Physical Properties of MTA Fillapex Sealer

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Abstract

Introduction: The aim of this study was to evaluate and compare several physicochemical properties including working and setting times, flow, solubility, and water absorption of a recent calcium silicate–based sealer (MTA Fillapex; Angelus, Londrina, Brazil) and an epoxy resin–based sealer (AH Plus; Dentsply, Konstanz, Germany). **Methods:** The materials were handled following the manufacturer's instructions. The working time and flow were tested according to ISO 6876: 2001 and the setting time according to American Society for Testing and Materials C266. For solubility and water absorption tests, the materials were placed into polyvinyl chloride molds (8 × 1.6 mm). The samples (n = 10 for each material and test) were placed in a cylindrical polystyrene-sealed container with 20 mL deionized water at 37°C. At 1, 7, 14, and 28 days, the samples were removed from the solutions and blotted dry for solubility and water absorption tests. The data were analyzed using 1-way analysis of variance with the Tukey test ($P < .05$). **Results:** MTA Fillapex showed the lowest values of flow, working and setting times, solubility, and water absorption ($P < .05$). The solubility and water absorption increased significantly over time for both materials in a 1- to 28-day period ($P < .05$). **Conclusions:** MTA Fillapex showed suitable physical properties to be used as an endodontic sealer. *(J Endod 2013;39:915–918)*

Key Words

Calcium silicate cements, endodontic sealers, epoxy resin sealers, mineral trioxide aggregate, solubility, water absorption

A hermetic 3-dimensional filling must avoid leakage from the oral cavity and/or periapical tissues, thereby reducing periapical inflammation (1). This filling is currently achieved using a combination of endodontic sealer and gutta-percha. Gutta-percha is widely used because of its good physical and biological properties, but the lack of adhesiveness and flow makes the association with endodontic sealers necessary (2).

An ideal endodontic sealer should flow along the entire canal wall surface, fill all voids and gaps between the core root filling material and dentin, and adhere to both dentin and gutta-percha (3). However, some studies have shown how adhesion of endodontic sealers to gutta-percha can be poor (4) and that all canal fillings may allow bacterial penetration over time (5).

A new calcium silicate–based sealer (MTA Fillapex; Angelus, Londrina, Parana) has been recently proposed as an endodontic filling material (6). The strong interest in developing mineral trioxide aggregate (MTA)-based endodontic materials is because of the excellent biocompatibility, bioactivity, and osteoconductivity of MTA (7). However, the results related to the biological response of MTA Fillapex are conflicting. When freshly mixed, this material showed high cytotoxicity and genotoxicity (8). Another study showed that when this sealer was implanted in subcutaneous tissues in rats, it remained toxic even after 90 days (9). However, a recent study showed that despite these initial toxic effects during setting, the cytotoxicity of MTA Fillapex decreases, and the sealer presents suitable bioactivity to stimulate nucleation sites for the formation of apatite crystals in human osteoblast-like cell culture (10).

MTA Fillapex is a sealer that is composed of MTA, salicylate resin, natural resin, bismuth oxide, and silica. A recent study showed that this sealer has suitable physicochemical properties, such as good radiopacity, flow, and alkaline pH (11). The manufacturer states that it has a great working time, low solubility, and easy handling (6). However, up to now, no scientific studies evaluating these physical properties have been published. Thus, the aim of the present study was to evaluate the working and setting time, flow, solubility, and water absorption of MTA Fillapex sealer compared with AH Plus (Dentsply, Konstanz, Germany).

Materials and Methods

Materials

The composition, manufacturers, and batch number of AH Plus and MTA Fillapex are shown in Table 1.

