

Research Article



The push-out bond strength of BIOfactor mineral trioxide aggregate, a novel root repair material

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Conflict of Interest

No potential conflict of interest relevant to this
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ABSTRACT

Objectives: The aim of this *in vitro* study was to evaluate the push-out bond strength of a novel calcium silicate-based root repair material-BIOfactor MTA to root canal dentin in comparison with white MTA-Angelus (Angelus) and Biodentine (Septodont).

Materials and Methods: The coronal parts of 12 central incisors were removed and the roots were embedded in acrylic resin blocks. Midroot dentin of each sample was horizontally sectioned into 1.1 mm slices and 3 slices were obtained from each root. Three canal-like standardized holes having 1 mm in diameter were created parallel to the root canal on each dentin slice with a diamond bur. The holes were filled with MTA-Angelus, Biodentine, or BIOfactor MTA. Wet gauze was placed over the specimens and samples were stored in an incubator at 37°C for 7 days to allow complete setting. Then samples were subjected to the push-out test method using a universal test machine with the loading speed of 1 mm/min. Data was statistically analyzed using Friedman test and *post hoc* Wilcoxon signed rank test with Bonferroni correction.

Results: There were no significant differences among the push-out bond strength values of MTA-Angelus, Biodentine, and BIOfactor MTA ($p > 0.017$). Most of the specimens exhibited cohesive failure in all groups, with the highest rate found in Biodentine group.

Conclusions: Based on the results of this study, MTA-Angelus, Biodentine, and BIOfactor MTA showed similar resistances to the push-out testing.

Keywords: Biodentine; Mineral trioxide aggregate; Push-out bond strength; Root repair

INTRODUCTION

Root perforations due to mechanical, iatrogenic, or pathological causes can increase the failure risk of endodontic treatment, and they can even result in the extraction of the affected tooth. A greater than 70% success rate can be achieved by repairing a tooth nonsurgically with a variety of repair materials [1]. Additionally, the perforation repair treatment time and the size and location of the perforation are crucial factors that influence the treatment success rate [2]. Moreover, a good seal at the perforated site with a biocompatible root repair material can help maintain the health of the periodontal tissues [3].

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Calcium silicate-based materials, with their many favorable properties, have become preferable for perforation repair. The traditional mineral trioxide aggregate (MTA), which is composed mainly of tricalcium silicate, dicalcium silicate, and bismuth oxide, has many advantages, including its biocompatibility, hard tissue induction capacity, and good sealing. However, MTA also has some major drawbacks, such as its long setting time, difficult handling characteristics, high cost, and tooth discoloration [4]. MTA-Angelus (Angelus Indústria de Produtos Odontológicos S/A, Londrina, PR, Brazil) is a commercial brand of MTA and has some advantages over ProRoot MTA (Dentsply Tulsa Dental, Tulsa, OK, USA) which is the first commercially available product of MTA. MTA-Angelus has been reported to be more economical than ProRoot MTA [5]. Additionally, the manufacturer of MTA-Angelus claims that this material has a setting time of 15 minutes which is shorter than ProRoot MTA's. However, other clinical problems like handling properties and tooth discoloration effects are still associated with clinical use of MTA-Angelus. The handling property of white MTA-Angelus has been reported to be grainy and sandy, making delivery to perforation site and compacting difficult [6]. Furthermore, the teeth filled with white MTA-Angelus revealed a grayish discoloration with evident dentin staining due to bismuth oxide content [7].

Biodentine (Septodont, St-Maur-des-fosses, France), a dentin substitute, is composed primarily of tricalcium silicate, dicalcium silicate, calcium carbonate, and zirconium oxide [8]. It has been shown to have good biocompatibility and bioactivity, while the improved handling characteristics and reduced setting time make Biodentine a promising alternative to MTA.

A new type of MTA, called BIOfactor MTA (Imicryl Dental, Konya, Turkey) has recently been introduced for pulp capping, pulpotomies, apexification, root perforation repairs, root end filling, and apical plug procedures. The BIOfactor MTA powder is composed of tricalcium and dicalcium silicate, tricalcium aluminate, and ytterbium oxide as a radiopacifier agent. The liquid consists of 0.5%–3% hydrosoluble carboxylated polymer and demineralized water. This cement can be prepared in a flowing or thick consistency, based on the treatment type. The manufacturer claims that BIOfactor MTA has a finer powder for faster hydration, easier handling properties, stronger sealing, and a shorter setting time, and that it does not cause tooth discoloration. Additionally, BIOfactor MTA seems to be a lower costing product.

