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ORIGINAL RESEARCH

Evaluation of the Physicochemical Properties and Push-Out Bond Strength of MTA-based Root Canal Cement

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ABSTRACT

Aim: This study investigated the flowability, setting time, pH, calcium release and bond strength of a MTA-based cement (MTA Fillapex®) compared to AH Plus and Sealapex.

Materials and methods: For the flowability test, the ISO 6876:2001 specification was utilized and for the setting time test, the ASTM C266-03 specification was utilized. For the pH and calcium release measurements, 10 samples were prepared for each group and analyzed for several different periods. For the push-out test, dentin disks were distributed into three groups, according to the cement utilized and into three subgroups, according to the root third (n = 10). After obturation, the specimens underwent push-out testing. The data were compared statistically using a significance level of 5%.

Results: The flowability of all materials was found to be similar ($p > 0.05$). The setting times were different among the groups tested (MTA Fillapex < Sealapex < AH Plus) ($p < 0.05$). At days 7 and 28, the MTA Fillapex presented the higher pH values ($p < 0.05$). At 24 hours and at 14 days, the calcium release of the MTA Fillapex was similar to that of Sealapex ($p > 0.05$). AH Plus presented the lowest pH and calcium release values ($p < 0.05$). In all root thirds, the adhesion to the dentin of the MTA Fillapex and Sealapex were significantly lower than that of AH Plus ($p < 0.05$).

Conclusion: MTA Fillapex and Sealapex presented several similar properties and both were found to be different than AH Plus.

Clinical significance: This study evaluated the physicochemical and mechanical properties of new MTA-based root canal cement, in order to use this scaler in root canal fillings. MTA Fillapex showed satisfactory properties for clinical use.

Keywords: Adhesion, Calcium, MTA, pH, Push-out.

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INTRODUCTION

Mineral trioxide aggregate (MTA) is recommended as a root-end filling material, treatment of root perforation, apexification and in conservative pulpal treatment due to its osteoconductive potential and sealing capacity.[1-3] Despite these favorable characteristics, MTA alone does not present ideal properties to be used as a root filling material.[4]

In an attempt to associate the physicochemical properties of a root canal cement with the biological properties of MTA, some experimental and commercially available endodontic cements with this compound have been developed, such as Endo CPM (EGEO SRL, Buenos Aires, Argentina) and I Root SP (Innovate BioCeramix Inc, Vancouver, Canada).[5-8] MTA Fillapex® (Angelus, Londrina, PR, Brazil) is a new MTA-based cement to be used in root canal filling. According to the manufacturer, its composition is basically MTA, salicylate resin, bismuth and silica.

MTA Fillapex presents effective antimicrobial activity against *E. faecalis* before setting, but not after setting, despite its high pH level.[9] Its bond strength to radicular dentin exhibits the lowest values when compared with another MTA-based cement (I Root SP).[10] Although the MTA Fillapex manufacturer claims that it has excellent radiopacity, easy handling and long working time, their physicochemical properties, such as flow and setting time are scientifically unknown.

Endodontic cements must have an adequate flowability to penetrate into small irregularities and ramifications of the root canal system and dentinal tubules.[11,12] The adequate handling of the cement is directly related to the setting time. The knowledge of the initial and final setting time is important to quantify the available timing for the inclusion of the cement into the root canal and to establish the time when the cement is not suitable for clinical purposes.[13]

It has been suggested, that in order to stimulate mineralization, all materials utilized should present satisfactory alkaline pH and calcium release abilities.[14,15] When evaluated in suspension with the crushed cement, the MTA Fillapex presented high alkaline pH values.[9] However, when immediately evaluated after mixing, the aforementioned pH value was significantly lower than ordinary gray or white MTA.[16] Nonetheless, there are no comparative studies with other routinely used cements for root canal filling. As of yet, there are no recorded studies performed regarding MTA Fillapex time-dependent calcium release.

The push-out test provides an adequate evaluation of the bonding strength of endodontic cement into the dentin.[10] The MTA Fillapex, associated with cold lateral compaction and utilizing gutta-percha, had the lowest push-out values to the root dentin when compared to I Root SP or AH Plus.[10] To evaluate the adhesion properties of cement to the root dentin, it could be interesting to evaluate the effect of the adhesion ability of the MTA Fillapex utilized without the presence of gutta-percha.

