



Push-out Bond Strength of Injectable Pozzolan-based Root Canal Sealer

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Abstract

Introduction: The present study aimed to rank the bond strength to root dentin of a new injectable pozzolan-based root canal sealer, EndoSeal MTA, as compared with MTA Fillapex and AH Plus. **Methods:** Eighteen dentinal slices (1 ± 0.1 mm) were obtained from the middle third of 6 maxillary incisors previously selected. Three canal-like holes with 0.8 mm diameter were drilled perpendicularly on the axial surface of each slice. Then, a standardized irrigation was applied for all holes that were subsequently filled with 1 of 3 test root canal sealers. After that, slices were stored in contact with phosphate-buffered saline solution (pH 7.2) for 7 days at 37°C before the push-out assay. Friedman test and Wilcoxon signed rank test with a Bonferroni correction were used to rank the results. Significance boundary was set at $\alpha = 5\%$. **Results:** Friedman test confirmed a significant dissimilarity in push-out ranks among the tested cements ($P < .01$). Wilcoxon signed rank test demonstrated AH Plus had significant superior resistance to dislodgment compared with Endo Seal ($P < .01$) or MTA Fillapex ($P < .01$), whereas MTA Fillapex presented the lowest push-out values as compared with Endo Seal ($P < .01$) or AH Plus ($P < .01$). **Conclusions:** EndoSeal presents satisfactory bond strength performance for application in endodontic therapy compared with MTA Fillapex, and although it displays a new alternative of injectable bio-tight root canal sealer, it is not able to improve adhesion compared with AH Plus. (*J Endod* 2016;42:1656–1659)

Key Words

MTA, pozzolan, push-out, root canal obturation, root canal sealer

Mineral trioxide aggregate (MTA) is a Portland cement–derived hydraulic material that has been widely used in a variety of applications in endodontics as supported by a broad body of evidence (1, 2). MTA has excellent physical and biological properties such as biocompatibility, bioactivity, and sealing ability

(1, 3). This sealing capacity is largely attributed to MTA's bioactivity and ability to release calcium ions and produce an apatite layer in the presence of phosphate-containing physiological fluids (4–7). The crystalline deposits produced by the interaction of MTA and physiological fluids positively influence the push-out bond strength of MTA. Conversely, MTA cement fails to present the physical properties required for a root canal sealer, which leads to increasing efforts to create an ideal MTA-based sealer with remarkable balance between its biological and physicochemical characteristics (1, 3, 8).

EndoSeal MTA (Maruchi, Wonju, Korea), a pozzolan-based MTA sealer, was recently introduced. It consists of a premixed and pre-loaded material confined into an air-tight syringe that permits its direct application into the root canals. During the injection, EndoSeal absorbs the environmental moisture from atmospheric air and sets without the need of previous powder/liquid or base/catalyst mixing (9, 10). This sealer contains pozzolan cement, which gets cementitious properties after the pozzolanic reaction with calcium hydroxide and water, allowing efficient flow of the pre-mixed substrate with adequate working consistency and reduced setting time (4, 11). The incorporation of small particle pozzolan cement, which is a mineral aggregate with watery calcium silicate hydration, resulted in fast-setting MTA without the addition of a chemical accelerator (4, 12).

Previous reports have demonstrated the capacity of EndoSeal MTA to induce dentinal tubule biomineralization (11), satisfactory biological and physical properties (10), favorable cytocompatibility (13), and superior sealer distribution (9). To date, however, no study has ranked the push-out bond strength of EndoSeal. Therefore, the present study aimed to investigate the bond strength to root dentin of this new injectable pozzolan-based root canal sealer by using MTA Fillapex (Angelus, Londrina, Brazil) and

Significance

EndoSeal MTA was previously demonstrated to induce dentinal tubule biomineralization, favorable cytocompatibility, and superior sealer distribution, as well as satisfactory biological and physical properties. The present finding adds to EndoSeal satisfactory bond strength performance for application in endodontic therapy and increases the overall knowledge about a material that has potential to become a clinical alternative of injectable bio-tight root canal sealer.

