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ORIGINAL ARTICLE

Effect of different concentrations of propylene glycol on the physical-chemical properties of MTA

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ABSTRACT

Aim: *This study evaluated the effect of different concentrations of propylene glycol (PG) on the dimensional change (DC), compressive strength (CS), solubility and pH of MTA.*
Methodology: *Groups were formed according to the proportion of distilled water (DW) and PG in MTA manipulation: G1 (100% DW); G2 (80% DW+20% PG); G3 (50% DW+50% PG); G4 (20% DW+80% PG). The tests were guided by ISO and BSI standards. DC and CS tests were performed after 24 h and 30 days of samples storage in water. In the solubility test, rings filled with the cements were weighed after setting and after 7 days. The pH was measured after 3 and 24 h, and 30 days. The data were analyzed with the ANOVA, Tukey's and Paired t tests (α=5%).*
Results: *After 24 h and 30 days, the DC and CS were similar among groups, except the CS for G3 in 30 days. After 7 days, G1 gained weight when hydrated, differing from G3 and G4 ($p<0.05$). When weighed dehydrated, G2 was different from the other groups ($p<0.05$). After 3 h, G1 had lower pH values in comparison with G2, G3 and G4 ($p<0.05$). At the 24-h period, G2 had higher pH values than G1 and G2 ($p<0.05$). After 7 days, there was no difference among groups.*
Conclusions: *PG did not affect the MTA dimensional change and compressive strength (except for G3). However, PG reduced MTA solubility (G2) and increased its pH after 3 hours.*

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KEYWORDS *compressive strength, dimensional change, physicochemical properties, propylene glycol, mineral trioxide aggregate*

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Introduction

Mineral Trioxide Aggregate (MTA) was initially developed as a retrograde filling material, and to seal the communications between the root canal system and periodontium. Gradually, MTA was also used in pulp-capping, pulpotomies, apexification, apexogenesis and as filling material, mainly due to its biological properties (1). Despite its wide indication, MTA presents poor handling properties, extended setting-time and wash-out possibility (2, 3). In order to improve its physical-chemical properties, the use of several additives in MTA manipulation have been investigated, and propylene glycol (PG) is one of them (4-11).

PG is a non-carcinogenic and non-genotoxic alcoholic vehicle widely used in dermatology (12). It is considered ideal for many topical preparations, used as solvent, skin conditioner, fluidizing, and in numerous fragrances compositions (13).

In endodontics, PG is frequently used as a vehicle for calcium hydroxide intracanal dressing, allowing a slower hydroxyl and calcium ions dissociation because of its consistency (14). Considering the benefits of mixing PG to calcium hydroxide, Holland et al. (4) used it as an MTA vehicle for root canal obturation. These authors investigated the influence of distilled water (DW) or PG on the periapical response of dog's tissues. Both mixtures had similar biological behavior and were biocompatible. In addition, other studies have reported that PG improved several properties of MTA, such as the sealing ability in furcal perforations against bacterial leakage after 30 days (5) and dentin bond strength (7). In the ratio of 80% DW/20% PG, this vehicle improved several properties of MTA, such as setting-time, flowability, pH, and calcium ions release (6).

Given the studies found in the literature, using PG as a MTA vehicle seems to be promising (4-11). Although, different ratios of DW and PG may affect the physical-chemical properties of MTA, since crystal hydration is an important factor for its setting reaction (2). The addition of high PG ratios results in a longer setting-time, leading to higher solubility and greater pores formation, compromising MTA mechanical strength (6, 15). Therefore, this study aimed to evaluate the effect of manipulating MTA with different concentrations of PG on its dimensional change, compressive strength, solubility and pH. The null hypothesis tested was that the different concentrations of PG would not interfere on the MTA properties mentioned above.

