
X-Ray diffraction analysis of White ProRoot MTA and Diadent BioAggregate

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BioAggregate is a new product that was formulated for root-end filling, perforation repair, and pulp capping. This study examined the chemical differences between white mineral trioxide aggregate (MTA) and BioAggregate in both powder and set forms using X-ray diffraction. The results showed that white MTA and BioAggregate have a similar chemical composition with some differences: BioAggregate contains a significant amount of tantalum oxide instead of bismuth oxide. In both groups, similar peaks were observed in the set and powder form, but sharper and stronger peaks were observed in the powder samples. (*Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2010;109:155-158)

Since mineral trioxide aggregate (MTA) was introduced in the 1990s, its application has broadened rapidly in areas such as vital pulp therapy, perforation repair, retrograde filling, and apexification. For the past 10 years, there have been many reports of the superior effects of MTA regarding sealability, biocompatibility, and hard tissue-forming capacity.¹⁻³ Originally, MTA had a gray formula, but the manufacturer now produces a tooth-colored formulation, white MTA. According to earlier studies,^{4,5} white MTA differs from the original gray MTA in the absence of iron. More recently, a Brazilian product, MTA Angelus (Angelus Soluções Odontológicas, Londrina, Brazil) was introduced and is used in certain regions. MTA Angelus is composed of 80% Portland cement and 20% bismuth oxide. It is possible that more materials modified from the original MTA will be developed in the near future.

BioAggregate (Innovative BioCeramix, Vancouver, Canada) is a relatively new product available in Canada and is in the process of receiving Food and Drug Administration approval in the U.S.A. Its recommended clinical applications include retrograde root

filling, perforation repair, vital pulp therapy, etc. It is comprised of off-whitish fine particles, and the powder is mixed with deionized sterile water. Most of the constituents are similar to those in MTA. BioAggregate contains several mixed synthetic components: calcium silicate hydrate, calcium hydroxide, hydroxyapatite (HA), tantalum oxide, and amorphous silicon oxide.⁶ The manufacturing company claims that it is free of aluminum, minimizing any toxic effects to the human body. Because BioAggregate has been only recently available, there are not many studies about this material.⁷

X-Ray diffraction (XRD) is used to identify the crystalline structures of cements. Islam et al.⁸ reported that the main constituents of MTA were tricalcium silicate, tricalcium aluminates, calcium silicate, and bismuth oxide. Song et al.⁵ reported the presence of bismuth oxide, calcium silicate oxide, calcium carbonate, calcium phosphate, and calcium silicate in white MTA. There have been no such studies on BioAggregate. Therefore, XRD was used to identify the existing chemical components to precede further studies of BioAggregate.

This study examined the chemical differences between white MTA and BioAggregate in both powder and set forms using XRD.

MATERIALS AND METHODS

Sample preparation and XRD analysis

ProRoot MTA with a tooth-colored formula (White MTA; Dentsply Tulsa Dental, Tulsa, OK) and BioAggregate were used. For the powder sample, 1.5 g of each specimen was placed into the sample holder and then packed with a sterile glass slide to provide a uniform surface. For the set specimen, 0.5 g of powder was mixed with the liquid included in the package accord-

