

The effect of final irrigation agents on push-out bond strength of calcium silicate-based cements to dentin

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Abstract

Aim: This aim of this study was to compare the effects of different chelating agents [ethylenediaminetetraacetic acid (EDTA), glycolic acid (GA) and citric acid (CA)] on the push-out bond strength (POBS) of two calcium silicate-based silicate cements (CSC) (Biodentine and PD MTA White).

Methodology: Dentin discs of 1 ± 0.2 mm thickness were taken from the middle root region of thirty-nine extracted mandibular premolar teeth and two holes (1 mm diameter) were drilled in each disc ($n = 78$ holes). The samples were then randomly divided into three groups ($n = 26$ holes) according to the final irrigation agents: Group 1: 20% CA, Group 2: 17% EDTA, Group 3: 10% GA. Then, two different materials were applied to the holes in each group ($n = 13$ holes): a: PD MTA White, b: Biodentine. POBS test was performed at a crosshead speed of 0.5 mm/min. The dentin discs were examined under stereomicroscope (25 \times) to assess the bond failure type. Data were analyzed using a two-way analysis of variance (ANOVA) and comparison of main effects was examined with the Bonferroni test, and multiple comparisons were analysed with the Tukey HSD test. The level of significance was 5%.

Results: Biodentine showed significantly higher POBS than PD MTA White ($p < 0.05$). There was no significant difference between the final irrigation agents ($p > 0.05$). CA - Biodentine group showed substantially higher POBS than EDTA - PD MTA White, CA - PD MTA White, and GA - Biodentine groups ($p < 0.05$).

Conclusion: While CA increased the POBS of Biodentine significantly, the POBS of PD MTA White was not affected by the final irrigation agents.

Keywords: Final irrigation agent, chelator, calcium silicate-based cement, push-out bond strength, Biodentine, PD MTA White

Introduction

Smear layer (SL) is a layer formed after root canal instrumentation, containing vital or necrotic pulp residues, bacteria, and dentin particles (1). The presence of this layer may prevent sealer penetration into the dentinal tubules and compromise the sealing of the filling materials (2). In addition, the SL can become infected, thereby protecting microorganisms in hard-to-reach areas such as the dentinal tubules and isthmus (3). The use of final irrigation agents has been suggested to remove the SL during chemical and mechanical preparation of root canals. While the most commonly used chelator in endodontics is EDTA; today, chelators such as etidronic acid, citric acid (CA), glycolic acid (GA), maleic acid and phosphoric acid are also used to remove the SL (4).

EDTA interacts with calcium ions present in dentin to form soluble chelates of calcium (5). However, it has disadvantages, like, a reduction in dentin microhardness and a reduction in the POBS of resin cement (6). GA, an organic compound belonging to the Hydroxy Acetic acid group, is commonly employed in the pharmaceutical industry and a readily biodegradable material. s widely employed in the pharmaceutical industry. It is a colorless, odorless, crystalline solid that is hygroscopic and highly soluble in water (7). GA, previously used for enamel and dentin etching in restorative treatment procedures, is less cytotoxic to fibroblasts than EDTA (8). In addition, the solution can maintain pH stability for up to 90 days and does not negatively affect dentin's flexural strength (8). The physical and biological properties of this solution, which is a suitable agent for removing the SL, are being studied (9). CA is a weak organic acid with properties comparable to EDTA in its antibacterial activity and low toxicity (10).

Calcium silicate-based cements (CSC) have a wide usage area in endodontics. Especially, perforations that occur in providing access to the canal can be repaired with these cements (11). Mineral trioxide aggregate (MTA) is considered the gold standard CSC material for perforation repair procedure (12). MTA derivatives are available from many manufacturers in the dental market (i.e. ProRoot MTA, MTA Angelus, MTA Plus, NeoMTA, MTA-Cem, MM-MTA, MTA Flow). PD MTA White (Produits Dentaires, Vevey, Switzerland) has been marketed as a CSC material with a particle size that facilitates wetting, and its properties do not change even in a humid environment (13). PD MTA White is a radiopaque root canal repair material and begins setting after 10 minutes and the final hardening time is 15 minutes. According to the manufacturer's statement, the superstructure filling can be applied onto this material at the end of the hardening period immediately (13). Biodentine (Septodont, Saint-Maur-des-Fossés, Creteil, France) is a fast-setting CSC that can replace dentin tissue. Biodentine has a wide usage area in endodontics as it hardens in a short time (12 minutes) and releases calcium ions. The physical properties of Biodentine are enhanced (when compared to MTA) by modifying the powder

