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## Push-out bond strength of MTA HP, a new high-plasticity calcium silicate-based cement

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**Abstract:** This study was designed to investigate the resistance to dislodgment provided by MTA HP, a new high-plasticity calcium silicate-based cement. Biodentine and White MTA Angelus were used as reference materials for comparison. Three discs  $1 \pm 0.1$  mm thick were obtained from the middle third of the roots of 5 maxillary canines. Three 0.8-mm-wide holes were drilled on the axial surface of each root disc. Standardized irrigation was performed. Then the holes were dried with paper points and filled with one of the three tested cements. The filled dental slices were immersed in a phosphate-buffered saline (PBS) solution (pH 7.2) for 7 days before the push-out assessment. The Kruskal-Wallis test was applied to assess the effect of each endodontic cement on the push-out bond strength. Mann-Whitney with Bonferroni correction was used to isolate the differences. The alpha-type error was set at 0.05. All specimens had measurable push-out values and no premature failure occurred. There were significant differences among the materials ( $p < 0.05$ ). The Biodentine specimens had the highest push-out bond strength values ( $p < 0.05$ ). MTA HP had significantly higher bond strength than White MTA ( $p < 0.05$ ). MTA HP showed better push-out bond strength than its predecessor, White MTA; however, Biodentine had higher dislodgment resistance than both MTA formulations.

**Keywords:** Endodontics; Dental Materials; Dental Cements.

**Declaration of Interests:** The authors certify that they have no commercial or associative interest that represents a conflict of interest in connection with the manuscript.

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## Introduction

Mineral trioxide aggregate (MTA) is considerable the gold standard material for several clinical procedures, as confirmed by a robust body of evidence supporting this material<sup>1,2,3,4,5,6</sup>. Nevertheless, traditional MTA compositions have some drawbacks, such as a long setting time, discoloration of the tooth and marginal gingiva, and difficult handling<sup>7,8</sup>. The difficult handling of MTA frequently reported by clinicians seems to be aggravated in such procedures as the filling of root-end cavities and furcation or root perforation<sup>1,5,9</sup>.

MTA HP (Angelus, Londrina, Brazil), a more recent silicate-based cement material, was developed based on the biological and physical properties of calcium-silicate cements, claiming improved performance compared with traditional MTA. MTA HP powder is composed mainly of tricalcium silicate, dicalcium silicate, tricalcium aluminate, calcium oxide, calcium carbonate (filler material) and calcium tungstate (radiopacifier), whereas the liquid supplied for

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mixing with the cement powder consists of water and a plasticizing agent<sup>10</sup>. According to the manufacturer, this new material has high-plasticity and improved physical properties, as compared with White MTA<sup>10</sup>.

The creation of a good seal is one of the major requirements of both root-end cavities and root perforation materials, and the bond with radicular dentin is directly dependent on the type of material used. Up to now, no study evaluated MTA HP push-out bond strength to dentine. Therefore, the present study was designed to investigate the resistance to dislodgment provided by MTA HP. Biodentine (Septodont, St. Maur-des-Fossés, France) and White MTA (Angelus, Londrina, Brazil) were used as reference materials for the comparison. The null hypothesis tested was that there is no difference in the resistance to dislodgement among the materials tested.

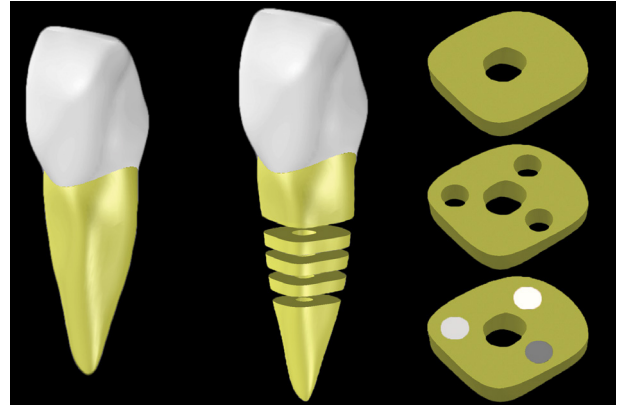
## Methodology

### Sample selection and preparation

This study was approved by the Local Ethics Committee. Five freshly extracted maxillary canines were selected. The teeth were cleaned by removing the hard deposits using curettes, and the soft tissues, by soaking in 5.25% NaOCl for 10 minutes. Sample preparation was performed by removing the coronal and apical segments of each tooth to leave the middle third measuring 10 mm in length. Three horizontal cross-sections ( $1 \pm 0.1$  mm thick) were created from this segment, using a low-speed saw (ISOMET, Buehler Ltd., Lake Buff, USA) with a diamond disc ( $\varnothing 125$  mm x 0.35 mm x 12.7 mm; Buehler Ltd. Lake Bluff, USA), under continuous water irrigation. The final thickness of each slice was measured with a digital caliper having an accuracy of 0.001 mm (Avenger Products, North Plains, USA). Fifteen root slices were produced following this protocol (Figure 1).

