

# Evaluation of the push-out bond strength of ProRoot MTA and Biodentine after removal of calcium hydroxide and triple antibiotic paste

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## Abstract

**Aim:** To evaluate the effect of calcium hydroxide (CH) and triple antibiotic paste (TAP) on the push-out bond strength of ProRoot® MTA and Biodentine®.

**Methodology:** Root canals of 120 human teeth were instrumented using rotary files. To obtain a standard diameter of 1.5 mm, the roots were instrumented with a #6 Peeso reamer. The reamers passed 1 mm beyond the apex point to simulate open apices. The roots were randomly assigned into the following groups: Group 1 was administered calcium hydroxide (CH); Group 2 was administered triple antibiotic paste (TAP); and Group 3 was a control group and had no medicament. The medicaments were removed after 3 weeks. Thereafter, the specimens were divided into two subgroups (n = 20) according to which calcium silicate-based cement, ProRoot MTA (Dentsply Maillefer, Ballaigues, Switzerland) or Biodentine (Septodont, France), was applied. The push-out test was performed, and the data were analyzed statistically using one-way ANOVA and by Tukey's post hoc test.

**Results:** In both the ProRoot MTA and Biodentine groups, pairwise comparisons between medicaments showed similar values to the control group ( $p > 0.05$ ). Overall, there was a predominance of cohesive failures between root dentin and the cements.

**Conclusions:** ProRoot MTA and Biodentine showed similar bond strength values, and prior application of TAP or CH did not affect the bond strength significantly.

**Keywords:** Biodentine, calcium hydroxide, mineral trioxide aggregate, ProRoot MTA, triple antibiotic paste

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## Introduction

Dental trauma or irreversible pulp inflammation to young teeth may result in pulp necrosis and incomplete root formation. The challenges of root canal treatment for immature teeth are cleaning, shaping and obturation because of open apices (1). In addition, root fractures may occur in these teeth due to thin and/or weakened root walls (2). A new technique called regenerative endodontic treatment (RET) involves tissue engineering in the root canal system of immature teeth (3). For successful results of RET, inflamed pulp tissue is completely removed and bacteria is eliminated from the root canal space; elimination is provided by effective applications with various irrigation solutions and medicaments (4, 5).

Antibiotic pastes and calcium hydroxide (CH) are frequently used medicaments for RET (6, 7). Triple antibiotic paste (TAP) is the most commonly used root canal disinfectant during RET since its introduction by Hoshino et al. in 1996. TAP consists of ciprofloxacin, metronidazole and minocycline and has effective antimicrobial properties (8). Also, CH is prevalently used as an endodontic intracanal dressing material because of its high pH and appropriate antibacterial effects (9). It has been successfully used to disinfect the canal during RET (7, 10).

After the RET period, mineral trioxide aggregate (MTA), which is a calcium silicate-based material, was generally placed on the coronal 3-4 mm section of the root canal. MTA has biocompatible, conductive and inductive properties which allows it to chemically bond to the root dentin wall (11, 12). Nevertheless, MTA remains subject to some concerns: it has a long setting time, is difficult to manipulate, has a low resistance to compression and flow capacity, causes discoloration of tooth structure and has a high cost (11, 13).

Alternative calcium silicate-based materials were introduced to replace MTA (14, 15). Biodentine (BD), one of these materials, is a tooth-coloured, bioactive cement used for apexification, repair of root perforations and retrograde root filling as an MTA (16).

In the literature, there is a limited number of studies evaluating push-out bond strength of BD using intracanal medicaments for the RET procedure (17). Therefore, the aim of this study was to evaluate the effect of intracanal medicaments (CH and TAP) used during RET on the push-out bond strength of an MTA and BD to root canal dentin.

