# The comparison 24-hour bonding performance of novel OrthoMTA and ProRootMTA on root dentin

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#### Abstract

Aim: Bioceramic cements act as an alternative dentin replacement materials. The aim was to compare 24h push-out bond strength of novel OrthoMTA (BioMTA, Seoul, Korea) cement, and ProRootMTA(Dentsply Sirona, Tulsa, OK, USA) cement on root dentin.

**Materials and Methods:** Twenty mandibular first premolar teeth were randomly divided into two groups. In each group, three 1-mm thick slices were obtained from the middle third of the root by cutting horizontally. Thirty root slices were obtained from ten roots. In the sectioned root slices, a cavity-like hole was prepared in the center and OrthoMTA and ProRootMTA were placed into the holes per groups. Samples were kept at 36°C, 100% humidity for 24h. The push-out bond strength was calculated (MPa). The critical point drying of all samples was performed followed by scanning electron microscopy evaluation.

**Results:** Adhesive-type of failure at the cement-dentin interface, cohesive-type of failure within the bioceramic cement, and combination of adhesive- and cohesive-type of failures termed as mixed-type of failure were observed. One-way analysis of variance followed by Tukey's test was performed (p<0.05).

**Conclusion:** Within the limitations of the in-vitro study the following conclusions can be drawn: 24h after hardening reaction, OrthoMTA presented superior bonding performance than that of ProRootMTA on root dentin (p<0.05) and the most common type of failure was mixed-type in both OrthoMTA and ProRootMTA.

Keywords: Adhesion; bioceramics; brittle materials; cements; debonding; OrthoMTA; ProRootMTA

### INTRODUCTION

Dental bioceramic cements are bioactive materials and they are indicated to use in multiple clinical conditions such as vital pulp treatments, tooth revitalization, immature roots, iatrogenic perforations and root canal obturation (1,2). ProRoot mineral trioxide aggregate (ProRootMTA) is the first introduced bioceramic cement to the dental market, as a perforation repair and retrograde grafting material (3). ProRootMTA could promote to osteogenic activity in resorptive defects, and apicoectomies (4,5). Moreover, ProRootMTA is the gold standard bioceramic cement for comparative studies in literature due to having long-term clinical results (2,6,7).

Bioceramic cements act as an alternative dentin replacement materials. Thus, the adhesion of the bioceramic cement to dentin is fundamental for sealing. Although root canal sealers or different adhesive strategies can establish firm adhesion of filling materials to the dentin substrate, the resinous-based relation may leave gaps (8). The contemporary approach in endodontic

materials is to assess various alternative obturation materials to gutta-percha and sealers. In this manner, a novel bioceramic cement, OrthoMTA (BioMTA, Seoul, Korea), was introduced for total root canal grafting (9). According to its manufacturer, OrthoMTA was synthesized by the gold standard mineral trioxide aggregate's active ingredient through a bioceramic manufacturing process (10). However, the composition of OrthoMTA has some differences between the ProRootMTA containing arsenic and chromium concentrations (11). Moreover, physical characteristics of OrthoMTA such as average particle size, curing duration are also different than ProRootMTA. Elemental composition and physical characteristics of bioceramic cements may affect its bioactivity and sealing properties (12). Thus, the purpose of this study was to compare the 24-hour push-out bond strength of OrthoMTA, as a novel canal grafting bioceramic cement, with gold standard ProRootMTA on the root dentin substrate. The 0 hypothesis was that there would not differences between OrthoMTA and ProRootMTA.

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#### MATERIALS and METHODS

The present study has been approved by the Human Ethics Committee of the Medicine Faculty of Eskisehir Osmangazi University. Twenty extracted mandibular first premolar teeth for orthodontic purposes were used. All human donors have been signed the written informed consent form for donation. Teeth with homogeneous sizes were selected. Standard periapical radiographs were acquired to confirm the single root canal of the root. Teeth were stored in 0.1% C10H140 media until they were used.

