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Research Article

Chemical Composition of Pozzolan-Based Mineral Trioxide Aggregate: An X-Ray Diffraction Analysis

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Abstract

Aim: The aim of this study was to determine the constitution of two pozzolan-based bioactive endodontic cements using x-ray diffraction analysis.

Materials and Methods: The materials analyzed were Endocem MTA and Endocem Zr (Maruchi, Wonju-si, Korea). XRD sample holders with internal dimensions of 0.1 mm high and a 20.0 mm diameter were used. Samples were presented in powder form on a single crystal sample holder to avoid unwanted diffraction peaks. X-ray diffractometer (XRD, Bruker D2 Phaser, Karlsruhe, Germany) used Ni-filtered CuKa radiation at 30 Kv and 10 mA Cu tube with 1,54184 Å wavelength. Scans were undertaken in the range 10°–70° 2θ. Phase identification was accomplished by use of search- match software utilizing International Centre for Diffraction Data database (ICDD, Pennsylvania, PA, USA).

Results: Endocem MTA analyzed by XRD were comprised of bismuth oxide (at 25.844°and 27,014° 2θ), tricalcium silicate oxide (at 29.541°and 32.207° 2θ), dicalcium silicate (at 32.186° 2θ) and calcium hydroxide (at 18.171° and 34.032° 2θ). Endocem Zr analyzed by XRD were comprised of tricalcium silicate oxide (at 29.564° and 32.215° 2θ), dicalcium silicate (at 32.171° 2θ) and calcium hydroxide (at 18.142° and 34.25° 2θ). Endocem Zr and Endocem MTA have a similar chemical composition with some differences: Endocem Zr contains a significant amount of zirconium and Endocem MTA contains bismuth oxide.

Conclusion: The XRD analysis showed that unhydrated MTA to be composed primarily of tricalcium silicate, dicalcium silicate and bismuth oxide. The formation of calcium hydroxide showed that MTA could start hydrating even in the absence of water, probably because of the atmospheric moisture.

Keywords: Bismuth Oxide; Endo CEM; Mineral Trioxide Aggregate; Pozzolan; Portland

Introduction

Mineral Trioxide Aggregate (MTA) is a calcium silicate-based bioactive material that is mainly composed of Portland cement and bismuth oxide [1]. MTA is a powder that consists of fine hydrophilic particles that harden in contact with water [2]. MTA is prepared as a mixture of powder and water and is used in a slurry form, which slowly hardens in the oral environment.

The main components of MTA are tricalcium silicate, dicalcium silicate, tricalcium aluminate and tetracalcium aluminoferrite [3].

Although MTA is considered to have ideal properties, it has some drawbacks such as its high-cost, difficult handling characteristics, long setting time and discoloration [1]. These shortcomings led to the continuous efforts in developing modified versions of MTA.

First modification was performed on the original gray formula (ProRoot MTA) by the manufacturer (Dentsply Tulsa Dental, Tulsa, OK, USA) producing a tooth-colored formulation in order to overcome the discoloration problem. Tooth-colored MTA mainly differs from the gray MTA by the absence of iron [4]. Later, MTA Angelus was introduced as an alternative to ProRoot MTA with its lower price and similar constituents [2]. The chemical difference between ProRoot MTA and MTA Angelus is that the latter one lacks calcium sulfate dehydrate as one of its main compounds, resulting in a shorter setting time than ProRoot MTA [5].

A pozzolan-based bioactive endodontic cement named Endocem MTA (Maruchi, Wonju, Korea) was recently introduced to the dental market. The manufacturer claims that Endocem MTA has rapid setting time up to 4 minutes and easier manipulation properties with a chemical composition comparable to that of commercially available portland-based MTA [6,7]. Endocem Zr (Maruchi, Wonju, Korea) was developed by the same company and contains the same base material as original Endocem MTA, with the addition of zirconium oxide as the radiopacifier.

X-ray diffraction (XRD) is a useful method to identify the crystalline structures and hydration properties of dental cements [2,8,9]. Islam., *et al*. [10]. reported that the main constituents of portland-based MTA were tricalcium silicate, tricalcium aluminates, calcium silicate, and bismuth oxide. Yet, to date there have been no such studies on pozzolan-based mineral trioxide aggregate. Therefore, the aim of this study was to identify the existing chemical components of Endocem MTA and Endocem Zr using X-ray diffraction analysis. The main hypothesis is that the crystalline structures of pozzolan-based MTA would be similar to that of the portland-based MTA. Also, the second part of the study compared the chemical differences between Endocem MTA and Endocem Zr using XRD analysis.

Materials and Methods

The materials analyzed were Endocem MTA and Endocem Zr (Maruchi, Wonju-si, Korea). XRD sample holders with internal dimensions of 0.1 mm high and a 20.0 mm diameter were used. Samples were presented in powder form on a single crystal sample holder to avoids unwanted diffraction peaks. Each specimen was placed into the sample holder and packed with a glass slide to provide a uniform surface.

X-ray diffractometer (XRD, Bruker D2 Phaser, Karlsruhe, Germany) used Ni-filtered CuKa radiation at 30 Kv and 10 mA Cu tube with 1,54184 Å wavelength. Scans were undertaken in the range 10°–70° 2Ө.