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TABLE 1. Materials Used

Materials	Composition (MSDS data)	Batch number
AH Plus	Paste A: bisphenol A epoxy resin, bisphenol F epoxy resin, calcium tungstate, zirconium oxide, aerosol, iron oxide	1110002296
	Paste B: dibenzyldiamine, adamantane amine, tricyclodecane-diamine, calcium tungstate, zirconium oxide, aerosol, silicon oil	1110001964
MTA Fillapex (Angelus, Londrina, Brazil)	After the mixture: salicylate resin, natural resin, diluting resin, bismuth oxide, nanoparticulated silica, MTA and pigments.	19595

Flow Evaluation

The flow test was performed following ISO 6876:2001 (12). Three determinations were made for each sealer tested ($n = 3$). The mean values and standard deviations were calculated and recorded (in millimeters) to obtain the flow rates.

Working Time Test

The working time was performed following the same procedure used in the flow test (ISO 6876:2001) (12), with the difference of the increase in the intervals between the initial mixing and the setting time. Measurements were performed at 10-minute intervals, and freshly mixed material was used each time. Three samples for each experimental sealer were made, and the mean was taken as the sample working time.

Setting Time Test

The setting time test was performed according to the C266-03 specification of the American Society for Testing and Materials (13). The initial and final setting times of each specimen ($n = 3$) were tested every hour until both times were reached.

Solubility

The solubility test was performed as described by Gandolfi et al (14, 15). Briefly, the samples ($n = 10$) were weighed (ie, the initial weight) and immersed in 20 mL deionized water at 37°C. At 1, 7, 14, and 28 days, the samples were removed from the solutions, rinsed with deionized water, blotted dry and placed in an incubator at 37°C for 48 hours, and then reweighed until they reached a constant weight (ie, the dry weight). The solubility (percentage weight variation) was calculated according to the following equation: solubility = ([dry weight at each time point − initial weight]/initial weight) × 100. Any specimen that showed evidence of disintegration was discarded, and the test was repeated.

Water Absorption

The water uptake was determined gravimetrically. The procedure was the same as that used in the solubility test. The samples ($n = 10$) were weighed (ie, the initial weight) and immersed in 20 mL deionized water at 37°C. At 1, 7, 14, and 28 days, the samples were removed and weighed. Each weight measurement was repeated 3 times, and the mean was recorded as the wet weight. Then, the samples were blotted dry at 37°C for 48 hours until weight stabilization (ie, the dry weight). The water absorption at each time point was calculated as follows: water absorption = ([wet weight at each time point − dry weight]/dry weight) × 100.

Statistical Analysis

For each test, the data were statistically analyzed by 1-way analysis of variance and the Tukey test. The significance level used was $P < .05$.

Results

Flow, Working, and Setting Times

The mean and standard deviation of flow, working time, and setting time are shown in Table 2. A considerable significant difference was found between the materials for all tests ($P < .05$). AH Plus showed the higher values of flow (37.97 ± 0.55 mm), working time (5.5 hours), and the initial (10.18 ± 0.1 hours) and final (18.11 ± 0.25 hours) setting times when compared with MTA Fillapex (flow = 29.04 ± 0.39 mm, working time = 0.5 hours, initial setting time = 2.27 ± 0.06 hours, and final setting time = 4.55 ± 0.05 hours).

Solubility

The solubility of AH Plus (1 day = −0.33 ± 0.03, 7 days = −0.36 ± 0.02, 14 days = −0.78 ± 0.05, and 28 days = −0.84 ± 0.03) was significantly higher than MTA Fillapex (1 day = −0.09 ± 0.06, 7 days = −0.15 ± 0.07, 14 days = −0.22 ± 0.08, and 28 days = −0.25 ± 0.08) for all tested time points ($P < .05$). The results are shown in Table 3. Both materials showed the highest values of solubility at 28 days ($P < .05$).

Water Absorption

AH Plus (1 day = 0.10 ± 0.01, 7 days = 0.12 ± 0.02, 14 days = 0.19 ± 0.02, and 28 days = 0.25 ± 0.01) absorbed significantly more water than MTA Fillapex (1 day = 0.05 ± 0.05, 7 days = 0.08 ± 0.07, 14 days = 0.15 ± 0.08, and 28 days = 0.20 ± 0.20) for all tested time points ($P < .05$). The results are shown in Table 3. The highest values of water absorption were found at 28 days for the 2 materials and the lowest values at 1 day ($P < .05$).

