ADDITION OF MTA IN A SALICYLATE-BASED RESIN ROOT CANAL SEALER TO RETROGRADE ROOT-END FILLING

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ABSTRACT

Aim: To evaluate the flow, pH and calcium ion release of white MTA (WMTA), salicylate-based resin root canal sealer (Sealapex; SEAL) and SEAL containing 10 (SE10) or 20% (SE20) (w/w) of MTA. **Methodology:** Flow test was performed according to ISO 6876 specification. The sealers samples (n= 10) were placed in polyethylene tubes and immersed in deionized water. After 24 hours and 7, 14, and 28 days, the water pH was determined with a pH meter, and calcium ion release was assessed by atomic absorption spectrophotometry. Anova one way and Tukey tests were used in all evaluations (p=0.05).
Results: SEAL and WMTA showed respectively the highest and lowest flow rate when compared with the other materials (p<0.05). SE20 showed the highest pH value only in 24h and 7 days periods (p<0.05). In 14- and 28-days periods, SEAL showed the lowest pH value (p<0.05), but there were no differences between other groups (p>0.05). In all periods, WMTA and SEAL respectively showed the highest and lowest calcium ion release (p<0.05).
Conclusions: SE20 proves to be an association with better flow and handling than WMTA, with satisfactory potential for alkalinization and calcium ion release.
KEYWORDS: Apical surgery. Calcium ion. Flow. Mineral trioxide aggregate. Root-end filling.

INTRODUCTION

The Mineral Trioxide Aggregate (MTA) was introduced in 1993 as an endodontic reparative material[1]. Since then, several studies indicate this cement as the material of choice for the treatment of perforations, radicular fractures, pulp capping and retrograde fillings[2,3].

Its composition is basically of calcium silicates and bismuth oxide as radiopacifier. It has excellent biological properties and induces bone repair by the formation of mineralized tissue[4,5]. However, its disadvantages such as difficulty handling[5], insertion, in addition to prolonged setting time, have motivated modifications for the development of new cement proposals based on MTA.

Among the alternatives, the manipulation of the material with different vehicles such as methylcellulose, calcium chloride, calcium lactate, hydrosoluble gel (KY gel) and propilene glycol were studied[6,7]. New formulations have also been proposed to improve material flow, such as MTA HP (Angelus Ind. Prod Odontológicos S/A, Londrina, PR, Brazil), MTA Flow and Biodentine (Septodont, Saint-Maur-des-Fossés, France)[8], however, these formulations have a high market value compared to MTA and Sealapex cement.

Endodontic sealers must have an adequate flow to penetrate small irregularities and root canal system isthmus and dentinal tubules. On the other hand, proper handling of the cement is directly related to the setting time of the material[9].

The presence of alkaline pH is associated with bactericidal and bacteriostatic properties and mineralized tissue deposition[10]. New root canal cements have been developed to incorporate the beneficial properties of calcium hydroxide, such as alkaline pH and release of calcium ions[9]. The Sealapex (Kerr, Orange, CA, USA) is a root canal cement incorporated with calcium hydroxide with the highest pH and release of calcium ions[10].

In a comparative study, Sealapex (Kerr, Orange, CA, USA) cement showed physical-chemical behavior similar to MTA, such as calcium ion release and pH[11]. It also demonstrated higher antimicrobial capacity than MTA[12]. However, it has a high flow rate[13-15], essential for filling root canals, but not for retrograde fillings.

Therefore, both materials have limitations of use and advantages if used properly. Our question in this study is whether adding different amounts of MTA to Sealapex endodontic sealer (Kerr, Orange, CA, USA) would improve physical-chemical properties, which would lead to beneficial results to be used clinically.

Therefore, the aim of this study was to evaluate the effect of incorporation of 10% and 20% of MTA to a salicylate-resin based cement (Sealapex) on the flow, pH and calcium ion release. The null hypothesis was that the incorporation of MTA to Sealapex would not modify its physicochemical properties.

Table 1 Constituents and manufacturers of the root canals sealers.

Sealer	Composition	Manufacturer
Sealapex	Isobutile Salicylate resin, bismuth trioxide, titanium dioxide, zinc oxide, polymeric resin, calcium hidroxide and pigments.	Kerr, Orange, CA, USA
White MTA	Silicon dioxide, Potassium oxide, aluminum oxide, sodium oxide, iron oxide, sulfur trioxide, calcium oxide, bismuth oxide, magnesium oxide, insoluble residues of calcium oxide, potassium and sodium per sulphate, and crystalline silica.	Angelus Ind. Prod Odontológicos S/A, Londrina, PR, Brazil.

