

Influence of the Addition of Calcium Hydroxide Powder on Some Physical and Chemical Properties of the Sealer MTA Fillapex

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ABSTRACT

Objective: This study evaluated the flow, pH and calcium release of MTA Fillapex (G1) or Fillapex plus 10% in weight of calcium hydroxide powder (G2), compared to AH Plus (G3) and Sealapex (G4).

Materials and methods: The flow test was performed according to ISO 6876:2001 requirements. The sealers were placed into plastic tubes and immersed in deionized water. After 24 hours, 7, 14 and 28 days, the water of each tube was removed and tested to evaluate the pH values and the level of released calcium. Calcium release values were analyzed statistically by Kruskal Wallis and Dunn tests and pH values analyzed by ANOVA and Tukey tests ($\alpha = 5\%$).

Results: G1 presented higher flow among all sealers. The addition of 10% calcium hydroxide into MTA Fillapex reduced the flow ($p < 0.05$) but, in a level, that is lower than the one recommended for ISO norms. G2 and G4 presented pH values and calcium release higher than G3 ($p < 0.05$) in all periods. G1 presented pH value higher than G3 ($p < 0.05$), except in 7 days period ($p > 0.05$). G4 presented higher pH values than G1 and G2, but the calcium release was similar for all periods ($p > 0.05$). G3 presented lower calcium release among all groups ($p < 0.05$).

Conclusion: The addition of 10% calcium hydroxide in MTA Fillapex caused reduction in flow and no negative interference in pH and/or calcium release. However, the obtained flow is different from ISO requirements.

Clinical relevance: MTA Fillapex presents levels of flow above the ISO norms. The addition of calcium hydroxide is a suggestion for solving this problem, but the impact of these procedures should be carefully evaluated.

Keywords: Calcium hydroxide, Calcium release, Flow, MTA, pH.

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INTRODUCTION

Mineral trioxide aggregate is a white or gray powder composed by hydrophilic particles whose main components are tricalcium silicate, tricalcium aluminate, tricalcium oxide and silicate oxide.¹ There are also small quantities of other

substances which enhance the physicochemical properties of the aggregate, such as bismuth oxide.²

Currently, it is used in conservative pulp therapy procedures, pulpotomy in primary teeth, in immature teeth treatment and for the resolution of several complications during endodontic treatment.³⁻⁷ MTA is also used for root canal filling with satisfactory biological results.⁸ However, when mixed with distilled water results in a material that is difficult to be inserted into the root canal.⁹

Recently, sealers containing MTA exclusively for use as root canal filling material were developed, such as Endo-CPM-Sealer (EGEO, Buenos Aires, Argentina), ProRoot Endo Sealer (Dentsply Maillefer, Ballaigues, Switzerland) and experimental cement, MTAS (mixture of 80% white Portland cement and 20% bismuth oxide) with an addition of water soluble polymer.¹⁰ A new formulation of sealer containing MTA (MTA Fillapex, Angelus Soluções Odontológicas, Londrina, PR, Brazil) was created to be used as root canal sealer, but few information are known about it.¹¹⁻¹⁵ According to the manufacturer, the composition of MTA Fillapex after mixing is MTA, salicilate resin, natural resin, bismuth and silica.

An adequate flow is a fundamental property to endodontic sealers.¹⁶ On the other hand, an excessive flow can favor apical extrusion of the sealer and consequently injure the periapical tissues.¹⁷ An evident clinical problem observed in the manipulation of MTA Fillapex is its excessive flow. To minimize this problem, some practitioners empirically have added 10% of calcium hydroxide into MTA Fillapex sealer (in w/w), with similar proposal for the AH Plus sealer.^{18,19} Although this procedure apparently reduces the flow, there are no studies that evaluate the impact of this method on some physical and chemical properties of MTA Fillapex, mainly in relation to pH and calcium release and if the obtained mixture is in accordance to ISO requirements.²⁰

Endodontic sealers that contain calcium hydroxide in their composition will only perform their biological and microbiological action, if calcium ion and hydroxyl release occurs.^{21,22} Thus, it is necessary to evaluate if the addition of calcium hydroxide in MTA Fillapex, in proportion of 10% in weight, interferes to these properties.

Therefore, the aim of this study was to evaluate the flow, pH and calcium ion release of MTA Fillapex, pure or incorporated with 10% calcium hydroxide (in weight), compared to that provided by AH plus (Dentsply De Trey, Konstanz, Germany), and Sealapex (SybronEndo, Glendora, USA) in periods of 24 hours, 7, 14 and 28 days.

