G It Endo - vol. 21 - n. 2, pp. 67-70
Aprile-Giugno 2007

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Pervenuto in Redazione il 31 gennaio 2007
Accettato per la pubblicazione il 28 marzo 2007

Comparative analysis of solubility, pH, and composition of white and grey mineral trioxide aggregate (MTA) and Portland cement

ABSTRACT

Aim: the purpose of this study was to evaluate and compare the chemical composition, pH and solubility of white and gray mineral trioxide aggregate (MTA) and Portland cement.
Methodology: materials underwent atomic absorption spectroscopy to quantify their components. pH values were assessed with a pH-meter using a temperature-compensated electrode. Six samples of each material were used for the analysis of solubility. Samples were weighed and stored in distilled water at 37°C for 7 days and then weighed again.
Results: the three materials showed the same chemical components, except bismuth oxide, which is not found in Portland cement. Statistical analysis of results revealed significant differences in the relative proportion of components in each material. All samples had pH values close to 12.0, and Portland cement had a pH result significantly less alkaline than the ones found for white and grey MTA samples. None of the materials tested had significant solubility.
Conclusions:
- The materials studied had similar chemical compositions, except for the presence of bismuth oxide in white and grey MTA.
- There were significant differences in the amount of components in the different samples.
- All samples had pH values close to 12 immediately after mixing; Portland cement had significantly lower alkalinity than white or grey MTA.
- There were no significant signs of solubility in the three materials tested.

Key words:
Mineral trioxide aggregate (MTA), Portland cement, chemical composition, solubility.

INTRODUCTION

Mineral trioxide aggregate (MTA) was introduced in 1993 (1) and was extensively studied by Torabinejad and colleagues, and has been indicated for several uses, such as root-end filling (2-7), repair of perforations (8-10), and conservative dental pulp treatment (11, 12).
MTA is a powder composed of trioxides combined with other hydrophilic mineral particles that crystallize in the presence of humidity (13). Its pH value is 10.2, but this increases to 12.5 three hours after mixing, which promotes alkalinization of the medium. Therefore, MTA plays the role of an antimicrobial agent (3-6).
MTA has physical and chemical properties which depend on the number of particles, water-powder *ratio*, and temperature and air humidity. Its setting time is about 2 hours and 45 minutes; in the presence of humidity, it undergoes slight expansion and converts to a colloidal gel that crystallizes and later expands, promoting marginal adaptation. It provides better sealing than amalgam, IRM®, and Super EBA® (3-7, 9, 14). Its radiopacity is greater than that of dentin or bone, and is close to that of gutta-percha, which makes its visualization easier for surgical radiographic control and follow-up regardless of its clinical applications (13).
The C 150 guideline of the *American Society for Testing and Materials* (ASTM), issued in 1991, defines Portland cement as a hydraulic aggregate produced by grinding clinker, which consists of hydraulic calcium silicate usually with one or more forms of calcium sulfate as an admixture. The cement is mostly composed of calcium silicates (tricalcium silicate and dicalcium silicate, both corresponding to about 75% of its total composition), and aluminates (tricalcium aluminate and tetracalcium aluminoferrite), in addition to other components, such as impurities and sulfates added to the mixture to regulate setting (15).
Recent studies have described similar microbiological characteristics for MTA and Portland cement, which is used in civil construction. In one of these studies, chemical analysis showed a similarity between most of the components in both materials (13).
The low cost of Portland cement compared to MTA suggests the possibility of using Portland cement for the same purposes that MTA is used. Possible differences between white and grey MTA become relevant in this context.
The purpose of this study was to evalua-

te the chemical composition, pH and solubility of white and grey mineral trioxide aggregate (MTA) and Portland cement.

MATERIALS AND METHODS

Analysis of Chemical Composition and pH

Samples were weighed on a precision scale reading to 0.0001 g (Analíticas Ohaus AP, Toledo, Pinais, PR, Brazil) and calibrated according to the manufacturer's reference standard. Three samples of each material under study were prepared: white MTA (Ângelus®, Londrina, Brazil), grey MTA (Ângelus®, Londrina, Brazil) and Portland cement (Cia de Cimento Itambé®, Balsa Nova–PR, Brazil). The powder cement of each weighed sample was kept in a previously labeled plastic container. A volumetric pipette was used to add 100 ml of Milli-Q to the powder in the container. The Milli-Q system was designed to provide type I water (18 Wcm resistivity at 25°C and total organic carbon –TOC- below 10 ppb), therefore free of any contaminant. Samples were diluted and stirred with plastic rods until a homogeneous solution was obtained.