Calcium silicate-based materials can be exposed to masticatory or dislocating forces during the cement condensation period of a perforation repair. Thus, resistance to these dislocating forces and the marginal adaptation with dentin are crucial factors for the success of various endodontic procedures [9]. The bond strength of the cement to the dentin can be assessed by using a push-out test. Therefore, the objective of this *in vitro* study was to evaluate the push-out bond strength of a novel calcium silicate-based root repair material (BIOfactor MTA) to the root canal dentin and compare it with the more contemporary calcium silicate-based materials (white MTA-Angelus and Biodentine).

MATERIALS AND METHODS

Sample size calculation

According to a previous study [10], sample size was calculated based on effect size = 0.30, power $b = 95\%$, and $\alpha = 5\%$ input into an F-test family for analysis of variance repeated measures (G*Power 3.1 for Windows) and 27 samples were required to perform this study. However, 36 samples were included in order to account for possible exclusions like premature failure.

Specimen preparation

This study was approved by the Human Ethics Committee of the Institutional Review Board (protocol No. 2018/06). The study included 12 maxillary central incisors extracted for periodontal reasons. Teeth with dentin defects, previous endodontic treatment, calcification, internal resorption, or immature apices were excluded from the study. The teeth were disinfected with a 2.5% sodium hypochlorite (NaOCl) solution and they were stored in distilled water until further processing. The apices of the teeth were fixed on sticky wax; then, the teeth were embedded in self-curing acrylic resin. The upper middle third of each root was sectioned into 1.1 mm slices under water cooling, and three slices were obtained from each root (**Figure 1**). In total, 36 root slices underwent further processing.

The methodological set-up was adopted from a recent study, with minor modifications [10]. Briefly, three canal-like standardized holes of 1 mm in diameter were created parallel to the root canal in each dentin slice using a diamond bur. The holes were located at the corners of the triangular-shaped slices, and they were 1 mm from root canal lumen. The root slices were immersed in 2.5% NaOCl for 15 minutes, in 17% ethylenediaminetetraacetic acid for 3 minutes, and in 2.5% NaOCl for 1 minutes to remove the smear layer. Finally, they were rinsed with distilled water for 5 minutes to remove any solution remnants. The holes were dried with paper points, and dentin slices were placed on a glass slab and each cavity was filled with one of the test materials (MTA-Angelus, Biodentine, or BIOfactor MTA). The root repair materials were prepared according to each manufacturer's instructions (**Table 1**). For the BIOfactor MTA samples, 3 scoops of powder were mixed with 1 drop of liquid. Wet gauze was placed over the specimens, and the samples were stored in an incubator at 37°C for 7 days in order to allow complete setting.

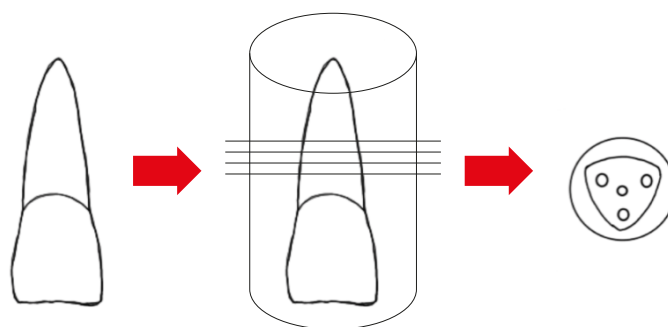


Figure 1. Schematic illustration of experimental design.