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AH Plus (Dentsply, DeTrey, Konstanz, Germany) as control materials for comparison. The following null hypotheses were tested:

1. There is no difference in dentin bond strength between the tested root canal sealers.
2. There is no difference in bond strength to root dentin between both MTA-derived materials (EndoSeal and MTA Fillapex).

Materials and Methods

Sample Size Calculation

According to a previous study (14), an effect size of 0.74 was added to power $\beta = 95\%$ and $\alpha = 5\%$ inputs into F test family for repeated-measures analysis of variance (G*power 3.1 for Macintosh). A total size of 9 slices samples was necessary to identify differences among the tested materials.

Sample Preparation

Local Ethics Committee approved the study. Six maxillary incisors were selected and cleaned by removing the hard deposits and the soft tissues with the aid of curettes and 5.25% NaOCl immersion for 10 minutes. After that, coronal and apical segments were removed from each tooth to obtain the middle third. Three horizontal cross sections (1 ± 0.1 mm thick) were obtained from this segment by using a low-speed saw (ISOMET; Buehler Ltd, Lake Buff, IL) with a diamond disk ($\text{Ø } 125 \times 0.35 \times 12.7$ mm; Buehler Ltd) under continuous water irrigation. The final thickness of each slice was checked with the aid of a digital caliper with accuracy of 0.001 mm (Avenger Products, North Plains, OR). Eighteen root slices were produced following this protocol (Fig. 1).

Preparation of Canal-like Holes for the Push-out Assay

Samples were drilled by using a 0.8-mm cylindrical carbide bur. Three canal-like holes were made parallel to the root canal in each root slice. The perforations were performed under constant water irrigation by using a vertical drill stand (Dremel Workstation 220, Mount Prospect, IL) to standardize the holes drilled perpendicular to the surface. A minimum distance of 1 mm was established between the holes drilled, the external cementum, and the root canal walls.

Thereafter, all samples were immersed in 2.5% sodium hypochlorite (NaOCl) solution for 15 minutes and further immersed for 1 minute in bidistilled water to neutralize the NaOCl solution. The smear layer was removed by using 17% EDTA for 3 minutes, bidistilled

water for 1 minute, 2.5% NaOCl for 1 minute, and a final flush with bi-distilled water for 1 minute. The holes were dried with absorbent paper points, and each of the 3 holes of every root slice was filled with 1 of the selected materials: EndoSeal, MTA Fillapex, or AH Plus. All the materials were mixed according to the manufacturers' instructions and were delivered into the holes. Bubble formation was avoided by gentle vibration while placing the material. Finally, the filled root slices were stored in contact with phosphate-buffered saline solution (pH 7.2) at 37°C for 7 days before the push-out assessment (Fig. 1).

Push-out Assessment

A plunger tip of 0.6-mm diameter was set up over the tested material, avoiding touching the surrounding dentin wall. Loading was performed on a universal testing machine (EMIC DL200 MF, São José dos Pinhais, PR, Brazil) at a head-speed of 0.5 mm/min^{-1} until the displacement of the material. The load was applied only in a coronal-apical direction. A load \times time curve was plotted during the test by using a real-time software program. The maximum load at failure, recorded in newtons, was divided by the area of the bonded interface, resulting in a bond strength expression in MPa^2 . The adhesion area of the root canal material was calculated by using the following formula: $\text{area} = 2\pi r \times h$, where π = 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)³.

Data Presentation and Analysis

After testing for data skewing (Shapiro-Wilk test, $P < .05$), the push-out from paired artificial holes was ranked by using a non-parametric Friedman procedure. Multiple comparisons were performed with the aid of a Wilcoxon signed rank test with Bonferroni correction. Significance boundary was set at $\alpha = 5\%$ (SPSS 17.0; SPSS Inc, Chicago, IL).

Results

Friedman test confirmed a significant dissimilarity in push-out ranks among the tested cements ($P < .01$). Wilcoxon signed rank test demonstrated AH Plus had significant superior resistance to dislodgment compared with Endo Seal ($P < .01$) or MTA Fillapex ($P < .01$), whereas MTA Fillapex presented the lowest push-out values as compared with Endo Seal ($P < .01$) or AH Plus ($P < .01$). Figure 2 displays a graphic representation of the findings.

