Influence of root canal irrigants on compressive strength and surface morphology of gray MTA Angelus®

Johnson Campideli **FONSECA**[1]
Luiz Fernando Ferreira de **OLIVEIRA**[2]

ABSTRACT

Objective: The present study aims to evaluate the influence of root-canal irrigants in the compressive strength and surface morphological characteristics of gray MTA Angelus using scanning electron microscopy (SEM). **Methods:** The MTA was mixed according to the manu instructions from manufacturer and packed incrementally into silicone cylindrical molds with an internal diameter of 2 mm and a height of 4 mm. After the initial setting, 30 samples were randomly divided into 3 groups (n=10). In Group C (control) the samples were immersed in a saline solution, in Group MC the samples were immersed in a 2.5% sodium hypochlorite solution and in Group MH the samples were immersed in a 2% chlorhexidine digluconate solution, all remaining for 1 hour. After rinsed, the compressive strength was measured in an Instron 4410 test machine with a crosshead speed of 0.5 mm / min. The surface morphological characteristics were determined by scanning electron microscopy (SEM). The statistical significance in compressive strength was evaluated by one-way analysis of variance (α=0,05). **Results:** The average compressive strength values (MPa) were 69.24±7.32 (Group C), 64.74±9.21 (Group MC) e 71.15±11.54 (Group MH), with no significant difference. The results of SEM had demonstrated that the microstructures from group C and MH were crystalline, composed of cubic crystals. A granular structure, in which crystals had not formed, was observed in the Group MC. **Conclusion:** The immersion in root-canal irrigants did not affect the compressive strength of MTA, with changes only in surface morphological characteristics. Further studies are needed to determine the clinical relevance of this crystal structure.

Keyhwords: Endodontics. Compressive strength. Microscopy.

How to cite this article: Fonseca JC, Oliveira LFF. Influence of root canal irrigants on compressive strength and surface morphology of gray MTA Angelus®. Dental Press Endod. 2011 Oct-Dec;1(3):34-40.

» The authors report no commercial, proprietary, or financial interest in the products or companies described in this article.

Received: September 19, 2011 / Accepted: September 31, 2011.

[1]Master and PhD in Dental Materials, FOP-UNICAMP. Full Professor, Unilavras.
[2]Scholar, Unilavras School of Dentistry.

Contact address: Johnson Campideli Fonseca
Rua Padre José Poggel, 506 Centenário – 37.200-000 – Lavras/MG – Brazil
E-mail: jcf.50@terra.com.br

Introduction

MTA (*Mineral Trioxide Aggregate*) has been introduced in the dental market in 1993 by Torabinejad (Loma Linda University), with the indication for primary use in repairing lateral perforations in root canals and sealing apical areas. Currently, its indications are varied, persisting as drawbacks the long working time and difficult manipulation.[1]

This material consists of a powder of fine particles, which main components are tricalcium silicate, tricalcium aluminate, tricalcium oxide and silicate oxide. Its working time is 3 to 4 minutes and the setting time 3 to 4 hours.[2] The pH at mixing is 10.2, changing to approximately 12.5 after setting, thereby providing an antimicrobial action. There is also a possibility that MTA may promote tissue regeneration at its site of insertion.[3] Additionally, MTA presents two additional advantages in relation to other sealing materials: Its biocompatibility and the possibility of use in the presence of moisture.[4]

In endodontic therapy, we aggregate mechanical procedures and use of chemicals for removal of pathogens and chemicals that are harmful to the living tissues. Solutions based on sodium hypochlorite have been largely used with positive results, but there is also the alternative of using chlorhexidine solutions.[5]

However, information on the effect of such substances on MTA are still scarce. Still, when they are used in cases of root perforation, MTA is invariably exposed to these irrigants.

The objective of this study was to evaluate the influence of irrigating solutions on gray MTA Angelus® by assessing the resistance to compression and qualitative analysis of surface morphology on micrographs obtained by SEM (scanning electron microscopy).