## Materials and Methods

One hundred twenty single-rooted human mandibular premolar teeth, which were freshly extracted for periodontal or orthodontic reasons, were selected and stored in 0.1% thymol until the commencement of the experiment. The teeth were examined under an operating microscope (Euromex, Arnhem, Germany) and excluded if there were any root caries, cracks or fractures. Preoperative radiographs were taken to validate a single canal without canal treatment, internal resorption or calcification. The specimens were decoronated using a diamond disk with water cooling to standardize root length to 15 mm.

The root canals were prepared using ProTaper rotary instruments (Dentsply Maillefer, Ballaigues, Switzerland) up to size F5. To achieve a standard internal diameter of 1.5 mm and to simulate immature teeth, reamers (Dentsply Maillefer, Ballaigues, Switzerland) between #1 and #6 were used in the root canals and passed 1 mm beyond the apex to simulate open apices. The canals were irrigated by using 2 ml of sodium hypochlorite (NaOCl) during instrumentation. After instrumentation, the canals were irrigated with 5 ml of NaOCl and 5 ml of 17% ethylenediaminetetraacetic acid (EDTA) solution. Finally, the root canals were flushed with 5 ml of distilled water and dried using paper points.

The roots were randomly assigned into three groups. Group 1 was treated with calcium hydroxide (CH); Group 2 was given triple antibiotic paste (TAP); and Group 3 acted as a control and had no medicament.

### Preparation of Intracanal Medicaments

Group 1: Calcium hydroxide powder (Kalsin; Spot Diş Deposu A.S, Izmir, Turkey) mixed with distilled water.

Group 2: A mixture of metronidazole (Flagyl® 500 mg tablet, AventisPharma S.A., France), ciprofloxacin (Cipro® 500 mg tablet, Biofarma İlaç San. ve Tic. A.Ş., Turkey) and minocycline (Minoz™ 50 mg tablet, Ranbaxy Lab. Ltd., India).

Group 3: No medicament.

The coronal access of the root canals was sealed with a cotton pellet and temporary filling material (Cavit; 3M ESPE, Seefeld, Germany). The specimens were stored at 37 °C in 100% humidity for 3 weeks.

Afterward, intracanal medicaments were removed by rinsing with 10 ml of NaOCL followed by 5 ml of distilled water. The specimens were divided into two subgroups (n = 20) according to the calcium silicate-based cement (ProRoot MTA or BD) applied. The subgroups were as follows:

CH + MTA Group

TAP + MTA Group

Control + MTA Group  
 CH + Biodentine Group  
 TAP + Biodentine Group  
 Control + Biodentine Group

ProRoot MTA (Dentsply Maillefer, Ballaigues, Switzerland) and BD (Septodont, Saint-Maur, France) were mixed according to the manufacturer's instructions. The test materials were placed into the canals with the aid of a lentulo spiral (Dentsply, Konstanz, Germany). By using pluggers (Dentsply, Konstanz, Germany), the materials were condensed vertically.

Approximately 3 mm of MTA or BD were placed in the coronal third of the canals and gently applied to the dentinal walls with a moistened cotton pellet. The cavities were filled with a temporary filling material. Finally, the specimens were stored at 37 °C in 100% humidity for one week.

Then, the specimens were cut horizontally from the coronal root region using a low-speed precision diamond saw (Micracut 201, Metkon, Bursa, Turkey) under water cooling to produce slices approximately 1 mm thick. The thickness of each slice was measured with a digital calliper (Teknikel, Istanbul, Turkey) with an accuracy of 0.001 mm. The push-out test was performed in a universal testing machine (Lloyd Instruments Ltd, Fareham, UK) at a crosshead speed of 1 mm/min by using cylindrical plungers (Figure 1). The diameter of each plunger was approximately 90% of the slice's diameter.

