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ORIGINAL ARTICLE

X-ray diffraction analysis of MTA mixed and placed with various techniques

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Abstract
Objectives The aim of this study was to evaluate the effect of various mixing techniques as well as the effect of ultrasonic placement on hydration of mineral trioxide aggregate (MTA) using X-ray diffraction (XRD) analysis.
Materials and methods One gram of ProRoot MTA and MTA Angelus powder was mixed with a 0.34-g of distilled water. Specimens were mixed either by mechanical mixing of capsules for 30 s at 4500 rpm or by manual mixing followed by application of a compaction pressure of 3.22 MPa for 1 min. The mixtures were transferred into the XRD sample holder with minimum pressure. Indirect ultrasonic activation was applied to half of the specimens. All specimens were incubated at 37 °C and 100% humidity for 4 days. Samples were analyzed by XRD. Phase identification was accomplished by use of search-match software utilizing International Centre for Diffraction Data (ICDD).
Results All specimens comprised tricalcium silicate, calcium carbonate, and bismuth oxide. A calcium hydroxide phase was formed in all ProRoot specimens whereas among MTA Angelus groups, it was found only in the sample mixed mechanically and placed by ultrasonication.
Conclusions Mechanical mixing followed by ultrasonication did not confer a significant disadvantage in terms of hydration characteristics of MTA.
Clinical relevance Clinicians vary in the way they mix and place MTA. These variations might affect their physical characteristics and clinical performance. For ProRoot MTA, the mixing and placement methods did not affect its rheological properties, whereas for MTA Angelus, mechanical mixing combined with ultrasonic placement enhanced the calcium hydroxide phase formation.

Keywords Calcium hydroxide · MTA · Mechanical mixing · Ultrasonic agitation · X-ray diffraction analysis · XRD

Introduction

Mineral trioxide aggregate (MTA) has been used in endodontics for the past two decades. MTA is a powder that consists of fine hydrophilic particles that harden when they come in contact with water [1]. The main components of MTA are tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetracalcium aluminoferrite [2]. On addition of water, calcium silicates undergo hydrolysis producing calcium hydroxide and the less basic calcium silicate hydrate [3]. MTA is mainly composed by an insoluble matrix of silica that maintains its integrity even in contact with water [4]. In an aqueous environment, MTA releases calcium hydroxide, which makes the hydrated cement highly alkaline [1]. After the placement of MTA in root canals and its gradual dissolution, hydroxyapatite crystals nucleate and grow, filling the microscopic space between the material and the dentinal wall [5]. With time, this mechanical seal leads to a chemical bond between the apatite layer and

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dentin [5]. The result is the creation of a chemical bond to dentin when placed against it [5].

The hydration process is a complex phenomenon that if modified might influence the biological, chemical, and physical properties of the resulting product [3]. Thus, any interference in the hydration process may adversely affect the clinical performance. In vitro investigations of simple parameters may draw a correlation of chemical and physical properties with clinical performance and can advise clinicians which cements need special care or entail particular risks during the mixing and placement process.

The physical characteristics of hardened MTA are influenced by several factors: the mixing liquid [6, 7], the amount of water used during mixing [1], the pressure used for compacting the material [8], the mixing procedure [9, 10], the humidity, and the ambient temperature [11]. Some of these factors are not easy to control; it is therefore difficult to standardize methods used to determine the properties of MTA [1]. In an attempt to achieve consistency, encapsulated pre-set proportions of MTA could confer advantages to the practitioner and the material [12].

In addition, ultrasonic energy has been used to enhance the mechanical properties of MTA such as compressive strength [9], surface microhardness [12], sealing ability [13], and fill density [14]. Ultrasonic energy can increase the total reactive surface by de-clustering the particles that are clogged to each other [14]. These changes in the rheological properties of the material might affect their physical characteristics [13].

To date, the effects of mixing and placement methods on the compressive strength [8, 9], flexural strength [15], push-out bond strength [16], surface microhardness [12], marginal adaptation [17], and porosity [15] of MTA have been investigated. Even though these mechanical tests on the physical properties of hydraulic cements can be used as indicators of the hydration and setting processes [3, 18], there is no direct information about the effect of mixing and placement methods on the hydration of MTA.