### Preparation of canal-like cavities for the push-out assay

A 0.8 mm cylindrical carbide bur was used to drill three canal-like cavities parallel to the root canal in each root slice. The cavities were drilled perpendicular to the surface under constant water irrigation, using a vertical drill stand (Dremel Workstation 220, Mount



**Figure 1.** Schematic representation of the root slice acquisition and filling.

Prospect, USA). A minimum distance of 1 mm between the cavities, the external cementum and the root canal wall was maintained<sup>11</sup>.

Afterwards, all the root slices were immersed in a 2.5% sodium hypochlorite (NaOCl) solution for 15 min and then immersed in doubledistilled water to neutralize the NaOCl solution. The smear layer was removed using 17% EDTA for 3 min, doubledistilled water for 1 min, 2.5% NaOCl for 1 min and a final flush with doubledistilled water for 1 min. The cavities were then dried with absorbent paper, and the three cavities of each root slice were randomly filled with one of the selected materials: Biodentine, MTA HP or White MTA. All the materials were mixed according to the manufacturers' instructions, and were delivered into the cavities. Bubble formation was avoided by gentle vibration while placing the material. Lastly, the filled root slices were incubated in contact with gauze moistened in phosphate buffered saline solution (PBS) (pH 7.2) at 37°C for 7 days, before the push-out assessment (Figure 1). This experimental setup allowed the three materials to be distributed in the same slice, reducing the impact of different dentine sources on the study design (Figure 1).

### Push-out assessment

A plunger tip (0.6 mm diameter) was positioned only over the test material. The load was always applied in a coronal-apical direction. Loading was performed on a universal testing machine (EMIC DL200MF, São José dos Pinhais, Brazil) at a speed of 0.5 mm min<sup>-1</sup> until material dislocation occurred. A load x time curve was plotted

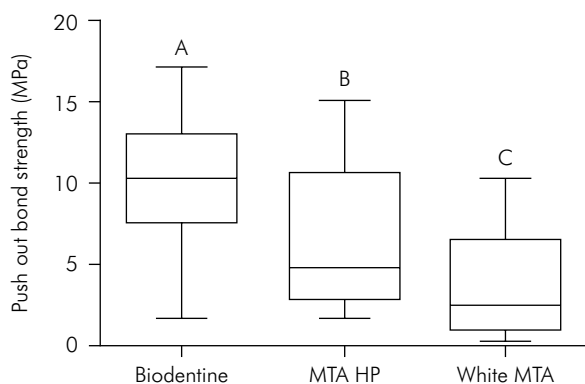
during the test, using a real-time software program. The load at failure (recorded in Newtons) was divided by the area of the bonded interface to express the bond strength in MPa<sup>12</sup>. The adhesion area of the root canal material was calculated using the following formula:  $\text{area} = 2\pi r \times h$ , where  $p =$  the constant 3.14,  $r =$  radius of the cavity with the root canal material (0.4 mm), and  $h =$  height of the material (1.0 mm)<sup>13</sup>.

### Data presentation and analysis

The main outcome variable in the present study was the push-out bond strength (MPa). Preliminary analysis of the raw pooled data was unable to reveal a Gaussian distribution (D'Agostino & Person omnibus normality test); therefore, a Kruskal-Wallis test was applied to assess the effect of each endodontic cement on the push-out bond strength. Mann-Whitney with Bonferroni correction was used to isolate the differences. The alpha-type error was set at 0.05. SPSS 11.0 (SPSS Inc., Chicago, USA) was used as the analytical tool.

### Results

All the specimens had measurable push-out values, and no premature failure occurred. There were significant differences among the materials ( $p < 0.05$ ). Biodentine specimens had the highest push-out bond strength values ( $p < 0.05$ ). MTA HP had significantly higher bond strength than White MTA ( $p < 0.05$ ). Box plots illustrating the variance of the push-out bond strength data in each experimental group are shown in Figure 2.



Different superscript letters indicate significant push-out differences among the materials ( $p < 0.05$ ).

