Effect of Various Endodontic Irrigants on the Push-out Bond Strength of Biodentine and Conventional Root Perforation Repair Materials

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Abstract

Introduction: The aim of this study was to evaluate the effect of various endodontic irrigants on the push-out bond strength of Biodentine (Septodont, Saint Maur des Fossés, France) in comparison with contemporary root perforation repair materials. **Methods:** Midroot dentin of canine teeth was horizontally sectioned into 1-mm-thick slices. The canal space of each dentin slice was enlarged with a diamond bur to 1.4 mm in diameter. The samples were divided into 5 groups (n = 40), and the following materials were placed, respectively: Biodentine, ProRoot MTA (Dentsply Tulsa Dental, Tulsa, OK), amalgam, Dyract AP (Dentsply DeTrey, Konstanz, Germany), and intermediate restorative material (IRM, Dentsply DeTrey). The samples were wrapped in wet gauze for 10 minutes and divided into 3 subgroups (n = 10) to be immersed into 3.5% sodium hypochlorite, 2% chlorhexidine gluconate (CHX), or saline for 30 minutes. No irrigation was performed in the controls (n = 10), and a wet cotton pellet was placed over each test material. After incubation for 48 hours, the dislodgement resistance of the samples was measured using a universal testing machine. The samples were examined under a stereomicroscope to determine the nature of the bond failures. **Results:** Biodentine showed significantly higher push-out bond strength than MTA ($P < .05$). The statistical ranking of push-out bond strength values was as follows: Dyract AP > amalgam ≥ IRM ≥ Biodentine > MTA. The push-out bond strength of Dyract AP, amalgam, IRM, and Biodentine was not significantly different when immersed in NaOCl, CHX, and saline solutions, whereas MTA lost strength when exposed to CHX. **Conclusions:** Biodentine showed considerable performance as a perforation repair material even after being exposed to various endodontic irrigants, whereas MTA had the lowest push-out bond strength to root dentin. *(J Endod 2013;39:380–384)*

Key Words

Biodentine, bond strength, irrigants, mineral trioxide aggregate, push-out

Furcation perforation is a procedural complication that can occur during endodontic treatment or post space preparation of teeth (1). An ideal perforation repair material should provide a tight seal between the oral environment and periradicular tissues. It also should remain in place under dislodging forces, such as mechanical loads of occlusion or the condensation of restorative materials over it (2–5). Although many dental materials have been tried including amalgam, Cavit (ESPE, Seefeld, Germany), composite resin, glass ionomer cement, calcium hydroxide, Super EBA (Harry J Bosworth, Skokie, IL), intermediate restorative material (IRM; Dentsply DeTrey, Konstanz, Germany), and mineral trioxide aggregate (MTA), most of these materials show significant shortcomings in 1 or more of the following areas: solubility, leakage, biocompatibility, handling properties, and moisture incompatibility (6–11).

Despite the numerous favorable properties of MTA that support its clinical use when compared with the traditional materials, there are several critical drawbacks such as the prolonged setting time, difficult handling characteristics, high cost, and potential of discoloration (9–12). A variety of new calcium silicate–based materials have been developed recently aiming to improve MTA shortcomings (13, 14). Biodentine (Septodont, Saint Maur des Fossés, France) is a high-purity calcium silicate–based dental material composed of tricalcium silicate, calcium carbonate, zirconium oxide, and a water-based liquid containing calcium chloride as the setting accelerator and water-reducing agent. Biodentine is recommended for use as a dentin substitute under resin composite restorations and an endodontic repair material because of its good sealing ability, high compressive strengths, short setting time (14, 15), biocompatibility, bioactivity, and biomineralization properties (16, 17).

Clinically, the operator should immediately repair the furcation perforations with an endodontic material in order to minimize the bacterial contamination and the irritation of periodontal tissues because of the usage of endodontic irrigants (18). After repairing the furcal perforation, endodontic treatment should be performed with various irrigants including 2% chlorhexidine gluconate (CHX) and sodium hypochlorite (NaOCl) solutions to disinfect the root canal system (19). However, this procedure causes unavoidable contact of irrigants with the repair materials. There is no information about the effect of endodontic irrigants on the push-out bond strength of Biodentine. Thus, the purpose of this *in vitro* study was to evaluate the effect of various endodontic irrigants (ie, 3.5% NaOCl, 2% CHX, or saline) on the push-out bond strength of Biodentine in comparison with other conventional perforation repair materials.

