Effects of calcium hydroxide addition on the physical and chemical properties of a calcium silicate-based sealer

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ABSTRACT

Recently, various calcium silicate-based sealers have been introduced for use in root canal filling. The MTA Fillapex is one of these sealers, but some of its physicochemical properties are not in accordance with the ISO requirements. Objective: The aim of this study was to evaluate the flowability, pH level and calcium release of pure MTA Fillapex (MTAF) or containing 5% (MTAF5) or 10% (MTAF10) calcium hydroxide (CH), in weight, in comparison with AH Plus sealer. Material and Methods: The flowability test was performed according to the ISO 6876:2001 requirements. For the pH level and calcium ion release analyses, the sealers were placed individually (n=10) in plastic tubes and immersed in deionized water. After 24 hours, 7 and 14 days, the water in which each specimen had been immersed was evaluated to determine the pH level changes and calcium released. Flowability, pH level and calcium release data were analyzed statistically by the ANOVA test (α=5%). Results: In relation to flowability: MTAF>AH Plus>MTAF5>MTAF10. In relation to the pH level, for 24 h: MTAF5=MTAF10=MTAF>AH Plus; for 7 and 14 days: MTAF5=MTAF10>MTAF>AH Plus. For the calcium release, for all periods: MTAF>MTAF5=MTAF10>AH Plus. Conclusions: The addition of 5% CH to the MTA Fillapex (in weight) is an alternative to reduce the high flowability presented by the sealer, without interfering in its alkalization potential.

Keywords: Calcium hydroxide. Dental cements. Endodontics. Physical and chemical properties. Silicate cement.

INTRODUCTION

Mineral trioxide aggregate (MTA) was developed to be used in endodontic complications, as well as in apical surgery. Actually, it is also recommended to be used in pulp conservative treatments and in obturation of the apical portion of immature teeth. MTA induces a new apical cementum deposition when used in root canals. However, its handling is difficult for use in root canal obturations. Several calcium silicate-based sealers were developed to be used as a root canal filling material, such as Endo-CPM-Sealer (EGEO, Temperley, Buenos Aires, Argentina), ProRoot Endo Sealer (Dentsply Maillefer, Ballaigues, Switzerland) and an experimental cement, MTAS. Recently, a new formulation of MTA-based cement (MTA Fillapex; Angelus Indústria de Produtos Odontológicos S.A., Londrina, Paraná, Brazil) was created to be used as a root canal sealer. The composition of MTA Fillapex after mixing is MTA, salicilate resin, natural resin, bismuth and silica, according to the manufacturer. MTA Fillapex has an antibacterial effect against E. faecalis before setting and adequate biocompatibility. Despite these biological properties, its physicochemical properties are controversial. MTA Fillapex had the lowest push-out values to root dentine compared with AH Plus and IRoot SP sealers. But in another study, MTA Fillapex presented acceptable resistance to dislodgement, which was similar to AH Plus. A possible reason
for this different result can be attributed to the difficulty in handling due to excessive flowability. This observation was previously confirmed in a pilot study.

Adequate flowability is a necessary property of endodontic sealers. Excessive flowability can favor apical extrusion and injure the periapical tissue, mainly in teeth with a wide foraminal opening. A suggestion in order to decrease the flowability is the addition of calcium hydroxide into the sealer.

On the other hand, the ideal endodontic sealer should be biocompatible and able to induce mineralized tissue formation. These properties are directly associated with the alkalization potential and calcium release of the materials. Calcium hydroxide provides an alkaline pH and promotes calcium release, leading to biochemical effects that culminate in the acceleration of the repair process.

The aim of this study was to evaluate the flowability, pH level and calcium ion release of MTA Fillapex, with the addition of 5% or 10% calcium hydroxide powder (in weight), compared to pure MTA Fillapex and AH Plus.

MATERIAL AND METHODS

Root canal sealers

The sealers used in this study were: AH Plus (Dentsply DeTrey, Konstanz, Germany) and the MTA Fillapex (MTAF) (Angelus Indústria de Produtos Odontológicos S.A., Londrina, Paraná, Brazil). The MTAF was used pure or with the addition of 5% (MTAF5) or 10% (MTAF10) calcium hydroxide (Labsynth, Diadema, São Paulo, Brazil), in weight. For the AH Plus, the base and catalyst pastes were used in equal parts, in the proportion of 1:1 (w/w).

Flowability

The root canal sealers were mixed according to conditions recommended by the manufacturer and the flowability test was performed according to the ISO 6876:2001 requirements.

Sample preparation

Forty polyethylene tubes measuring 10 cm in length and 1.5 mm in internal diameter were filled with the sealers to be evaluated. For the pH level and calcium ion release evaluation, 10 samples were prepared from each material studied. Immediately after manipulating the materials, the tubes were filled and weighed to check the standardization of the amount of sealer in each tube (±0.002 g) and placed in polypropylene flasks (Injeplast, São Paulo, Brazil) containing 10 mL of deionized water. The tubes were kept at 37°C (Fanem, São Paulo, São Paulo, Brazil) during the entire study. After 24 h, 7 and 14 days, the water was assessed for pH levels and calcium release. Previous to the immersion of the specimens, the pH level and calcium ion concentration of the deionized water were verified, attesting a pH level of 6.8 and a total absence of calcium ions. The tubes containing the sealers were placed in new flasks with 10 mL of deionized water for further analyses in the different time periods.

pH level test and calcium release analysis

Measurements were performed with a pH meter Q400AS (Quimis, Diadema, São Paulo, Brazil) in constant temperature (25°C). After 24 h of immersion, the tubes were carefully removed and placed into another flask with an equal amount of new deionized water. This procedure was repeated for a total of 336 h with the solution changed at 24 h, 7 and 14 days. The pH values were compared by the ANOVA and Tukey tests, at a 5% significance level.