The aim of this study was to evaluate the effect of various mixing techniques including mechanical and manual mixing as well as the effect of ultrasonic agitation during placement on hydration of MTA using X-ray diffraction analysis. It was hypothesized that mechanical mixing followed by ultrasonic agitation would not affect the hydration of MTA.

Materials and methods

The materials investigated were tooth-colored ProRoot MTA (Dentsply Maillefer, Ballaigues, Switzerland) and white MTA Angelus (Angelus Soluçoes Odontologicas, Londrina, Brazil).

Sample preparation

One gram of each powder was mixed with a 0.34-g aliquot of distilled water. A total of eight groups were prepared using manual mixing or mechanical mixing with either conventional placement or ultrasonic agitation. ProRoot MTA was used in groups 1 to 4, and MTA Angelus was used in groups 5 to 8. Groups 1 and 5 were mixed mechanically and placed with ultrasonic agitation. Groups 2 and 6 were mixed mechanically and placed without ultrasonic agitation. Groups 3 and 7 were mixed manually and placed with ultrasonic agitation. Groups 4 and 8 were mixed manually and placed without ultrasonic agitation.

Stainless steel XRD sample holders with internal dimensions of 2.0 ± 0.1 mm high and a 15.0 ± 0.1-mm diameter were used.

Mechanical mixing

One gram of MTA powder and 0.34 g of liquid were placed into an empty amalgam capsule with a plastic rod-like pestle to facilitate mixing [12]. The capsules were sealed and mixed for 30 s at 4500 rpm by using an amalgamator (Promix TM; Dentsply Caulk, York, PA). A total of 3 g of MTA slurries was loaded with minimal pressure in the circular depression of the XRD sample holder.

Manual mixing

An aliquot of 0.34 g distilled water was added to 1 g MTA powder until it was saturated. A total of 3 g of mixture was transferred into the XRD sample holder with minimum pressure. Using a custom-made device (Medical Physics and Clinical Engineering, Cardiff and Vale UHB), the material was then subjected to 3.22 MPa vertical pressure for 1 min.

Ultrasonication

Indirect ultrasonication was applied by placing a CPR-2D tip (Obtura Spartan, Fenton, MO) of the ultrasonic device (Suprasson P5; Satelec, Merignac, France) in contact with the outer surfaces of the mold, avoiding contact with the material inside the mold and then activated for 30 s at scale 5. The ultrasonic energy was transferred throughout the outer surfaces of the mold, as was previously stated by Basturk et al. [15]. Extruded material was removed without causing any visible indentation on the surface of the material, a wet cotton pellet was placed on the exposed surface of each specimen, and the molds were transferred to a plastic container that was sealed

and stored at a temperature of 37 °C and fully saturated humidity for 4 days.

XRD analysis

One specimen, containing 3 g of MTA, from each group were analyzed. The specimen surfaces were polished with 1200-grit fine grain sandpaper (3M; St Paul, MN, USA) to ensure that the surface of the sample was leveled with the holder surface. X-ray diffractometer (XRD, Panalytical X'pert pro, Almelo, Netherlands) used Ni-filtered CuKa radiation at 40 Kv and 40 mA. The crystalline structure of the materials was determined by passing a beam of X-rays of known wavelength into the specimen while rotating it through an angle . The samples were rotated to ensure the effects of preferred orientation are limited. Scans were undertaken in the range 10°–80° 2 .

Each constituent has characteristic diffraction patterns, and there may be multiple peaks in each sample owing to the cements consisting of several chemical compounds. Phase identification was accomplished by use of search-match software utilizing International Centre for Diffraction Data database (ICDD, Pennsylvania, PA, USA).

Results

All specimens analyzed by XRD were comprised of bismuth oxide (α-Bi_2O_3, ICDD 00-027-0053) indicated by the strongest peaks at 27.39°, 33.03°, and 33.24° 2 , tricalcium silicate (Ca_3SiO_5, ICDD 00-055-0738) indicated by the peaks at 29.425°, 32.60°, and 34.37° 2 , dicalcium silicate (Ca_2SiO_4) indicated by the strongest peaks at 32.05° 2 , and calcium carbonate ($CaCO_3$ ICDD 00-005-0586) indicated by the peaks at 29.46°, 39.48°, and 48.61° 2 (Fig. 1).