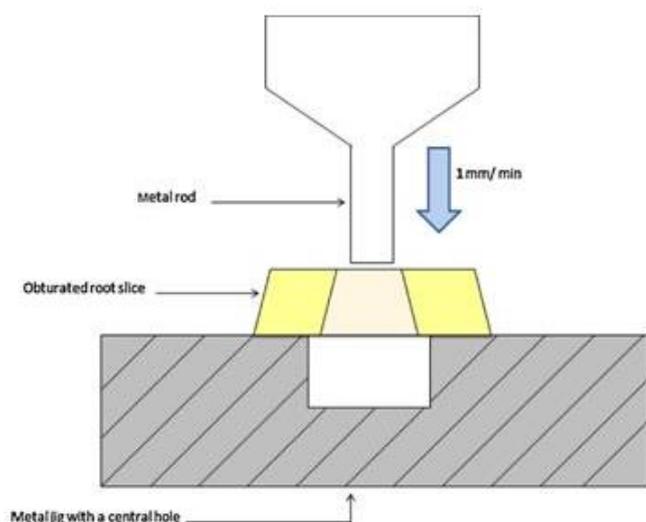


Figure 1. Schematic drawing of the push-out test.

The maximum load applied to the filling material before failure was recorded in newtons (N) and converted to megapascals (MPa) according to the following formula:

Push-out bond strength (MPa) = Maximum load (N)/Adhesion area of root filling (A) (mm<sup>2</sup>).

The adhesion surface area of the root canal filling (A) was calculated by the following:

$$(\pi r^1 + \pi r^2) \times L, \quad (\text{Eq. 1})$$

where  $\pi = 3.14$ ;  $r^1$  and  $r^2 =$  smaller and larger radii, respectively;  $L = \sqrt{(r^1 - r^2)^2 + h^2}$ . The h equals the thickness of the slice in mm (18).

After the test procedure, each specimen was examined under an operating microscope (Euromex, Arnhem, Germany) at 30× magnification to determine the mode of fracture. Three types of failure were categorized: adhesive, between the filling materials and root dentin; cohesive, within the filling materials or root dentin; and mixed, a combination of cohesive and adhesive failures (19).

## Statistical Analysis

The data were analysed using one-way ANOVA, and Tukey's post hoc test was performed for multiple comparisons. The significance level was set at  $p < 0.05$ . All data were processed using SPSS 16.0 statistical software (SPSS Inc., Chicago, IL, USA).

## Results

The mean and standard deviation of the push-out bond strength values are shown in Table 1. The statistical analysis indicated that bond strength values of both silicate cements were not significantly affected by the intracanal medicaments ( $P > 0.05$ ). For BD and MTA materials, the highest push-out bond strength values were obtained with TAP groups (TAP + MTA and TAP + Biodentine). The bond strength of TAP + Biodentine was significantly higher than CH + Biodentine, CH + MTA and the control + MTA groups ( $p < 0.05$ ).

The modes of failure are listed in Table 2. Cohesive failures were observed most frequently in all groups.

**Table 1.** Push-out bond strength values (MPa, mean, standard deviation) of calcium silicate cements with respect to intracanal medicaments

Groups	Bond strength
Control +MTA Group	8.31±2.01
CH + MTA Group	8.14±2.44
TAP + MTA Group	8.98±1.87
Control + Biodentine Group	8.65±2.99
CH + Biodentine Group	7.70±2.40
TAP + Biodentine Group	10.11±2.25

**Table 2.** Failure modes for each group

Groups	Failure Modes		
	Adhesive	Cohesive	Mixed
Control +MTA Group	1	12	7
CH + MTA Group	2	14	4
TAP + MTA Group	1	15	4
Control + Biodentine Group	1	15	4
CH + Biodentine Group	2	16	2
TAP + Biodentine Group	1	17	2

## Discussion

After RET, calcium silicate-based materials were used to form bacteria-tight seals in the pulp space (11). MTA has been widely used for RET (11, 20). Nevertheless, MTA has some disadvantages; therefore, BD could be an alternative material used with RET (14, 15). The push-out test has been shown to be a reliable method for evaluating the displacement resistance of the filling material during operative procedures and mastication (21-23). Furthermore, pulp scaffolding may be affected by the force applied to the coronal barrier during the placement of restorative material (20).