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Materials and Methods

Freshly extracted single-rooted human canine teeth were used. The crowns of all teeth were removed, and the midroot dentin was sectioned horizontally into slices with a thickness of 1.0 mm by using a water-cooled low-speed IsoMet diamond saw (Buehler, Lake Bluff, NY). In each slice, the space of the canal was enlarged with a 1.4-mm-diameter diamond bur. The root sections were randomly divided into 5 groups (n = 40), and the following test materials were used: group 1: Biodentine liquid from a single-dose container was emptied into a powder-containing capsule and mixed 30 seconds at 4,000–4,200 rpm, group 2: MTA (ProRoot; Dentsply Tulsa Dental, Tulsa, OK) was hand mixed with sterile water at a powder to liquid ratio of 3:1 in accordance with the manufacturer's instructions, group 3: amalgam (Cavex Avalloy; Cavex Holland BV, Haarlem, Netherlands), group 4: Dyract AP compomer (Dentsply DeTrey, Konstanz, Germany), and group 5: IRM.

The test materials were incrementally placed into the canal spaces of the dentin slices and condensed. Excess material was trimmed from the surface of the samples with a scalpel. Subsequently, the samples were wrapped in wet gauze, placed in an incubator, and allowed to set for 10 minutes at 37°C with 100% humidity. Immediately after incubation, the samples were divided into 3 subgroups (n = 10) to be immersed into 3.5% NaOCl (Caglayan Kimya, Konya, Turkey), 2% CHX (Klorhex; Drogsan, Ankara, Turkey), or saline solution (IE Ulagay, Istanbul, Turkey). After 30 minutes of immersion, all samples were removed from the test solutions, rinsed with distilled water, and allowed to set for 48 hours at 37°C with 100% humidity in an incubator. As a control group, a wet cotton pellet was placed over each test material without any irrigation and allowed to set for 48 hours (n = 10).

Push-out Test

The push-out bond strength values were measured by using a universal testing machine (Instron Universal test machine; Elista, Istanbul, Turkey). The samples were placed on a metal slab with a central hole to allow the free motion of the plunger. The compressive load was applied by exerting a download pressure on the surface of the test material in each sample with the Instron probe moving at a constant speed of 1 mm/min. The plunger had a clearance of approximately 0.2 mm from the margin of the dentinal wall to ensure contact only with the test materials. The maximum force applied to materials at the time of dislodgement was recorded in newtons. The push-out bond strength in megapascal (MPa) was calculated by dividing this force by the surface area of test material (N/2prh), where p is the constant 3.14, r is the root canal radius, and h is the thickness of the root dentin slice in millimeters. The nature of the bond failure was assessed under a stereomicroscope (SZTP; Olympus Optical Co, Tokyo, Japan) at 10× magnification. Each sample was categorized into 1 of the 3 failure modes: adhesive failure at test material and dentin interface, cohesive failure within test material, or mixed failure (Fig. 1*A–C*). Data were analyzed by using 1-way analysis of variance and post hoc Tukey tests. The level of statistical significant was set at .05.

SEM Analysis

One specimen from each group was randomly chosen for scanning electron microscopic (SEM) examination. A magnification considered adequate to characterize the microstructure (10,000×) was selected, and the irrigant-treated surface characteristics of the samples were recorded with the digital imaging system.

