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 ($P < .05$).