composition, adding setting accelerators and softeners, and formulating a predosed capsule for use in a triturator. Flexural strength and elastic modulus Biodentine, are also higher than those of MTA and similar to dentine, with Biodentine being denser and less porous than MTA (14). The marginal compatibility and POBS of these CSCs to dentin is vital, as functional loads on the tooth or different materials applied to the cement may displace the CSC after perforation repair (15). Moreover, cements must be resistant to displacement forces to ensure a hermetic seal (16).

No study in the literature compares the POBS of Biodentine and PD MTA White on the dentin to which three different final irrigation agents (CA, EDTA, and GA) are applied. Since EDTA is the most commonly used chelator in endodontics, it will act as a control group for comparing the efficacy of GA and CA. Thus, this study's aim was to compare the effects of final chelating agents on the POBS of CSCs on the simulated perforation cavities. The null hypotheses of this study can be listed as follows: (i) There is no difference between CSCs in terms of the POBS; and (ii) There is no difference between different final irrigation agents in terms of the POBS.

Materials and Methods

Sample size calculation

Based on a similar study, the sample size was calculated using G*Power 3.1 software (Heinrich Heine University, Dusseldorf, Germany) (17). As a result of the calculation, it was found that at least 10 dentin holes should be included in each group with 80% confidence ($1-\alpha$), 80% test power ($1-\beta$) and $f = 0.490$. Considering the 25% dropout rate, 13 dentin holes per group were included in the study. Thus, a total of thirty-nine teeth (78 holes) for 6 groups were included in our study.

Sample selection, preparation and grouping

Study ethical approval was obtained from the Ethical Committee of the Dicle University, Faculty of Dentistry, Diyarbakır, Türkiye (Decision no: 2023-01). Thirty-nine freshly extracted human single-rooted mandibular premolars for orthodontic or periodontal reasons, without caries, resorption, and cracks, with a single canal and with similar root lengths were selected. Deposits on the tooth surface were removed with a scaler and the teeth were washed in an ultrasonic bath with distilled water for 10 minutes.

The crowns of the teeth were removed with a diamond disc (Metkon, Microcut Precisioncutter, Bursa, Türkiye) on a water-cooling cutting device (Isomet 1000, Buehler, Lake Bluff, IL, USA). Remaining root lengths were measured with a digital calliper with an accuracy of 0.001 mm. The roots were embedded vertically in acrylic (Imicryl Ltd., Konya, Türkiye). The middle third of the root was defined on the acrylic model and then

marked. Thirty-nine root dentin slices were obtained by taking middle sections of 1 ± 0.1 mm thickness perpendicular to the long axis of the tooth. Two holes of 1 mm in diameter were created in each dentin disc with a cylindrical carbide bur (Fig.1).

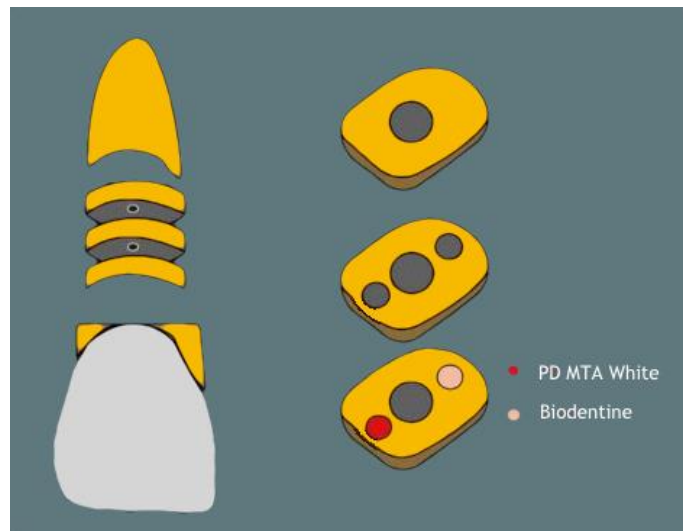


Figure 1. Schematic representation of the dentine slice acquisition and holes with materials.