The intensity of bismuth oxide in ProRoot MTA groups was higher than those of MTA Angelus, regardless of the mixing and placement methods applied.

ProRoot MTA samples shown in Fig. 1a indicated that the formation of the calcium hydroxide phase was not affected by mechanical mixing, manual mixing, or ultrasonication given that the peaks from calcium hydroxide occurred in all groups. The formation of the calcium hydroxide phase (ICDD 00-044-1481) did not follow any particular pattern for MTA Angelus samples. Among MTA Angelus groups, the calcium hydroxide phase was found only in the sample, which was mixed mechanically and placed by ultrasonic agitation (Fig. 1b). The highest intensity of tricalcium silicate among MTA Angelus samples was also observed in that group.

Discussion

Diffraction patterns of cementitious materials provide phase, chemical, and crystal structure information data that are needed to understand the cement performance [19]. The use of XRD permits the identification of the major constituents or compounds present in a material [20]. It has been used for the phase composition [21], chemical analysis [7], and hydration characteristics [22] of mineral trioxide aggregate.

X-ray diffraction analysis was used in this study to determine the effect of mixing and placement methods on the hydration process of ProRoot MTA and MTA Angelus. Bismuth oxide (bismite), tricalcium silicate (alite), dicalcium silicate (belite), and calcium carbonate were observed in all specimens. Tricalcium silicate exhibited peaks at 29.4°, 32°, and 34° 2 . Using XRD, Islam et al. [20] and Lee et al. [18] demonstrated the presence of multiple peaks of tricalcium silicate for unhydrated MTA. They observed sharp peaks at 2 = 27.3° and multiple peaks at 32° and 34°. Grazziotin-Soares et al. [23] presented bismuth oxide peaks at 2 = 27° for both hydrated and unhydrated MTA Angelus samples, which corroborated our findings. Song et al. [24] suggested no obvious differences in the composition and crystalline structure between the hydrated and un-hydrated forms. XRD analyses which were performed on powder and hydrated MTA samples revealed that a similar pattern was observed in both groups, with the powder form showing sharper and stronger peaks [18, 19, 25]. On the other hand, calcium hydroxide can be identified only in the hydrated samples [24, 25]. In this study, manual mixing, mechanical mixing, or ultrasonic activation did not affect the formation of calcium hydroxide phase formation of ProRoot MTA, which is in accordance with previous findings [12]. Yet, in MTA Angelus groups, the formation of the calcium hydroxide phase only occurred in the samples prepared by mechanical mixing and ultrasonication. This is unusual for MTA, as Nekoofar et al. [7] reported the only instance where samples of MTA did not show the formation of calcium hydroxide was in the samples prepared with blood.

In hydrated ProRoot samples, Nekoofar et al. [7] and in hydrated MTA Angelus samples, Grazziotin-Soares et al. [23] reported that a calcium hydroxide peak was detected at 18° 2 . However, Camilleri et al. [26] reported that MTA Angelus did not exhibit a calcium hydroxide peak after 1 day of testing and speculated that MTA Angelus might vary from one batch to another as a result of its mineralogy. In accordance with these studies, all ProRoot MTA samples and only mechanically mixed and ultrasonicated MTA Angelus samples that were tested in our study revealed a sharp calcium hydroxide peak at 18° 2 . Tricalcium silicate and dicalcium silicate are the main crystalline phases