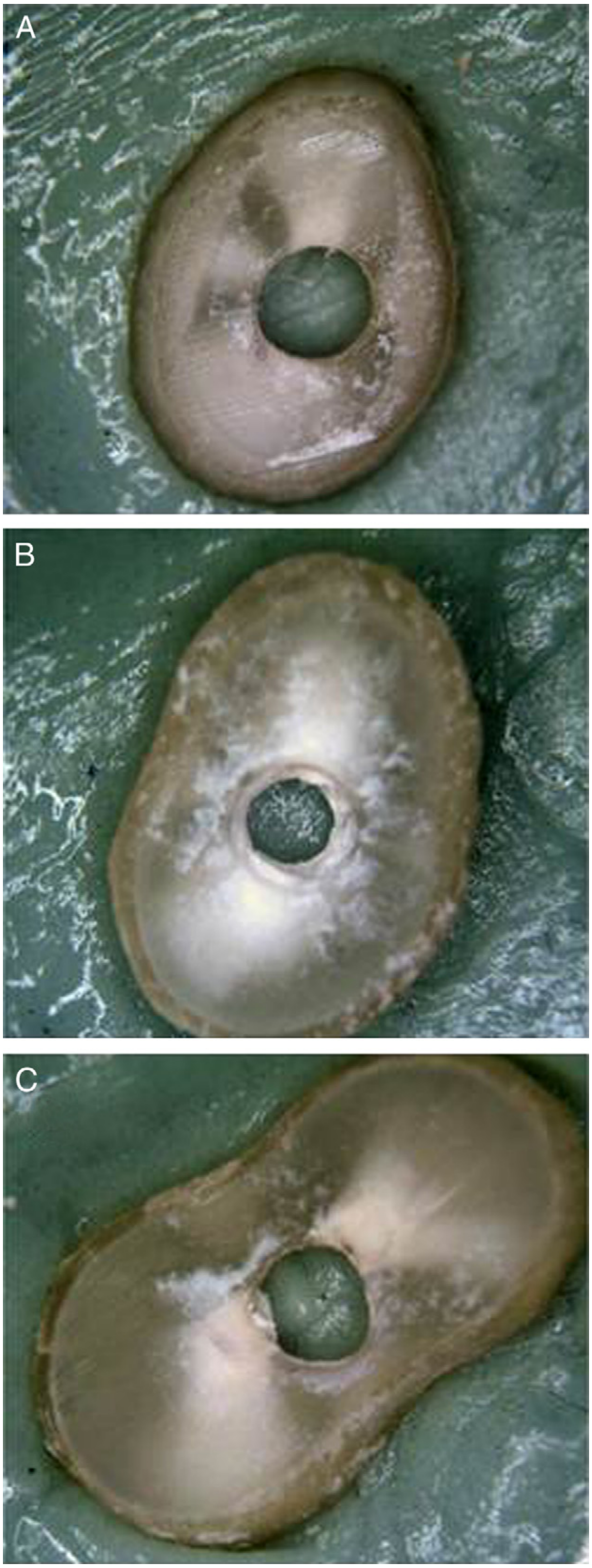

Figure 1. Inspection of the samples under a stereomicroscope at 10× magnification and various failure modes of Biodentine samples. (*A*) Adhesive failure; note the clean canal wall. (*B*) Cohesive failure within Biodentine. (*C*) Mixed failure; there are remnants of Biodentine inside the canal.

Results

Push-out Test

Table 1 shows the mean values and standard deviations of the push-out bond strength (MPa) and the distribution of failures of all groups. The lowest push-out bond strength was observed in the MTA group ($P < .05$). Biodentine displayed a significantly higher resistance to displacement than the MTA group, whereas the mean push-out bond strength value was lower than that observed in the Dyract AP, amalgam, and IRM groups ($P < .05$).

The mean values of push-out bond strength (MPa) for the subgroups of each test material are shown in Table 2. Exposure to NaOCl, CHX, and saline solutions did not affect the resistance to displacement of the Biodentine, amalgam, Dyract AP, and IRM groups ($P > .05$). However, the mean values of the push-out bond strength of the saline-treated MTA group were greater than the MTA control group ($P < .05$). MTA lost strength after exposure to CHX solution, and the bond strength of the CHX-treated MTA group was the lowest. The percentages of the failure modes of the samples are presented in Table 1. The contact with the test irrigants did not have a significant effect on the failure types of the tested materials.

SEM Analysis

In the SEM examination, the Biodentine control group showed large irregular and hexagonal crystals, and the surfaces of the irregular structures appeared uneven (Fig. 2*A*). The NaOCl solution–treated Biodentine surface showed little surface crystalline formation. The crystals morphed into an undeveloped hexagonal structure with a marked decrease in size and an increase in number when compared with the control group (Fig. 2*B*). The saline-treated Biodentine group showed a relatively smooth surface, which consisted of small and globular crystals without the hexagonal plates (Fig. 2*C*). The crystallized structure, which formed after exposure to CHX solution, presented a typical cluster of globular crystalline with its round and prickly shaped structure (Fig. 2*D*).