Therefore, the aims of this study were to evaluate the physicochemical properties such as flowability, setting time, pH and calcium release, and push-out bond strength of MTA Fillapex® (a MTA-based cement) in comparison with AH Plus (an epoxy-based cement) and Sealapex (a calcium-based cement).

MATERIALS AND METHODS

The proportions used for endodontic cements were in a 1:1 ratio. The MTA Fillapex and AH Plus jet (Dentsply Caulk, Milford, DE, USA) are automix systems. The proportion used for Sealapex (SybronEndo, Glendora, USA) was a 1:1 ratio (in weight). The cements were manipulated in accordance to the manufacturers' instructions.

The AH plus is an epoxy-based cement, which is composed of two pastes systems: paste A (bisphenol-A epoxy resin, bisphenol-F epoxy resin, calcium tungstate, zirconium oxide, silica and iron oxide pigments) and paste B (dibenzyldiamine, aminoadamantane, tricyclodecane-diamine, calcium tungstate, zirconium oxide, silica and silicone oil). Sealapex is calcium-based cement which is also composed of two pastes: a catalyzer (isobutyl salicylate resin, silicon dioxide, bismuth trioxide, titanium dioxide pigment) and a base (N-ethyl toluene sulphonamide resin, silicon dioxide, zinc oxide and calcium oxide). The MTA Fillapex composition is basically MTA, salicylate resin, bismuth and silica.

Flowability and Setting Time

The flowability test was performed in accordance to ISO 6876:2001 specifications and the initial and final setting time were performed in accordance to ASTM, C266-03 specifications.[12,17-19] The results obtained were submitted to ANOVA and Tukey testing, at a 5% significance level.

pH Measurement and Calcium Release

One hundred twenty polyethylene (60 for pH testing and 60 for calcium release testing) tubes measuring 1.0 cm in length and 1.5 mm in internal diameter were filled with the cements. For the pH and calcium release evaluations, 10 samples were prepared for each group. The tubes filled with fresh mixtures were weighed to standardize the cement quantity. They were placed into polypropylene flasks (Injeplast, São Paulo, SP, Brazil) containing 10 ml of deionized water and kept at 37°C (Farmen, São Paulo, SP, Brazil). Prior to immersion of the specimens, both pH and calcium concentrations of the deionized water were verified, with the pH reading 6.9 and calcium being totally absent.

The pH and calcium release evaluations were carried out after 24 hours, 7, 14 and 28 days. The specimens were placed into new flasks with deionized water for each period analyzed. The pH measurement was performed with a pH meter (model Q400I; Quimis, Diadema, SP, Brazil), previously calibrated with solutions of known pH level (4, 7 and 14), at a constant temperature (25°C). The flasks were then placed in a shaker (251, Farmen, São Paulo, SP, Brazil) for 5 seconds before obtaining the pH measurement.

The calcium release concentrations in the deionized water (in mg/l) were measured using an atomic absorption spectrophotometer (AA6800, Schimadzu, Tokyo, Japan).[19] Lanthanum oxide was added to all samples to eliminate ionic interferences. Solutions containing calcium concentrations of 0, 1, 2, 3, 4 and 5 ppm were utilized to create a standard calibration curve. For the pH and calcium release dosage, the polyethylene tubes and deionized water were independents. The results obtained were submitted to ANOVA and Tukey testing, at a 5% significance level.

Push-out Test

The study protocol was reviewed and approved by the local Ethics' Committee (67/10). Thirty extracted human maxillary canines with similar anatomic characteristics were selected. The crowns were sectioned at the cement-enamel junction using a water-cooled diamond bur. The root length was adjusted to 13 mm, and the working length established 1 mm short of the apex.

All teeth were instrumented using ProTaper rotary instruments (Dentsply Maillefer, Ballaigues, Switzerland) until ProTaper F4. The root canals were irrigated with 5 ml of 2.5% NaOCl between each instrument. The final rinse was done with 5 ml of 17% EDTA for 1 minute, followed by 10 ml of 2.5% NaOCl as a final irrigation. All root canals were dried with paper points.

In sequence, the specimens were vertically positioned in a circular plastic matrix (16.5 in width × 15.0 mm in length) and embedded in polyester resin (Maxi Rubber. Diadema, SP, Brazil), maintaining 1 mm of root length, extending beyond the top of the polyester resin. All specimens remained intact for 24 hours to allow resin polymerization.