Material and Methods

The matrices were obtained using devices consisting of an aluminum ring and a central base with a pin. Once these parts were positioned, addition silicone (Splash L - Discus Dental) was used to fill the rings and copied the cylindrical pins. After the setting of the silicone, the aluminum rings were removed (Figs 1A, B). Thirty samples were prepared inside the silicone matrices. The cylindrical cavities had a 2 mm diameter and a 4 mm height (Figs 1C and 1D). The material (Gray MTA Angelus - Lot 2302 - Angelus, Brazil) was handled under standard conditions (temperature 25 ± 2 °C and relative air humidity at 55%) using the proportion recommended by the manufacturer. The amount of powder was measured with a precision scale and the volume of the liquid dispensed with a micropipette.

The material was inserted into the matrix in reduced portions with a spatula and an amalgam condenser. The cavities were filled with an excess of the cement, with a constant vertical pressure of approximately 3 MPa applied for 1 minute in order to standardize the procedure.[6] The spilled material was carefully removed and the matrices were kept in containers at a temperature of 37 °C and 100% relative humidity for 72 hours. After storage, all samples were removed from the silicone matrix, checked for possible surface irregularities and, according to each experimental group (n = 10), immediately immersed for 1 hour in the following solutions:

» Group C: Control - saline solution.
» MC Group: 2% solution of chlorhexidine.
» MH Group: 2.5% solution of sodium hypochlorite.

Later, they were all rinsed in deionized and distilled water for 5 minutes and kept in a container with 100% relative humidity until the mechanical testing. The tests were performed in a test machine (Instron, model 4410, Norwood, MA, USA) at a crosshead speed of 0.5 mm / min. Compressive strength values were recorded in MPa.

In addition, two extra cylinders of equal size for each group were subjected to the experimental procedures previously described and stored in a container with 95% relative humidity. They were further evaluated in a scanning electron microscope model LEO EVO 40 (Carl Zeiss AG, USA), with low vacuum (0.05 to 2.0 Torr) in the region of the electrode, column and specimen chamber.[7,8] The images were recorded on file in TIFF (Tagged Image File Format) format in order to avoid errors caused by digital image compression, noise generation and distortion.

The original values obtained in the compressive strength test were subjected to analysis of variance, considering the model with one factor (dipping environment) and $\alpha = 0.05$, with the result described in the appendix. We used the software Microsoft Excel® with the add-in Analysis Tools (Microsoft Office® 2003).

