Influence of powder-liquid ratio on the physicochemical properties of MTA Repair HP cement

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ABSTRACT

Introduction: MTA Repair HP has been recently developed. The aim of this study was to evaluate setting time, radiopacity and solubility of HP Repair MTA in powder-liquid ratio according to manufacturer's instructions (MTA HP+, 0.8 g powder and liquid 320µl) or less powder (MTA HP- 0.7 g powder and liquid 320 µl) compared to MTA Angelus. **Material and methods:** The radiopacity was assessed by radiographs of materials in relation to an aluminum scale. Setting time was evaluated by Gilmore needles; and solubility, after immersing the material in distilled water (7 days). Data were analyzed by using ANOVA and Tukey tests with a 5% significance level. **Results:** The initial and final setting times were longer for MTA HP- than MTA HP+ and MTA Angelus ($p < 0.05$). MTA HP+ and MTA HP- had higher solubility than the MTA Angelus ($p < 0.05$). The radiopacity values of MTA HP+ and MTA HP- were lower than the MTA Angelus ($p < 0.05$). **Conclusions:** Decreasing the amount of powder in powder-liquid ratio of MTA HP results in longer setting time, with no change in other properties evaluated. MTA HP had lower radiopacity than MTA Angelus.

Keywords: Endodontics. Root Canal Preparation. Dental Pulp Cavity.

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Introduction

Mineral Trioxide Aggregate (MTA) is indicated for root perforation repair,[1] as well as for retrofillings, internal resorptions, pulpotomy, pulp protection and apexification.[2] The main component of MTA is Portland cement, which is basically composed of tricalcium silicate and dicalcium silicate,[3] in addition to small quantities of calcium sulphate and tricalcium aluminate[4]. The radiopacifier is bismuth oxide[5,6] that provides a radiopacity value in accordance with the ANSI/ADA Specification No.57/2000.[7] It is presented in the form of powder and liquid (distilled water), and after manipulation, the setting time is approximately 3-4 hours.[8] The properties of MTA depend on the particle size, powder-liquid ratio, temperature and adequate manipulation.[9,10] Some properties such as the "sandy" and granular consistency[11] may be enhanced, as well as the disadvantages attributed to bismuth oxide, such as pigmentation[5] and reduced mechanical stability.[12]

Recently, a new formulation denominated MTA Repair HP (MTA "High Plasticity") was developed; and according to the manufacturer, it presents greater plasticity than MTA Angelus. MTA Repair HP is composed of tricalcium silicate, dicalcium silicate, tricalcium aluminate, calcium oxide and calcium tungstate, which replaced the radiopacifier, bismuth oxide, in MTA Angelus. The liquid is based on water and a plasticizer. According to the manufacturer, the setting time is approximately 15 minutes when it is kept in a humid environment. Few studies have been developed on the powder-liquid ratio in the formulation of MTA[13,14,15,16] and relative to MTA Repair HP, there are no studies in the literature. Nevertheless, it is well known that changing the powder-liquid ratio affects the physicochemical properties of the cement.[17] Cavenago et al.[13] (2014) observed that the reduction of powder quantity in the powder-liquid ratio resulted in an increased setting time, solubility and pH; whereas the increase of powder quantity resulted in an increased radiopacity.

The aim of this study was to evaluate the physicochemical properties that include the setting time, radiopacity and solubility of MTA Repair HP in comparison with MTA Angelus, both in the powder-liquid ratio indicated by the manufacturer, and a modification to MTA Repair HP, in an attempt to facilitate manipulation and increase the working time. The null hypothesis was that the addition of less powder to the liquid would result in a significant improvement in the physicochemical properties.

Material and methods

The materials used in this study were divided into the following groups: MTA Angelus (Angelus Soluções Odontológicas, Londrina, PR, Brazil) manipulated in accordance with the manufacturer's instructions; and the groups for MTA Repair HP (Angelus Soluções Odontológicas, Londrina, PR, Brazil) were denominated MTA HP+, manipulated in accordance with the manufacturer's specifications (0.8 g powder to 320µl liquid); and denominated MTA HP-, manipulated with a smaller quantity of powder and the same amount of liquid (0.7 g powder to 320µl liquid) (Table 1).

Table 1. Composition and proportions used in Groups MTA Angelus, MTA HP+ and MTA HP-.

Material	Quantity of powder	Quantity of liquid	Composition
MTA Angelus	0,8 g	320µl	Powder: Tricalcium silicate, dicalcium silicate, calcium sulphate, tricalcium aluminate and bismuth oxide Liquid: Distilled Water
MTA HP +	0,8 g	320µl	Powder: Tricalcium silicate, dicalcium silicate, calcium sulphate, tricalcium aluminate and bismuth oxide, calcium tungstate Liquid: Water, plasticizer
MTA HP -	0,7 g	320µl	

Setting Time

The materials were manipulated and inserted into metal molds with an internal diameter of 10 mm and 1 mm high (n=6). According to the ISO 6876:2002 specifications, the initial setting time was determined by using a Gilmore needle with a mass of 100 ± 0.5 g and diameter of 2 ± 0.1 mm supported on the cement surface; while to obtain the final setting time a Gilmore needle with a mass of 456 ± 0.5 g and 1 ± 0.1 mm in diameter was used. During the tests the molds were kept in an oven at 37°C and 95% humidity. The setting time of each cement was determined based on the mean time between manipulation of the cement and time elapsed until each needle no longer marked the surface of the test specimens.