A total of seventy-eight dentin holes were obtained. Then, all root slices were kept in 2.5% sodium hypochlorite (NaOCl) solution for 10 minutes and then immersed in distilled water for 5 minutes. All discs were

numbered and randomly divided into three groups (www.random.org) according to the final irrigation agent (n = 26 holes in each group).

Group 1: Final irrigation was performed with 20% CA (pH = 1.45) for 1 minute, then distilled water for 2 minutes, 2.5% NaOCl for 5 minutes, and distilled water for 2 minutes.

Group 2: Final irrigation was performed with 17% EDTA (pH = 7.13) for 1 minute. The remainder of the protocol was the same as for Group 1.

Group 3: Final irrigation was performed with 10% GA (pH = 2.24) for 1 minute. The remainder of the protocol was the same as for Group 1.

Samples of each group were divided to one of the subgroups based on the CSC material (n = 13 in each group). Calcium silicate-based PD MTA White (Produits Dentaires, Vevey, Switzerland) and Biodentine (Septodont, St-Maur-des-Fossés, France) were mixed according to the manufacturer's instructions. Then, two separate CSCs were placed in two holes drilled into the same dentin disc to ensure standardization of the samples. CSCs were applied using a plugger. The subgroups were as follows:

- a: PD MTA White
- b: Biodentine

The dentin holes were sealed with wet cotton wool and the specimens were stored for 1 day at 37 °C in 100% relative humidity to simulate the clinical situation. The flow chart of the study is given in Figure 2.