Table 1. Compositions of tested root repair materials and instructions for use according to manufacturer

Product and manufacturer	Composition	Instructions for use
MTA-Angelus (Angelus Industria de Produtos Odontologicos, Londrina, Brazil)	Powder: tricalcium silicate, dicalcium silicate, tricalcium aluminate, calcium oxide, bismuth oxide Liquid: distilled water	Mix 1 scoop of powder with 1 drop of distilled water for 30 seconds
Biodentine (Septodont, St-Maur-des-fosses, France)	Powder: tricalcium silicate, dicalcium silicate, calcium carbonate, calcium oxide, zirconium oxide as a radiopacifier Liquid: water, calcium chloride, hydrosoluble polymer (plasticizing agent)	Mix 1 capsule of powder with 5 drops of liquid for 30 seconds using amalgam mixing machine
BIOfactor MTA (Imicryl Dental, Konya, Turkey)	Powder: tricalcium silicate, dicalcium silicate, tricalcium aluminate, ytterbium oxide as a radiopacifier Liquid: 0.5%–3% hydrosoluble carboxylated polymer, demineralized water	Mix 3 scoops of powder with 1 drop of liquid until having a homogeneous consistency

Push-out testing

The samples were subjected to push-out testing using a universal testing machine (Devotrans Quality Control Test Equipment, Istanbul, Turkey) with a 0.6 mm in diameter cylindrical stainless-steel plunger tip and a loading speed of 1 mm/min until dislocation occurred. The maximum load applied for dislodging the cement was recorded in newtons (N) and transformed to megapascals (MPa) by dividing the load by the adhesion surface area of the root repair material. The bonded area was calculated using the following formula: $2 \times \pi \times r \times h$, where r is the root canal radius, h is the thickness of the root dentin slice in millimeters, and π is fixed at 3.14 [11].

Failure mode analysis

After the push-out testing, the samples were examined under a stereomicroscope (SZTP; Olympus Optical Co., Tokyo, Japan) at $\times 25$ magnification in order to evaluate the following failure modes: adhesive failure at the dentin-material interface, cohesive failure within the material, or mixed failure (a combination of adhesive and cohesive failures).

Scanning electron microscopy

Two dentin slices were selected for the scanning electron microscopy (SEM) evaluation. After the push-out testing, the samples were air dried overnight at room temperature, and then they were sputter coated with gold/palladium for 3 minutes. The specimens were examined particularly with the intention to observe the interface between dentin and the root repair material. The observations were conducted using SEM (Hitachi SU-1510; Hitachi High-Technologies Corp., Tokyo, Japan) under 20 kV and $50\times$ magnification.

Statistical analyses

Measurable push-out values could not be obtained from six of the specimens due to premature failure; therefore, only 30 specimens were involved in the statistical analyses. IBM SPSS Statistics for Windows version 22.0 (IBM Corp., Armonk, NY, USA) was used to analyze the results. The Shapiro-Wilks test indicated that the data was not normally distributed; therefore, a nonparametric Friedman analysis of variance was performed to compare the push-out values of the paired materials. The significance level was set at $p < 0.05$. Wilcoxon signed rank test with Bonferroni correction was performed as a post hoc test if the variance analysis test result was significant [12]. The level of statistical significance was set at $p < 0.017$ ($0.05/3$) after Bonferroni adjustment.

RESULTS

No statistically significant differences in the push-out bond strength were found between the three groups of materials ($p > 0.017$). The mean and standard deviation of push-out bond strength value was 15.77 ± 8.22 MPa for MTA-Angelus, 17.55 ± 5.67 MPa for Biodentine, and 18.24 ± 13.19 MPa for BIOfactor MTA (**Figure 2**).

Under the stereomicroscope, all of the groups showed cohesive failure majorities rather than mixed failures (**Table 2** and **Figure 3A**). An adhesive failure was observed in only one specimen in the MTA-Angelus group.

Representative scanning electron microscopic images of root repair material–dentin interfaces were shown in **Figure 3**. After the push-out testing, no signs of debonding was observed at

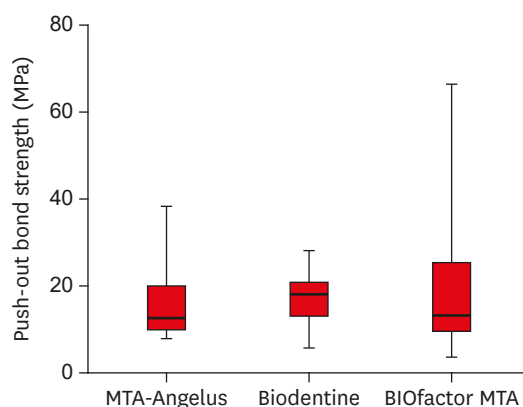


Figure 2. Push-out bond strength values for MTA-Angelus, Biodentine, and BIOfactor MTA.