Solubility

According to Carvalho-Junior et al.[18] (2007), the use of smaller samples may be a feasible alternative for evaluating the solubility of retrofilling cements, based on ANSI/ADA Specification No. 57.[7] Based on these statements, the samples (n = 10) were fabricated with standardized dimensions of 1.5 mm thickness and 7.75 mm in diameter, containing a nylon thread incorporated into the mass of the material. The initial mass of each sample was determined by using a precision balance of 0.0001 g (HM - 200 A & D Engineering, Inc., Bradford, MA, USA). Then, the specimens were immersed in flasks containing 10 mL distilled water, and remained stored in an oven at 37°C for 7 days. After this period, the samples were removed from the receptacles, placed in a dehumidifier, and weighed daily until a stable mass was obtained. The loss of mass was expressed in percent of initial mass.

Radiopacity

The radiopacity test was based on the ISO 6876 specifications.[19] The samples (n = 5) of each material were fabricated and standardized, measuring 10 mm in diameter and 1 mm thick. Radiopacity was determined by means of a digital radiographic sensor (RVG 6100 Digital Radiography System, Kodak, France). One sample of each material was placed on the radiographic sensor together with the millimetric aluminum scale, and the set was radiographed by using an X-Ray appliance (Focus 50540 Instrumentarium Dental, Tusula, Finland) operated at 60 kV, 7 mA, 0.32 pulses per second and focal distance of 33 cm. The images obtained were evaluated by using the Image J software, with the purpose of determining the radiopacity equivalence of each sample in relation to the aluminum scale (mm Al).

Statistical Analysis

The results were statistically analyzed by ANOVA and the Tukey post-test at a 5% level of significance.

Results

Setting Time

The initial and final setting times of MTA HP- were longer than those of MTA HP+, and both showed a longer setting time than MTA Angelus, presenting statistically significant difference ($P < 0.05$) (Table 2).

Table 2. Mean and standard deviation of solubility (%); radiopacity (mm Al), and setting time (minutes) for the Groups MTA Angelus, MTA HP+ and MTA HP-.

Material	Solubility (%)	Radiopacity (mm Al)	Setting Time (minutes)	
			Initial	Final
MTA Angelus	0,0300 ± 0,07348[a]	5,967 ± 0,7474[a]	24,33 ± 4,179[a]	170,7 ± 1,633[a]
MTA HP +	1,028 ± 0,3362[b]	2,724 ± 0,2266[b]	47,67 ± 6,831[b]	208,0 ± 30,05[b]
MTA HP -	1,371 ± 0,4360[b]	2,330 ± 0,09381[b]	87,00 ± 0,0[c]	240,0 ± 0,0[c]

* Different subscript letters indicate statistically significant difference at 5% between the materials.

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Solubility

The cements MTA HP+ and MTA HP- showed no significant difference between them (P > 0.05) and higher than those of MTA Angelus (P < 0.05) (Table 2).

Radiopacity

MTA HP- and MTA HP+ showed no significant difference (P > 0.05). However, both showed lower radiopacity than that of MTA Angelus (P < 0.05) (Table 2).

Discussion

The setting time observed for MTA Repair HP was significantly longer than MTA Angelus. This may be explained because in addition to distilled water, the liquid of MTA Repair HP contains a thickening agent. When manipulated with a smaller quantity of powder than that recommended, the setting time was significantly longer. Similar results were observed in studies with MTA, which observed a shorter setting time with a increased powder quantity in relation to water.[9,10] This may be related to the smaller quantity of liquid required for hydrating the cement.[20] Cavenago et al.[13] (2014) observed that the setting time was shorter when a smaller proportion of water was used during the manipulation of MTA Angelus.

The solubility values of all the materials evaluated were in accordance with the ANSI/ADA 57 specifications,[7] in which an endodontic cement must not present solubility exceeding 3%. The solubility of both MTA Repair HP groups was higher than MTA Angelus. However, MTA HP- showed no significant difference when compared with MTA HP+. These results differed from those found by Cavenago et al.[13] (2014), who observed that the solubility value presented by MTA manipulated in the powder-liquid ratio of 2:1 was significantly higher when compared with that of the ratio 3:1, in accordance with the manufacturer's specification. Fridland & Rosado[21] (2003) observed that a higher quantity of water in the manipulation of the cement promoted greater solubility and porosity. This means that the quantity of water used in manipulation has a direct effect on solubility when the material is in contact with an aqueous environment. Modified powder-liquid ratios may improve handling, but a smaller quantity of water available for hydration may promote non-reacted particles in the hydrated cement.[4]

The radiopacity observed for MTA HP+ and MTA HP- were lower than those of MTA Angelus. This may be justified by the radiopacifying agent, because MTA Angelus contains bismuth oxide and MTA Repair HP, calcium tungstate. Hungaro Duarte et al.[6] (2009), studied different radiopacifying agents added to Portland cement in the proportion of 20% by weight of radiopacifying agent to 80% cement, and found that the radiopacity provided by the association with calcium tungstate was considerably inferior to that observed for the association with bismuth oxide. Calcium tungstate as radiopacifying agent is a good alternative to bismuth oxide, because it does not present cytotoxicity,[22] however, the quantity of this radiopacifier present in MTA HP does not make it possible to obtain radiopacity values higher than 3 mm Al in compliance with that established by ISO 6876.[19] Bosso-Martelo et al.[23] (2016) evaluated the physicochemical properties of calcium silicate cements associated with different radiopacifying agents and observed that calcium tungstate showed a similar radiopacity to bismuth oxide when used in the proportion of 30% of radiopacifier to 70% of calcium silicate cement.

Conclusion

In conclusion, the alteration in the powder-liquid ratio of MTA HP resulted in a longer setting time, but did not change the other properties evaluated. MTA HP showed a lower radiopacity than MTA Angelus. Further studies must be conducted to verify other physicochemical and biologic properties, including in vitro and in vivo tests.

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