Discussion

Because of the good properties of calcium silicate MTA cements, MTA-based endodontic sealers for root canal obturation have been

TABLE 2. Flow, Working Time, and Initial and Final Setting Times (mean ± standard deviation, $n = 3$ for each material)

Material	Flow (mm)	Working time (h)	Initial setting time (h)	Final setting time (h)
AH Plus	37.97 ± 0.55^a	5.5^a	10.18 ± 0.10^a	18.11 ± 0.25^a
MTA Fillapex	29.04 ± 0.39^b	0.5^b	2.27 ± 0.06^b	4.55 ± 0.05^b

Data followed by different letters are statistically different ($P < .05$).

TABLE 3. Solubility and Water Absorption (mean ± standard deviation, n = 10 for each material) Expressed as the Percentage Weight Variation (%)

Material	1 day	7 days	14 days	28 days
Solubility				
AH Plus	-0.33 ± 0.03^{aD}	-0.36 ± 0.02^{aC}	-0.78 ± 0.05^{aB}	-0.84 ± 0.03^{aA}
MTA Fillapex	-0.09 ± 0.06^{bD}	-0.15 ± 0.07^{bC}	-0.22 ± 0.08^{bB}	-0.25 ± 0.08^{bA}
Water absorption				
AH Plus	0.10 ± 0.01^{aD}	0.12 ± 0.02^{aC}	0.19 ± 0.02^{aB}	0.25 ± 0.01^{aA}
MTA Fillapex	0.05 ± 0.05^{bD}	0.08 ± 0.07^{bC}	0.15 ± 0.08^{bB}	0.20 ± 0.20^{bA}

Data followed by different small letters in columns and capital letters in rows are statistically different ($P < .05$).

developed recently (16), and different MTA-based sealers were marketed (iRoot; Innovative BioCreamix Inc, Vancouver, Canada, and Tech Biosealer; Isasan, Rovello Porro, Italy). The present study investigated the physical properties of MTA Fillapex as the latest calcium silicate–MTA–based proposed as an endodontic sealer. The working and setting times, flow, solubility, and water absorption have been assessed and compared with those of AH Plus.

The working time is the period of time measured between the start of mixing until it is no longer possible to handle the sealer without promoting adverse effects on its properties (12). This time is directly linked with the setting time. Both times are dependent on the constituent components, their particle size, the ambient temperature, and relative humidity (15, 17). The setting reactions are complex, and even though the sealer surface becomes hard, the inner mass may remain soft for an extended period (18).

Because the extended setting time is advantageous to root canal sealers, different studies showed the possibility to modify the composition of MTA-based materials in order to delay the setting time (16, 19–21). In this study, C266-03 was used because this specification provided initial setting and final setting time evaluations; on the other hand, ISO 6876:2001 only provides final setting time evaluation. MTA Fillapex showed initial and final setting times of 2.27 and 4.55 hours, respectively. Both values are shorter than those of AH Plus; however, they are suitable for an endodontic sealer. The initial setting time of AH Plus obtained in this study was in agreement with previous studies (22–24) reporting an initial setting time of 10 hours and a final setting time of 18 hours. AH Plus is a paste-to-paste material in which a slow polymerization reaction of epoxy resin amines with a high molecular weight including bisphenol A and bisphenol F occurs. Then, the conversion of monomers into polymers occurs slowly (22, 25); this might explain the long times associated with this material. On the other hand, MTA Fillapex is a paste-to-paste, which when in contact promotes 2 chemical reactions that are responsible to set material: the progressive hydration of the orthosilicate ions (SiO_4^{4-}) and the reaction between MTA and salicylate resin. The MTA reacts with salicylate, creating an ionic polymer (26). When calcium silicate particles of MTA react with water, a nanoporous amorphous calcium silicate hydrate gel forms on the cement particles (27). The gel polymerizes and hardens, forming a solid network. The differences of setting times between MTA Fillapex and AH Plus also could be related to the resin/filler ratios because a higher resin/filler ratio leads to longer working and setting times.