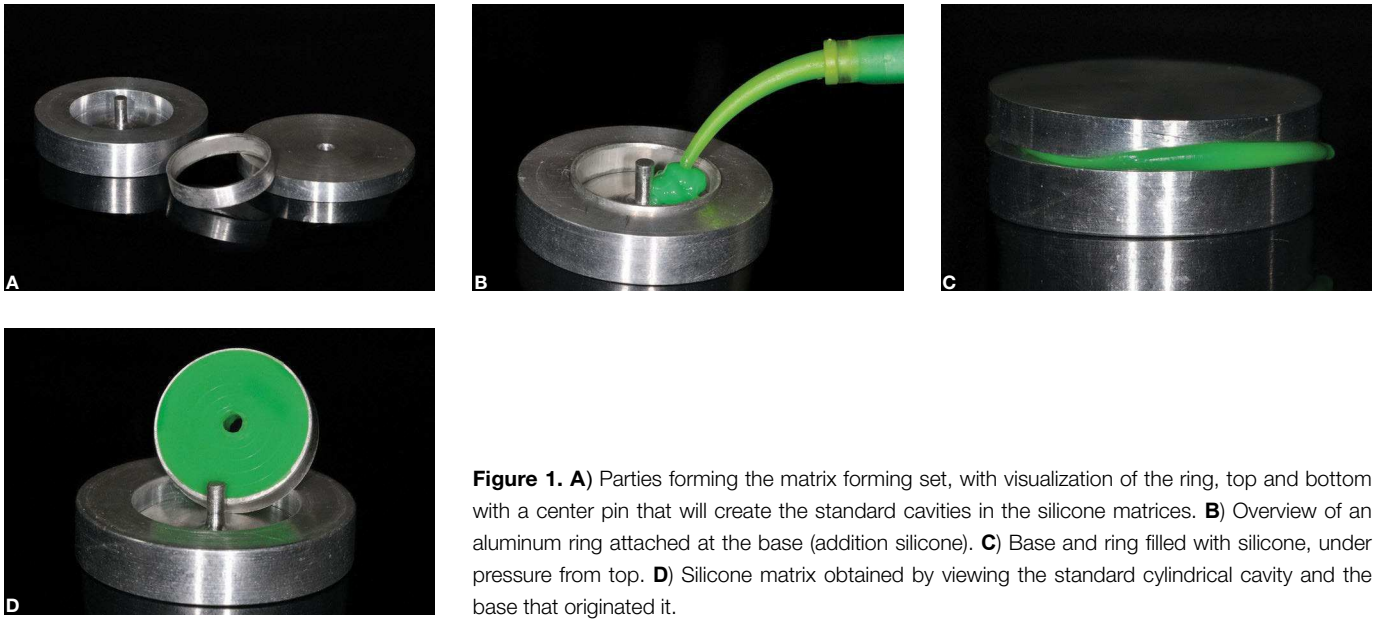

Figure 1. A) Parties forming the matrix forming set, with visualization of the ring, top and bottom with a center pin that will create the standard cavities in the silicone matrices. **B**) Overview of an aluminum ring attached at the base (addition silicone). **C**) Base and ring filled with silicone, under pressure from top. **D**) Silicone matrix obtained by viewing the standard cylindrical cavity and the base that originated it.

Results

After obtaining the values of compressive strength, no statistically significant difference between the groups was observed (Table 1):

Figures 2 - 7 show the photomicrographs used for qualitative analysis involving the samples surface. In Figure 2 it is noticeable the presence of cubic crystals with a greater variety of sizes and major proximity, increasing the compression on the surface, features which are more evident under higher magnification (Fig 3).

In the photomicrographs for the MC group, it is shown a major filling and partial recoating of the cubic crystals and gaps if compared to Group C (Fig 4). It can be seen with an even higher magnification (Fig 5) that gaps still remain on the surface, consistent with dissolution of less stable phases. These areas became less cohesive, what may be the cause for lower mechanical strength.[9]

The photomicrographs relative to Group MH showed less gaps along the analyzed surface (Fig 6) and a greater presence of smaller size cubic crystals among the larger crystals (Fig 7). This may increase the packing factor and the resulting structure. This fact allows a more sparse distribution of gray MTA Angelus particles in relation to the particles size. However, this feature may come to affect the homogeneity of the newly manipulated material.[6]

Discussion

The dental materials are constantly evolving and their clinical use should be preceded by a greater knowledge of their physical, chemical and biological

Table 1. Experimental groups, mean values of compressive strength and standard deviation as a function of immersion solutions.

Group	Mean Value	Standard Deviation
C	69.24[a]	7.32
MC	64.04[a]	9.21
MH	71.15[a]	11.54

Results are expressed in MPa. Mean values followed by same letter vertically do not show statistically significant difference (α = 0.05).

properties.[10] Among these materials, MTA stands out due to its excellent repair properties, biocompatibility and tolerance to moisture.[1,2,4,11] Additionally, it shows a sealing ability within the standards, with the advantage of radiopacity provided by the presence of bismuth oxide.[12]

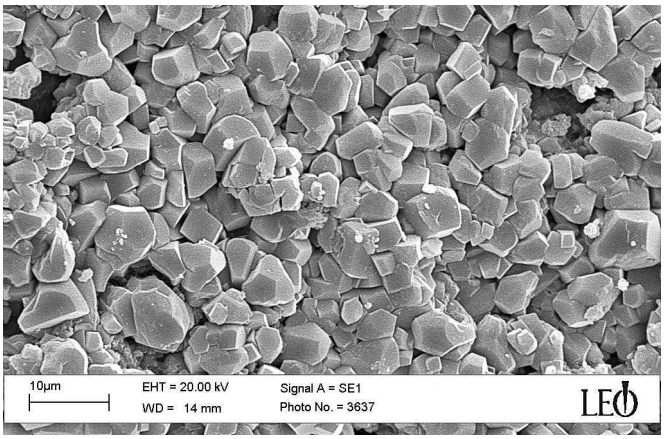

Figure 2. Photomicrograph displaying the surface for a representative sample of the group C (4000 x)