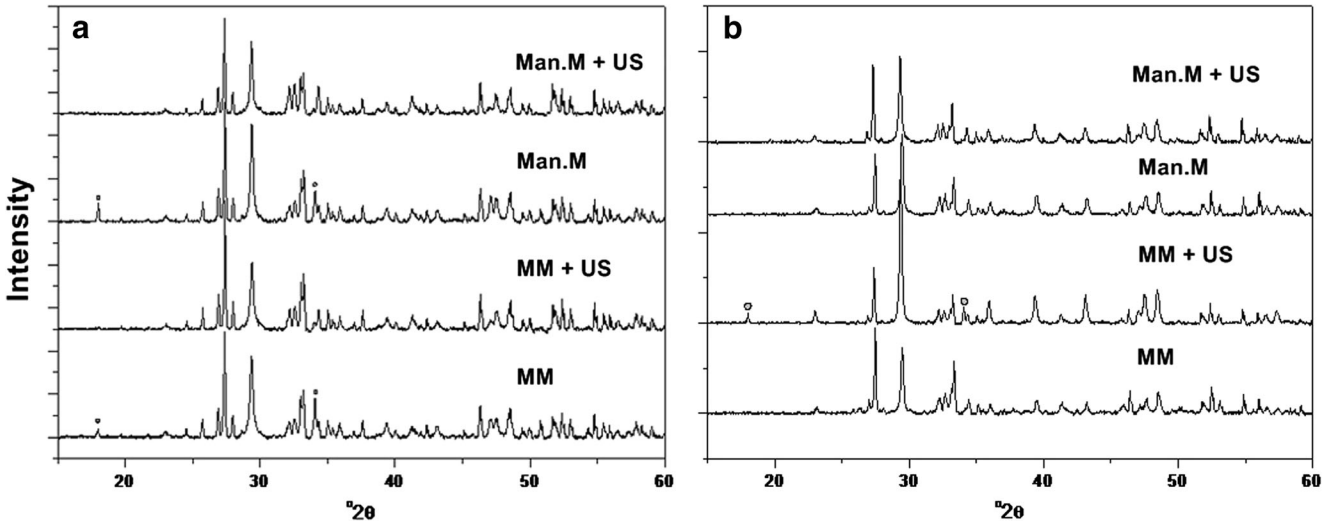

Fig. 1 X-ray diffraction analysis of **a** ProRoot MTA and **b** MTA Angelus. Calcium hydroxide phases were highlighted by circles. Man.M manual mixing, US ultrasonication, MM mechanical mixing

involved in the hydration of MTA [20, 21]. This can explain why the highest tricalcium silicate intensity among MTA Angelus groups was from the only group that revealed calcium hydroxide phase formation.

Calcium silicate hydrate and calcium hydroxide are by-products of hydration reaction of MTA [3]. The former component is not easily detectable by XRD because it does not have a long-range order, as a result of the amorphous nature of calcium silicate hydrate. On the other hand, calcium hydroxide can be easily identified through its marker band [27]. In the present study, the formation of calcium hydroxide was examined because it is directly related to the hydration progress. Therefore, lack of the calcium hydroxide phase formation might indicate a problem in the hydration reaction of MTA Angelus. Gandolfi et al. [27] observed calcium sulfate in ProRoot MTA, whereas it was absent in MTA Angelus. They reported that it would affect cement's setting time, since the presence of ettringite crystals, rapidly formed by reaction of tricalcium aluminate and calcium sulfate, retard the setting reaction. Nekoofar et al. [12] also reported that MTA Angelus set more rapidly and had a significantly lower surface microhardness value compared to ProRoot MTA and speculated that it might indicate inadequate hydration of the material, which is in accordance with the results of our study. This result is reasonable because the amount of sulfate phase is related to the fineness of the cement [21]. In this regard, the importance of particle size and shape characterization of MTA particles should be indicated [21]. MTA Angelus had a wide range of particle size distribution and low circularity compared to ProRoot MTA [28]. Therefore, ProRoot MTA appeared to have more homogeneous chemical composition than MTA Angelus [24, 29]. The influence of particle size and shape characterization on the hydration of hydraulic cements is important. The rate of hydration reaction, with enhanced formation of by-products, can be increased by fine grinding of the cement [30]. Smaller particle size implies a greater contribution of effects of surface area, causing a potential increase in the reactivity of the calcium silicate particles to form calcium hydroxide and calcium silicate hydrate gel [28]. Particle size might also have an influence on the liquid to powder ratio [28]. Smaller particles have a greater predisposition to absorb moisture [31].

Ultrasonic vibration has a dispersing effect on the particles of the material, which frequently cluster together [12]. It was speculated that mechanical mixing followed by ultrasonic activation may create a less grainy mixture with fewer unhydrated particles resulting in better water diffusion [9]. The stable cavitation and associated acoustic microstreaming promoted by ultrasonic activation [32] might reduce the size of cement particles and consequently increase the area available to react with water during the hydration process. Duque et al. [33] also reported that ultrasonication produced smaller particle sizes and more homogeneous distribution of particles in calcium silicate cements.