Globular structures of variable sizes were observed in the MTA control group (Fig. 2*E*). Little surface crystalline formation with a marked decrease in size was observed after NaOCl treatment (Fig. 2*F*). The crystals showed an increase in size in the MTA saline group (Fig. 2*G*). When compared with the MTA control group, there were fewer and smaller sized globular structures on the surface of CHX-treated MTA with the signs of erosion (Fig. 2*H*).

Discussion

After repair of the furcal perforation, the success of the endodontic therapy depends on a well-placed coronal restoration as well as the resistance of the repair material to displacement forces happening while undergoing condensation of permanent restorative materials. The amalgam condensation force could reach up to 8.9 ± 2.4 MPa and 5.5 ± 1.8 MPa with a small and large amalgam plugger, respectively (20). Such pressure could lead to the dislodgement of the furcal repair materials (4, 5, 21, 22). Thus, the bond strength of the perforation repair materials is an important factor in clinical practice. To assess the bond strength, the push-out bond test has been shown to be efficient, practical, and reliable (2, 4, 18, 19, 21–25). This study is the first to evaluate the push-out bond strength between Biodentine, a new calcium silicate–based material, compared with other repair materials before and after exposure to endodontic irrigants.

TABLE 1. Mean Push-out Bond Strength Values with Standard Deviations and Failure Modes of Each Test Material

Groups	Number	Mean (MPa)	Failure mode, % (A/C/M)
Biodentine	40	7.18 ± 3.11^{c}	2.5/92.5/5
MTA	40	3.86 ± 3.22^{d}	45/25/30
Amalgam	40	9.72 ± 3.68^{b}	80/5/15
Dyract AP	40	14.90 ± 5.74^{a}	0/100/0
IRM	40	8.61 ± 3.06^{bc}	5/77.5/17.5

A, adhesive failure along the material-dentin interface; C, cohesive failure within the material; M, mixed failure.

Groups identified by the same superscript letters are not significantly different ($P > .05$). Different letters identify significantly different groups ($P < .05$).

TABLE 2. Mean Push-out Bond Strength Values and Standard Deviations of All Test Groups

Groups	Subgroups	Number	Mean (MPa)
Biodentine	NaOCl	10	7.23 ± 4.22^{a}
	CHX	10	7.13 ± 2.17^{a}
	Saline	10	7.22 ± 3.14^{a}
	Control	10	7.12 ± 3.10^{a}
MTA	NaOCl	10	3.49 ± 3.02^{ab}
	CHX	10	2.45 ± 1.99^{b}
	Saline	10	6.18 ± 3.80^{a}
	Control	10	3.03 ± 1.28^{ab}
Amalgam	NaOCl	10	9.94 ± 1.83^{a}
	CHX	10	9.96 ± 4.82^{a}
	Saline	10	9.23 ± 3.32^{a}
	Control	10	9.74 ± 2.79^{a}
Dyract AP	NaOCl	10	14.02 ± 5.95^{a}
	CHX	10	15.30 ± 3.23^{a}
	Saline	10	14.45 ± 7.66^{a}
	Control	10	15.82 ± 3.80^{a}
IRM	NaOCl	10	9.21 ± 2.90^{a}
	CHX	10	8.07 ± 2.79^{a}
	Saline	10	8.18 ± 2.78^{a}
	Control	10	8.99 ± 2.71^{a}

Subgroups identified by the same superscript letters are not significantly different in each group ($P > .05$). Different letters identify significant differences within subgroups ($P < .05$).

Amalgam, reinforced zinc oxide–eugenol–based cements, composite resins, and MTA seem to be proper materials for perforation repair and have been sufficiently investigated (26). The results of our study indicated that Dyract AP, amalgam, and IRM showed greater push-out bond strength than MTA and Biodentine. Despite their higher bond strengths, the adequate sealing abilities of these materials for furcation repair should be questioned. Because of the toxicity, corrosion, potential for tissue staining, and marginal leakage, the use of amalgam as a perforation repair material is a controversial topic (11, 27). Because perforation repair materials are in contact with periradicular tissues, biocompatibility is one of the other essential factors when choosing a repair material. Huang et al (27) found that compomer was cytotoxic to human gingival fibroblasts. IRM, which is a reinforced zinc oxide–eugenol cement, can cause mild to moderate toxicity when it is freshly mixed, which is probably because of its eugenol component. Furthermore, IRM may show solubility over time (11). For these reasons, MTA is still the gold standard when compared with amalgam, compomer, and IRM.