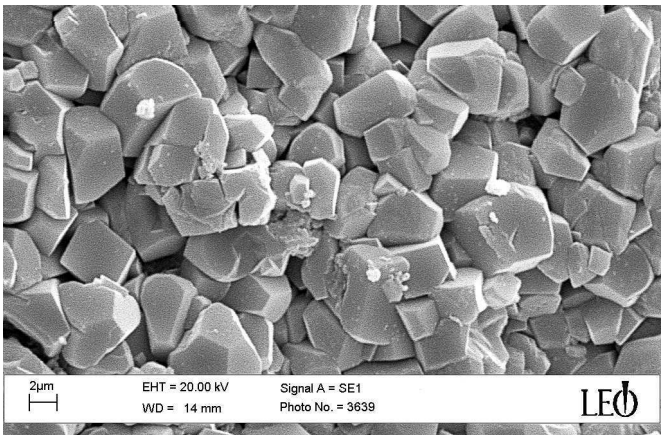

Figure 3. Photomicrograph displaying the surface for a representative sample of the group C (7000 x).

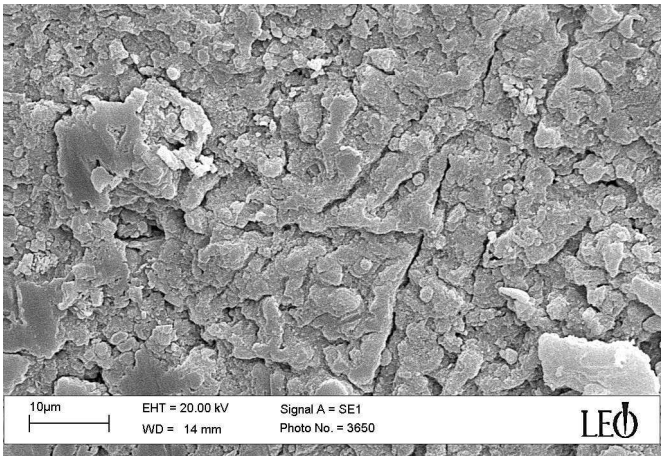

Figure 4. Photomicrograph displaying the surface for a representative sample of the group MC (4000 x).

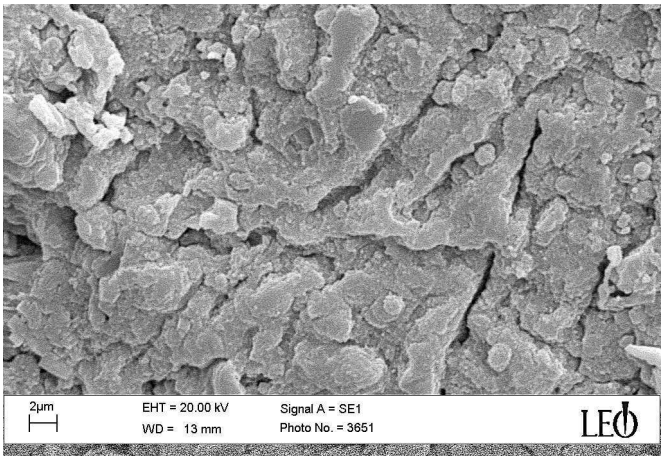

Figure 5. Photomicrograph displaying the surface for a representative sample of the group MC (7000 x).