**Figure 2.** Box-plot with the median, the range and the minimum and maximum push-out values of the tested materials.

### Discussion

The material used to seal the external surface of the teeth should be able to prevent leakage and remain in place under dislodging forces, such as functional pressures or the application of other restorative materials<sup>9</sup>. To the best of our knowledge, this study is the first to evaluate the push-out bond strength of MTA HP, a modified tri-calcium silicate-based material, in comparison with Biodentine and White MTA. The present results pointed out a significantly different performance among the tested materials. Biodentine produced higher bond strength values to root dentin than both MTA HP and White MTA ( $p < 0.05$ ). Therefore, the null hypothesis was clearly rejected. In fact, Biodentine outperformance cannot be regarded as a novelty, since it has been well documented in endodontic literature<sup>14,15,16</sup>. Biodentine biomineralization ability can most likely be attributed to the formation of tags, and may be the cause of its superior dislodgement resistance herein demonstrated. Han and Okiji<sup>17</sup> showed that calcium and silicon ion uptake into dentin, leading to the formation of tag-like structures in Biodentine, was higher than in MTA, and may have a role in the overall adaptation to root dentin. Moreover, the different particle sizes of MTA and Biodentine may affect their penetration into the dentinal tubules, with consequences to displacement resistance. Atmeh et al.<sup>18</sup> demonstrated that Ca-Si cements facilitate the permeation of Ca and OH ions into the dentine, due to their caustic effect. It can be assumed that the improved ability to release remineralizing Ca and OH ions by Biodentine<sup>19</sup> is responsible for the improved formation of apatite at the interface<sup>19,20</sup>, and for micromechanical anchorage<sup>21</sup>.

MTA HP showed improved push-out bond strength values compared with those of White MTA ( $p < 0.05$ ). The substitution of bismuth oxide for calcium tungstate as a radiopacifier agent in the MTA HP could explain the better results of this cement in comparison with White MTA. Calcium tungstate contributes to higher calcium release, promoting higher biomineralization<sup>22</sup>. Moreover, the high-plasticity of MTA HP may positively affect the marginal adaptation of the cement to the root walls, and this can be associated with higher bond strength.

However, unlike previous studies, the current study has a methodological aspect that deserves attention. Artificial, standard canal-like holes were machined in dentin slices to assure a more reliable baseline. Moreover, cements and time points were further compared using the same dentine sample, in which routine confounding factors such as tooth age, scleroses, hardness, canal shape and others factors had a slight and better-controlled effect due to baseline balance<sup>11</sup>. Furthermore, a final standardized cylindrical shape with a 0.8 mm diameter for all cavities was used to allow standardization of the internal root canal anatomy among the experimental groups, inasmuch as this anatomy is a wellknown critical confounding factor and biological bias. The cavities were prepared with a 0.8-mm drill bit, producing cavities with parallel sides (versus tapered sides). This ensures that the force placed on the material/dentine interface is purely shear force. This procedure is also a requisite for the equation used for calculating bond strength to be valid, since it is based on the assumption of constant diameter along the entire height of the cavity. A pillar drill was used to ensure that the axis of the drilled cavity was exactly perpendicular to the bottom surface of the tooth.

The moistening of calcium silicate cements during setting is particularly important, since these cements have greater comprehensive strength when kept in a moist environment<sup>23</sup>. In addition, the retention characteristic and push-out strength of calcium silicate cements increases over time if kept under moist conditions<sup>24</sup>. In the present study, all the samples were kept in a PBS-moist environment for 7 days, before

the push-out assessment. Considering the clinical conditions, these materials are likely to come in contact with blood. The previous literature has evaluated the effect of blood contamination on the push-out bond strength values of endodontic materials<sup>25,26,27</sup>. The results of these studies are conflicting, showing that blood could improve, decrease or make no difference in the push-out values. However, the use of blood during the storage period is not usual in endodontic studies. For the purpose of standardizing the study conditions and in agreement with the endodontic literature, it was decided that the PBS would be kept in contact with the samples during the storage period.

Calcium-silicate based materials are known to interact with dentine to promote intratubular Ca and Si incorporation<sup>17</sup>, as well as dentine remineralization<sup>28</sup>, intrafibrillar apatite deposition<sup>29</sup> and the formation of tag-like structures<sup>17,24</sup> in the presence of PBS. The nucleation of apatite at the interface increases the sealing ability by reducing the interface voids<sup>20</sup> and improving the dislocation resistance<sup>21</sup>. The use of PBS promotes a condition closer to a clinical condition.

## Conclusions

MTA HP showed better push-out bond strength than its predecessor, White MTA; however, Biodentine showed better results than both MTA formulations.

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