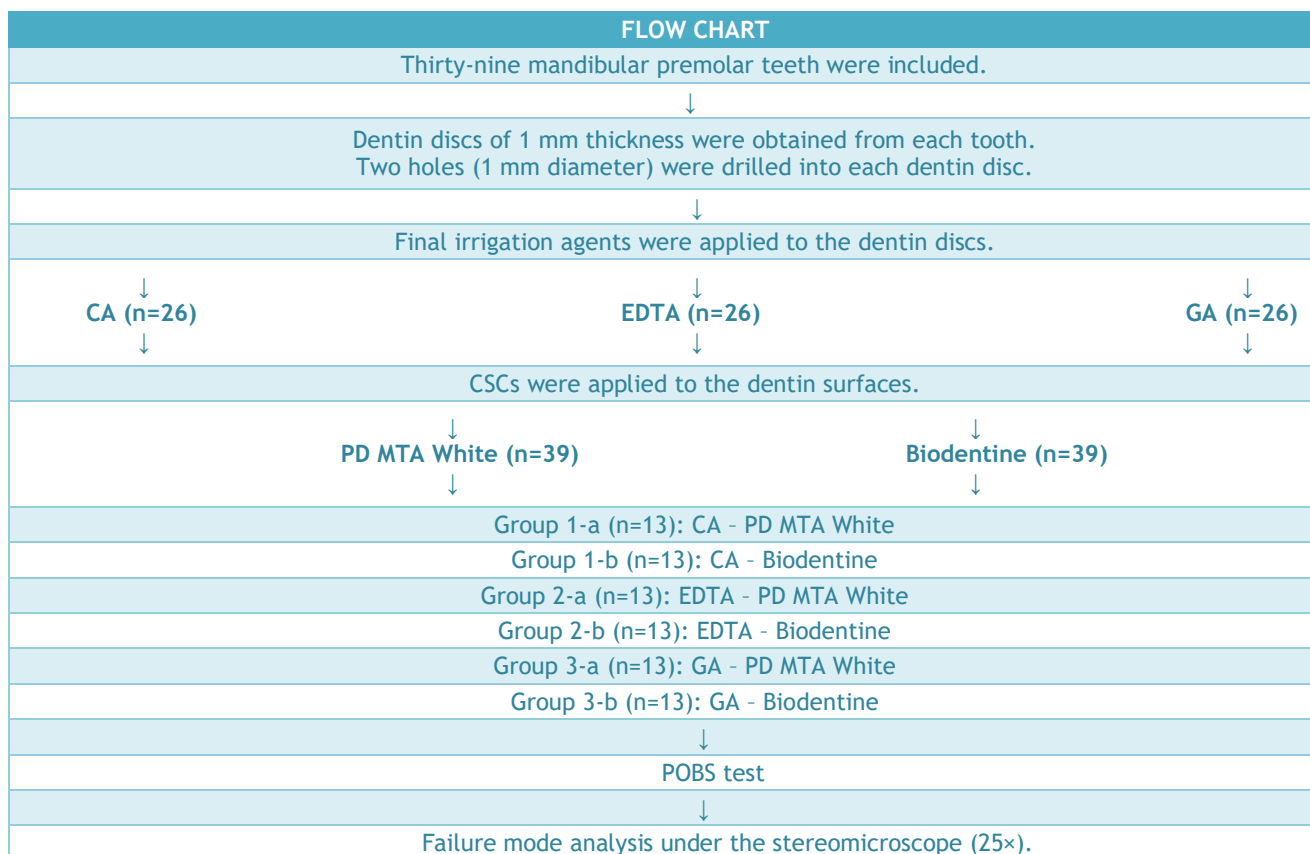


Figure 2. Flow chart of this study.

Push-out bond strength (POBS) test

The 0.8 mm diameter piston tip was placed over the tested CSCs. Loading was carried out on a universal testing machine (Lloyd™ LRX-plus; Lloyd Instruments, Fareham, UK) at a speed of 0.5 mm per minute until the material displaced. The POBS was obtained in MPa by dividing the load at fracture (Newton) by the area of the bonded interface (16).

Failure type analysis

The dentin holes were examined under stereomicroscope at 25× magnification to determine the failure mode. Each sample was recorded in one of three failure modes: “adhesive failure” between CSC and dentine, “cohesive failure” within the CSC, “mixed failure” in both the CSC and dentine (Fig. 3).

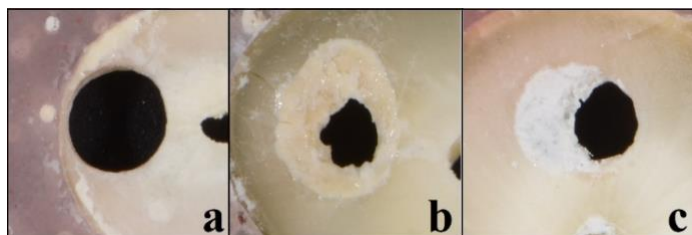


Figure 3. Failure mode types in CSC materials. (a) Adhesive failure between CSC and dentine, (b) cohesive failure within the CSC, (c) mixed failure in both the CSC and dentine.

Statistical analysis

The POBS values were analysed with SPSS Statistics (IBM SPSS Statistics version 23, IBM Inc., Armonk, NY, USA).

The normality of the data was confirmed by the Shapiro Wilk and Kruskal Wallis H tests according to the number of samples. The effect of the final irrigation agent and CSC on the POBS was analysed by two-way analysis of variance (ANOVA). Comparison of main effects was examined with the Bonferroni test, and multiple comparisons were analysed with the Tukey HSD test for interactions. Analysis results were presented as mean ± standard deviation. The significance level was set at $p < 0.05$.

Results

Table 1 shows the effect of the interaction of final irrigation agent, CSC, and final irrigation agent*CSC on the POBS. The main effect of the final irrigation agent on POBS was statistically insignificant ($p = 0.213$). The mean POBS value was 9.19 MPa in CA, 8.06 MPa in EDTA, and 7.44 MPa in GA. The main effect of CSC was significant on POBS ($p = 0.003$). While the mean POBS value of Biodentine was 9.49 MPa, that of PD MTA White was 6.97 MPa. Biodentine showed significantly higher POBS than PD MTA White ($p = 0.003$). The final irrigation agent and CSC interaction significantly impacted POBS ($p = 0.012$). CA - Biodentine group showed substantially higher POBS than EDTA - PD MTA White, CA - PD MTA White, and GA - Biodentine groups ($p < 0.05$). There was no significant difference between EDTA - PD MTA White, CA - PD MTA White, and GA-Biodentine groups ($p > 0.05$). There was no significant difference between CA - Biodentine, EDTA - Biodentine, and GA - PD MTA White groups ($p > 0.05$) (Fig. 4) (Table 2).