Decoronation was performed using a precision diamond disc (Isomet 15 LC, Lot#110069939D16, Buehler, IL, USA) 1 mm below the cemento-enamel junction under water cooling and crowns were discarded. The soft tissue remnants were removed by barbed-broaches (Antaeos, VDW GmbH, Munich, Germany) and an excavator (17EX17; Deepeler S.A. Rolle, Switzerland). The smear layer on the root dentin surfaces was removed using the 17% EDTA solution (Lot# 190804, Promida Co. Eskişehir, Turkey) by 1 min continuous flushing. The pulp tissues were removed using an excavator, then the pulp chamber was cleaned with 5.25% with sodium hypochlorite (Lot#190800, Promida Co. Eskişehir Turkey), was disinfected with 2% chlorhexidine (2% ProChex Lot# 190802, Promida Co., Eskişehir Turkey). Then samples were rinsed with 5 mL of distilled water and dried with gentle stream of oil-free compressed air for 5 s, at the distance of 10 cm to remove excess water.

Ten roots were randomly selected for each bioceramic cement presented in Table 1. In each main group, three slices were obtained from the middle region of the root by cutting horizontally using the diamond saw under water cooling. The mean thickness of each slice was 1±0.1mm (Data was not shown). In each main group, 30 slices were obtained from 10 teeth. Slices were classified as coronal, middle or apical according to the proximity of the slices to the crown or apex (Graphical abstract).

Table 1. Information of bioceramic cements tested in this study							
Group	Powder	Powder Color	Liquid	Lot #	Manufacturer's mixing instruction	Manufacturer	
OrthoMTA	Tricalcium silicate,				Sterile water was added into OMTA tube then was mixed	BioMTA,	
	Dicalcium silicate,						
	Tricalcium aluminate,	White	Distilled	0M1502D07			
	Tetracalcium aluminoferrite, water water	01011 303001	Excess water was removed	ocoul, Koled			
ProRoot Mineral Trioxide Aggregate	Free calcium oxide,				with a cotton swab PMTA powder is mixed with supplied sterile water in a 3:1 powder/liquid ratio	Dentsply Sirona, Tulsa, OK, USA	
	Bismuth oxide						
	Tricalcium silicate,	White	Distilled water	14050801			
	Dicalcium silicate,						
	Tricalcium aluminate,						
	Tetracalcium aluminoferrite,						
	Free calcium oxide,						
	Bismuth oxide						



**Figure 1.** Schematic representation of the push-out set-up. a; Plunger tip positioned over the bioceramic cement, b; Geometric parameters of the set-up as force (F), the diameter of plunger tip (d1), the diameter of bioceramic cement (d2), the thickness of dentin slice (t)

In the sectioned root slices, a cavity-like hole was drilled using size #2 Peeso Reamer (VDW GmbH) using an endodontic hand piece attached on a dental surveyor

at 800 rpm. Drilled root slices were rinsed with distilled water for 60s to remove remnants and dried with ISO #90 paper points (DiaPaper, Diadent Group International, Almere, Netherlands). Both bioceramic cements were then hydrated with a constant powder: liquid ratio as 3:1 and they carried into the prepared cavity-like holes using a MTA carrier (MTA+ Applicator, Cerkamed, Stowola-Wola, Poland). Grafted cements were gently condensed into the cavities with non-vigorous pressure (Figure 1). To simulating physiologic conditions, slices were then placed in a preheated universal laboratory incubator (EC 160 - Nüve Laboratory & Sterilization Technology, Ankara, Turkey) at 36°C, 100% humidity for 24 hour. The stereomicroscopic examination was performed on each slice to assess any procedural errors before the attachment to the universal test machine Figure 2 (MOD Dental - Esetron Mekatronik, Ankara, Turkey). Previously described push-out bond strength test parameters where

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the plunger diameter was 0.7mm were used in the present study (13).



Figure 2. A representative image of a sample attached to the device

The critical point drying at 36°C of each deboned slice was performed followed by scanning electron microscopy evaluation to identify visualization of failure types at 100X magnification (Gemini FESEM 500, Carl Zeiss NTS Ltd. Cambridge, UK). Furthermore, both OrthoMTA and ProRootMTA cements were prepared separately, and they placed into Teflon cylindrical molds (the dimension of the molds was 2 mm radius, 1 mm thickness). Eightmold were produced by computer-aided manufacturing. To monitoring OrthoMTA and ProRootMTA at very high magnification after 24-hours, filled molds were kept at 100% humidity and 36°C for 24-hours.

Data were analyzed using statistical software (Prism 6.0, GraphPad Software, La Jolla, CA, USA). Data were normally distributed. Therefore, one-way variance analysis followed by Tukey's test was performed to compare regional pushout bond strength data in each group. However, statistical analogy amongst the root regions was observed. Thus, regional data were collected into a single data pond for each main group and the comparisons was performed using unpaired t-test between the OrthoMTA and ProRootMTA. Alpha set at 0.05

## RESULTS

The statistical comparisons of bioceramic cement groups and mean and standard deviations of push-out bonding strengths (MPa) were given in Table 2. Twenty four-hour after hardening reaction, unpaired t-test showed that OrthoMTA presented higher push-out bond strength than that of ProRootMTA on root dentin (p<0.05).