The same samples were used for the chemical composition and pH tests. The pH-meter (CG 840, Schott-Mainz, Germany) was calibrated with pH 7 and pH 10 buffer solutions, and the pH of each sample was then measured 15 minutes following mixing. The pH reading membrane was immersed in Milli-Q water every time it was moved from one sample to the other to avoid interference with the results. The samples then underwent atomic absorption spectroscopy. The presence of the following metals was investigated: *silicon, magnesium, iron, aluminum* and *calcium*. These were the chemical components with the best reading potential through this technique.

The spectrophotometer (Perkin-Elmer 4000, Perkin-Elmer Corp, Wellesley, MA, USA) was optimized with standard solutions for the reading of each metal. For the analysis of calcium, the equipment was optimized with Merck's Calcium Standard Solution (Merck & Co., Whitehouse Station, NJ, USA). Three samples of each material were individually placed in the spectrophotometer to obtain the reading. Each metal was analyzed individually in each sample. A total of 45 calibrations were made (5 elements x 3 samples x 3 materials).

Results and data obtained in chemical composition and pH tests were analyzed statistically with Student's *t* test or one-way ANOVA, followed by the Duncan test in the cases for which F was significant. SPSS software was used. Significance was established at $P < 0.05$.

Solubility Test

Materials were measured and mixed by a single operator according to manufacturer's directions. After mixing, each material was poured in to a cylindrical mold of about 6 mm x 2 mm and fixed between two boards. Six disks of each material were made and tested. The discs were placed in a container protected from heat, light and humidity for 3 hours to set. On the next day, the samples were individually stored in glass containers with 50 ml distilled water at 37°C. After one day, they were removed from the water and dried for one hour at 37ºC. Each disk was weighed in an electronic precision scale and returned to the same container, without changing the water, for the final period of tests. Drying and weighing were performed at one and seven days.

RESULTS

Results of the chemical analysis are shown in Table 1. Atomic absorption spectroscopy showed that silicon, magnesium, iron, aluminum and calcium are found in white MTA, grey MTA and Portland cement. Measurements showed that calcium was the major component in all samples. There were significant differences between the amounts of metals found in the three materials. Portland cement had 3.6 times more silicon than white MTA and 2.6 times more than grey MTA. (Value of ANOVA F (2.8) = 307.972; $P < 0.001$).

Portland cement had 2.45 times more magnesium than white MTA and 1.5 times more than grey MTA. (Value of ANOVA F (2.8) = 24.095; $P < 0.001$)

The amount of iron in white MTA was significantly greater than the amount in Portland cement (Value of ANOVA F (2.8) = 4.807; $P = 0.057$), and there was no significant difference between the amount of iron in Portland cement and in grey MTA.

White MTA had 2.7 times more aluminum than grey MTA and 1.2 times more than Portland cement. Portland cement had 2.3 times more aluminum than grey MTA. (Value of ANOVA F (2.8) = 188.927; $P < 0.001$)

Mean amount of calcium found in grey MTA was significantly greater than the amounts in Portland cement (1.3 times greater) and white MTA (1.2 times greater). (Value of ANOVA F (2.8) = 12.401; $P = 0.007$)

The results of the pH analysis are shown in Table 2. All samples had a pH close to 12 immediately after mixing, and this value was constant. The comparison between mean pH values revealed that the pH of Portland cement was significantly lower (Value of ANOVA F (8.2) = 43.988; $P < 0.0001$) than that of white MTA or grey MTA.

Table 3 shows the results of the solubility test for the samples of white and grey MTA and Portland cement. Samples were weighed before and after immersion in water. White and grey MTA and Portland cement in this study did not have statistically significant signs of solubility.