The flowability of an endodontic sealer is a very important property in relation to its ability to penetrate into the irregularities and accessory canals of the root canal system. Conversely, if the flow is excessive, the risk of material extravasation to the periapex is increased, which could damage periodontal tissues (2). The ability to penetrate into the root canal system depends on the particle size, temperature, setting time, rate of shear, rate of insertion, and internal diameter of the canals (18).

The flow of a material can be evaluated using several methods including viscosity, penetrability, degree of flattening, and extrusion. The method used in this study (in accordance with the ISO 6876:2001) was the degree of flattening or extensibility, which was defined as being the mean area obtained when the material is subjected to a constant load for a certain period of time. The present study showed higher values of flowability for AH Plus (37.97 mm) than MTA Fillapex (29.04 mm), and both were in agreement with ISO 6876:2001 requirements (ie, >20 mm) (12). The reported values for AH Plus were higher than the values reported by both Silva et al (11) and Versani et al (23) but lower than values reported by Almeida et al (2) and Duarte et al (28). This could be explained by variations in the physicochemical properties of AH Plus depending on the portion of the tube where the sealer was taken in relationship with the findings of Baldi et al (29), which showed higher flow values when the sealer was collected from the beginning of the tubes in which they were supplied. The flow value of MTA Fillapex was similar to the value obtained by Silva et al (11). This is one of the pioneer studies using MTA Fillapex, so it was difficult to compare the results obtained with previous studies. Moreover, the manufacturer of MTA Fillapex does not provide any information about the composition ratio. A high resin/MTA ratio could lead to a high flow material formation.

The dissolution of a sealer may cause the release of chemical compounds that could irritate the periapical tissues (30) and induce the formation of gaps between root canals and filling materials, resulting in an increase in bacterial leakage over time. Despite the fact that ISO 6786:2001 suggests evaluating the solubility only at 24 hours, in the present study, the solubility test was performed at 1, 7, 14 and 28 days following a modification previously proposed by Gandolfi et al (14, 15). The findings showed that the solubility of MTA Fillapex was statistically lower than that of AH Plus ($P < .05$) despite the fact that the solubility of both sealers was within the ISO 6876:2001–recommended range (less than 3%) at all time points. Such results are in accordance with previous studies that showed a low weight loss in the solubility test for both AH Plus (at different times) and MTA Fillapex (at 24 hours) (30–32). This is the first time that a long-term solubility test was performed on MTA Fillapex. It is important to point out that the standard test methods for solubility recommend immersion of the materials in water only after complete setting (or at least 70% of the initial setting). However, this situation is impossible to be achieved clinically because the materials are immediately placed into contact with fluids and blood. Therefore, the solubility values in a clinical scenario are probably higher than the ones found in *in vitro* tests (33).

Water diffusion into cements may result in the deterioration of their physical/mechanical properties, decreasing the life expectancy of the interfaces by hydrolysis and microcrack formation (34). However, water absorption could be beneficial because it promotes an expansion of the material, which may promote proper sealing. In the current study, AH Plus showed higher water sorption values than MTA Fillapex at all time points ($P < .05$). In general, water absorption properties of the resinous materials are affected by many factors including the chemical composition, the presence of hydrophilic constituents in the resin matrix (14), and the structural parameters of the polymeric network such as cross-linking density and porosity (35). A more porous polymeric network with distant polymeric chains may explain the higher water absorption results for AH Plus.

MTA Fillapex has a shorter but clinically acceptable working time associated with a shorter setting time. MTA Fillapex had a lower flow than AH Plus. MTA Fillapex also showed lower solubility and water absorption than AH Plus. Solubility and water absorption decreased in rate over 28 days. Both materials showed values in accordance to ISO 6876:2001. In conclusion, the results of this study showed that the MTA-based endodontic sealer MTA Fillapex showed suitable physical properties to be used in endodontic therapy.

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

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