Adhesive-type of failure at the cement-dentin interface, cohesive-type of failure within the cement, and combination of adhesive- and cohesive-type of failures termed as mixed-type of failure were observed in both bioceramic cements Figure 3 and the most common type of failure was mixed-type in both OrthoMTA and ProRootMTA cements (Table 3). After 24-hours, both bioceramic cements have different surface characteristics Figure 4. OrhoMTA presented denser or bulkier structure than ProRootMTA at 50,000X magnification.

Table 2. The mean and SD of the bond strengths values (MPa) of bioceramic cements tested in this study						
Groups	Total number of slices	Mean ± SD				
OrthoMTA	30	9.809 ± 2.176*				
ProRootMTA	30	7.103 ± 2.145				

'Unpaired t-test p< 0.0001



**Figure 3.** Scanning electron micrographs show representative failure modes; a, adhesive-type of failure original magnification x77; b, cohesive type of failure at original magnification x125; c, mixed type of failure original magnification x100

Table 3. Distribution of failure modes of OrthoMTA and ProRootMTA								
Groups	Failure modes	Coronal	Middle	Apical	Total			
ProRootMTA	Adhesive	-	-	-	0			
	Cohesive	2	3	3	8 (36%)			
	Mixed	8	7	7	22 (64%)			
OrthoMTA	Adhesive	-	-	-	0			
	Cohesive	1	-	1	2 (6%)			
	Mixed	9	10	9	28 (94%)			



**Figure 4.** FESEM visualisation of OrthoMTA(top) and ProRootMTA(bottom) at 50,000X magnification

## DISCUSSION

In the present study was tested the 24-hour push-out bond strength performance of novel OrthoMTA and was compared with the gold standard ProRootMTA. There were statistically significant differences were observed between the tested biocereamic cements; thus, the 0 hypothesis was rejected.

To test the bonding performance of both bioceramic cement, the push-out test was used in the present study. The technique is recommended for assessing the bonding strength of root canal material at the dentin substrate interface (14). To standardize the volume of OrthoMTA and ProRootMTA, standard cavity-like holes were prepared in all root slice samples by parallel reamers. In agreement with the previous report, cavity-like holes were prepared inside the root canal for simulating the actual clinical condition (15).

The final geometry of the cavity was cylindrical-shaped and the diameters were as equal. Thus, bioceramic cements were grafted to standard 1-mm thick cavities regardless of the manipulation effect of an operator per groups, recommended by Dawood et al (16). Moreover, conical geometry-related problems were prevented during the displacement action of the push-out test (17). The push-out bond strength values could be affected by previously described geometric parameters by Chen et al. (18) as follows: The plunger diameter, the cavity diameter, and the thickness of the slice. In the present study, dimensions of the push-out test model were designed in accordance with the previous suggestions.

Principally, bioceramic cements contain hydrophilic particles (3). When these particles contact with water, tricalcium-silicate-hydrate which is a kind of less soluble gel formation occurs (19). The insoluble gel has a short tag-like form at the cement-dentin interface and it could be seen after 4 hours for hydrated ProRootMTA (20,21) reported that the tricalcium-silicate-hydrate gel has been the adhesive property of bioceramic cement to dentin substrate in the short-term. The gel formation followed by biomineralization or hydroxyapatite aggregation reactions is seen in the long-term. These reactions were observed in both ProRootMTA (20,21) and OrthoMTA (13,22) after weeks for hydrated cement. Although the long-term biomineralization reactions could improve the dislocation resistance or the bonding strength of the bioceramic cement, the short-term 24-hour push-out bonding performance was evaluated in the present study for obtaining a baseline data for the novel OrthoMTA. Thus, the adhesion property of OrthoMTA was tested using the push-out bonding test instead of monitoring only the interface at high-magnifications.