MATERIALS AND METHODS

Flow Test

The root canal sealers used, their formulations and manufacturers, are listed in **Table 1.** Material samples were divided in four groups: white MTA (WMTA), Sealapex (SEAL), Sealapex containing 10% of MTA (SE10), and Sealapex containing 20% of MTA (SE20). White MTA (Angelus Ind. Prod Odontológicos S/A, Londrina, PR, Brazil) and Sealapex were mixed according to manufacturer's instructions. Based in the Sealapex sealer, two experimental sealers were prepared by adding proportionally 10 and 20% (w/w) white MTA (Angelus Ind. Prod Odontológicos S/A, Londrina, PR, Brazil) during spatulation and 0.05$\pm$0.005mL of sealer was placed on the centre of a glass plate with a graduated syringe. After 3 minutes, a second plate weighing 120g was placed on the sealer. After 7 additional minutes, the average of the major and minor diameter of the disc of sealer was recorded in millimeters using a digital caliper with a resolution of 0.01mm (Mitutoyo MTI Corporation, Tokyo, Japan). The mean of three measurements for each sealer was taken as the flow rate of the material. All these procedures were realized according to ISO 6876; 2002, described by Camilleri et al.[16] (2009).

Table 2 Flow rate (in mm) in the materials studied.

WMTA	SEAL	SE10	SE20
13.06 $\pm$ 0.48^c	29.40 $\pm$ 1.05^a	17.08 $\pm$ 0.26^b	16.80 $\pm$ 0.71^b

The comparison amongst groups (p<0.01) is indicated by different superscripts (a, b and c).
MTA, mineral trioxide aggregate.

Analyses of pH and Calcium Ion Release

Material samples were divided in the same four groups: white MTA (WMTA), Sealapex (SEAL), Sealapex containing 10% of MTA (SE10), and Sealapex containing 20% of MTA (SE20). White MTA (Angelus Ind. Prod Odontológicos S/A, Londrina, PR, Brazil) and modified Sealapex (10 and 20%) were mixed according to above conditions. Thirty polyethylene tubes measuring 10mm in length and 1.5mm in internal diameter were filled with the fresh mixtures of evaluated sealers, using a Lentulo spiral (Maillefer, Ballaigues, SW). For pH and calcium ion release evaluation, 10 samples were prepared from each studied material. The tubes filled with the fresh mixtures were weighed to check the standartization of cement amount. They were placed in polypropilene flasks (Injeplast, São Paulo, SP, BR) containing 10mL of neutral pH deionized water and kept in an oven at 37ºC (Farmen, São Paulo, SP, BR). Previous to the immersion of specimens, the pH and calcium concentration of deionized water were verified, attesting pH 6.8 and total absence of calcium. Evaluations of pH and calcium ion release were carried

Table 3 pH values found in the materials in different experimental periods.

EP	WMTA	SEAL	SE10	SE20
24 hours	9.52 ± 0.28[b]	9.01 ± 0.74[b]	9.49 ± 0.35[b]	11.11 ± 0.15[a]
07 days	9.84 ± 0.73[b]	8.99 ± 0.92[b]	9.87 ± 0.31[b]	11.21 ± 0.11[a]
14 days	9.92 ± 0.34[a]	8.14 ± 0.27[b]	9.42 ± 0.75[a]	9.62 ± 0.55[a]
28 days	9.82 ± 0.61[a]	8.01 ± 0.42[b]	9.34 ± 0.22[a]	9.14 ± 0.12[a]

The comparison amongst groups ($p<0.05$) is indicated by different letters (a, b) in the various lines.
EP= experimental periods; MTA, mineral trioxide aggregate.

out after 24 hours, 7, 14 and 28 days. After each period of immersion, the tubes were removed and placed into another flask with the same volume of new deionized water.

Measurement of pH was performed with a pH meter (Quimis, São Paulo, SP, BR) previously calibrated with solutions of known pH (4, 7, and 14) in constant temperature (25ºC). After removal of the specimens, the flasks were placed in a shaker (251, Farmen) for 5 seconds, before pH measurement. The control procedure included measuring the pH of the water in which no samples were immersed.

The calcium ions release was measured using an atomic absorption spectrophotometer (AA6800, Shimadzu, Tokyo, JPN), equipped with a calcium specific hollow cathode lamp as described by Vasconcelos et al.[17] (2009).

Statistical analysis

The date was submitted to ANOVA one-way to all experimental evaluation ($p=0.05$).