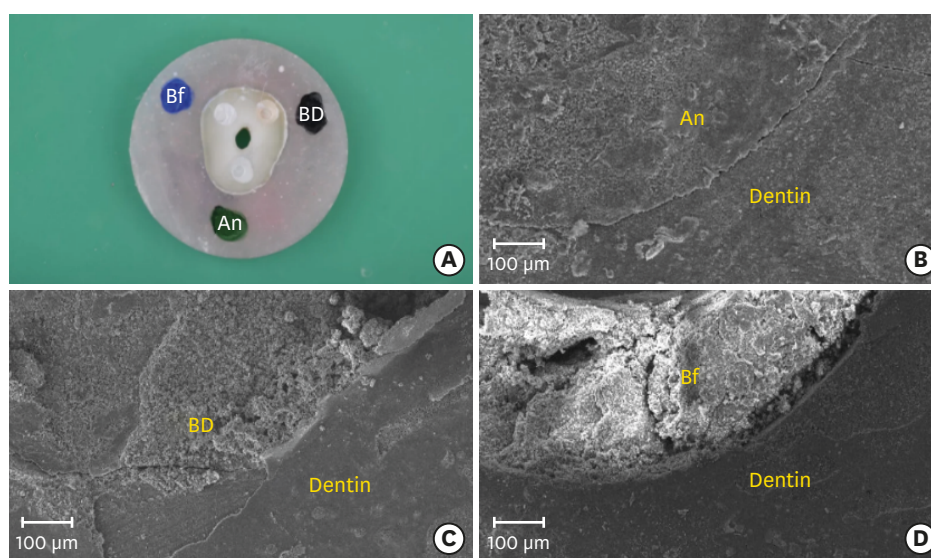


Figure 3. (A) Cohesive failure modes in randomly selected dentin disc (original magnification, $\times 25$) and (B-D) representative scanning electron micrographs of root repair material-dentin interface after push-out testing; (B) MTA-Angelus (An), (C) Biodentine (BD), and (D) BIOfactor MTA (Bf) (original magnification, $\times 50$).

Table 2. Failure mode distribution after the push-out test for root repair materials

Failure type	MTA-Angelus	Biodentine	BIOfactor MTA
Adhesive	1 (3.3)	0 (0)	0 (0)
Cohesive	27 (90.0)	29 (96.6)	24 (80.0)
Mixed	2 (6.6)	1 (3.3)	6 (20.0)

Values are presented as number (%).

the MTA-Angelus/dentin (**Figure 3B**) and Biodentine/dentin interfaces (**Figure 3C**), indicating good adaptation of the materials. However, few interfacial gaps could be seen between dentin and BIOfactor MTA (**Figure 3D**).

DISCUSSION

The perforation repair material's adhesion to the root dentin is one of the major requirements of a repair, because providing a good seal maintains the integrity of the filling material-dentin interface, increasing the overall success rate of an endodontic treatment.

However, the type of material can directly affect the quality of the dentin bonding. Therefore, this study was designed to evaluate and compare the bond strengths of three different calcium silicate-based materials used for perforation repairs.

In the present study, push-out testing was used to evaluate each calcium silicate-based material's resistance to dislocating forces. Previously, the push-out testing method has been shown to be efficient, reliable, and practical [13]. Furthermore, push-out testing exhibits certain advantages, including the fact that it more closely simulates clinical stress, permits accurate specimen standardization, and produces purer shear forces [14,15]. An innovative modification of the push-out test that was first presented by Scelza *et al.* [10] was chosen for this study. Because of its morphological and physical variations, human dentin is a complex bonding substrate [16], and thus, many variables related to the dentin substrate could alter the bond strength values. This new set-up enabled us to produce more reliable baseline values using the same dentin sample, while the confounding factors, such as the tooth age, mineralization, hardness, and elastic modulus, could be controlled better [10]. Some previous researches have applied this new methodology for testing root canal sealers and they have reported the reliability of this new set-up [17,18]. Other studies have also confirmed the usefulness of this method on determining push-out bond strength of calcium silicate-based cements [19,20].