MATERIALS AND METHODS

The sealers used in this study were: MTA Fillapex (Angelus Ind Prod Odontológicos S/A, Londrina, PR, Brazil) (G1), AH Plus (Dentsply De Trey, Konstanz, Germany) (G3) and Sealapex (SybronEndo, Glendora, USA) (G4). All sealers were mixed according to manufacturers instructions, except the groups MTA Fillapex (Angelus Ind Prod Odontológicos S/A, Londrina, PR, Brazil), in which total volume of the sealer was added 10% (weight) of calcium hydroxide powder (G2).

The flow of the sealers were evaluated in accordance to ISO 6876 requirements, as described by Camps et al.^{16,20}

After flow analysis, forty polyethylene tubes measuring 10 mm in length and 1.5 mm in internal diameter were filled with the mixtures to be evaluated. For pH and calcium ion release evaluation, 10 specimens were prepared from each material studied. Immediately after manipulating the materials, the tubes were filled and weighed to check the standard amount of sealer in each specimen (± 0.002 gm) and placed into polypropylene flasks (Injeplast, São Paulo, Brazil) containing 10 ml of deionized water. All specimens were kept at 37°C (Farmen, São Paulo, Brazil).

After 24 hours, 7, 14 and 28 days, the water was assessed for pH and calcium release. Previous to the immersion of specimens, the pH and calcium ion concentration of deionized water were verified, attesting pH 6.8 and total absence of calcium ions. The tubes containing the cements were placed in new flasks with 10 ml of distilled water for further analysis in the different time periods.

The pH measurements were conducted with a pH meter (model DM, 22 Digimed, São Paulo, Brazil), as described by Vivan et al.²³ The pH values were compared by ANOVA test ($\alpha = 5\%$).

The release of calcium ions was measured using an atomic absorption spectrophotometer (AA6800, Shimadzu, Tokyo, Japan) equipped with a calcium-specific hollow cathode lamp, as described Vivan et al.²³ The calcium release values were compared by Kruskal Wallis and Dunn tests ($\alpha = 5\%$).

RESULTS

The mean flow values for the sealers are described in Table 1 (in mm). MTA Fillapex had higher flow than the other sealers ($p < 0.05$). Moreover, the addition of 10% of calcium hydroxide into MTA Fillapex provided a significant reduction in flow ($p < 0.05$), but this value is in disagreement to ISO requirements. The flow of AH Plus and Sealapex were similar ($p > 0.05$).

The mean pH values and standard deviations for the several sealers evaluated in the different experimental periods are described in Table 2. In 24 hours, G4 presented higher pH values than other sealers ($p < 0.05$). G1 and G2 presented similar pH values ($p > 0.05$) and both presented higher pH values than G3 ($p < 0.05$). In 7 days, G4 presented higher pH values than other sealers ($p < 0.05$). G2 presented higher pH values than G1 and G3 ($p < 0.05$). G1 and G3 presented similar pH values ($p > 0.05$). In 14 days, G4 and

Table 1: Mean and standard deviation of the flow value for the sealers (in mm)

	Groups			
	G1	G2	G3	G4
	29.4 (1.05) ^a	16.80 (0.71) ^c	21.91 (0.36) ^b	21.11 (0.40) ^b

^{a,b,c}Different letters in each period indicate significant difference ($p < 0.05$); G1: MTA Fillapex; G2: MTA Fillapex with 10% of calcium hydroxide powder; G3: AH Plus; G4: Sealapex

Table 2: Mean and standard deviation of the pH value for the sealers, in the different periods

Groups	G1	G2	G3	G4
24 hours	9.39 (0.30) ^b	9.43 (0.32) ^b	8.10 (0.29) ^c	9.82 (0.18) ^a
7 days	7.68 (0.23) ^c	8.75 (0.45) ^b	7.67 (0.52) ^c	10.05 (0.29) ^a
14 days	8.89 (0.54) ^b	9.42 (0.05) ^a	6.80 (0.52) ^c	9.77 (0.35) ^a
28 days	9.15 (0.48) ^b	9.34 (0.22) ^b	6.11 (0.58) ^c	9.83 (0.10) ^a

^{a,b,c}Different letters in each period indicate significant difference ($p < 0.05$); G1: MTA Fillapex; G2: MTA Fillapex with 10% of calcium hydroxide powder; G3: AH Plus; G4: Sealapex

Table 3: Rank means of the calcium release values for the sealers in the different periods