DISCUSSION

Several studies have investigated the physical properties of MTA (3-7, 9, 12, 13, 16, 17). However, the data on the properties of this material are still insufficient. Most studies of MTA do not specify the color of the material used. A report on the comparison of the results of white and grey MTA used in dental pulps of dogs demonstrated that grey MTA gave better results than white MTA (18). Another biocompatibility study found that differences between grey and white MTA, like the color of cement after final set, and a shorter time to final set for the white MTA sample, did not affect their similar behavior and mechanism of action (19).

Most comparative studies of the chemical composition of MTA and Portland cement have been conducted by means of the X-ray spectrophotometer (20), in which a point in the sample is selected for evaluation. In the atomic absorption technique used in this study, the sample was mixed and therefore it was possible to conduct a global analysis of it, which provides more accurate results about metals in the sample.

A comparative study of the chemical composition of MTA and Portland cement (13) using X-ray spectrophotometer reported that both materials have the same components, except that MTA also contains bismuth. The components found in Portland cement and MTA were: CaO (58.5%), SiO (17%), AlO (4.5%), MgO (3.3%), SO (3.0%), FeO (2.9%), KO (0.9%), NaO (0.2%).

Our results are similar to these reported, and confirm the presence of the same materials in the chemical composition of white and grey MTA and Portland cement. Metal components are: calcium (the largest relative proportion), silicon, aluminum, iron and magnesium. However, statistical analysis of the results revealed significant differences for the amounts of each component in the different materials.

A chemical analysis to quantify bismuth in the samples was not possible in this study because of the method used. However, previous studies (1) showed that this is the component responsible for MTA's radiopacity, and that it is not found in Portland cement.

MTA has a high pH, similar to that of calcium hydroxide cement and may induce hard tissue formation when used as a capping or filling material (21). The pH value when MTA is first exposed to humidity is 10.2, and it increases to 12.5 about 3 hours after mixing, which promotes both the alkalinization of the medium in which it is used and its antimicrobial action (3-6).

When Portland cement is mixed with water, its pH has initial values close to 7, but gradually increases to up to 12.9 after 3 hours (22).

White MTA showed the largest intergroup weight value range. Being from the same stock, it could be speculated that the ideal homogeneity of this material was not achieved. This lack of uniformity was not found in a different study using ProRoot MTA (23).

This study found that all samples had a pH greater than 11. However, in contrast to other results in the literature, these pH values were observed immediately after mixing and did not take 3 hours to reach stability. As the pH measurement method was the same as used in all other studies, this difference may be explained by the greater solubilization of the samples in this study. However, pH is believed to be constant and not to require so much time to stabilize. Statistical analysis of pH results showed that the alkalinity of Portland cement was significantly lower than that of white or grey MTA. This differs from other studies in the literature (24, 25), and this could be explained by the different brands of MTA as well as Portland cement used. The differences in pH profile during and after setting (24), and from different manufacturers (25), have only relative clinical relevance, because they all show levels of pH high enough to provide alkalinity to the tissues in contact.

The risk of presence of impurities in Portland cement is still a matter of concern (23, 25), and this should be taken into consideration when deciding which materials would consistently give reliable results.

This study used MTA Angelus, which is different from ProRoot MTA. This can explain some differences in the literature with regards to amount of iron content (20, 26, 27). Also, this study did not detect, in MTA composition, substances found in other studies, such as $TiO2$ (20) and $SO3$ (27).

White and grey MTA and Portland cement in this study did not have statistically significant signs of solubility, which confirmed their desirable properties as a filling material. However, other long term solubility tests involving MTA have been reported in the literature (28), with results showing the potential of this material to solubilize up to 31% especially if the water-powder *ratio* is increased.

The constituents of MTA (29) and its biological properties (30) have influenced many operators to use in various clinical situations. However, as any material used in dentistry, MTA and Portland cements have variations depending on the manufacturers. Understanding their differences allows clinicians to make their choices considering all desirable and undesirable factors in each of these materials. Therefore, the study of MTA is far from being exhausted and further questions will arise with regards to its physical and biological properties.

CONCLUSION

The materials studied had similar chemical compositions, except for the presence of bismuth oxide in white and grey MTA.

There were significant differences in the amount of components in the different samples.

All samples had pH values close to 12 immediately after mixing; Portland cement had significantly lower alkalinity than white or grey MTA.

There were no significant signs of solubility in the three materials tested.

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