Table 1. Analysis of the effect of final irrigation agent and CSC on POBS.

	F	P	Partial Eta Squared
Final irrigation agent	1.578	0.213	0.042
CSC material	9.464	0.003	0.116
Final irrigation agent * CSC material	4.686	0.012	0.115

Two-way ANOVA, $R^2=0.181$

Table 2. Representation of descriptive statistics (mean and standard deviation) of POBS according to final irrigation agent and CSC (in MPa).

Final irrigation agent	CSC material		Total
	PD MTA White	Biodentine	
CA	6.69 ± 3.52 ^a	11.70 ± 5.09 ^b	9.19 ± 4.99
EDTA	6.33 ± 2.71 ^a	9.78 ± 2.69 ^{ab}	8.06 ± 3.18
GA	7.89 ± 3.92 ^{ab}	6.98 ± 3.17 ^a	7.44 ± 3.52
Total	6.97 ± 3.40	9.49 ± 4.18	8.23 ± 3.99

Two-way ANOVA and post-hoc Tukey test. a-b: There is no difference between columns and rows with the same letter

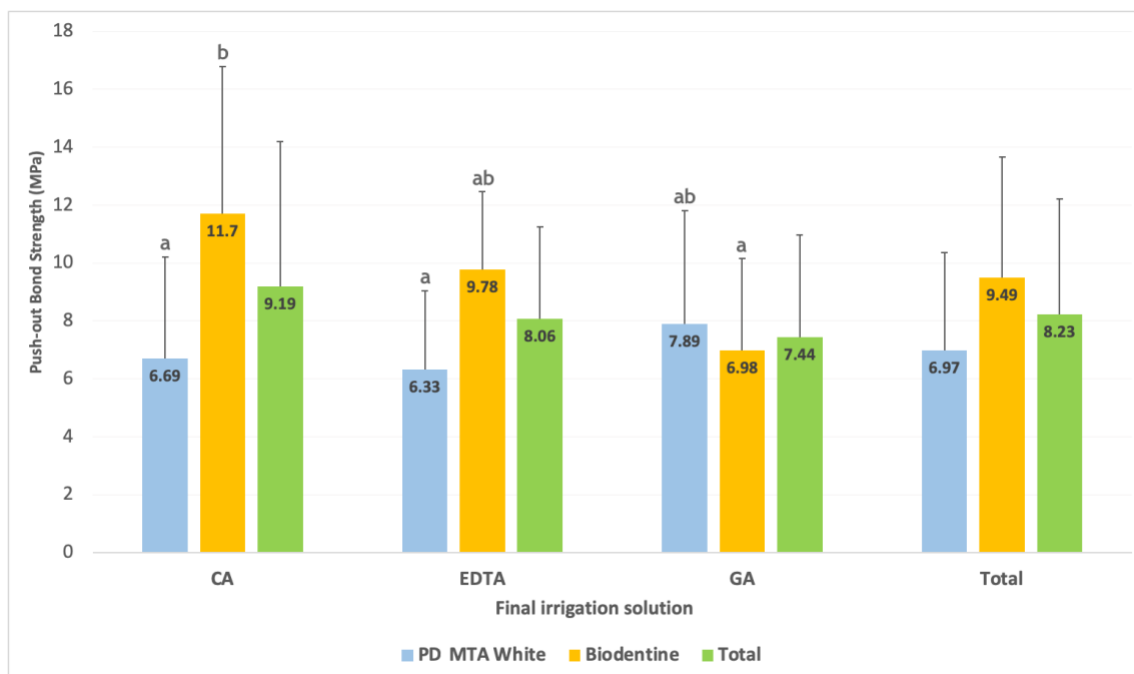


Figure 4. Graphical representation of the distribution of POBS values. a-b: There is no difference between columns and rows with the same letter.

