

# Shear bond strength of novel calcium aluminate-based cement (EndoBinder) to root dentine

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## ABSTRACT

**Objective:** To evaluate the shear bond strength of a novel calcium aluminate-based cement, EndoBinder (EB), to dentine in comparison with Grey and White Mineral Trioxide Aggregate (MTA). **Materials and Methods:** Root canal hemi-sections obtained from 30 extracted molar teeth were embedded in self-polymerized acrylic resin and were grounded wet in order to obtain a flat dentine surface. Next, the roots were randomly assigned into three groups ( $n = 10$ ), according to the cement used, as follows: EB: EndoBinder; WMTA: White MTA and GMTA: Grey MTA. The shear bond strength test was performed using a Universal Testing Machine (0.5 mm/min) and the data were submitted to statistical analysis (1-way ANOVA and Tukey tests,  $P < 0.05$ ). **Results:** EB presented the highest shear bond strength values; however, there was no statistically significant difference in comparison with GMTA ( $P > 0.05$ ). WMTA presented the lowest mean values, which were significant in comparison with EB ( $P < 0.05$ ). **Conclusions:** The novel calcium aluminate-based cement presented higher shear bond strength than WMTA, and should be considered as a promising alternative in endodontic therapy.

**Key words:** Calcium aluminate cement, mineral trioxide aggregate, shear bond strength

## INTRODUCTION

Mineral Trioxide Aggregate (MTA) was originally developed as a cement for retrograde filling and root perforation treatment.<sup>[1]</sup> However, because of its adequate clinical performance, it has been used in several other applications such as pulpotomy,<sup>[2]</sup> direct pulp capping,<sup>[3]</sup> apexification,<sup>[4]</sup> retrograde filling after apicectomy or, in selected cases, the entire root canal.<sup>[5]</sup> MTA is available in two forms, Grey MTA (GMTA) and White MTA (WMTA), with White MTA having reduced iron oxide concentration.<sup>[6]</sup>

Despite the low concentration of iron oxide in WMTA, some negative properties still remain in

both versions of the cement, such as the high rates of dental structures staining,<sup>[7,8]</sup> long-setting time,<sup>[9]</sup> poor handling characteristics,<sup>[10]</sup> high solubility in moist environment,<sup>[11,12]</sup> low flow capacity<sup>[13]</sup> and low adhesion to the root canal walls compared with other cements used for perforations repair,<sup>[14]</sup> and should be taken into consideration.

These facts justify constant changes in the composition of mineral aggregate cements and the development of new materials, such as a novel calcium aluminate-based cement, EndoBinder (EB) (Binderware, São Carlos, SP, Brazil), which has adequate biological<sup>[15-17]</sup> and physico-chemical properties<sup>[12,18,19]</sup> and clinical

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applications similar to MTA however without its negative features.

According to Castro-Raucci *et al.*,<sup>[15]</sup> the exposure of primary osteogenic cell cultures to EB promoted greater osteoblastic cell differentiation than samples exposed to MTA. Furthermore, EB presented, among its characteristics, superior mechanical properties than MTA,<sup>[18]</sup> absence of dental structure staining<sup>[8]</sup> and increased flowability, allowing its application within the pulp chamber and root canal areas where the access is difficult.<sup>[20]</sup> However, the adhesion ability of the material to dentine has not been tested yet.

Therefore, the aim of this *in vitro* study was to evaluate the adhesiveness of this novel calcium aluminate-based cement to root dentine in comparison with GMTA and WMTA by means of a shear bond strength test. The hypothesis tested was that there would be no significant difference in bond strength among cements.

## MATERIALS AND METHODS

### Teeth collection

Thirty freshly extracted permanent human (maxillary) molars from the Tooth Bank of FORP/USP, with approval of the Ethics Committee of the institution (Protocol n° 2010.1.553.58.3 - CAAE n° 0040.0.138.000-10) and according to Helsinki Declaration principles, were selected for the shear bond strength tests. The teeth were kept in chloramine solution (0.5%) at 4°C for 48 h for disinfection process and, next, washed in running water for 24 h.