The roots were then sectioned perpendicular to the longitudinal axis of the root utilizing a slow-speed Diamond saw (Isomet; Buehler Ltd, Lake Bluff, IL, USA) under water-cooling. Three sections were prepared at a thickness of 2.0 mm ± 0.1, in the apical, middle and coronal thirds of each root. The coronal sections were taken 1 mm in distance after the coronal side of the root, the middle sections were taken 5 mm in distance of the coronal side, and the apical sections were taken 8 mm in distance of the coronal side of the root.

To standardize the diameter of the root canals, a metallic platform containing a cylindrical well was affixed on the base of the delineator to removable prosthesis (Bio Art Equipamentos Odontológicos, São Carlos, SP, Brazil), perpendicular to a 703 bur adapted in handpiece. The thickness of the metal platform was configured so that the bur penetrated in the cylindrical wheel for the same depth every time in all specimens.

After the standardization of the root canals, the specimens were immersed in 2.5% NaOCl, for 15 minutes and 17% EDTA for 1 minute followed by copious irrigation with distilled water. All specimens were dried in paper towel and randomly distributed into 3 experimental groups, according the endodontic cement. Each group was subdivided into three subgroups, according to the root thirds.

The specimens were filled with respective cement and maintained at 37°C and 100% humidity for 7 days, to allow for the setting of the sealer. After this period, the cement excess was removed from the specimens and ground with 1.200 grit silicone carbide papers (3M, Sumaré, SP, Brazil). The root filling in each specimen was submitted to loading using a universal testing machine (EMIC, São José dos Pinhais, PR, Brazil). The loading speed was 0.5 mm min^{-1} until dislodgement of the filling material occurred.

The values at the time of dislodgment were recorded in N and transformed into tension (MPa) using the formula: MPa = F/CL. CL was calculated using the following equation: $CL = \pi \cdot (R + r) \cdot g$, where CL = cement adhesion area; R = radius of coronal canal, in mm; r = radius of apical canal, in mm; g = height relative to the tapered inverted cone, in mm. The coronal and apical diameters of the specimens were individually obtained through measurements with a stereomicroscope at 20 × (Leica Microsystems, Wetzlar, Germany). The g value was obtained with the following the equation: $g^2 = (R - r)^2 + (2.0)^2$. The results were obtained for each cement and the third root, and they were submitted to ANOVA and Tukey testing, at a 5% significance level.

After the push-out test, each specimen was examined with a stereomicroscope at 20 × (Leica Microsystems, Wetzlar, Germany), to determine the mode of failure that occurred to the 3 cements, which were classified as: adhesive failure along the cement-dentine interface; cohesive failure within the cement, and mixed failure that consisted of partial failure along the dentinal walls and partial cohesive failure within the cement.

RESULTS

The flowability (in mm) of the MTA Fillapex, Sealapex and AH Plus were: 20.95 ± 0.83; 20.46 ± 0.22 and 22.44 ± 1.07, respectively. No significant statistical differences were observed among the groups ($p > 0.05$). All cements presented flowability in accordance with the ISO 6876:2001 specifications.

The initial and final setting times were different among all cements tested ($p < 0.05$). MTA Fillapex presented the shorter and AH Plus presented the longer setting time. Table 1 shows the means and standard deviation of the initial and final setting times of the cements.

By 24-hour and 14-day periods, the pH values means of the MTA Fillapex and Sealapex were statistically similar ($p > 0.05$). At days 7 and 28, the MTA Fillapex presented the higher pH values. AH Plus presented the lower pH values in all periods tested ($p < 0.05$). Table 2 shows the means and standard deviations of the pH values of the cements, for all periods of analysis.

By the 24-hour and 14-day periods, the calcium release (mg/l) of the MTA Fillapex and Sealapex were statistically similar ($p > 0.05$). At days 7 and 28, Sealapex presented the highest calcium release among all the groups tested. AH Plus

Table 1: Means, standard deviations and statistical comparison of initial and final setting times (in minutes) for each endodontic cement

	MTA Fillapex	*Sealapex*	*AH Plus*
Initial setting	192.0 (31.04)[a]	414.33 (0.57)[b]	1,893.33 (2.30)[c]
Final setting	445.0 (13.22)[a]	3,214.33 (16.74)[b]	4,503.33 (2.30)[c]