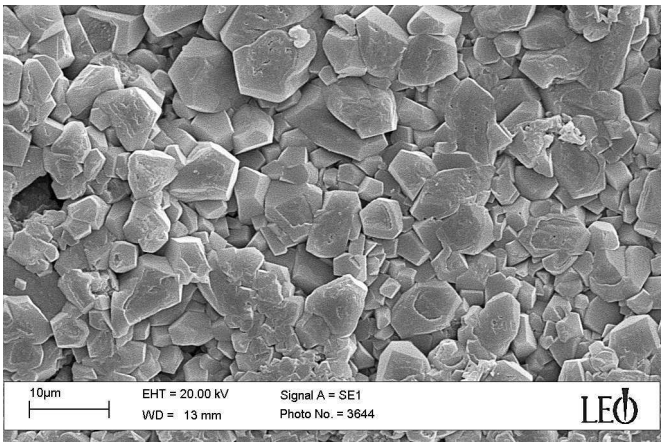

Figure 6. Photomicrograph displaying the surface for a representative sample of the group MH (4000 x).

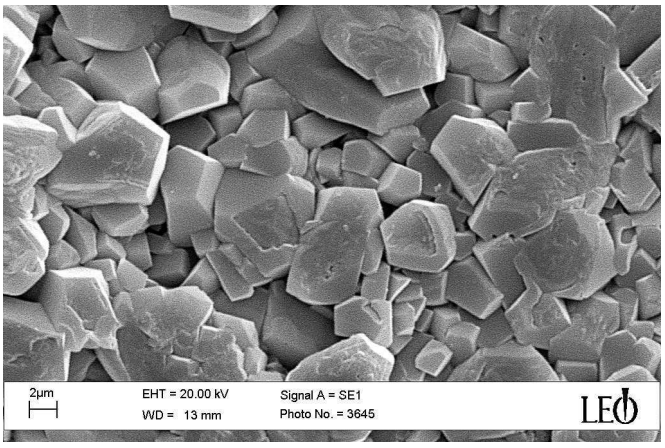

Figure 7. Photomicrograph displaying the surface for a representative sample of the group MH (7000 x).

Characteristics related to mechanical strength may not be a factor of utmost importance when a material is inserted into cavities that do not directly support high stresses (e.g.: a retrofiller). However, one should consider that in situations such as furcation perforations in molars and direct pulp capping, this may be significant due to the occlusal loads.[13] In this study, the compressive strength was selected with the purpose of representing such clinical situation. It should be emphasized that applying a correct methodology in laboratory tests shows a direct relation to the standardization and validity of the results.[14] For instance, in compressive strength tests, the presence of internal and external failures can lead to the occurrence of complex loads. This may occur if the sample is too short or too long. To avoid bending, the sample should have a height twice its diameter.[15]

Because MTA has a long setting time, patients should be informed to avoid strong chewing on the MTA-treated tooth for at least 4 hours after placement. Research has confirmed that in cases of root perforations, the placement of the MTA should be performed via root canal with the aid of an amalgam holder and be gently compressed with moistened cotton pellets.[16,17]

In studies that address the relation between the insertion and mechanical strength of MTA,[18,19,20] there is frequent mention of the time of 72 hours that should be awaited in order to present satisfactory values of mechanical strength and resistance to displacement.

In this study, the mean compressive strengths (MPa) were 69.24, 64.04 and 71.15 respectively for the groups C, MC and MH. These values differ from those described in the literature, e.g., approximately 40 MPa after 21 days of insertion up to 67 MPa, which corresponds to those of IRM and Super EBA.[21] The manufacturer of the evaluated product (gray MTA - Angelus) describes the value of 42 MPa for the compressive strength after 28 days. Nevertheless, in literature there are mean values of 70 MPa for Gray MTA,[19] which are similar to those obtained here.

Given the variations found in the values of compressive strength, it should be considered that different protocols were used. It has been shown that the setting reaction of MTA is highly susceptible to the environment in which it occurs (presence of chemical substances and pH values).[22,23,24]

Looking for similarity between laboratory testing and clinical performance, solutions such as 2% chlorhexidine and 2.5% sodium hypochlorite were used in this study. This was performed to simulate the clinical situation in which the MTA would be exposed to these substances during endodontic treatment.[5,25] Such substances, either due to their composition or pH, change the structure and the surface morphology of the MTA,[8,17,24] which was actually demonstrated in this study.