### Sample preparation

The cements tested in this study are described in Table 1. The palatine roots were sectioned perpendicular to the long axis of the tooth, next to the cemento-enamel junction, using a double-face diamond disk #7020 (KG Sorensen, Barueri, SP, Brazil) coupled to the handpiece at low-speed rotation (MRS 400, Dabi Atlante, Ribeirão

Preto, SP, Brazil), producing root cylinders of 7 mm. Next, the root cylinders were sectioned longitudinally with a low-speed diamond disk under water coolant (150 Low Speed Diamond Saw, MTI Corporation Marcal, San Francisco, CA, USA) in order to obtain two root canal hemi-sections. The hemi-sections were centered into a polyethylene matrix (15 mm in diameter) and embedded in self-polymerized acrylic resin (Jet Classic, São Paulo, SP, Brazil), with the dentine root canal surface upwards. After polymerization of the acrylic resin, the dentine surfaces were flattened in polishing machine (Struers, Ballerup, Denmark) with water abrasive sandpaper (Norton, Sao Paulo, SP, Brazil) in decreasing order of granulations (100, 320, 600, 800 and 1000) to obtain a flat dentine surface and to produce a thin and delicate smear layer. The samples were separated randomly into three groups ( $n = 20$ ) according to the cements applied on the dentine surfaces as follows: EB, WMTA and GMTA. Then, the dentine surfaces were treated with 1 mL of 1% NaOCl solution (Biodinâmica, Ibioporã, PR, Brazil) and 17% EDTA (Biodinâmica), which was applied for 1 min for smear layer removal. Stainless steel matrices with an orifice of 4.5 mm<sup>2</sup> were placed on the dentine surface and were used as molds. After manipulation according to manufacturers' recommendations, with the proportion of 1 g of powder to 0.21 mL of distilled water for EB, and one dose of powder to 1 drop of distilled water for both MTA,<sup>[8]</sup> the cements were placed into the matrix [Figure 1].

### Shear bond strength test

After a period corresponding to three times the setting time of each cement, according to the manufacturers' recommendation, the matrices were removed and the test specimens were coupled vertically in a Universal Testing Machine (EMIC-DL, São José dos Pinhais, PR, Brazil) to perform the shear bond strength test. The test specimens were attached to a stainless steel support to align as close as possible the shear-loading device with the adhesive interface. Each test specimen was loaded with a cross-head speed of 0.5 mm/min to produce a shearing force that promoted displacement of the root hemi-section along the cements' interface [Figure 2]. The force, in Newtons (N), required to displace the cement was recorded and then the shear bond strength (Megapascal - MPa) was calculated by dividing the shear force (N) by the adhesion area (4.5 mm<sup>2</sup>).

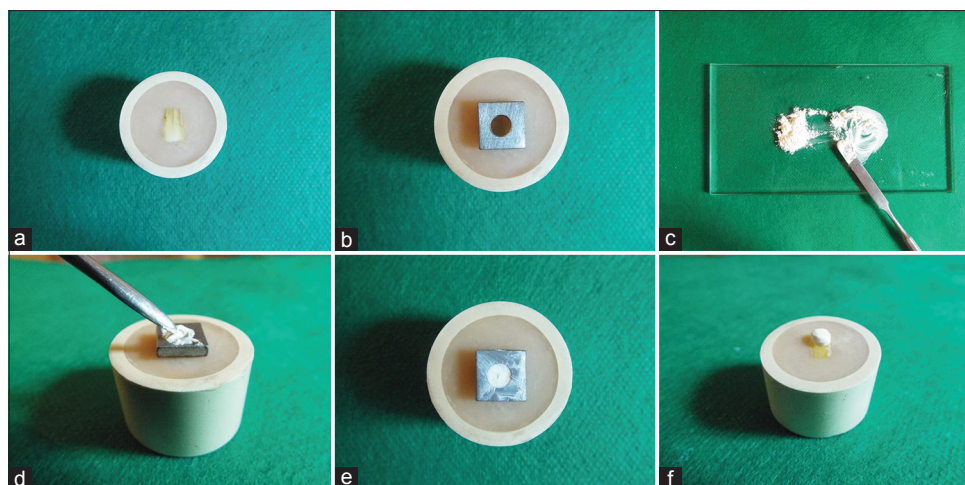
### Statistical analysis

The normal distribution of data was tested by the Shapiro-Wilks test and the values of shear bond