[a,b,c]Different letters indicate statistically significant difference on the same line ($p < 0.05$)

Table 2: Means and standard deviations of the pH values in the different experimental periods

	MTA Fillapex	*Sealapex*	*AH Plus*
24 hours	10.11 (0.20)[a]	10.44 (0.38)[a]	7.93 (0.38)[b]
7 days	9.07 (0.87)[a]	7.98 (0.21)[b]	7.22 (0.42)[c]
14 days	8.44 (0.61)[a]	8.32 (0.88)[a]	7.31 (0.24)[b]
28 days	8.52 (0.63)[a]	7.96 (0.10)[b]	7.35 (0.25)[c]

[a,b,c]Different letters in each period indicate statistically significant differences ($p < 0.05$)

presented the lowest calcium release means than any of the other cements for all periods tested ($p < 0.05$). Table 3 shows the means and standard deviations of calcium release of the cements, for the different periods of analysis.

In relation to the push-out results, statistically significant differences among the groups were detected ($p < 0.05$). In all thirds analyzed, AH Plus revealed significantly higher bond strength values than MTA Fillapex and Sealapex ($p < 0.05$). No statistically significant difference was observed between MTA Fillapex and Sealapex, independently of the thirds evaluated. Table 4 shows the means and standard deviations of the push-out strength values (in MPa) for the displacement of the cements from the specimens in the different thirds. Graph 1 presents the frequency (%) of failure modes among the three cements. No cohesive failure was observed for the MTA Fillapex and Sealapex. Most cases regarded adhesive failure. For AH Plus, the failure mode was mainly cohesive and mixed.

DISCUSSION

Sealapex and AH Plus are routinely used as materials for comparative studies with other endodontic cements.[10,20-22] The physicochemical properties of MTA Fillapex were different than AH Plus, with the exception of flowability. The flowability and dentin adhesion of MTA Fillapex and Sealapex were similar. In some periods, the pH and calcium release values were similar among these two cements.

The MTA Fillapex presented similar flowability to that of Sealapex and AH Plus and all the materials were in accordance to the ISO 6876 specifications. According to the ISO-6876 specifications, after the endodontic cement is mixed, the diameter provided by the cement should be greater than 20 mm.[11,12] When the Sealapex is mixed as recommended by the manufacturer, in equal parts of two pastes, variations in the proportion may occur. This may be the cause of different flowability results, even under the ISO specifications.[11] In our study, Sealapex catalyst and base paste were used in mass (w/w) ratio in order to standardize the proportional measurements among the paste.

Setting time is a control test on the behavior of a product and is dependent on its components, particle size, room temperature and relative air humidity.[13] In the present study, the initial and final setting times of the MTA Fillapex were lower than the Sealapex and AH Plus cements. On the other hand, with regards to the function of the slow and gradual polymerization reaction of the epoxy resin amines with high molecular composition (Bisphenol A and Bisphenol F), the AH plus had the longest setting time.[13,23]

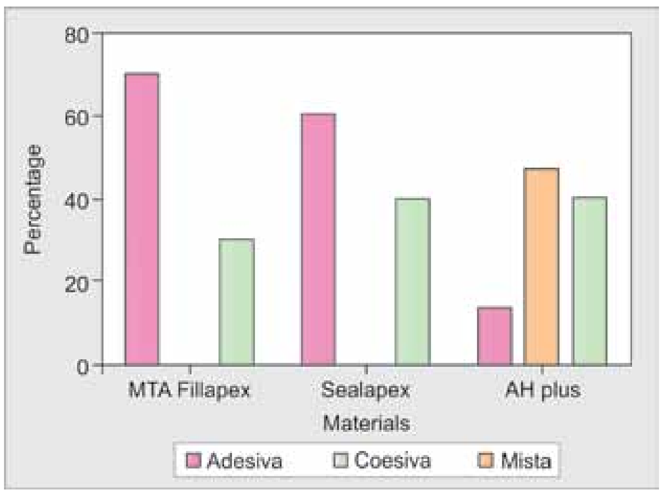
The methodology for evaluating the pH and calcium release levels is well established in the literature.[16,24] The present study consisted of filling standardized polyethylene tubes with the sealers to be tested and immersing them into deionized water. After, a specific period the pH level was determined with the use of a pH meter and the calcium release value was measured with an atomic absorption spectrophotometer with a cathode lamp.