Materials and Methods

White MTA Angelus (Angelus, Londrina, Brazil) was manipulated with different concentrations of DW and PG, and distributed into four groups, as follows: G1 (100% DW); G2 (80% DW and 20% PG); G3 (50% DW and 50% PG) and G4 (20% DW+80% PG). The DW-PG ratio was determined by volume, and the powder-liquid ratio was the same for all groups (1.0 g of MTA powder to 0.4 mL of liquid). After the cement was prepared as determined in each group, the physicochemical properties were evaluated according to the tests described below.

Dimensional Change

Twelve cylindrical samples for each group were fabricated in Teflon molds (3.58 mm in height, 3 mm in diameter), according to the methodology of Carvalho-Júnior et al. (16). The molds were placed on a glass plate (1 mm thick, 25 mm wide, 74 mm long) covered by a cellophane paper strip. Then, each mold was filled with the cements (MTA/different PG-DW ratios), leaving a slight material excess on the upper side. Another glass plate, also covered with cellophane paper strip, was placed on the top of each mold and the set was held tightly together by a "C" clip. All samples were transferred to a laboratory oven at 3± 1 °C and 95% relative humidity, five minutes after the manipulation has started. After 24 h, all samples were removed from the laboratory oven and their surfaces were polished with a 600-wet sandpaper (3M, Sumaré, SP, Brazil) under copious water irrigation. The initial length of each sample was measured with a thickness digital caliper (Mitutoyo America Corporation, Chicago, IL, USA). Next, the samples were immersed in individual flasks containing 2.24 mL of DW at 37±1 °C for 24 h and 30 days (16). Six samples from each group were used for both periods. After the experimental periods, they were removed from the flasks, dried with absorbent paper and measured again with the digital caliper. The dimensional change was calculated by the following formula:

$$[(C_{FINAL} - C/C)] \times 100$$

where C_{FINAL} is the final length of the sample (after the experimental periods), and C the initial length after setting (17).

Compressive Strength

The same samples (n=12) used in the dimensional change test were used to verify the mechanical strength of the cements. After 24 h and 30 days, the samples were removed from the flasks, dried with absorbent paper and the compressive strength was then evaluated using a Universal testing machine (Instron, Model 4444, Instron Corp., Canton, MA,USA) at a cross-head speed of 1 mm/min. The maximum load required to reach the failure point was recorded.

Compressive strength was calculated in MegaPascal (MPa) according to the equation: $C=4P/\pi D^2$, where P is the maximum load recorded by the testing machine in Newton (N), D is the sample diameter in millimeters (mm), and C is the maximum load in MPa (18).

Solubility

Twelve teflon rings molds (1.5 mm in thickness, 7.75 mm in internal diameter) were used following the methodology described by Carvalho-Júnior et al. (16). In each ring, a carbide drill nº 2 was used to create a through hole in the rings for posterior introduction of a nylon thread, which later allowed the suspension of the rings. Each ring was filled with the different MTA-like cements and kept in a laboratory oven at 37±1 °C and 95% relative humidity for a period equivalent to three times the setting-time. Then, the samples were polished with a 600-wet sandpaper, gently dried in absorbent paper and weighed (initial hydrated weight) on a precision balance (AND model GR-202, Tokyo, Japan). After 24 h in a desiccator with silica, the rings were re-weighed (initial dehydrated weight). The samples were suspended by a nylon thread and placed in flasks containing 7.5 mL of DW and placed in a laboratory oven at 37±1 °C for 7 days (16). Following, the rings were removed from the flasks, gently dried and weighed (final hydrated weight). Samples were placed in a desiccator for 24 h and then, re-weighed (final dehydrated weight). The solubility corresponded to the samples weight loss, expressed as percentage, in comparison with the original samples weight.

pH

The pH of the DW in which the samples were immersed in the dimensional change test was measured in triplicate at 3 and 24 h, and 7 days. The measurements were obtained using a pHmeter (Micronal, PH-1700, São Paulo, Brazil) calibrated according to the buffer solution (pH 7.0 and 9.0) at 25±2 ºC temperature, which was kept constant using an air conditioner.