The size of the cylindrical plunger used in the studies was adjusted to be closest fitting without touching the canal walls and mostly 3 sizes between 0.4 and 1.2 mm, usually in increasing increments of 0.2 mm were chosen [21]. As the hole diameter was 1 mm in the present study, 0.6 mm sized plunger was preferred. A previous study has investigated the effect of plunger diameter on push-out bond strength of different root filling materials and it has been showed that different plunger: canal size ratios (50%–83% of canal diameter) did not significantly affect the push-out bond strength [22]. More recent research has revealed lower push-out bond strength values when the plunger size was approximately 50% of the canal diameter [23]. According to these previous researches, the plunger size used in this study (60% of canal diameter) appears not to affect the push-out bond strength values. Additionally, 0.6 mm sized plunger could be easily positioned without contacting the dentin and maintained centralized.

The results of the present study indicated similar bond strength values for the MTA-Angelus, Biodentine, and BIOfactor MTA. Our findings are in agreement with previous research that revealed no significant differences between the bond strengths of the MTA-Angelus and Biodentine [24]. The similarities in the bond strengths among these calcium-silicate based materials may be due to the similarities in their basic composition, according to the manufacturers' information. However, Biodentine's higher resistance to dislodgement has been demonstrated previously [10], and it could be attributed to Biodentine's high biomineralization ability. This may have caused the uptake of calcium and silicon ions into the dentin, leading to the formation of tag-like structures [25] and increasing the material's adaptation to the dentin. Besides, MTA-Angelus has been shown to release high levels of calcium [26]. This higher calcium ion release may lead to more calcium phosphate precipitates and tag-like structures, resulting in high dislodgement resistance.

The BIOfactor MTA displayed at least as high of a bond strength as the MTA-Angelus and Biodentine. The finer Biodentine and BIOfactor MTA particles could have contributed the formation of the tag-like microstructure, thus improving the cement's penetration into the dentin tubules and providing better micromechanical adhesion [27]. Unlike other calcium

silicate-based materials, ytterbium oxide was added to the BIOfactor MTA as a radiopacifier agent. The effects of the addition of ytterbium oxide to the calcium silicate-based material on the material's physicochemical properties are unknown. However, ytterbium trifluoride has been incorporated into calcium silicate-based materials, and it was determined that the ytterbium trifluoride slightly increased the compressive strength of the portland cement, while enhancing the porosity of the material [28]. Further studies are required to determine whether the substitution of ytterbium oxide could affect the chemical bonding of the BIOfactor MTA to the dentin. Moreover, another BIOfactor MTA constituent, tricalcium aluminate, has been reported to be the most reactive component of portland cement [29]. Although tricalcium aluminate reacts vigorously with water, its contribution to the strength is minimal [29].

Our findings demonstrated that the bond failures observed in all the experimental groups were predominantly within the material (cohesive failure). These results are in accordance with a previous study that reported that the calcium silicate-dentin bond failures were usually cohesive [30]. The cohesive mode of fracture indicates that the bonding of these calcium silicate-based materials to the dentin was stronger than the cohesive strength of the materials. The cohesive failure percentage in the Biodentine group was the highest in our study, and previous studies have shown that the Biodentine-dentin bond failures were predominantly cohesive [31-33]. However, there are conflicting results for the MTA-Angelus; some previous studies reported adhesive failures [31,34], while other revealed mixed failures [24]. The differences in the MTA-Angelus failure modes could be attributed to the new methodological design used in the present study or different storage conditions. Incidentally, the BIOfactor MTA bond failure mode has not been determined in any other studies. Overall, the tubule penetration and micromechanical anchorage, which improve the retention of the material to the dentin, could explain the cohesive failure majorities in all of the groups [34].

One of the limitations of this *in vitro* study was that the samples were incubated in a moist environment for seven days before the push-out testing. However, in clinical conditions, the bond strengths of the perforation repair materials would be influenced by contamination with blood, irrigation solutions (including mostly NaOCl), and an acidic environment. Therefore, further long-term studies are suggested to evaluate the effects of both ageing and unavoidable environmental factors during setting on novel calcium silicate-based cements.

CONCLUSIONS

Within the limitations of this *in vitro* study, the MTA-Angelus, Biodentine, and BIOfactor MTA exhibited similar resistances to the push-out testing. All of the materials showed predominantly cohesive failures, with the highest rate found in the Biodentine samples. Overall, the BIOfactor MTA seemed appropriate for use as a root repair material with regard to its bond strength; however, its other characteristics, including its biocompatibility, bioactivity, and physicochemical properties, should be investigated further.

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