinal erosion and reduction in microhardness, which negatively affects bonding [14]. The combination of EDTA and HEDP appears to provide an optimal balance between smear layer removal and dentin preservation.

The highest bond strength observed in the Bioceramic + EDTA + HEDP group suggests that favorable dentin surface characteristics enhance biomineralization and sealer adaptation. These findings are consistent with previous studies that reported superior performance of calcium silicate–based sealers [15,16].

Push-out bond strength is an important parameter that reflects resistance to dislodgement forces during obturation and functional loading [2]. Strong adhesion reduces the risk of sealer displacement and microleakage, which are critical factors influencing long-term endodontic success [1].

From a clinical perspective, the use of bioceramic sealers in combination with HEDP may improve treatment outcomes by enhancing sealing ability and durability.

However, this study has certain limitations. Being an in vitro study, it does not fully replicate intraoral conditions such as thermal fluctuations, occlusal stresses, and aging. Future studies incorporating thermocycling and fatigue loading are recommended to better simulate clinical conditions [17].

5. Conclusion

Within the limitations of this in vitro study, it can be concluded that both the type of root canal sealer and the final irrigation protocol significantly influence push-out bond strength.

Bioceramic sealers demonstrated superior bonding performance compared to resin-based sealers. This can be attributed to their bioactive nature, chemical bonding ability, and formation of hydroxyapatite at the sealer–dentin interface, which enhances both micromechanical retention and chemical adhesion.

The use of HEDP as a final irrigant significantly improved bond strength in both sealer groups. Its mild chelating action preserves dentin microstructure while effectively removing the smear layer, thereby creating a favorable substrate for sealer adhesion.

The combination of bioceramic sealer with HEDP irrigation resulted in the highest bond strength, suggesting that this protocol may offer improved sealing ability and resistance to dislodgement forces.

From a clinical perspective, selecting an appropriate irrigation protocol in conjunction with advanced sealer materials can enhance the long-term success of endodontic treatment by reducing microleakage and improving obturation stability.

However, as this study was conducted under controlled laboratory conditions, the results should be interpreted with caution. Clinical factors such as thermal cycling, occlusal loading, and aging may influence long-term performance. Future studies incorporating these variables are recommended to validate the findings.

6.References

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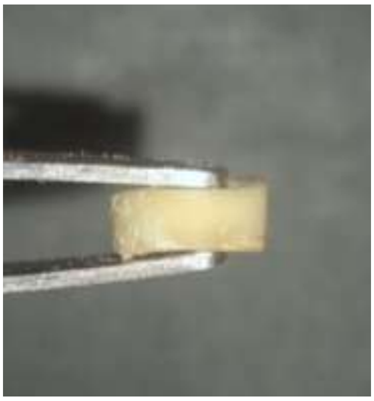
Figure

(a) Root slices embedded in acrylic resin blocks after sectioning into 2-mm thick samples.

(b) Close-up view of a prepared root slice showing the root canal space filled with sealer material.

(c) Sectioned dentin slice positioned for push-out testing.



(d) Schematic diagram illustrating the push-out bond strength test setup, showing the plunger applying

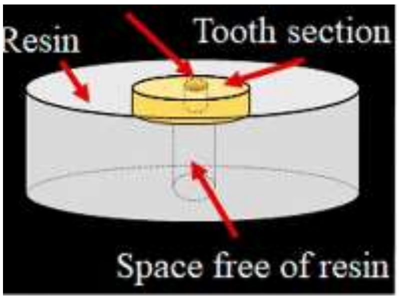


Table 1:

Group	Final Irrigation Protocol	Sealer Type	Mean ± SD (MPa)
Group I	EDTA	Resin-based (Sealmax-R)	2.8 ± 0.3
Group II	HEPB	Resin-based (Sealmax-R)	3.4 ± 0.35
Group III	EDTA	Bioceramic (SMARTSEAL)	4.2 ± 0.4
Group IV	HEBP	Bioceramic (SMARTSEAL)	4.9 ± 0.45

Mean push-out bond strength values (MPa) of resin-based and bioceramic sealers under different final irrigation protocols. The highest bond strength was observed in Group IV (Bioceramic sealer with

HEBP), while the lowest was recorded in Group I (Resin-based sealer with EDTA). Values are expressed as mean ± standard deviation.

Figure 1:

Bar graph representing the mean push-out bond strength (MPa) of different experimental groups based on sealer type and final irrigation protocol. Group IV (HEBP + Bioceramic sealer) exhibited the highest bond strength, followed by Group III (EDTA + Bioceramic sealer), Group II (HEBP + Resin-based sealer), and Group I (EDTA + Resin-based sealer), which showed the lowest values. Error bars represent standard deviation.