The calcium release was measured using an AA7000 atomic absorption spectrophotometer (Shimadzu, Nakaygio-ku, Kyoto, Japan), in accordance to the manufacturer’s instructions. For the reading, 6 mL of the standard solutions or water samples were associated with 2 mL of lanthanum nitrate solution. For the white solution, 6 mL of deionized water was associated with the same amount of lanthanum nitrate solution. With the standard solutions, the white solutions and the prepared sample, the reading was carried out using an atomic absorption spectrophotometer. The

Figure 1- Flowability values from each sealer evaluated (in mm). Different letters indicate statistically significant differences (p<0.05)
calcium release was calculated by the equation of the line of the standard curve. The reading of the calcium release was taken in the same periods used for measuring the pH level. The calcium release values were compared by the ANOVA and Tukey tests, at a 5% significance level.

RESULTS

The representative mean and standard deviation for flowability values from each sealer are presented in Figure 1 (in mm). MTAF showed higher flowability (29.4 mm) than the other sealers (p<0.05).

Figure 2- pH from each sealer studied in the different experimental periods. Different letters indicate statistically significant differences (p<0.05)

Figure 3- Calcium release (mg/L) from each sealer studied in the different experimental periods. Different letters indicate statistically significant differences (p<0.05)
The flowability of the MTAF5 was lower than the MTAF and AH Plus (p>0.05); however, it was in accordance with the ISO 6876:2001 requirements. The MTAF10 showed the lowest flowability in relation to the other sealers (p<0.05) and this value was below the ISO 6876:2001 requirements (p<0.05). The flowability means and standard deviations (in mm) for the MTA, MTAF5, MTAF10 and AH Plus were 29.40 (1.05), 20.75 (0.89), 16.80 (0.71) and 21.91 (0.36), respectively.

The representative mean and standard deviation of pH values and calcium release from each sealer in different periods are presented in Figures 2 and 3, respectively. For all periods, the AH Plus showed the lowest pH value (p<0.05). At 24 h, the MTAF, MTAF5 and MTAF10 showed similar pH values (p>0.05). At 7 and 14 days, the MTAF5 and MTAF10 showed similar pH values (p>0.05) and higher pH level than the MTAF (p<0.05).

For all periods, the AH Plus showed the lowest means of calcium release (p<0.05). On the other hand, the MTAF5 and MTAF10 showed similar calcium release (p>0.05) and lower calcium release than the MTAF.

DISCUSSION

The addition of 5% calcium hydroxide to the MTAF provided a reduction of the flowability, in accordance with ISO requirements. Its pH level was alkaline in all periods but the calcium release was lower than pure MTA Fillapex. Despite the addition of 10% calcium hydroxide to MTA Fillapex, it provided a reduction in flowability, and this mixture is in disagreement with the ISO 6876:2001 requirements.

The flowability test results revealed that the addition of calcium hydroxide provides a reduction in flowability of the MTAF. This can be explained by an increase in the powder/liquid ratio also promoted, in accordance with that observed in a previous study23. Except for the MTAF10, all sealers were in accordance with the ISO 6876:2001 requirements18.

It has been suggested that in order to stimulate mineralization, a material should have an alkaline pH level and calcium release10. The methodologies used for the pH level and calcium release evaluations were similar to other studies6,19,31. The MTAF5 and MTAF10 presented pH levels similar to the MTAF, but only in a 24 h period. After this time, the pH values were higher than any other sealer.

For all periods, the MTAF showed a higher calcium release despite the fact that calcium hydroxide was added in the MTAF5 and MTAF10. Hosoya, et al.17 (2004) observed that the addition of 10% calcium hydroxide in some sealers causes variations in setting time. Therefore, the addition of calcium hydroxide in the MTAF could also have reduced the setting time of the sealer and consequently its solubility, consequently reducing the calcium release. In contrast with this result, the addition of calcium hydroxide did not alter the AH Plus setting time8. However, the compositions of these sealers are different. AH Plus sealer presented lower calcium release than the other sealers, although it contains tungstate calcium in its composition. These results are consistent with other studies also carried out with atomic absorption spectrophotometry7,12.

Through a comparative analysis of the compositions described by the manufacturers, it was observed that the MTA and Sealapex (SybronEndo, Orange, California, USA) contain several similar substances in their formulations. Thus, it is possible that many reactions and effects described in the Sealapex also occur with the MTA. Eldeniz, et al.9 (2007) report that a possible reason for the high calcium release provided by the Sealapex is related with the presence of 24% calcium hydroxide. Since the sealer has a porous matrix with a low dimensional stability and high water absorption, the calcium is easily released2,24.

On the other hand, the addition of substances with calcium in the MTA promotes a reduction of the setting time10. Thus, the addition of calcium hydroxide in the MTA could have interfered in the setting time and consequently in the calcium release. This could be a possible reason for the higher calcium release provided by MTAF when compared to the MTAF5 and MTAF10.

MTA has established properties and indications for use in several endodontic procedures25. Its use as an endodontic sealer requires further adjustments, since its handling is very difficult. MTA is a new calcium silicate-based sealer whose flowability is in accordance with the ISO 6876:2001 requirements18; clinically, however, it is very high, which can favor the extrusion of the material to the apical tissues. The purpose of adding 5% calcium hydroxide (in weight) to the total weight of the sealer is to obtain an adequate alternative. Further studies are necessary to biologically evaluate this purpose.

CONCLUSION

Based on the methods and conditions employed in the present study, it is concluded that the addition of 5% calcium hydroxide (in weight) to the MTAF provides a reduction in the flowability of the sealer, in conditions required by the ISO 6876:2001 specifications and favors a higher alkaline pH.
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