Statistical Analysis

Data were analyzed for normal distribution and homoscedasticity using the Kolmogorov-Smirnov's and Levene's test, respectively, followed by parametric statistical tests. Data from the groups in each period was analyzed using ANOVA and Tukey's tests. Data from the same group over time were analyzed using the t-test. All tests used an $\alpha=0.05$ significance level and all analyses were carried out using the SPSS 21 software (IBM SPSS Inc.; Chicago, IL, USA).

Results

The dimensional change and compressive strength mean values are expressed in Table 1.

Regarding the dimensional change anal-

Table 1

Mean values and standard deviation (SD) for dimensional change (%) and compressive strength (MPa) of the different groups at the two periods of analysis

Test/Groups	100% DW	20% PG	50% PG	80% PG
Dimensional change*	Mean SD	Mean SD	Mean SD	Mean SD
24 h	0.05±0.03^{Ab}	0.00±0.04^{Ab}	0.11±0.03A^{a}	0.11±0.11^{Aa}
30 days	0.17±0.03^{Aa}	0.24±0.02^{Aa}	0.29±0.13^{Aa}	0.25±0.06^{Aa}
Compressive strength*				
24 h	27.75±19.05^{Aa}	41.03±14.57^{Aa}	29.75±8.40^{Ab}	36.93±15.13^{Aa}
30 days	42.67±3.06A^{Ba}	42.72±6.19A^{Ba}	52.52±7.57^{Aa}	30.46±8.21^{Ba}

**Capital letters represent a significant difference among the groups (ANOVA and Tukey's test) and lowercase letters between the periods (t-test) (α=5%).*

DW: distilled water, PG: propylene glycol.

ysis, there was no statistical difference among groups in each evaluated period (p>0.05). Over the course of time, the samples from all groups presented increase in their dimensions, mainly for G1 (100% DW) and G2 (20% PG), which had a significant expansion from 24 h to 30 days (p=0.0007 and p=0.0002, respectively).

At the 24-hour period, all groups have similar compressive strength values (p>0.05). After 30 days, there was significantly different among the groups. G3 (50% PG) had the highest mean value, differing from G4 (80% PG) (p=0.0008). When the analysis was performed between the experimental periods in the same group, G3 (50% PG) increased its resistance significantly at the 30-day period (p=0.0099).

Table 2

Mean values and standard deviation (SD) for solubility (%) of the different groups. Negative values mean weight loss

Groups	7 days (hydrated)		7 days (dehydrated)	
	Mean	SD	Mean	SD
100% DW	0.0160^{Aa}	0.0097	-0.0032^{Bb}	0.0017
20% PG	0.0074A^{Ba}	0.0020	0.0048^{Aa}	0.0020
50% PG	-0.0017^{Ba}	0.0129	-0.0032^{Ba}	0.0129
80% PG	-0.0013^{Bb}	0.0039	-0.0057^{Ba}	0.0039

The capital letters represent a significant difference among the groups (in each column, ANOVA and Tukey's tests) and lowercase letters between the conditions (hydrated and dehydrated, t-test) (α=5%).

DW: distilled water, PG: propylene glycol.

The mean solubility values are shown in Table 2. After 7 days, G1 (100% DW) gained weight when hydrated, with statistically significant difference in comparison with G3 (50% PG) and G4 (80% PG), which lost weight (p=0.0194 and p=0.0221, respectively). When the dehydrated samples were compared among each other, it was observed that G2 (20% PG) gained weight, being statistically different from the other groups (p<0.05).

The pH mean values are expressed in Table 3. Statistical difference among groups after 3 and 24 h was observed (p<0.05). After 3 h, G1 (100% DW) had lower pH in comparison with G2 (20% PG), G3 (50% PG) and G4 (80% PG) (p=0.004, p=0.000, and p=0.000, respectively). At the 24-hour period, G3 (50% PG) had higher pH values, with statistical difference when compared to G1(100%) and G2 (20% PG) (p=0.002 and p=0.009, respectively). At the 7-day period, there was no statistical difference among groups.