The results show that at 7 and 28 days after the manipulation of the cements, the MTA Fillapex presented the

Table 3: Means and standard deviations of the calcium release (mg/l) values in the different experimental periods

	MTA Fillapex	*Sealapex*	*AH Plus*
24 hours	7.66 (1.16)[a]	7.45 (1.41)[a]	1.56 (1.20)[b]
7 days	8.95 (2.43)[a]	12.90 (2.05)[b]	1.14 (0.30)[c]
14 days	9.68 (3.00)[a]	9.30 (0.63)[a]	0.38 (0.06)[b]
28 days	8.17 (1.98)[a]	10.99 (1.86)[b]	0.33 (0.07)[c]

[a,b,c]Different letters in each period indicate statistically significant differences ($p < 0.05$)



	Materials		
	Adhesive	*Cohesive*	*Mixed*
MTA Fillpaex	70%	0%	30%
Sealapex	60%	0%	40%
AH Plus	13.3%	46.7%	40%

Graph 1: The frequency of the failure modes of the cements after the push-out test

Table 4: Means and standard deviations of the push-out strength values (in MPa) for the displacement of the cements from the specimens in the different thirds

		MTA Fillapex	*Sealapex*	*AH Plus*
Push-out strength values	Cervical	1.21 (0.42)[a]	0.92 (0.28)[a]	5.03 (1.87)[b]
(in MPa) for the displacement of the cements	Middle	1.22 (0.41)[a]	1.20 (0.37)[a]	3.65 (1.53)[b]
	Apical	1.19 (0.56)[a]	1.36 (0.40)[a]	10.15 (4.36)[b]

[a,b]Different letters in each third indicate statistically significant differences for the push-out strength test ($p < 0.05$)

highest pH values, with significantly statistical differences from Sealapex and AH Plus. With regards to the calcium release value, Sealapex presented the highest values at 7 and 28 days. These results confirm the satisfactory performance of both cements in terms of the physicochemical properties studied.[16,24,25]

The possible reason for the MTA Fillapex's lower calcium release is due to the percentage of mineral trioxide aggregate present in this cement. Although the AH Plus contains calcium tungstate in its formula, the pH and calcium release values were lower than the others cements tested.[25]

The adhesive strength of endodontics cements has been frequently examined by the push-out method.[10,21] However, the tendency of the gutta-percha to deform when a compressive load is applied during testing is a problem of the push-out test.[26] Another problem is the risk of contact of the gutta-percha with the dentinal wall. To overcome these limitations, in our study the root canal space was obturated only with the endodontic cement.

AH Plus showed a higher bond strength when compared with Sealapex and MTA Fillapex, in all thirds analyzed. Similar results were observed when this material was compared with two MTA-based cements.[10] The higher bond strength obtained with the AH Plus, may be explained by its ability to react with exposed amino groups in collagen in order to covalent form bonds among the resin and the collagen.[27] Sealapex and MTA Fillapex do not display this characteristic. In our findings, these cements had low bond strength to the dentin, which may have been caused by the low tensile cohesive strength of the self-cured cements that contained calcium in its composition.[21]

As previously described, the methodology used in the root canal obturation to evaluate the shear bond adhesion of endodontic cements onto the root dentin, interferes with the results.[28] Another study which compared the MTA Fillapex and AH Plus, utilizing the lateral condensation technique for the obturation procedures, observed similar dentin adhesion among the MTA Fillapex and AH Plus, but lower than Endo-CPM.[29]

MTA Fillapex is promising endodontic cement. Its physicochemical properties are similar to Sealapex's. The main problem of this cement is the dentinal adhesion, which is significantly lower than AH Plus. Our study demonstrated the importance of knowledge of the physicochemical properties of the endodontic cements and consequently provides the possible corrections in the formulations for an adequate clinical use.

CONCLUSION

The physicochemical properties studied for MTA Fillapex were similar to Sealapex's, except with regards to setting time and the pH and calcium release values, at days 7 and 28. However, its properties are different from AH Plus, except for flowability. Its bond strength to the root dentin is lower than AH Plus, independent of the thirds evaluated.

CLINICAL SIGNIFICANCE

This study evaluated the physicochemical and mechanical properties of new MTA-based root canal cement, in order to use this sealer in root canal fillings. MTA Fillapex showed satisfactory properties for clinical use.

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