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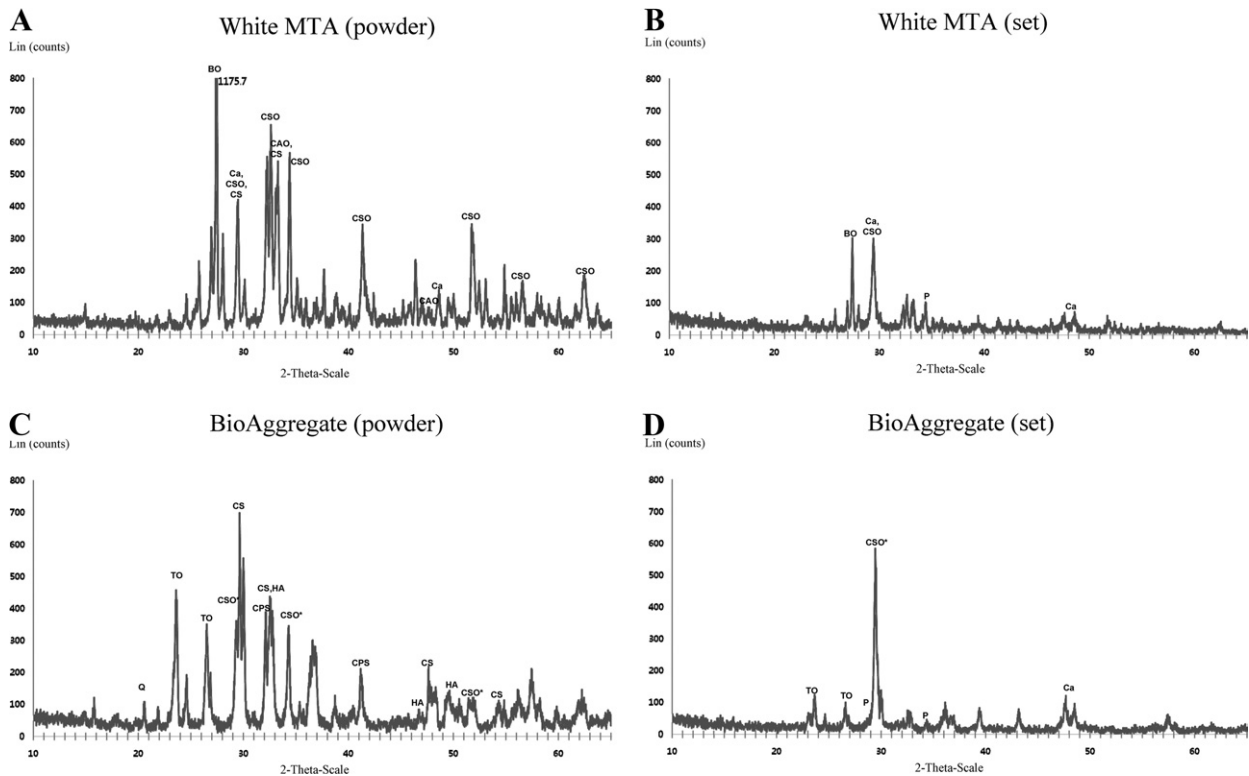


Fig. 1. X-Ray diffraction patterns of White MTA (A, B) and BioAggregate (C, D) in the powder (A, C) and set (B, D) forms. *BO*, bismuth oxide (bismite; $\alpha\text{-Bi}_2\text{O}_3$); *Ca*, calcite (CaCO_3); *CAO*, calcium aluminum oxide ($\text{Ca}_3\text{Al}_2\text{O}_6$); *CPS*, calcium phosphate silicate ($\alpha'\text{Ca}_2\text{SiO}_4 \cdot 0.05\text{Ca}_3(\text{PO}_4)_2$); *CSO*, calcium silicate oxide ($\text{Ca}_3(\text{SiO}_4)\text{O}$); *CSO**, calcium silicate oxide (Ca_3SiO_5); *HA*, hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$); *P*, portlandite ($\text{Ca}(\text{OH})_2$); *Q*, quartz ($\alpha\text{-SiO}_2$); *TO*, tantalum oxide (Ti_2O_5).

ing to the manufacturer's instructions. One-half gram of Biaggregate powder was mixed with 0.19 mL of liquid provided. The slurry mixed cement was placed onto a glass slide and the upper surface of the specimen was swept with a plastic spatula. The samples were set for 3 days at 37°C and 100% humidity in the incubator. Both powder and set materials were mounted for XRD analysis. A thin-film X-ray diffractometer (D8 advance; Bruker AXS, Madison, WI,) was set to run at 40 kV and 40 mA in continuous mode. The scan range was $10\text{-}65^\circ 2\theta$ with a scan speed of $2^\circ 2\theta$ per minute.

Data interpretation

Each constituent has characteristic diffraction patterns, and there may be multiple peaks in each sample owing to the cements consisting of several chemical compounds. Diffrac-Plus Evaluation software (version 9.0; Bruker AXS) was used for the analysis. The peaks of the diffraction patterns of each sample were matched with those of the standard data in the powder diffraction files (PDF).

RESULTS

Figure 1 shows the XRD patterns of the samples. The XRD patterns of set and powder forms showed similar peaks in both the White MTA and BioAggregate groups. However, the intensities of peaks in the set forms had decreased after hydration.

In the White MTA group, large peaks representing bismuth oxide, calcium silicate oxide, calcite, were observed at 27.395 , 29.425 and $29.406^\circ 2\theta$, respectively. Calcium aluminum oxide and calcium silicate were also identified.