MTA is a type of hydraulic cement which sets in the presence of fluid. When MTA comes into contact with a fluid, it continues to dissolve and hydroxyapatite-like crystals form between the MTA and dentinal walls (12, 16, 24). In the present study, the specimens were stored for 1 week in a moist environment to simulate clinical conditions.

BD has similar properties to MTA (except for zirconium oxide and calcium chloride, a setting accelerator water reducing agent). In both materials, a calcium silicate hydrate colloidal gel layer and apatite deposits form on the surface; these interact with tissue fluids and enhance the sealing ability and bond strength

of the materials (25). The setting time of BD is relatively short (12 min) and has favourable handling characteristics; its placement is less time-consuming than MTA. Thus, coronal restoration can be performed at the same appointment (26, 27).

In the present study, both CH + MTA and CH + BD groups displayed lower mean bond strength values than other groups. This could be explained by CH's reaction with dentin and may cause the disruption of dentin collagen. The high alkalinity of CH deteriorates the bond between hydroxyapatite and collagenous fibrils and reduces the dislocation resistance of MTA and BD to root dentin (28, 29). On the contrary, the push-out bond strength of MTA and BD was not affected by CH in the study of Nagas et al. and Centenaro et al. (17, 30).

The present study has shown that BD displayed higher push-out bond strength values compared to MTA, except for the CH + BD group. Nagas et al. have evaluated the dislocation resistance of BD and MTA to the root dentin after applying various intracanal medicaments (CH, TAP, Augmentin, Ledermix); BD yielded higher bond strength values than MTA. This result was explained as BD having a potential to enhance penetration into the medicament-free dentinal tubules because of its smaller particle size, leading to improved bond strength (17). Also, the study

of Guner et al. showed that BD established significantly stronger bonds to root dentin than MTA after being exposed to various endodontic irrigants (31). Similarly, Centenaro et al. stated that BD yielded higher push-out bond strength values compared with those of MTA, regardless of the use of CH (30). On the contrary, the study of Alsubait et al., which evaluated push-out bond strength of ProRoot MTA and BD without medicaments, showed that bond strength of BD is like MTA (14). This may be due to the absence of any medicaments or irrigants.

The results of the present study showed that intracanal medicaments (CH and TAP) did not significantly affect the dislocation resistance of MTA and BD to root dentin. This result is similar to the study of Topcuoglu et al. They stated that CH and TAP did not influence the dislocation resistance of MTA to root dentin (20). Conversely, in the study of Nagas et al. compared with the control groups, TAP decreased the debonding force of MTA and BD (17). The difference in the removal procedures may have led to the differing results. In the present study we did not use EDTA for removal of medicaments.

In the present study, highest bond strength values were observed in TAP groups. In accordance with these results, the study of Akçay et al. found that the TAP group displayed higher bond strength values than CH and a double antibiotic paste (DAP) group (18). This may be due to the binding of residual minocycline to calcium ions via chelation, which could increase bond strength after application of TAP (32). On the other hand, in the study of Oktay et al. the push-out bond strength of the CH group was found significantly higher than the TAP group of a calcium-phosphate-silicate-based cement (EndoSequence root repair material) (33). Also, Felipe et al. and Bidar et al. found that CH had a positive effect on the bond strength of calcium silicate-based sealers (34, 35). Nagas et al. stated that the reaction of calcium silicate-based cements with residual calcium hydroxide resulted in improvement of the marginal adaptation (17).

## Conclusions

Within the limitations of this study, BD and ProRoot MTA showed similar bond strength values, and it can be concluded that prior application of TAP and CH had no significant negative effect on the bond strength of the materials.

When previous literatures were evaluated, there was found a need for new studies regarding the influence of antibiotic medicaments and CH on the bond strength of calcium silicate-based cements.

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