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Original research

Effect of different water-to-powder ratios on the dimensional stability and compressive strength of mineral aggregate-based cements

Purpose
The aim of this study was to evaluate the effect of different water-to-powder ratios on the dimensional stability and compressive strength of Portland cement and Mineral Trioxide Aggregate (MTA).

Materials and Methods
Five different volumes of distilled water (0.26; 0.28; 0.30; 0.33 and 0.35 mL) were used for every 1 g of the cements. Twelve samples (12 mm long x 6 mm in diameter) were prepared in Teflon molds. After measuring the initial length, the specimens were stored in distilled water for 24 hours or 30 days. At the end of these time intervals, the specimens were measured again, and the dimensional change was calculated. The same samples used in the previous test were submitted to compression in a universal test machine (1 mm/min^{-1}).

Results
Analysis of the dimensional stability results showed no statistical difference between the cements, proportions and time intervals tested, or between their interactions. After 24 hours, MTA was more resistant than Portland cement ($p<0.05$). At 30 day-period, both cements had similar, and significantly higher resistance than they did at 24 hours ($p<0.05$).

Conclusion
The powder/water ratio had no influence on the dimensional stability of cements. Compressive strength of Portland cement was affected at the proportions of 0.30 and 0.35 mL/g.

Keywords: *Portland cement; mineral trioxide aggregate; dimensional stability; compressive strength*

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Introduction

An ideal retrofilling material must be capable of sealing the pathological communications between root canal system and the surrounding tissues, and the presence of moisture must not interfere in its sealing capacity (1-3). This material should be biocompatible, easy to handle, have adequate radiopacity, be minimally insoluble and dimensionally stable (4-6). In spite of the evolution of cements for endodontic application have presented over the last few years, there is still no material that meets all these requirements (7).

Mineral Trioxide Aggregate (MTA) was developed in the 1990s in Loma Linda, California (4), as a retrofilling cement and perforation sealant, and it was subsequently used in other diverse clinical applications due to its excellent physicochemical and biological characteristics (5,8). Studies

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have shown that MTA can induce mineralized hard tissue deposition (9), in addition to being well tolerated by living tissues (10). It is still outstanding among the other retrofilling cements because it presents good sealing capacity, expands during setting, releases calcium ions and because it is possible to use it in environments with relative moisture (2,11-13).

Conversely, MTA has some inconvenient features such as a long setting time that favors its solubilization and/or disintegration; or could even lead to its displacement from the retrograde cavity; and its sandy consistency, which makes it difficult to inset into the retropreparation and in areas with perforations (14,15).

Despite not being a Food and Drug Administration (FDA) approved commercial product to be used for medical purposes, Portland cement is widely used as an alternative material to MTA in laboratory studies due to its availability and low cost (11). Considering the similar characteristics between MTA and Portland cement, different vehicles and additives have been proposed to be used in association with these cements with the purpose of improving their physicochemical properties (14-16). In addition, the final consistency of both cements is similar, and it is directly related to the water/powder ratio used in the mixture (15). Fridland and Rosado (15) demonstrated the larger the quantity of water used in the manipulation, the greater would be the solubility of MTA.

The manufacturers of MTA recommend a water-to-powder ratio of 3:1, which hinders the cement's manipulation (17,18). Therefore, the ideal water/powder ratio still is a controversial point among researchers, which could be determinant in obtaining a better consistency and make the cement easier to manipulate. Moreover, it may directly influence the physical and mechanical properties of the cement in either a negative or positive manner.

The aim of this study was to evaluate the influence of different powder/water ratios on the dimensional stability and compressive strength of Portland cement and MTA. The null hypothesis tested was that the different water/powder ratios would not interfere in the physical-mechanical properties of the cements.

Materials and Methods

Specimen preparation

The cements tested in this study were the following: White Portland Cement (Irajazinho, Votoratin, São Paulo, SP, Brazil) (WPC) and White MTA (Angelus Soluções Odontológicas, Londrina, PR, Brazil - Lot nº 21584) (WMTA). As WPC has no radiopacifying agent, bismuth oxide was added to its formula at 20% by weight, the same percentage found in MTA.