Studies with similar methodology described the formation of cubic crystals in the MTA, when exposed to a neutral pH solution, a fact which corroborates the results of this study. However, there is a description of the presence of acicular crystals, which was not observed in the experimental conditions herein. Differences in the type of MTA and methodology may have been responsible for this variation.[17,26]

The analysis of the photomicrographs regarding the control group (Figs 2 and 3) showed characteristic images of cubic and compact crystals, with an approximate width of 5 mm and size and shape variations, which were consistent with the literature.[27,28] In Figure 3 the cubic crystals are presented juxtaposed, without the presence of an amorphous layer covering them.

In Figure 5 gaps still remain on the surface, consistent with dissolution of the less stable phases, related to lower mechanical strength.[9] However, the fact that they occur in small scattered regions causes them to not be representative in order to influence the compressive strength, a fact evidenced by the mechanical test performed.

The photomicrographs for the MH group exhibited less gaps along the analyzed surface (Fig 6) and a greater presence of cubic crystals with smaller size among the larger crystals (Fig 7), thus increasing the packing factor and the resulting structure. This fact provides a more sparse distribution of gray MTA Angelus particles in relation to the particles size. However, this feature may come to affect the homogeneity of the newly manipulated material.[6]

Despite the fact that the solutions did not influence the values of compressive strength, the clinical performance of a material is grounded in a satisfactory set of properties, acting in a synergistic way.

Assessments involving surface relations under the tested conditions are necessary, in order to clarify whether such changes affect the interaction with other restorative materials and mechanical strength in the long-term.

Conclusions

Before the experimental conditions, we may conclude that:

» There was no statistically significant difference in compressive strength among the experimental groups.

» The SEM analysis showed variation on the surfaces, with a similar pattern between groups C and MH, and a granular structure occurring only in group MC.

Acknowledgements

To Angelus for the unrestricted support for research; the Faculty of Dentistry of Piracicaba (FOP-UNICAMP) for providing the test machine; to UFLA and Professor. Eduardo Alves for their support in the use of scanning electron microscope (Federal University of Lavras, Department of Phytopathology).