RESULTS

Flow rate

The results showed that the mean diameters of the materials WMTA, SEAL, SE10 and SE20 were 13.06mm, 29.40mm, 17.08mm and 16.80mm, respectively (**Table 2**). SEAL had the highest flow rate compared with the other materials ($p<0.05$). There was no significant difference between SE10 e SE20. WMTA had flow rate significantly lower than the other ($p<0.05$).

pH and Calcium Ion Release

The mean pH values for the materials evaluated in the different experimental periods are described in **Table 3**. SE20 presented the highest pH value compared with the other materials in 24 hours and 7 days periods ($p<0.05$). In 14 and 28 dyas, SEAL showed the lowest pH level ($p<0.05$), but WMTA, SE10 and SE20 were similar each other ($p>0.05$). **Table 3** shows the mean pH values for the materials in the different experimental periods

In relation to calcium ion release, SE20 and WMTA showed similar results in all periods and the higher values than SEAL and SE10 ($p<0.05$). **Table 4** shows the mean calcium ion release values for the materials in the different experimental periods.

DISCUSSION

In the present study, MTA (Mineral Trioxide Aggregate) was added to salicylate-resin based cement (Sealapex) in different proportions in order to evaluate flow rate, calcium ion release and pH. The incorporation of MTA into Sealapex modified the properties of this cement, so the null hypothesis (H0) was rejected.

Salicylate-resin based cements have limitations when used in places with moisture, such as the perirradicular region[3,4,7]. Therefore, calcium silicate-based materials have indications to be used in humid areas, because in contact with moisture, calcium oxide converts into calcium hydroxide[13-15,18], releasing calcium ions in the region[19]. However, the difficulty of manipulation and the relatively long setting time make its use clinically impossible[20].

To perform retrograde filling, it is desirable that the flow of the material is reduced, due to local humidity and so that solubilization and displacement of the retrograde cavity do not occur[13-15]. In this study, only the SEAL group presented high flow (above 20mm in diameter), according to specifications of ADA nº 57 and ISO[21.22], ideal for the purpose of endodontic filling. However, for use in retrograde cavities, it is possible that there is a solubilization of this cement, due to non-adaptation to the cavity[13]. The proposed alternative of the incorporation of the MTA,

Table 4 Calcium ion release (in mg/L) observed in the materials studied after different experimental periods.

EP	WMTA	SEAL	SE10	SE20
24 hours	17.65 ± 3.17[a]	3.51 ± 2.03[c]	7.51 ± 1.03[b]	15.32 ± 1.96[a]
07 days	18.46 ± 4.84[a]	3.12 ± 1.07[c]	7.88 ± 1.07[b]	17.61 ± 1.62[a]
14 days	13.24 ± 2.60[a]	2.98 ± 0.48[c]	7.70 ± 0.48[b]	12.52 ± 0.70[a]
28 days	12.41 ± 1.37[a]	2.31 ± 0.95[c]	7.34 ± 0.35[b]	11.09 ± 0.62[a]

The comparison amongst groups ($p<0.05$) is indicated by different letters (a, b, and c) in the various lines.
EP= experimental periods; MTA, mineral trioxide aggregate.

regardless of the quantity, to Sealapex cement reduced the flow values, making it feasible to be used in retrograde fillings.

In contact with tissue fluid, MTA is converted into calcium hydroxide and then dissociates into calcium and hydroxyl ions, resulting in increased pH[23]. The alkaline pH is associated with antimicrobial properties[10] and the absence of microorganisms in the periapical region facilitates the formation of the apical barrier[24,25]. In the present study, the groups in which 20% of MTA was added to Sealapex cement, evaluated 24h and 7 days after treatment, demonstrated the most alkaline pH values. However, in the later analyses, all groups in which the MTA was present showed pH values above 7.0. This is interesting because the maintenance of an alkaline pH for a prolonged period favors the formation of mineralized tissue in the apical region[26,27].

Studies show that the maintenance of alkaline pH allows the neutralization of lactic acid from osteoclasts and contributes to the formation of mineralized tissue by the activation of alkaline phosphatases and a positive influence on osteoblastic cell metabolism [24,28,29].

Calcium ion is the main chemical compound released by MTA when this material encounters aqueous medium[24,30,31]. In this study, regardless of the evaluation period, the group that was added 20% of MTA to Sealapex demonstrated the highest value of calcium ions, essential for the mineralization of hard tissue[32].

Further studies are necessary to evaluate other endodontic sealers associated with MTA, different amounts of the incorporation of MTA into resin-based cements, the length of permanence of the alkaline pH in the periapical region, bone formation and apical permeability of the mixture of materials used in this work.

CONCLUSION

We concluded that the incorporation of 20% of MTA into Sealapex sealer proved to be an association with better flow and handling than WMTA, with satisfactory potential for alkalinization and calcium release.

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