Groups	G1	G2	G3	G4
24 hours	29.6 ^a	20.1 ^a	5.6 ^b	26.7 ^a
7 days	23.8 ^a	17.6 ^a	5.9 ^b	34.7 ^a
14 days	31.1 ^a	19.6 ^a	5.5 ^b	25.8 ^a
28 days	20.4 ^a	22.6 ^a	5.5 ^b	33.5 ^a

^{a,b}Different letters in each period indicate significant difference ($p < 0.05$); G1: MTA Fillapex; G2: MTA Fillapex with 10% of calcium hydroxide powder; G3: AH Plus; G4: Sealapex

G2 presented similar pH values ($p > 0.05$) and higher results than other sealers ($p < 0.05$). G1 presented higher pH values than G3 ($p < 0.05$). In 28 days, the pH values were equal to that presented in 24 hours period.

As calcium release values no presented a normal distribution, a nonparametric statistical analysis was used. In all periods, the G3 presented calcium release lower than other groups ($p < 0.05$). The calcium release values of G1, G2 and G3 were similar in all periods ($p > 0.05$). The rank means of the calcium release for the several sealers evaluated, in the different experimental periods, are described in Table 3.

DISCUSSION

The addition of calcium hydroxide to MTA Fillapex, in proportion of 10%, had no negative interference in pH value and calcium release, in all analyzed periods. When compared to AH Plus sealer, this mixture presented significantly higher pH values and calcium release. Despite the significantly reduced flow in relation to MTA Fillapex alone, the final result is not compatible to the ISO requirement.

An adequate flow allows the sealer to fill the empty spaces unfilled by gutta-percha cone.²⁴ The methodology used in the present study is in accordance with ISO 6876 requirements related to endodontic sealers.^{16,20,25} The flow values found were higher than the minimum required by the ISO 6876 (≥ 20 mm), except to G2. The flow reduction of MTA Fillapex can be attributed to use of calcium hydroxide powder, once the relation powder/liquid interferes with the flow property.^{16,24} The impact of this reduction in flow of MTA Fillapex needs further assessments.

It has been suggested that to mineralization stimulus, materials should have an alkaline pH level and release calcium.^{21,22} The methodology used to evaluate pH and calcium release consisted in filling standardized polyethylene tubes, with the same dimensions (1.0 cm in length \times 1.5 mm in internal diameter) to that used by Scarparo et al.²⁶ After specific periods, the pH values of deionized water were determined with pH meter and the calcium

release measured with atomic absorption spectrophotometer. Similar methods were employed in other studies.^{19,21,23}

Sealapex presented the highest release of hydroxyl ions in all analyzed periods, in agreement with those obtained by Duarte et al.²¹ MTA Fillapex showed a decrease in pH values during the experiment, increasing only in final period. Probably, it is related to the time of immersion of the specimens in deionized water. The addition of calcium hydroxide prevented the reduction of the pH value of the MTA Fillapex, mainly in the period of 14 days, similar to the Sealapex. However, in 28 days, probably due to the final setting, there was a decline in pH value, returning to resemble the pH of the MTA Fillapex. Similar results were observed, when the calcium hydroxide was added in AH Plus sealer.¹⁹

The calcium release provided by MTA Fillapex alone or added with calcium hydroxide and Sealapex were similar in all analyzed periods. As described by the manufacturers, the similarity between the composition of the sealers may have contributed to similar the calcium release.^{11,21,26} On the other hand, AH Plus could not provided the release of calcium, as also observed by Duarte et al.¹⁹

MTA Fillapex has satisfactory biological results.¹⁴ But its high flow may cause overfilling beyond the apical foramen, which is avocate for some endodontic sealers.¹⁷ The addition of calcium hydroxide powder in the sealer is a suggestion to correct excessive flow.¹⁹ In relation to the MTA Fillapex, the addition of calcium hydroxide powder did not affect the properties of alkalization and calcium release of sealer, but is not compatible ISO 6876 requirements. Therefore, further studies with other proportions of calcium hydroxide are required, to obtain the best consistency to MTA Fillapex, without the occurrence of changes in its physical and chemical properties.

CONCLUSION

The results of calcium release and pH observed after addition of 10% calcium hydroxide powder (in weight) in the MTA Fillapex are similar to the cement pure and Sealapex, with lower flow than the original composition but in conditions not recommended by ISO requirement. This proportion is not recommended for clinical use, and proportions with less than 10% of calcium hydroxide powder should be assessed.

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