**Table 1: Cements used in the study**

Cements	Composition (% weight)	Manufacturer
EndoBinder	Al <sub>2</sub> O <sub>3</sub> (≥68.0), CaO (≤31.0), SiO <sub>2</sub> (0.3-0.8), MgO (0.4-0.5), Fe <sub>2</sub> O <sub>3</sub> (<0.3)+Bi <sub>2</sub> O <sub>3</sub> (20.0)	Binderware, São Carlos, SP, Brazil
White MTA	SiO <sub>2</sub> (20.0), CaO (68.0), Al <sub>2</sub> O <sub>3</sub> (4.7), MgO (0.48), Fe <sub>2</sub> O <sub>3</sub> (1.82), CaSO <sub>4</sub> (5.0)+Bi <sub>2</sub> O <sub>3</sub> (20.0)	Ângelus, Londrina, PR, Brazil
Grey MTA	SiO <sub>2</sub> (20.0), CaO (63.82), Al <sub>2</sub> O <sub>3</sub> (4.7), MgO (0.48), Fe <sub>2</sub> O <sub>3</sub> (6.0), CaSO <sub>4</sub> (5.0)+Bi <sub>2</sub> O <sub>3</sub> (20.0)	

MTA: Mineral trioxide aggregate



**Figure 1:** (a) Root hemi-section embedded in self-polymerized acrylic resin. (b) Stainless steel matrix (4.5 mm<sup>2</sup>) placed on dentine surface to be used as mold. (c) Cement manipulation. (d) Cement being placed into the matrix. (e) Cement inside the matrix. (f) After a period corresponding to three times the setting time of each cement, the stainless steel matrix was removed

strength (1-way ANOVA and Tukey's tests,  $P < 0.05$ ) were statistically analyzed with the aid of Graphpad Prism 4.0 Software (GraphPad Software, La Jolla, CA, USA).

## RESULTS

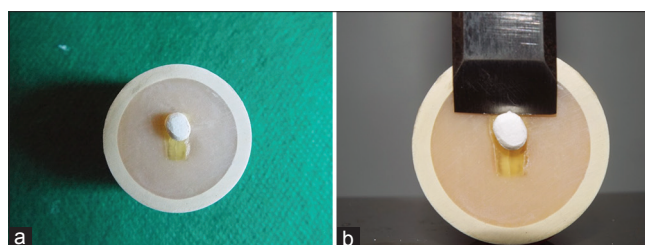
The mean values obtained in the shear bond strength test and their comparisons are presented in Figure 3.

EB presented the highest shear bond strength mean values however with statistically significant difference only for WMTA ( $P < 0.05$ ). GMTA and WMTA did not present statistically significant difference between them ( $P > 0.05$ ).

## DISCUSSION

The present study evaluated the shear bond strength of a novel calcium aluminate-based cement, EB, to root dentine in comparison with GMTA and WMTA. Based on the results obtained, it can be stated that the tested hypothesis was partially accepted as EB presented higher bond strength values only for WMTA.

Several negative features of MTA, such as adhesiveness, compromise retrograde filling after apicectomy and root perforations' treatment, which justify the development of new materials. The adhesion to dentine is a fundamental condition for a cement does not displace when submitted to load.<sup>[21]</sup> However, the American Dental Association<sup>[22]</sup> does not determine which is the minimum bond strength required for such cements can be considered effective. Furthermore, tests that evaluate the bond strength



**Figure 2:** (a) Sample after cement setting. (b) Sample coupled in the Universal Testing Machine to perform the shear bond strength test



**Figure 3:** Graphic representation of the mean values (MPa) and standard deviations of the shear bond strength test ( $n = 20$ ). Different lowercase letters over columns indicate statistically significant difference. Tukey's test categories with the same letter are not statistically different from each other ( $P < 0.05$ ) 1-way ANOVA and Tukey's tests,  $P < 0.05$   $P = 0.0803$

of filling materials are not standardized due to the difficulty of testing materials, which, despite being used for the same purpose, have different natures and thus different elastic modulus, leading to controversial results.<sup>[23,24]</sup>

Among several tests used to evaluate the bond strength of filling materials, the push-out and micropush-out tests, performed directly in the center of the filled root canal reproducing the clinical condition in which the material is used,<sup>[24]</sup> and the shear bond strength test where the displacement force is applied parallel to the interface material/dentine, are widely used.<sup>[25]</sup>

Despite reproducing the clinical condition accurately, the push-out test frequently does not provide reliable results, as dentine morphology is not uniform along the root canal, presenting many differences mainly due to the biomechanical preparation that is performed prior to placing the filling material.<sup>[26]</sup> Thus, one of the greatest advantages of shear bond strength tests is the capacity of samples' standardization, in addition to allowing not only evaluation of the bond between cement and dentine but also between cement and other surfaces, such as gutta-percha.<sup>[27]</sup>

One of the main negative features of MTA is its low-flow capacity, which makes its application into deep cavities, retrograde filling after apicectomy and root canal system filling (in selected cases), especially in the middle and apical thirds, difficult.<sup>[5]</sup> Such fact restricts the use of the push-out tests, as the accommodation and penetration of cement inside the dentinal tubules to promote its mechanical retention is compromised.<sup>[24]</sup> For this reason, shear bond strength tests are more appropriate for this type of material.