WMTA and WPC were mixed using the water/powder (WP) ratio of 0.26, 0.28, 0.30, 0.33, and 0.35 mL of distilled water, establishing the following experimental groups: G1 - WMTA+0.26, G2 - WMTA+0.28, G3 - WMTA+0.30, G4 - WMTA+0.33, G5 - WMTA+0.35, G6 - WPC+0.26, G7 - WPC+0.28, G8 - WPC+0.30, G9 - WPC+0.33, and G10 - WPC+0.35 (n=6). Each sample contained 1.00 g of WMTA or WPC powder measured on an analytic balance. Using a micro-pipette, the appropriate amount of distilled water described above was added to each sample to achieve the proper WP ratio. The cements were hand-mixed on a nonabsorbent pad in a standardized fashion, totaling 120 specimens. Sixty specimens were used to perform the tests (dimensional stability and compressive strength) at the 24-hour period, and the other sixty specimens were used at the 30-day-period.

Dimensional stability

For each group, 12 cylindrical samples were obtained, measuring 12 mm high by 6 mm in diameter, in accordance with the Specification No. 57 of the American Dental Association (ADA) (19). For this purpose, Teflon molds were placed on a glass slide measuring 1 mm thick by 25 mm wide and 75 mm long, covered with a strip of cellophane paper. After this, the molds were filled with the manipulated cements, so that a slight excess of material could be verified at their upper extremity. After filling, another glass slide, also covered with a strip of cellophane paper was placed over the top surface of the mold. The set was kept firmly united by means of a C-shaped clip. After the elapse of 5 minutes from the time of starting the mixture, the set was transferred to an oven at 37 ± 1 ºC, with relative humidity of 95%. After 24 hours, the set was removed from the oven and the sample surfaces were smoothed with water abrasive paper #600 (3M, Sumaré, SP, Brazil), under abundant cooling with distilled water. On conclusion of this stage, the samples of each group were removed from their molds, their lengths measured with a digital pachymeter (Digimess, São Paulo, SP, Brazil), and the measurements were recorded. Right after this, the samples were placed in individual receptacles containing 30 mL of distilled and deionized water, identified by sample group and number, and were kept in the oven at 37 ± 1ºC, for 24 hours or 30 days. Afterwards, the samples were removed from the receptacles, the excess water was removed with the aid of absorbent paper, and a new length measurement was made.

The dimensional change was calculated using the following formula: $[(C_{24\ hours\ or\ 30\ days} - C \times 100/C)]$, where $C_{24\ hours\ or\ 30\ days}$ is the sample lengths after elapse of 24 hours or 30 days, and C is the initial sample length (16). The dimensional change of the groups was established by means of the arithmetic mean of 6 repetitions performed. The variables considered for analysis were: cement (WMTA and WPC); time intervals (24 hours and 30 days) and WP ratios (0.26; 0.28; 0.30; 0.33 e 0.35 mL).

Compressive strength

The compressive strength of samples was determined by the method recommended by Standard Specification 6039:1981 of the British Standards Institution (BSI) (20).

The same samples used in the previous test were used, considering the same variables. After each time interval, they were removed from the receptacles, the excess water was removed with the aid of an absorbent paper towel, and the compressive strength was determined in a Universal test machine (Instron, Model 1334, Instron Corp., Canton, MA, USA) at a speed of 1 mm/min^{-1}. The maximum load necessary to fracture each sample was obtained and recorded. The compressive strength was calculated in megapascal (MPa) according to the following formula: $C = 4P / \pi D^2$, where "P" represented the maximum load recorded by the machine in Newtons (N) and "D" the diameter of the sample in millimeters (mm).

Statistical analysis

After verifying the normality of the sample (Shapiro-Wilk test), the values obtained in the dimensional stability and compressive strength tests were statistically compared with three-way analysis of variance (ANOVA), and the Tukey test ($p<0.05$). The statistical analysis was performed using the Graphpad Prism 4.0 Software program (GraphPad Software, La Jolla, CA, USA).