However, scientific literature indicates that lower pH environments may affect MTA's various physical and chemical properties (4, 28). Exposure to 2% CHX, even though it is not an acid, may result in a reduced surface hardness (24, 28), a decreased sealing ability, a slower setting time (3, 28), and a lower resistance to dislodgement forces (24, 29). We also observed that immersing MTA in CHX after 10 minutes of setting resulted in a statistically significant decreased push-out bond strength (ie, from 3.03 to 2.45 MPa). This result was consistent with the results of Hong et al (24), who showed that 2% CHX reduced the push-out strength of accelerated MTA. Nandini et al

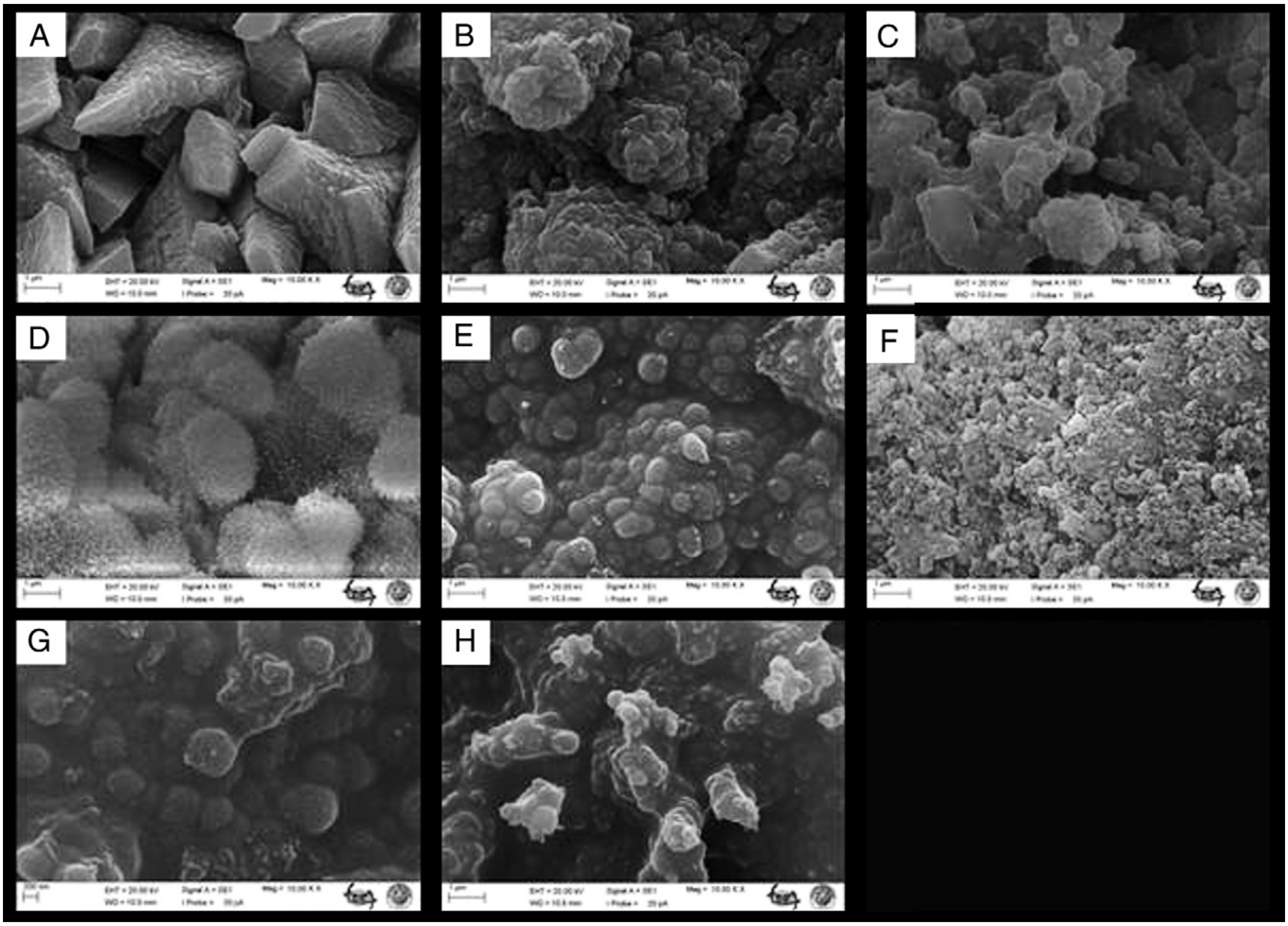

Figure 2. SEM pictures of Biodentine and MTA samples (10,000×). (*A*) Large irregular and hexagonal crystals of the Biodentine control group. (*B*) An undeveloped hexagonal crystal structure was seen after NaOCl treatment when compared with the control group. (*C*) Small and globular crystals without the hexagonal plates in the Biodentine saline group. (*D*) A typical cluster of globular crystalline with its round and prickly shaped structure in the Biodentine CHX group. (*E*) Globular structures observed in the MTA control group. (*F*) The MTA NaOCl group showed little surface crystalline formation with a marked decrease in size when compared with the MTA control group. (*G*) The MTA saline group showed larger crystals. (*H*) CHX changed the surface morphology of MTA; there were fewer globular structures and they were smaller in size and less in amount.

(28) showed that 2% CHX decreased the surface hardness of set white MTA significantly and suggested that CHX irrigation within 24 hours of placement of white MTA should be avoided. Aggarwal et al (30) found that 2% CHX reduced the microhardness and flexural strength of MTA. According to our SEM examinations, CHX altered the surface morphology of MTA with the signs of erosion. The amount and the size of globular structures on the MTA surface were decreased after 30 minutes of CHX immersion. Hong et al (24) detected silicon along with calcium, oxygen, and carbon, which proved the absence of calcium hydroxide crystals. These findings may explain why CHX significantly reduced the push-out bond strength of MTA. On the contrary, CHX could not erode the surface of Biodentine as it could on the MTA surface.

Saline-treated MTA samples resisted dislodgement more efficiently than the MTA control group, which was in accordance with a previous report (18) that indicated that the compressive strength of MTA increased when immersed in saline solutions because of the remaining unreacted mineral oxides, which may be solidified after additional supplied hydration and may result in the increased strength of the material. Although NaOCl might have an effect on the higher push-out bond strength values of MTA as it has been reported previously (3, 18, 24), this effect was not statistically significant in our study. Therefore, one may speculate that NaOCl or saline solution can be considered for single-visit procedures if MTA will be used as a repair material.