Discussion

The use of endodontic sealers during root canal obturation yields the sealing between gutta-percha and dentinal walls, acting against bacterial leakage that may lead to endodontic failures (15, 16). Every currently available root canal sealer presents limitations regarding the ideal properties of an endodontic sealer (17). Therefore, new sealers are constantly being developed, especially calcium silicate-based or bioceramics materials—MTA and BioAggregates—because of their biological and sealing properties (3, 8, 18). A relevant physical aspect of a newly developed injectable pozzolan-based sealer (EndoSeal MTA), the dentinal bond strength, was ranked in comparison with MTA Fillapex and AH Plus.

The first null hypothesis was not accepted because a significant difference in push-out was observed among the materials. Both MTA-based sealers, EndoSeal and MTA Fillapex, produced inferior dentinal bond strength values compared with AH Plus. The present result is in accordance with several previous studies in which AH Plus was also associated with significantly higher bond strength values when compared with other sealers (19–23). The superior bond strength property of AH Plus may be largely attributed to the capacity of

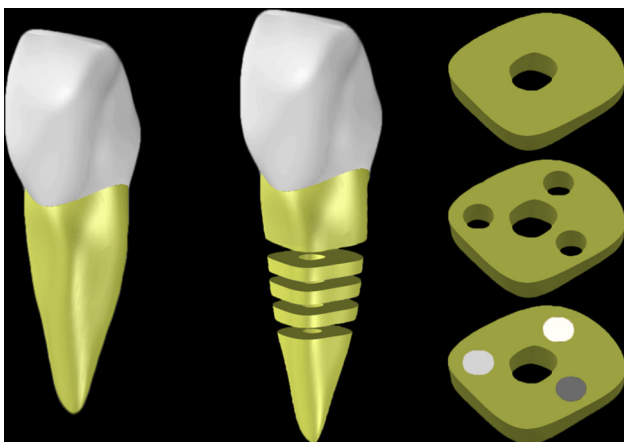


Figure 1. Schematic representation of sample preparation.

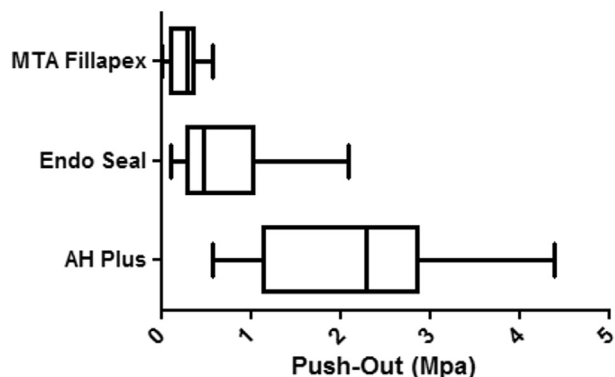


Figure 2. Horizontal box plot of the central tendency push-out values displayed by the 3 cements. Significant difference among the 3 tested sealers was detected by Friedman and Wilcoxon signed rank tests ($P < .05$).

producing a covalent bond by mixing diepoxide compounds and polyamine paste during its manipulation so that each amine group in the collagen network reacts with an open epoxide ring, resulting in a heavily cross-linked polymer that is rigid and strong (24, 25). In addition, low polymerization stress, long-term dimensional stability, and efficient cohesion between molecules had already been reported to increase root dentin micro retention of AH Plus (19, 20, 24).