Each crystalline substance has a unique diffraction pattern with several X-ray peaks. The peaks at that intensity representing the diffraction patterns of the tested materials were matched with the standard Powder Diffraction Files (PDF) found in the International Centre for Diffraction Data (ICDD) database.

Results

Endocem MTA analyzed by XRD were comprised of bismuth oxide (α-Bi_2O_3, ICDD 00-027-0053) showed the strongest peaks at 25.844°and 27,014° 2Ө, tricalcium silicate (Ca_3SiO_5, ICDD 00-055-0738) with peaks at 29.541°and 32.207° 2Ө, dicalcium silicate (Ca_2SiO_4) indicated by the strongest peaks at 32.186° 2Ө, calcium carbonate ($CaCO_3$ ICDD 00-005-0586) with major peaks at 29.368 and 36.017 2Ө and calcium hydroxide [$Ca(OH)_2$ ICDD 00-044-1481] indicated by the strongest peak at 18.171° and 34.032° 2Ө (Figure 1).

Figure 1: XRD Analysis of Endocem MTA reveals A) Bismuth Oxide peaks B) Calcium Hydroxide peaks C) Absence of Zirconium oxide peaks and D) Calcium Carbonate peaks

Endocem Zr analyzed by XRD showed major peaks of tricalcium silicate at 29.564° and 32.215° 2Ө, dicalcium silicate at 32.171° 2Ө and calcium hydroxide at 18,142° and 34.25° 2Ө (Figure 2).

Endocem Zr and Endocem MTA have a similar chemical composition with some differences: Endocem Zr contains a significant amount of zirconium and Endocem MTA contains bismuth oxide.

Figure 2: XRD Analysis of Endocem Zr reveals A) Absence of Bismuth Oxide peaks B) Calcium Hydroxide peaks C) Zirconium oxide peaks and D) Calcium Carbonate peaks

Discussion

X-ray diffraction analysis is a useful tool in identifying the major constituents or compounds present in a material [10]. This will help us understand a material's physical, chemical and mechanical properties. We used X-ray diffraction analysis to detect the chemical composition of two pozzolan-based mineral trioxide aggregate, namely; Endocem MTA and Endocem Zr.

Mineral trioxide aggregate contains a significant amount of Portland cement, whose major components have been extensively studied [2,8]. One of the main phases of unhydrated cements is tricalcium silicate, accounting for a large portion of the MTA [1,11] and Portland cement powder [12,13]. Our study revealed that it is also one of the main components of pozzolan-based MTA.

Pozzolan based MTA appears to be a modified version of MTA. Endocem MTA and Endocem Zr contain high amount of calcium oxide. A clear difference was noticed in the finding that Endocem Zr contained a significant amount of zirconium oxide instead of bismuth oxide as a radiopacifier. Bismuth oxide, which is one of the main compounds of MTA, inhibits cell growth and interferes with the bioactivity of the material [8, 14]. Camilleri [15] reported that the calcium hydroxide phase formation is reduced in hydrated MTA when compared with a Portland cement with no Bi_2O_3. Whereas other radiopacifies work as fillers, Bi_2O_3 in MTA is not inert and affects the MTA hydration process [14]. Bismuth oxide has the potential to decrease the mechanical strength and increase the porosity of Portland cement, as a result of the larger amount of unreacted water [16]. Camilleri., *et al*. [17] reported that the zirconium oxide can be preferred to the bismuth oxide since zirconium oxide was an inert compound [18]. These, along with bismuth oxide's potential to cause tooth discoloration [19] may be the reasons why manufacturers develop forms of MTA with other radiopacifies such as zirconium oxide that is present in Endo Cem Zr.

The studies upon the hydration of MTA revealed that Portlandite, which is crystalline calcium hydroxide, was identified only in the hydrated samples). The only instance where calcium hydroxide formation is absent in a hydrated MTA is when the material was mixed with blood [20]. Grazziotin-Soares., *et al*. [14] reported that the presence of $Ca(OH)_2$ in the unhydrated powder form was detected yet it was unexpected. Our study showed that the unhydrated powder form of MTA also revealed calcium hydroxide, indicating that the material could start hydrating even in the absence of water, probably because of the atmospheric moisture [21].

Both MTA forms include similar constituents; tricalcium silicate, dicalcium silicate and calcium carbonate. Endocem MTA has bismuth oxide as a radiopacifier while Endocem Zr included zirconium oxide instead. The powder Endocem MTA and Endocem Zr exhibited peak of calcium hydroxide, which is considered unusual, yet we believe that the hydrophillic particles of the material interacts with the environmental humidity quite easily. Thus, the material should not be further used once the lid is opened.

Conclusion

The XRD analysis showed that unhydrated Endocem MTA was composed primarily of tricalcium silicate, dicalcium silicate and bismuth oxide. Endocem Zr contains zirconium oxide instead of bismuth oxide as a radiopacified. The formation of calcium hydroxide in an unhydrated MTA showed that the material could start hydrating even in the absence of water, probably because of the atmospheric moisture.

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