Ultrasonication has been used to enhance the mechanical properties of MTA, such as compressive strength [9], surface microhardness [12], sealing ability [13], push-out bond strength [10], and fill density [14]. To date, two different methods were introduced for the placement of MTA, hand condensation, and ultrasonic placement [14, 15, 34]. Matt et al. [35] reported that apical barriers placed with indirect ultrasonication resulted in fewer voids than barriers placed without ultrasonic energy. Basturk et al. [9] reported that indirect ultrasonic activation enhanced the compressive strength of the specimens regardless of the mixing regime applied. Yeung et al. [14] advocated that indirect ultrasonication resulted in better fill density than direct ultrasonication, as even the smallest ultrasonic tip was unable to extend to the full length

of the curved canal. In the present study, indirect ultrasonic placement was applied in accordance with previous studies [9, 12]. The main objective of indirect ultrasonic activation was to agitate MTA without contacting the ultrasonic tip directly to the material, as direct ultrasonication may cause voids inside MTA [36]. Previous in vitro studies [14, 15, 17, 36] used indirect ultrasonication techniques by transferring the ultrasonic energy through an endodontic instrument or the outer surface of the mold. However, it is difficult to directly compare the results of the present study with the available literature because the parameters used, such as ultrasonic activation time and application methods vary.

In the present study, to standardize the effect of spatulation, MTA was condensed with the use of a compaction machine which applied a constant 3.22 MPa force for 1 min. The condensation pressure might have packed the powder molecules closer together, limited the formation of microchannels, compromising the ingress of water to hydrate MTA Angelus adequately. This might cause a reduction in crystalline formation due to lack of sufficient space for water molecules [8]. However, this is not in accordance with ProRoot MTA, which was not affected by the mixing regime applied.

Mechanical mixing of encapsulated materials resulted in a more thorough wetting of the powder particles compared with conventional vibratory action [37]. Moreover, the combination of mechanical mixing and indirect ultrasonic activation reduced porosity when compared with manual mixing and hand condensation [17]. Porosity in cement systems can stem either from water which was not consumed in the setting reaction or, from dry spots and air bubbles [38]. Coomaraswamy et al. [38] have shown that porosity increase was strongly related to bismuth oxide content as a result of more unused water in the setting reaction. In an attempt to overcome this internal flaw, Grazziotin-Soarez et al. [23] evaluated a modification of MTA Angelus without bismuth oxide. They reported that the lack of bismuth oxide was associated with a large amount of calcium and a morphology characteristic of calcium hydroxide in EDX/SEM analysis. However, our results revealed that the intensity of bismuth oxide in ProRoot MTA groups was higher than those of MTA Angelus. Therefore, bismuth oxide cannot be merely responsible for the missing calcium hydroxide phase formation in some MTA Angelus groups. In an EDX analysis, Camilleri et al. [26] reported that MTA Angelus displayed the presence of free calcium oxide, aluminum oxide, and silicon dioxide in the unhydrated cement. This indicates that the clinkering was not complete mostly due to a too low maximum sintering temperature, leading to a potential important variability in its mineralogy depending on the sintering conditions. Thus, from one batch to another, the clinker of MTA Angelus is expected to be variable with as a consequence to its properties. This might also explain why calcium hydroxide phase formation was detected only in the mechanically mixed and ultrasonicated MTA Angelus group, whereas it was observed in all ProRoot groups.

One limitation inherent to our study is that under test conditions, the whole surface of the specimen was in contact with water whereas in clinical situations, only a small part of the MTA would be in contact with a wet cotton pellet or the periapical tissues. Instead of phosphate-buffered solution (PBS) or Hank's Balanced Salt Solution (HBSS), the MTA samples in this study were in contact with water. However, our results were in agreement with previous studies [26, 39] who reported similar peaks in MTA samples which were in contact with HBSS. Furthermore, Camilleri et al. [30] reported that the hydration of the core material was not affected by contact with the different solutions, namely water and HBSS. Therefore, our results may at least be qualitatively transferable to a clinical situation, where MTA releases calcium hydroxide as it comes in contact with water [1]. Also, in order to minimize the potential to miss the calcium hydroxide crystals formed on the surface, samples used in this study were not subjected to grinding prior to XRD analysis in accordance with Nekoofar et al. [7] and Song et al. [24].