We observed the bond failures in all MTA groups predominantly at the MTA-dentin interface (adhesive type). This finding is in agreement with previous studies (4, 21, 22, 24) that reported that MTA-dentin bond failures were usually adhesive. The adhesive mode of failure may be caused by the short storage time before the evaluation of the bond strength, which was 2 days in the present study and 3 and 7 days in the studies performed by Saghiri et al (21) and Vanderweele et al (31). In contrast, almost all Biodentine samples revealed a cohesive bond failure. The different failure types of MTA and Biodentine may be explained by the particle size of these materials, which affects the penetration of cement into dentinal tubules. A smaller particle size and uniform components might have a role in better interlocking of Biodentine with the dentin, which finally causes cohesive failure inside the cement. The adhesion of Biodentine to dentinal tubules may also result from the tag-like structures within the dentinal tubules leading to a micromechanical anchor (32).

Biodentine was more resistant to dislodgement forces than MTA in the present study. The biomineralization ability of Biodentine, most likely through the formation of tags, may be the reason of the dislodgement resistance. Han and Okiji (14) showed that calcium and silicon ion uptake into dentin leading the formation of tag-like structures in Biodentine was higher than MTA. Biodentine also displayed a remarkably consistent performance even after exposure to 3.5% NaOCl, 2% CHX, and saline solutions despite the affected surface morphology in the present study. The alteration on the physical properties of Biodentine in CHX solution should be studied further before advocating the clinical application of Biodentine successively with CHX.

Based on this *in vitro* study, it can be concluded that the force needed for the displacement of Biodentine from root dentin was significantly higher than MTA. Saline solution increased the push-out bond strength of MTA, whereas CHX reduced it. However, endodontic irrigants did not influence the resistance to the dislodgement of Biodentine. It is recommended that care should be taken to prevent the contact of CHX solution with MTA in single-visit endodontic therapy procedures. Further research is needed to warranty clinical usage of Biodentine.

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

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