References

1. Arens ED, Torabinejad M. Repair of furcal perforations with mineral trioxide aggregate. Oral Surg Oral Med Oral Pathol Oral Radiol Endod. 1996;82(1):84-8.
2. Lee SJ, Monsef M, Torabinejad M. Sealing ability of a mineral trioxide aggregate for repair of lateral root perforations. J Endod. 1993;19(11):541-4.
3. Abedi HR, Ingle JL. Mineral trioxide aggregate: a review of new cement. J Calif Dent Assoc. 1995;23(12):36-9.
4. Pitt Ford TR, Torabinejad M, McKendry DJ, Hong CU, Kariyawasam SP. Use of mineral trioxide aggregate for repair of furcal perforations. Oral Surg Oral Med Oral Pathol Oral Radiol Endod. 1995;79(6):756-63.
5. Yamashita JC, Tanomaru Filho M, Leonardo MR, Rossi MA, Silva LA. Scanning electron microscopic study of the cleaning ability of chlorhexidine as a root-canal irrigant. Int Endod J. 2003;36(6):391-4.
6. Nekoofar MH, Aseeley Z, Dummer PM. The effect of various mixing techniques on the surface microhardness of mineral trioxide aggregate. Int Endod J. 2010;43(4):312-20.
7. Shipper G, Grossman ES, Botha AJ, Cleaton-Jones PE. Marginal adaptation of mineral trioxide aggregate (MTA) compared with amalgam as a root-end filling material: a low-vacuum (LV) versus high-vacuum (HV) SEM study. Int Endod J. 2004;37(5):325-36.
8. Kayahan MB, Nekoofar MH, Kazandag M, Canpolat C, Malkondu O, Kaptan F, et al. Effect of acid-etching procedure on selected physical properties of mineral trioxide aggregate. Int Endod J. 2009;42(11):1004-14. Epub 2009 Sep 1.
9. Shie MY, Huang TH, Kao CT, Huang CH, Ding SJ. The effect of a physiologic solution pH on properties of white Mineral Trioxide Aggregate. J Endod. 2009;35(1):98-101.
10. Torabinejad M, Hong CU, McDonald F, Pitt Ford TR. Physical and chemical properties of a new root-end filling material. J Endod. 1995;21(7):349-53.
11. Holland R, Otoboni Filho JA, Souza V, Nery MJ, Bernabé PFE, Dezan Jr E. Mineral trioxide aggregate repair of lateral root perforations. J Endod. 2001 Apr;27(4):281-4.
12. Moraes SH. Aplicação clínica do cimento de Portland no tratamento de perfurações de furca. JBC: J Bras Clín Odontol Int. 2002;6(33):223-6.
13. Bernabé PFE, Holland R. Cirurgia parendodôntica: como praticá-la com embasamento científico. In: Estrela C. Ciência Endodôntica. São Paulo: Artes Médicas; 2004. v. 2, p. 657-797.
14. Nekoofar MH, Adusei G, Sheykhrezae MS, Hayes SJ, Bryant ST, Dummer PM. The effect of condensation pressure on selected physical properties of mineral trioxide aggregate. Int Endod J. 2007;40(6):453-61. Epub 2007 Apr 24.
15. Craig RG. Materiais dentários: propriedades e manipulação. 7ª ed. São Paulo: Ed. Santos; 2002.
16. Torabinejad M, Chivian N. Clinical applications of mineral trioxide aggregate. J Endod. 1999;25(3):197-205.
17. Sluyk SR, Moon PC, Hartwell GR. Evaluation of setting properties and retention characteristics of mineral trioxide aggregate when used as a furcation perforation repair material. J Endod. 1998;24(11):768-71.
18. Herzog-Flores DS, Velázquez LMA, González VM, Rodrígues FJM, Gómez MVB, Barrientos MVG. Análisis fisicoquímica del Mineral Trióxido Agregado (MTA) por difracción de rayos X, calorimetria y microscopía electrónica de barrido. Rev ADM. 2000;57(4):125-31.
19. Schmitt D, Lee J, Bogen G. Multifaceted use of ProRoot MTA root canal repair material. Pediatr Dent. 2001;23(4):326-30.
20. Darvell BW, Wu RC. "MTA" — an hydraulic silicate cement: review update and setting reaction. Dent Mater. 2011;27(5):407-22. Epub 2011 Feb 26.
21. Torabinejad M, Hong CU, McDonald F, Pitt Ford TR. Physical and chemical properties of a new root-end filling material. J Endod. 1995;21(7):349-53.
22. Watts JD, Holt DM, Beeson TJ, Kirkpatrick TC, Rutledge RE. Effects of pH and mixing agents on the temporal setting of tooth-colored and gray mineral trioxide aggregate. J Endod. 2007;33(8):970-3. Epub 2007 May 23.
23. Parirokh M, Torabinejad M. Mineral Trioxide Aggregate: a comprehensive literature review - Part I: chemical, physical, and antibacterial properties. J Endod. 2010;36(1):16-27.
24. Torabinejad M, Parirokh M. Mineral Trioxide Aggregate: a comprehensive literature review - Part II: leakage and biocompatibility investigations. J Endod. 2010;36(2):190-202.
25. Stowe TJ, Sedgley CM, Stowe B, Fenno JC. The effects of chlorhexidine gluconate (0.12%) on the antimicrobial properties of tooth-colored ProRoot mineral trioxide aggregate. J Endod. 2004;30(6):429-31.
26. Lee YL, Lee BS, Lin FH, Yun Lin A, Lan WH, Lin CP. Effects of physiological environments on the hydration behavior of mineral trioxide aggregate. Biomaterials. 2004;25(5):787-93.
27. Komabayashi T, Spångberg LS. Particle size and shape analysis of MTA finer fractions using Portland cement. J Endod. 2008;34(6):709-11. Epub 2008 Apr 15.
28. Hwang YC, Kim DH, Hwang IN, Song SJ, Park YJ, Koh JT, et al. Chemical constitution, physical properties, and biocompatibility of experimentally manufactured Portland cement. J Endod. 2011;37(1):58-62. Epub 2010 Nov 12.