The chemical compositions of White MTA and BioAggregate were similar, but there were some noticeable differences. White MTA contained a significant amount of synthetic bismuth oxide (bismite). There were no peaks for bismuth oxide in the BioAggregate sample. Instead, it showed strong peaks at 23.580 and $26.587^\circ 2\theta$, representing tantalum oxide. Other than tantalum oxide, the major components in the BioAggregate group were calcium silicate oxide and calcium silicate, which has characteristic peaks at 34.250 and $29.626^\circ 2\theta$. Peaks for

HA, quartz, calcium phosphate silicate, and calcite were also observed. Calcite was identified in the set form of BioAggregate but not in the powder sample.

Portlandite (calcium hydroxide) was identified in the set form of both cements. However, it was not observed in the powder forms.

DISCUSSION

Mineral trioxide aggregate contains a significant amount of Portland cement, whose major components have been investigated and identified. Earlier studies reported that its principle compounds are tricalcium silicate, tricalcium aluminate, tricalcium oxide, silicate oxide, and bismuth oxide.^{4,9} The results from the White MTA samples are generally in agreement with those studies. Synthetic bismuth oxide, calcium silicate oxide, and calcite demonstrated strong peaks. There were also multiple small peaks in the White MTA sample that were not clearly identified in the powder and set forms. Song et al.⁵ reported that white MTA contained 56.7% bismuth oxide and 34.1% calcium silicate oxide. Quantitative analysis was not carried out in the present study.

BioAggregate appears to be a modified version of MTA. BioAggregate does not contain calcium aluminum oxide, calcium magnesium aluminum oxide, or bismuth oxide. A clear difference was noticed in the finding that BioAggregate contained a significant amount of tantalum oxide instead of bismuth oxide as a radiopacifier. Tantalum oxide is not commonly used in the field of endodontics. Tantalum and tantalum oxide have been used as sutures, plates, and membranes in orthopedics on account of their inertness.^{10,11} In the 1990s, tantalum was evaluated as a possible radiopacifier in composite resin.¹² Steinemann reported a strong inhibition zone when osteoblasts were grown, whereas fibroblasts proliferated well on the tantalum disc.¹³ Because tantalum oxide is the major difference between MTA and BioAggregate, it may be important to examine the differences between bismuth oxide and tantalum oxide in terms of toxicity and biocompatibility. The effects of containing tantalum oxide instead of bismuth oxide are unknown. Bismuth oxide, which is one of the main compounds of MTA, may not contribute to its excellent biocompatibility. Camilleri et al.¹⁴ reported no cell growth over bismuth oxide. However, they could not infer whether those results were due to surface roughness and/or the chemical nature of the material.

The other noticeable difference between the products was the existence of HA. In orthopedics, HA particles have been used as the components of various bioactive cements, and they are known to strengthen the cement.¹⁵ Based on the information provided by the man-

ufacturing company,⁶ Bioaggregate contains HA. However, peaks for HA in set form of Bioaggregate were not noticeable in the present study. Since peaks from set Bioaggregate were generally weak, it might be difficult to catch the exact signal, especially when the component is minor.

Lee et al.¹⁶ performed XRD of powder and hydrated MTA samples. Their results revealed that a similar XRD powder pattern was observed in the hydrated samples, with decreased intensities for the peaks after hydration. Song et al.⁵ suggested no noticeable differences in the composition and crystalline structure between the powder and set forms. The present results showed similar patterns: Similar peaks were observed in the set and powder form, with the powder form showing sharper and stronger peaks.

Portlandite, which is crystalline calcium hydroxide, was identified only in the set samples. These results are in agreement with Lee et al.,¹⁶ where Portlandite was identified only in the hydrated MTA.

A modified or newly developed bioactive material inspired from the existing one can be used if it is proved to be as good or can overcome a weakness of its predecessor. Indeed, MTA was modified successfully from Portland cement. However, meticulous studies will be needed before newly introduced products or materials modified by adding a different component are applied clinically. The findings from Owadally and Pitt Ford¹⁷ showed indirectly that newly formulated products should be investigated to determine if they have compatible properties even when the majority of its constituents are similar to the existing materials. For example, some studies comparing white and gray MTA showed conflicting results, even though both products are almost identical except for a few components.^{14,18} Therefore, intensive studies of BioAggregate may be essential in the future.

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