Adhesive failure was not observed in groups using Biodentine, whereas adhesive failure was observed in two samples in the GA - PD MTA White group. The number of samples with cohesive failure was higher for both CSC materials. Mixed failure was observed in one sample in the EDTA group of PD MTA White, while mixed failure was observed in two samples each in the CA and GA groups in which Biodentine was used (Table 3).

Table 3. Distribution of failure types according to the CSC material and final irrigation agent

CSC material	Failure type	CA	EDTA	GA	Total
PD MTA White	Adhesive	-	-	2	2
	Cohesive	13	12	11	36
	Mixed	-	1	-	1
Biodentine	Adhesive	-	-	-	-
	Cohesive	11	13	11	35
	Mixed	2	-	2	4
Total	Adhesive	-	-	2	2
	Cohesive	24	25	22	71
	Mixed	2	1	2	5

Discussion

In this study, when the main effect of Biodentine and PD MTA White were compared for all final irrigation agents applied to dentin, Biodentine's POBS to dentin was significantly higher. Therefore, we reject the first null hypothesis of our study. In one study, the authors attributed this to the fact that a tag formation between Biodentine and dentin increases the resistance of Biodentine to dislodgement (18). In another study investigating the dislodgement resistance of Biodentine, authors attributed the high POBS of Biodentine to its small particle size (19).

PD MTA White's POBS was unaffected by the final irrigation agents in our study. According to the information given by the manufacturers, PD MTA White reaches its final hardness in 15 minutes (13). For this reason, the short setting time of this brand may have prevented the material from being affected by the final irrigation agents. In this case, we also rejected the second null hypothesis of our study.

Although the adhesion of the CSCs to the dentine depends on the material's properties, it affects the connection in the SL on the dentine. Al-Hiyasat et al. have shown that Biodentine had more POBS than MTA in the presence and the absence of a SL. Plus, they reported that Biodentine was unaffected by the SL. In this study, authors stated that MTA showed better POBS in the presence of the SL (20). El-Ma'aïta et al. reported that the SL was actively involved in the mineral interaction in the interface (21). In contrast to the study

of Al-Hiyasat et al., in our study, the POBS of Biodentine decreased according to EDTA in the GA group, while there was no difference in PD MTA White in the groups used by the three chelation agents. Although we tried to remove the SL with chelation agents in our study, we do not know how much was removed. The functioning of the calcium-binding mechanisms of the chelating agents may have affected the POBS.

There are only a few studies on PD MTA White in the literature (22, 23). In one of these studies, the POBS of root-end filling materials to the dentin walls was examined with three different condensation methods (condenser, 30 seconds ultrasonic agitation, 60 seconds ultrasonic agitation), and because of the study, TotalFill and Biodentine showed higher POBS than PD MTA White for all methods. This result parallels our study in which we placed all materials with a condenser (22).

In another study, the POBS of three different CSCs (Biodentine, PD MTA White and K-Biocer) applied in furcation repair after exposure to three different irrigation agents (BioAKT, NaOCl and distilled water) was investigated. According to the study's results, while BioAKT increased the POBS of Biodentine, it did not affect the POBS of PD MTA White in the three irrigation solutions (23). In our study, CA significantly increased the POBS of Biodentine to dentin compared to GA. Although GA increased the POBS of PD MTA White, this increase was not significant compared to CA and EDTA. That is, the POBS of PD MTA White was not affected by the change of the final irrigation agent. Our study results were similar to these two studies on PD MTA White.

In a study of GA (5, 10, and 17%), EDTA (17%), and CA (10%), Dal Bello et al. examined the mechanical and cytotoxic effects of CA (10%) (7). According to the results of this study, they did not find a significant difference in coronal and middle sections related to the smear removal. In addition, comparison of to 10% GA, 10% CA, and 17% EDTA did not show a significant difference between the surface roughness of the dentin. They stated that surface roughness could increase the micromechanical connection.

Machado et al. reported that 17% EDTA and 10% CA efficacies were similar in SL removal and tubular sealer penetration (24). Contrary to the results of these studies, it has been shown in a few studies that 10% CA has a higher decalcifying effect and chelation potential than EDTA (25, 26). In our study, we did not observe any difference in POBS between chelation agents regardless of the CSC materials.