The main objective of our study was to analyze the hydration products of MTA to see if the mixing or placement regime had a significant role on the hydration mechanism. For ProRoot MTA, the mixing and placement methods did not confer any significant difference, whereas for MTA Angelus, mechanical mixing combined with ultrasonic placement enhanced the calcium hydroxide phase formation.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest..

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent For this type of study, informed consent is not required.

References

1. Fridland M, Rosado R (2003) Mineral trioxide aggregate (MTA) solubility and porosity with different water-to-powder ratios. J Endod 29:814–817
2. Camilleri J, PittFord TR (2006) Mineral trioxide aggregate: a review of the constituents and biological properties of the material. Int Endod J 39:747–754
3. Camilleri J (2007) Hydration mechanisms of mineral trioxide aggregate. Int Endod J 40:462–470

4. Fridland M, Rosado R (2005) MTA solubility: a long term study. J Endod 31:376–379
5. Sarkar NK, Caicedo R, Ritwik P, Moiseyeva R, Kawashima I (2005) Physicochemical basis of the biologic properties of mineral trioxide aggregate. J Endod 31:97–100
6. Holt DM, Watts JD, Beeson TJ, Kirkpatrick TC, Rutledge RE (2007) The anti-microbial effect against enterococcus faecalis and the compressive strength of two types of mineral trioxide aggregate mixed with sterile water or 2% chlorhexidine liquid. J Endod 33: 844–847
7. Nekoofar MH, Davies TE, Stone D, Basturk FB, Dummer PMH (2011) Microstructure and chemical analysis of blood-contaminated mineral trioxide aggregate. Int Endod J 44:1011–1018
8. Nekoofar MH, Adusei G, Sheykhrezae MS, Hayes SJ, Bryant ST, Dummer PM (2007) The effect of condensation pressure on selected physical properties of mineral trioxide aggregate. Int Endod J 40: 453–461
9. Basturk FB, Nekoofar MH, Günday M, Dummer PM (2013) The effect of various mixing and placement techniques on the compressive strength of mineral trioxide aggregate. J Endod 39:111–114
10. Küçükkaya Eren S, Görduysus MÖ and Şahin C (2017) Sealing ability and adaptation of root-end filling materials in cavities prepared with different techniques. Microscopy Research and Technique
11. Torabinejad M, Hong CU, McDonald F, Pitt Ford TR (1995) Physical and chemical properties of a new root-end filling material. J Endod 21:349–353
12. Nekoofar MH, Aseeley Z, Dummer PMH (2010) The effect of various mixing techniques on the surface microhardness of mineral trioxide aggregate. Int Endod J 43:312–320
13. Lawley GR, Schindler WG, Walker IIIWA, Kolodrubetz D (2004) Evaluation of ultrasonically placed MTA and fracture resistance with intracanal composite resin in a model of apexification. J Endod 30:167–172
14. Yeung P, Liewehr FR, Moon PC (2006) A quantitative comparison of the fill density of MTA produced by two placement techniques. J Endod 32:456–459
15. Basturk FB, Nekoofar MH, Gunday M, Dummer PM (2014) Effect of various mixing and placement techniques on the flexural strength and porosity of mineral trioxide aggregate. J Endod 40:441–445
16. Shahi S, Rahimi S, Yavari HR, Samiei M, Janani M, Bahari M, Abdolrahimi M, Pakdel F, Aghbali A (2012) Effects of various mixing techniques on push-out bond strengths of white mineral trioxide aggregate. J Endod 38:501–504
17. Sisli SN, Ozbas H (2017) Comparative micro–computed tomographic evaluation of the sealing quality of ProRoot MTA and MTA Angelus apical plugs placed with various techniques. J Endod 43:147–151
18. Lee Y-L, Lee B-S, Lin F-H, Lin AY, Lan W-H, Lin C-P (2004) Effects of physiological environments on the hydration behavior of mineral trioxide aggregate. Biomaterials 25:787–793