Results

Dimensional stability

Table 1 shows the mean dimensional stability values of each group.

The positive and negative mean values indicate expansion and contraction of the cements, respectively. In general, WPC and WMTA had expansion in 24 hours. However, after 30 days, WPC had contraction in all the WP ratios tested; and WMTA, expansion, with exception of G7 (0.28 mL). In spite of the different behavior between the two cements after 30 days, there was no significant differences among the variables tested, or between their interactions, demonstrating that the dimensional stability between the cements and the WP ratios tested were equivalent in all the situations .

Compressive strength

Table 2 shows the mean compressive strength values of each group.

Both cements tested with the different WP ratios had significant increase in compressive strength over the course of time, with the exception of G6 (0.26 mL), G9 (0.33 mL) and G10 (0.35 mL). WPC and WMTA had similar compressive strength at 30 day-period, however, the mean values of WMTA were significantly higher at 24 hours in comparison with WPC ($p<0.05$), except for G7 (0.28 mL) . The WP ratios in the G3 and G5 significantly diminished the compressive strength of WPC cement in comparison with G8 and G10 (WMTA), at the 24 hour-period ($p<0.05$).

Discussion

The aim of this study was to evaluate the influence of different WP ratios on the dimensional stability and compressive strength of Portland cement and MTA. According to the results obtained in this study, the null hypothesis tested was partially accepted, since the two cements presented similar dimensional stability, however, the compressive strength of the cements was affected by the WP ratio.

The microstructure of hydraulic cements, such as Portland cement and MTA, is basically formed of pores and channels that serve to diffuse water within the cement mass, guaranteeing its continuous process of hydration until final hardening (21,22). The characteristics of this microstructure may be influenced by diverse factors, such as the pressure used during manipulation of the cement, the method of mixture, and the water/powder ratio, and these factors are difficult to control (15). During manipulation of the cement, for example, the greater the force applied in the cement, the more compacted it will be, and consequently, the less presence of canals that form its microstructure there will be (23). Moreover, the less incorporation of bubbles of air there will be, thus diminishing the presence of porosities (15,23). The absence or diminishment of these structures will make hydration of the cement difficult, compromising its performance (22).

During the setting process of hydraulic cements, the water is not only incorporated into the powder, but it initiates a process in which its molecules bind chemically to diverse phases of these materials (22). Portland cement and MTA set, and become more resistant as time passes, presenting a faster initial hardening, but one that lasts, and becomes

Table 1. *Mean values and standard deviation (SD) for dimensional stability (%) of the different groups at the two-time intervals of analysis.There was no significant difference among groups (cements/WP ratios) (3-way ANOVA, the Tukey test, p<0.05). n=6.*

Cements	WPC					WMTA				
	1 (0.26mL)	2 (0.28mL)	3 (0.30mL)	4 (0.33mL)	5 (0.35mL)	6 (0.26mL)	7 (0.28mL)	8 (0.30mL)	9 (0.33mL)	10 (0.35mL)
24 h	-0.334	0.000	0.048	0.145	0.236	0.048	-0.096	-0.191	0.001	-0.048
	(0.334)	(0.183)	(0.214)	(0.352)	(0.215)	(0.117)	(0.148)	(0.148)	(0.256)	(0.217)
30 days	-0.284	-0.050	-0.328	-0.292	-0.190	0.145	-0.429	0.375	0.967	0.239
	(0.178)	(1.250)	(0.212)	(0.261)	(0.394)	(0.623)	(0.398)	(0.384)	(3.731)	(0.492)

Table 2. *Mean values (MPa) and standard deviation (SD) for compression strength of the different groups at the two-time intervals of analysis. Different capital letters in columns and lowercase letters in lines indicate statistically significant difference (3-way ANOVA, the Tukey test, p<0.05). n=6.*