In a study examining the connection of dentin and cement interfaces, tubular diffusion was observed in dentin thanks to the denaturation feature of Biodentine, and the formation of a "mineral infiltration zone" between intertubular dentin and Biodentine was demonstrated (27). Moreover, the effects of dentin roughness, CSC particle size, and removal of the SL on the bonding of CSCs have been investigated in the literature (28-30).

Biodentine's higher POBS is thought to result from its small particle size, which increases its penetration into dentinal tubules (30). It has been reported that

increasing the pH of the chelator increases surface roughness (29). The CA we used in our study had a concentration of 20% and high acidic pH (1.45). Although statistically insignificant, thanks to its high concentration, CA (9.19 MPa) may have removed the SL better and increased micromechanical adhesion better than EDTA (8.06 MPa) and GA (7.44 MPa). In this study, the POBS of Biodentine was significantly higher than PD MTA White in the CA irrigation agent group. Considering all these aspects, the fact that CA used in our study was the chelator with the highest pH, the small particle size of Biodentine, and the formation of a mineral infiltration zone between Biodentine and dentin may have increased the bond strength.

EDTA has been shown to reduce the stiffness and flexural strength of MTA (31). It has also been demonstrated that final irrigation with EDTA reduces the sealing ability of MTA (32, 33). Similarly, PD MTA White exhibited the lowest POBS compared to CA (6.69 MPa) and GA (7.89 MPa) when EDTA (6.33 MPa) was used as the final irrigation agent, although it was not statistically significant in our study.

In one study, MTA-Angelus was shown to release higher calcium levels than ProRoot MTA, thereby increasing dislodgement resistance by precipitating more calcium phosphate and forming tag-like structures (34, 35). There may be less adhesion to the canal walls because PD MTA White has less calcium release than Biodentine in our study. Of course, this situation is related to the amount of calcium silicate cement in its content. While the tri-calcium silicate content of Biodentine is 80.1%, the tri- and di-calcium silicate content of PD MTA White is between 50% and 72%. Moreover, PD MTA White may not be adapted due to the particle size of the tubular width provided by chelation agents. Since the particle size of the material was not tested, this can only go up to a comment. For this reason, further studies are needed to fully understand the mechanism of the adhesion of the PD MTA White.

The most dominant type of failure for all solutions in both the PD MTA White and Biodentine groups was cohesive failure. Cohesive failure indicates that materials have reached maximum strength in adhesiveness, whereas adhesive failure occurs due to poor bonding and is an undesirable type of failure (36). Adhesive failure was seen only in two samples in the GA - PD MTA White group. This group's lowest POBS value is consistent with the failure type.

In this study, confounding factors such as dentin hardness, sclerotic dentin, and root canal diameter arising from different dentin samples were eliminated by applying two CSC materials to two standardized slots opened on a single dentin disc. This is one of the strengths of the methodology of this study. Although the incubator tries to imitate the oral conditions, the inability to mimic the chewing forces acting on the teeth and the fact that the effectiveness of the irrigation solutions can change in the intraoral conditions on different root canal levels are the limitations of this study.

Conclusion

In cases where CA is used as the final irrigation agent, choosing Biodentine as a repair material increases the POBS by increasing infiltration into the dentinal tubules. GA acid unexpectedly decreased the POBS of Biodentine. Although unaffected by the final irrigation agent, PD MTA White showed lower POBS values than Biodentine. However, interestingly, GA acid decreased the POBS of Biodentine compared to the EDTA. The effect of chelators on the POBS of CSCs to dentin was variable. More studies are needed on this topic.

Disclosures

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Ethical Approval: Ethics committee approval was received for this study from Dicle University, Faculty of Dentistry, Research Ethics Committee, in accordance with the World Medical Association Declaration of Helsinki, with the approval number: 2023/01.

Peer-review: Externally peer-reviewed.

Author Contributions: Conception - M.Y.Ö.; Design - M.Y.Ö., S.F.; Supervision - M.Y.Ö., M.G.K.; Materials - M.Y.Ö., M.B.; Data Collection and/or Processing - M.Y.Ö., M.B.; Analysis and/or Interpretation - M.Y.Ö.; Literature Review - M.Y.Ö., Ö.A.; Writer -M.Y.Ö.; Critical Review - Ö.A.

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