19. Camilleri J (2008) Characterization of hydration products of mineral trioxide aggregate. Int Endod J 41:408–417
20. Islam I, Chng H, Yap A (2006) X-ray diffraction analysis of mineral trioxide aggregate and Portland cement. Int Endod J 39:220–225
21. Belío-Reyes IA, Bucio L, Cruz-Chavez E (2009) Phase composition of ProRoot mineral trioxide aggregate by X-ray powder diffraction. J Endod 35:875–878
22. Camilleri J (2010) Hydration characteristics of calcium silicate cements with alternative radiopacifiers used as root-end filling materials. J Endod 36:502–508
23. Grazziotin-Soares R, Nekoofar M, Davies T, Bafail A, Alhaddar E, Hübler R, Busato A, Dummer P (2014) Effect of bismuth oxide on white mineral trioxide aggregate: chemical characterization and physical properties. Int Endod J 47:520–533
24. Song J-S, Mante FK, Romanow WJ, Kim S (2006) Chemical analysis of powder and set forms of Portland cement, gray ProRoot MTA, white ProRoot MTA, and gray MTA-Angelus. Oral Surg Oral Med Oral Pathol Oral Radiol Endodontol 102:809–815
25. Park J-W, Hong S-H, Kim J-H, Lee S-J, Shin S-J (2010) X-ray diffraction analysis of white ProRoot MTA and Diadent BioAggregate. Oral Surg Oral Med Oral Pathol Oral Radiol Endodontol 109:155–158
26. Camilleri J, Sorrentino F, Damidot D (2013) Investigation of the hydration and bioactivity of radiopacified tricalcium silicate cement, biodentine and MTA Angelus. Dent Mater 29:580–593
27. Gandolfi MG, Van Landuyt K, Taddei P, Modena E, Van Meerbeek B, Prati C (2010) Environmental scanning electron microscopy connected with energy dispersive X-ray analysis and Raman techniques to study ProRoot mineral trioxide aggregate and calcium silicate cements in wet conditions and in real time. J Endod 36: 851–857
28. Komabayashi T, Spångberg LSW (2008) Comparative analysis of the particle size and shape of commercially available mineral trioxide aggregates and Portland cement: a study with a flow particle image analyzer. J Endod 34:94–98
29. Dammaschke T, Gerth HU, Züchner H, Schäfer E (2005) Chemical and physical surface and bulk material characterization of white ProRoot MTA and two Portland cements. Dent Mater 21:731–738
30. Camilleri J, Formosa L, Damidot D (2013) The setting characteristics of MTA plus in different environmental conditions. Int Endod J 46:831–840
31. Ha WN, Kahler B, Walsh LJ (2014) Particle size changes in unsealed mineral trioxide aggregate powder. J Endod 40:423–426
32. Roy RA, Ahmad M, Crum LA (1994) Physical mechanisms governing the hydrodynamic response of an oscillating ultrasonic file. Int Endod J 27:197–207
33. Duque J, Fernandes S, Bubola J, Duarte M, Camilleri J and Marciano M (2017) The effect of mixing method on tricalcium silicate-based cement. Int Endod J
34. Parashos P, Phoon A and Sathorn C (2014) Effect of ultrasonication on physical properties of mineral trioxide aggregate. Biomed Res Int 2014
35. Matt GD, Thorpe JR, Strother JM, McClanahan SB (2004) Comparative study of white and gray mineral trioxide aggregate (MTA) simulating a one- or two-step apical barrier technique. J Endod 30:876–879
36. Aminoshariae A, Hartwell GR, Moon PC (2003) Placement of mineral trioxide aggregate using two different techniques. J Endod 29:679–682
37. Fleming G, Marquis PM, Shortall A (1999) The influence of clinically induced variability on the distribution of compressive fracture strengths of a hand-mixed zinc phosphate dental cement. Dent Mater 15:87–97
38. Coomaraswamy KS, Lumley PJ, Hofmann MP (2007) Effect of bismuth oxide radioopacifier content on the material properties of an endodontic Portland cement–based (MTA-like) system. J Endod 33:295–298
39. Camilleri J, Sorrentino F, Damidot D (2015) Characterization of unhydrated and hydrated BioAggregate™ and MTA Angelus™. Clin Oral Investig 19:689–698