When comparing the pH values among the experimental periods, at 7-days period, G1 (100% DW) presented significant higher pH mean values than in 24 h ($P = 0.0062$). G2 (20% PG) and G3 (50% PG) had higher pH values in the initial periods (3 and 24 h) than in the 7-day period of analysis (p<0.05).

Table 3

Mean values and standard deviation (SD) for pH of the different groups at the three periods of analysis

Groups	3 h		24 h		7 days	
	Mean	SD	Mean	SD	Mean	SD
100% DW	10.62^{Aab}	0.03	10.97^{Ba}	0.06	10.18^{Ab}	0.59
20% PG	10.79^{Ba}	0.06	11.00^{Ba}	0.16	9.93^{Ab}	0.68
50% PG	10.85^{Ba}	0.06	11.25^{Aa}	0.07	10.07^{Ab}	0.56
80% PG	10.86^{Ba}	0.08	11.07^{ABa}	0.08	10.42^{Aa}	0.73

Capital letters represent a significant difference among the groups (in each column) and lowercase letters among the periods (in each line) (ANOVA and Tukey's tests, α=5%).

DW: distilled water, PG: propylene glycol.

Discussion

This study evaluated the dimensional change, compressive strength, solubility and pH of MTA prepared with different DW/PG ratios. Based on the obtained results, the null hypothesis tested was partially accepted, since the mixture of different DW/PG ratios to MTA affected the evaluated properties, except for dimensional change.

The PG dilutions in water and the powder-liquid ratio used in the present study followed those used by Duarte et al. (6). However, only four of the five proportions proposed by them were used. As the cement never reached its complete setting when it was manipulated with 100% PG (6), it was decided to exclude this group from the study.

Although the proportion 80%DW/20%PG in MTA is the most investigated in the literature (6, 8, 9), it may not yet be assert that it is the best choice, due to some negative effects (9). For this reason, more studies with different PG proportions should be carried out, and also other properties need to be evaluated.

In this study, the effect of PG on the dimensional change of MTA was investigated for the first time. The experimental groups manipulated with PG had similar dimensional change among each other and in comparison with G1 (100%DW) in both periods of analysis. Although no statistical difference among groups was found, it was noted that the groups with PG had higher expansion mean values. This result relies on the humectant ability of PG, which keeps the hydration process of MTA for a longer period, leading to a late expansion (2, 9). Camilleri (2) showed that after a few hours, the hydration process of mineral aggregate-based cements promotes a significant volumetric change in this type of materials. The hydration process also influences the setting-time, and it has been shown that the more PG is added to the mixture, the longer the setting-time of the MTA (6, 8).

There are no published data available on the the dimensional change of MTA when PG was used as a vehicle; therefore, direct comparison with other studies was not possible. However, in relation to G1, several studies have reported an average percentage of expansion for MTA mixed with DW of 0.08% after 24 h (19), and 0.28% (20) and 0.30% (21) after 30 days, differing from the findings of the present study, which were inferior (0.05% in 24 h and 0.1% in 30 days). The possible explanation for the controversial results relies on the use of samples of smaller dimension in the present study, in addition to the different MTA commercial brand used to perform the tests. To evaluate the expansion of MTA is an interesting issue, as this property may increase the cement's sealing ability (21), and consequently, to reduce the possibility of leakage (20). However, an excessive expansion is undesirable, especially if the material is used as a root-end filling material, because it may lead to microcracks at the apical portion of the root (20).

ISO specification (17) states that the mean dimensional change in length of a sealer should not exceed 1.0% in shrinkage or 0.1% in expansion. In this study, G3 and G4 at the 24 h-period, and all groups after 30 days had greater dimensional change than the ISO recommendation (17).