EndoSeal displayed significantly superior bond strength performance than MTA Fillapex. Hence, the second null hypothesis was also rejected. The chemical composition of a root canal sealer has substantial impact over adhesion capacity. The base material of EndoSeal is calcium silicate, which presents a similar chemical composition to that of MTA cement. Hence, it is expected that EndoSeal would display comparable biological and physical properties to this cement. Indeed, recent studies confirmed that EndoSeal produces consistent dentinal tubule biomineralization beyond the sealer tag (11) and presents biocompatibility, solubility, radiopacity, and high alkalinity in similar levels as commercially available MTA (10, 13). In this context, it seems that EndoSeal contains a significant percentage of MTA that yields bioactivity and may explain its satisfactory bond strength results. In addition, according to the manufacturer, EndoSeal presents outstanding flowability, which may be related to the sealer penetration into dentinal tubules, anatomic irregularities, or accessory canals and thus results in increase in sealing ability and bond strength. EndoSeal also has excellent dentinal wall distribution (9) that may result from its injection-type, self-setting use that has a clinical advantage in terms of application. It is important to point out that it was not possible to compare the values of dentin bond strength of EndoSeal with previous results because this is a pioneer evaluation of this subject.

MTA Fillapex produced the worst results in the present study. This is in agreement with previous research that also observed poor bond strength values for MTA Fillapex (16, 22, 23). This is a paste-to-paste sealer that, whenever mixed, establishes 2 important chemical reactions that are responsible for the material's setting and physical-mechanical characteristics, the progressive hydration of the orthosilicate ions and the reaction between MTA and salicylate resin (26). A 1:1 ratio of MTA:salicylate resin would be required to achieve an appropriate setting. This allows the formation of an ionic polymer containing calcium silicate particles that reacts with water (1, 8, 27). The final reaction leads to the formation of calcium hydroxide and a nanoporous amorphous calcium silicate hydrate gel that polymerizes and creates a solid network (1, 27). However, it has been speculated that MTA Fillapex displays a higher ratio of salicylate resin than MTA, which affects the

chemical reaction among these components and explains the extended setting time of this material (8). During setting time, endodontic sealers with salicylate resin experience an initial volumetric shrinkage that increases the contraction factor (28). Hence, higher amounts of salicylate resin associated with extended setting time increase dimensional changes and formation of gaps between root canals and filling materials. This affects the bond strength of a root canal sealer both directly and indirectly because it may interfere with other properties such as flowability and solubility that are also associated with dentinal wall sealing. Therefore, the extended setting time consequent of an unbalanced resin/MTA ratio may explain the unenthusiastic MTA Fillapex results regarding push-out bond strength.

Calcium silicate-based sealer bioactivity represents the spontaneous production of an apatite layer, dentin intratubular calcium, and silicon ions incorporation (29), as well as an intrafibrillar apatite deposition (5) and production of tag-like structures at sealer-dentin interface (5, 6, 29) that lead ultimately to dentin remineralization (30) when in contact with phosphate-containing physiological fluids (27, 31). Apatite nucleation resulting from bioactivity processes improves the displacement resistance of filling materials by producing a micromechanical bonding system to dentin that decreases gaps at the interface (7). The ability to nucleate calcium phosphate is strictly correlated to environmental conditions, the material's chemical composition, and calcium-releasing properties (32). Although MTA Fillapex is an MTA-based sealer, the particle size and composition of several types of MTA differ. MTA Fillapex contains higher amounts of resins and less than 20% MTA particles, which may be an insufficient proportion to demonstrate the full biological and sealing characteristics of this cement (25, 33). Furthermore, MTA Fillapex has bismuth oxide as the radiopacifier, which is associated with increase in deterioration of mechanical strength, porosity, and material degradation, which was mostly replaced by zirconium oxide in EndoSeal (13, 34).

The present study was set to improve the reliability of the push-out model proposed. To reduce confounding factors such as mineralization and tooth age and hardness (14, 35), the same dentin source was provided to all root canal sealers. A cylindrical final shape was established for all canal-like holes, providing the standardization of size and internal root canal anatomy that ultimately improves biological standardization of the holes at the baseline that allows a paired mathematical evaluation to be taken, which reduces the amount of samples required for a test. The root canal space was filled only with the sealer to obtain an application of the compressive load over the sealer without the presence of a resilient gutta-percha material and to provide better control of the type of failure mode that could lead to misinterpretations of the results.

On the basis of the results, EndoSeal MTA presents superior bond strength performance compared with MTA Fillapex, although it is not comparable to a traditional epoxy resin-based sealer and thus may be taken as a new alternative of injectable bio-tight root canal sealer.

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The authors deny any conflicts of interest related to this study.

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