Cements	WPC					WMTA				
Groups	1	2	3	4	5	6	7	8	9	10
Periods	(0.26mL)	(0.28mL)	(0.30mL)	(0.33mL)	(0.35mL)	(0.26mL)	(0.28mL)	(0.30mL)	(0.33mL)	(0.35mL)
24 h	38.86^{A,a}	52.07^{A,a}	34.35^{A,a}	45.82^{A,a}	36.50^{A,a}	60.06^{A,b}	40.06^{A,a}	52.87A,b	56.08^{A,b}	62.93^{A,b}
	(6.44)	(4.96)	(5.46)	(5.05)	(5.57)	(9.09)	(19.06)	(9.58)	(15.15)	(13.77)
30 days	74.90^{B,a}	73.56^{B,a}	56.95^{B,a}	75.30^{B,a}	50.20^{B,a}	57.32^{A,a}	58.08^{B,a}	59.76^{B,a}	59.51^{A,a}	53.63^{A,a}
	(12.33)	(6.22)	(10.94)	(20.70)	(14.95)	(9.89)	(10.99)	(10.38)	(13.12)	(8.16)

slower during the following days (24). The longer the time of hydration, the more organized and rigid will be the crystalline microstructure that can be formed, improving the physicochemical properties of these cements (24).

Numerous studies have demonstrated that the compressive strength of MTA was significantly lower than that of amalgam after 24 hours (4,5). However, after 3 weeks, there was no significant difference between the materials relative to compressive strength (4,5). These results corroborated the findings of the present study, because Portland cement and MTA presented significantly higher compressive strength values at 30 days in comparison with the initial period (24 hours).

The two cements are basically composed of tricalcium silicate ($3CaO\ SiO_2$) and dicalcium silicate ($2CaO\ SiO_2$) (11), with the addition of bismuth oxide (Bi_2O_3) to give them radiopacity (25). Because the hydration of dicalcium silicate is slower than that of tricalcium silicate, the compressive strength and resistance to displacement attain their maximum values several days after their mixture (26,27).

The two cements compared in the present study presented similar compressive strength values at 30 days, however, MTA showed higher strength values in the initial period of analysis. According to Kao et al. (28), Portland cement has a more delicate microstructure than MTA, due to the different temperatures at which the cements are sintered, leading to the formation of distinct phases and oxides from those found in conventional MTA. Furthermore, the raw materials used in their purification process, guarantee that MTA has advantages in comparison with other mineral aggregate-based cements (28).

Considering the different WP ratios, there was no statistically difference among the experimental groups, except for G3 (0.30 mL) and G5 (0.35 mL), which significantly decrease the strength values of Portland cement in comparison with the WMTA groups at the 24 hour-period. Studies have previously reported that higher ratios of water lead to greater porosity in the final microstructure of the cement, which at first sight would be beneficial, because of network of intercommunicating pores within the cement would allow greater diffusion of water molecules, and consequently, a better and more accentuated hydration during the setting process (15,22). However, Basturk et al. (29) demonstrated a negative correlation between the quantity of pores present and the mechanical strength of mineral aggregate-based cements.

Basturk et al. (30) demonstrated that the increase in the WP ratio from 0.34 mL to 0.40 mL was sufficient to significantly diminish the compressive strength of MTA (30). Shojaee et al. (31) also reported that higher WP ratios (0.40 and 0.50) lead to lower compressive strength of mineral aggregate-based cements. According to Fridland & Rosado (15), a ratio higher than 0.33 mL to 1 g of MTA powder is incapable of producing a sufficiently viscous mass to be manipulated in a clinically adequate manner, making it difficult to insert the cement into the area to be treated. On the other hand, a ratio lower than 0.26 mL did not allow a cement with adequate physicochemical properties to be obtained (15). However, in the present study, these situations were only observed for Portland cement, in which the WP ratios of 0.30 mL and 0.35 mL significantly diminished the strength values of the cement between the time intervals of 24 hours and 30 days.