A recent study has reported that the push-out bond strength of OrthoMTA was  $15.08 \pm 4.17$ MPa in the middle region of the root after 14-day (13). The same study has also reported adhesive-type of failure was seen the most type of failure in OrthoMTA in the middle

region of the root.13 In the present study, the push-out bond strength of OrthoMTA had 9.80 ± 2.17MPa and the type of failure was mostly mixed-type in both OrthoMTA and ProRootMTA. The differences between the recent report and the present study might be originated from observation period differences. A previous study has observed 24-hour dislodgment resistance of ProRootMTA (23). Authors emphasized that ProRootMTA was still setting at 24-hour evaluation period (23). Evaluation at periods longer than 24-hour revealed that push-out bond strength of ProRootMTA increased which can be based on biomineralization ability of bioceramic cements (23-25). In agreement with the previous reports, predominantly mixed-type failure of OrthoMTA and ProRootMTA may be explained by ongoing hardening and bioactivity of the cements. One could expect more adhesive failures and less cohesive failures as cements get harder.

Previous studies showed dentin tubule penetration and biomineralization ability of ProRootMTA (20,21) and OrthoMTA (26). Authors emphasized that dentinal tubules and orifices were sealed by short and long tags which indicated biomineralization ability of the material. Komabayashi & Spangberg (27) reported that the particle size and shape of bioceramic cements could affect its adhesive properties to the dentin substrate. According to the Mjor et al (28) the diameter of the dentin tubule ranges from 2µm to 5µm therefore, smaller particles may penetrate the tubules (27). The median of the particle size of OrthoMTA is 2µm (10,29) whereas, the particle size of ProRootMTA ranges between 1.5-10µm (30). Therefore, one could expect better sealing with OrthoMTA than ProRootMTA, since bioceramic cements with smaller particles have better contact with the distilled water used for hydration reaction; thus, result in improved handling properties and higher early strength of the hydrated bioceramic cement (2). This could partially explain the higher bond strengths obtained with OrthoMTA.

A recent study has investigated the chemical composition and porosity characteristics of various calcium silicatebased endodontic cements (31). It has been reported that the specific surface area of ProRootMTA has been smaller than OrthoMTA in the porous test (31). In agreement with the recent study, OrhoMTA presented denser or bulkier structure than ProRootMTA after 24-hours in the present study.

The physicochemical properties of TCSs are also influenced by different types of storage media. Several studies evaluating the properties of TCSs used body fluid simulation solutions such as phosphate-buffered saline (16,20,21), Hank's balanced salt solution (32) to store the setting cement before testing. These phosphatecontaining synthetic tissue fluids could negatively affect the setting of TCSs (33). Different TCSs react differently and might exhibit different properties in phosphatecontaining synthetic tissue fluids than in water (32). It was speculated that hydration and setting behavior of TCSs in physiologic solutions cannot be predicted (34). Therefore, in this study, we kept the specimens in contact with sterile gauze moistened in distilled water to prevent potential negative effects. Since no perforation model or apical sealing model was used in our study, it was not necessary to keep samples in simulated body fluid environment.

The adhesive properties of the TCS are influenced by many factors such as cement type, cement thickness. powder/liquid ratio, environmental humidity and pH, and condensation pressure (2). Although hydrated TCS does not require a precise ratio, inconsistent powder/liquid ratio, insufficient condensing or dehydration may cause macro and micro porosities of hydrated PMTA which can lead to leakage (35). If not all powder particles are hydrated during mixing, the ultimate strength of the material could be reduced. Thus, it is important to obtain a standardized mixed cement. It may be difficult to obtain an accurate water-powder ratio for PMTA during mixing which could lead to the dissolution of bioactive components and material porosity (35,36). The amount of porosity in mixed cement is related to the amount of water added to make a cement paste, entrapment of air bubbles during the mixing procedure. According to its manufacturer, initial hydration of the OMTA powder could be standardized as follows; sterile distilled water is added into a tube containing the OMTA powder up to a marked level then it is mixed for 20 s. Excess, moisture is then absorbed with a cotton swab applied into the mixed content in the tube. This method ensures that every mixed OMTA has standardized water content. This might partly explain the bond strength differences between PMTA and OMTA.

## CONCLUSION

The present study investigated that the push-out bond strength of a novel OrthoMTA, designed specifically for root-canal obturation. Within the limitations of the study, it can be concluded 24-hour push-out bond strength of OrthoMTA is higher than that of ProRootMTA. The higher bond strength for OrthoMTA was probably to be due to its finer particle size. Bond strengths of both bioceramic cement were not affected by the root dentin regions. These findings may be a reference for further studies investigating the orthograde filling technique with OrthoMTA.

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