Among several biological properties of MTA, the most important is its reparative capacity, which stimulates the pulp to form mineralized tissue and dentine bridges.<sup>[2,3]</sup> However, studies demonstrate that such a property is unable to promote an effective bond between dentine and cement, the same being observed for EB.

Sarkar *et al.*<sup>[28]</sup> suggested a chemical bond between dentine and MTA as the result of a physico-chemical interaction between the cement and the substrate. After application of MTA on the dentine, hydroxyapatite crystals grow around the particles of the cement, filling the microscopic space between the material and the substrate.<sup>[28]</sup> The hydroxyapatite formed is deposited within the collagen fibrils, promoting controlled mineral nucleation on the dentine surface and forming structures similar to resin tags however with shorter length and without adhesion capacity, which does not guarantee an adequate bond strength.<sup>[29]</sup> The same could be said with regard to EB, as the material is

also capable of releasing calcium and hydroxyl ions, leading to the formation of hydroxyapatite crystals in contact with the dentine.<sup>[20]</sup> Despite the higher mean values obtained for EB compared with WMTA, this phenomenon was not able to promote an effective bond strength.

Another factor that should be taken into consideration is the pre-treatment of dentine surface with chelating and irrigating solutions. The smear layer removal procedure before root canal system filling can increase the capacity of cements to penetrate into the dentinal tubules, thereby increasing their bond strength.<sup>[30]</sup> According to Mönika and Fröner,<sup>[31]</sup> the most effective procedure to remove the smear layer is the application of 1% NaOCl solution associated with 17% EDTA. In the present study, pre-treatment of the dentine was performed to simulate root canal treatment. Such fact is a major concern about the results of this study, as the bond strength values of the tested cements were smaller than the smear layer cohesive strength, which is about 5 MPa.<sup>[32]</sup> However, Yan *et al.*<sup>[32]</sup> reported that the use of NaOCl and chlorhexidine solutions prior to root canal filling, which are widely used in biomechanical preparation, was not able to significantly increase the bond strength between MTA and dentine, which leads us to conclude that its low-flow capacity is the main reason for such results.

Despite EB and GMTA not having any difference regarding bond strength, it was observed that WMTA obtained significantly lower values than EB. Several studies report conflicting results regarding the same properties for GMTA and WMTA, although the main difference between them is the iron oxide concentration.<sup>[10,11]</sup> The poor handling characteristics of MTA is considered one of the main factor responsible for the controversial results, as such difficulty leads to incorporation of air bubbles and induces the formation of pores in the cement after manipulation, making its microstructure unstable.<sup>[10,11]</sup> Such fact could explain the conflicting behavior of WMTA and GMTA in the present study regarding bond strength.

According to Garcia *et al.*,<sup>[18]</sup> EB has a more homogeneous microstructural arrangement than MTA, with globular particles similar in size and shape allowing better flow and stress distribution by the cement, which may have been decisive for the highest bond strength observed in this study. Furthermore, the significant lack of regularity in particle size and shape between GMTA and WMTA<sup>[18]</sup> may justify

the different rheological properties of the cements, compromising penetration of the cements into the root dentine surface and, consequently, the bond strength.

Hydration of MTA powder during handling results in a colloidal gel that solidifies into a rigid structure after the end of its setting time.<sup>[33]</sup> However, the microstructure characteristics can be influenced not only by the material's handling but also by its powder/liquid ratio,<sup>[33]</sup> humidity, temperature and pH of the environment<sup>[34]</sup> and time elapsed between manipulation and application.<sup>[35]</sup> According to Fridland and Rosado,<sup>[11]</sup> such factors are not easily controlled and, therefore, different results can be obtained in studies concerning the physico-chemical properties of MTA.

Despite the limitations of this study, it was concluded that EB presented highest bond strength values than WMTA; however, these values were similar to the gray version of the cement. This novel calcium aluminate-based cement should be considered as an alternative to MTA in endodontic therapy. However, further studies related to other physico-chemical and biological properties of the cement must be performed prior to its application in humans.

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