When analysing the compressive strength test, at the 24-h period, PG groups were statistically similar to G1. After 30 days, G3 increased significantly its compressive strength (from 29.75 to 52.52 MPa) and had the highest mean value, differing only from G4 (30.46 MPa). Despite the statistical similarity with G1 (42.67 MPa) and G2 (42.72 MPa), G3 had the most appropriate mechanical performance. This finding is in agreement with the results of other studies, where the compressive strength of MTA significantly increased when the cement was manipulated with 50%PG (10, 11). It is valid to emphasize that a proper compressive strength is fundamental for MTA, as this material may be used for pulp-capping or as a coronal restorative material, because of the occlusal forces in which the cement is submitted (22).

The solubility of a specific material is the loss of mass during a period of immersion in water (16). Materials with high solubility may release irritant by-products into the medium and increase leakage risks (23). In the present study, all tested cements had a solubility percentage inferior than 3%, following the ISO recommendation (17).

When weighted hydrated after 7 days, G1 gained weight differing from G3 and G4, which lost weight. When the samples were weighted dehydrated, G2 gained weight being different from the other groups. These results were different from those found by Marciano et al. (9), where the group 20% PG had solubility of 0.25%. These findings might be explained by the different size of the samples used in each study. The authors of the mentioned study (9) used polytetrafluoroethylene rings (1.5 mm in thickness, 20 mm in internal diameter) filled with the cements and a nylon thread was placed inside the material. In the present study, Teflon rings with a smaller dimensions (1.5 mm, 7.75 mm) were used, as suggested by other studies (3, 16, 24). Also, the nylon thread passed through the holes created in the rings, and not within the material. The larger contact area between the material and the liquid, and the placement of the nylon thread inside the material probably induced the different results. The reduction of the dimensions of the Teflon rings aimed to decrease the amount of material used to perform the test, and showed to be compatible with the ISO standards (17).

ISO (17) also recommends the weight of the residues released by the cement's samples after the water in which they were immersed evaporates, not considering the possible absorption by the material. This feature is extremely significant for MTA, which has high water absorption capacity (2, 25). For this reason, samples were weighed before being placed in the desiccator, providing a weight value of the water absorbed (initial and final hydrated weight), showing the absorbent capacity of the cements (3, 25).

At the 3h-period, PG groups showed higher pH mean values than G1 (100% DW), and this result is in accordance with a previous study (6). After 24 h, the pH averages remained high, with emphasis on G3 (50%PG). At the 7-day period, there was no statistical difference among groups, in agreement with the results of previous studies (6, 9). When only the values were observed, this study showed higher pH values than those found by Duarte et al. (6). This variation might be attributed to the samples and solutions contact area. The authors used artificial resin teeth containing root-end preparations filled with the cements to perform the test. The methodology restricts MTA contact area with water, and consequently, lower pH values were found.

One of the main reasons for changing the MTA vehicle is to improve its consistency, which restricts it from being used as a filling material and also makes it difficult to insert it into cavities (3, 4). MTA sets by a hydration reaction to form a calcium silicate hydrate gel and calcium hydroxide. Calcium hydroxide will release Ca^{2+} ions and the decomposition of calcium silicate hydrate will generate an alkaline pH (2). Mixing PG to MTA reduces the amount of water available for the hydration reaction, resulting in a slower setting-time, and that process is probably sustained for a longer period (6, 8, 9). According to these authors, the interference with the setting-time might result in higher solubility, greater formation of pores and have a negative impact on the mechanical strength of the material. The present study has reported that the tested PG concentrations did not negatively affect the mechanical strength of MTA.

Conclusions

Considering the present results and the inherent limitations of the methodology used, it was concluded that the PG did not affect the MTA dimensional change and compressive strength (except for G3). However, PG reduced the MTA solubility (G2) and increased the pH of the medium after 3 hours.

Clinical Relevance

The ideal quantitity of propylene glycol to manipulate mineral aggregate-based cements still is a controversial point. This study investigated the effect of mixing different ratios of distilled water and propylene glycol on the physical-chemical properties of MTA in order to elucidate this matter.

Conflict of Interest

The authors deny any conflicts of interest related to this study.

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