Considering the dimensional stability, the different WP ratios caused no significant changes in the cements tested. According to the results obtained in this study, the positive values indicated expansion of the cements, and negative values, the opposite. After the initial 24 hours had elapsed, both cements presented expansion, a common fact, because the hydration process of cements leads to the diffusion and chemical bonding of the water molecules with the cement particles, forming a semi-solid mass of colloidal silica that continues to expand as time passes (22). After 30 days MTA continued to present expansion, however, Portland cement presented contraction, irrespective of the WP ratio tested. The process of expansion of these cements during their setting time is water-dependent and is directly associated with the cement capacity to absorb water from the medium and the use of water in the formation of hydrates during their manipulation (32). The greater this hydration capacity, the more complete will be the cement hardening, and the more satisfactory will be its properties. Although there were no differences between the dimensional stability values of the cements, the expansion of MTA, even after the initial time interval of analysis may have influenced the compressive strength results, which were significantly higher in the initial 24 hours, in comparison with Portland cement. It is worth pointing out that only for the ratio of 0.28 mL was there no significant difference in the compressive strength values of the cements; the same proportion in which MTA presented contraction, 30 days after its manipulation.

Due to the variety of types and commercial brands of mineral aggregate-based cements existent on the market, in-depth comparisons between the studies conducted up to now, have been difficult to carry out in an appropriate manner. Furthermore, samples of Portland cement may present even greater variations, particularly due to the considerable number of manufacturers, limiting the comparisons between scientific findings. In spite of these difficulties, it is justifiable to emphasize that different WP ratios may affect certain properties of this class of cements, however, others do not appear to present significant changes. Such information is crucial for the clinicians when they use mineral aggregate-based cements in areas that receive forces from the condensation of restorative materials or occlusion, as the compressive strength of this type of material may be affected by WP ratio (30).

Conclusion

Based on the results obtained, and considering the limitations of this study, it may be stated that the different water/powder ratios did not influence the dimensional stability of the cements tested, however, MTA was more resistant to compression than Portland cement in the initial 24 hours. Other variables such as time and manner of manipulation, and methods of application of the cement must be investigated before these results are extrapolated to clinical situations.

Türkçe öz: *Farklı toz/su oranlarının mineral agregat esaslı simanların boyutsal stabilite ve basınç dayanımları üzerindeki etkileri. Amaç: Bu çalışmanın amacı, farklı toz/su oranlarının Portland simanı ve mineral trioksit agregat'ın (MTA) boyutsal stabilite ve basınç dayanımları üzerindeki etkilerinin araştırılmasıdır. Gereç ve Yöntem: Simanların 1'er gramı 5 farklı hacimde (0,26; 0,28; 0,30; 0,33; ve 0,35 mL) distile su ile karıştırılmıştır. Teflon kalıplar içinde 12'şer örnek (12 mm yükseklik X 6 mm çap) hazırlanmıştır. Başlangıçtaki yükseklikler ölçüldükten sonra, örnekler 24 saat veya 30 gün distile su içinde saklanmıştır. Bu sürelerin sonunda, örnekler*

tekrar ölçülmüş ve boyutsal değişim hesaplanmıştır. Daha sonra Universal test cihazı yardımıyla örneklerin basınç dayanımı ölçülmüştür (1 mm/dak-1). Bulgular: Boyutsal stabilite sonuçları değerlendirildiğinde, simanlar, orantılar, süreler ve bunların interaksiyonları arasında istatistiksel bir fark saptanmamıştır. 24 saat sonunda MTA'nın Portland simanından daha dayanıklı olduğu gözlenmiştir (p<0,05). 30 gün sonunda her iki simanın benzer basınç dayanımı sonuçları verdiği ve 24 saat sonundakine kıyasla anlamlı ölçüde daha dayanıklı oldukları bulunmuştur (p<0,05). Sonuç: Toz/su oranı simanların boyutsal stabilitesini etkilememiştir. 0,30 ve 0,35 mL/g oranlarında karıştırıldığında, Portland simanının basınç dayanımının etkilendiği gözlenmiştir. Anahtar kelimeler: Portland simanı; mineral trioksit agregat; boyutsal stabilite; basınç dayanımı

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