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X-RAY Diffraction Analysis of Indigenously Developed Bioceramic Pulp Capping Agent Ceremagnum Plus with Conventional One: An Invitro Study

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pulp capping, crystalline, bio activity, ceramics, phase identification

ABSTRACT

Objective: This study aimed to compare the crystallographic properties of a novel bioceramic material, Ceremagnum Plus, with MTA-Angelus, using X-ray diffraction (XRD) analysis to evaluate their phase composition, crystallinity, and amorphous content.

Methods: Ceremagnum Plus was synthesized using KH_2PO_4 and MgO in a 1:1 molar ratio, sintered at 900°C, and combined with $CaSiO_3$, cerium oxide (CeO_2), zirconium oxide (ZrO_2), and NaF. MTA-Angelus was used as the control. The materials were prepared, incubated at 37°C with 100% humidity for three days, and then analyzed using XRD. The diffraction patterns were matched with the International Centre for Diffraction Data (ICDD) database for phase identification.

Results: MTA-Angelus exhibited 72.4% crystallinity, with distinct peaks at $2\theta \approx 29.0°$, 34.4°, and 39.4°, corresponding to calcium silicate phases (C_3S, C_2S) and calcium hydroxide ($Ca(OH)_2$), with bismuth oxide (Bi_2O_3) acting as a radiopacifier. In contrast, Ceremagnum Plus showed 25.5% crystallinity and 74.5% amorphous content, with dominant peaks at $2\theta \approx 42.45°$, suggesting a composition of phosphate-based glass-ceramics, calcium silicate, and magnesium phosphate phases. The presence of cerium oxide (CeO_2) at 62.26° and zirconium oxide (ZrO_2) at 28.55° was also confirmed.

Conclusion: MTA-Angelus demonstrated a more ordered crystalline structure, contributing to its stability and long-term bioactivity, whereas Ceremagnum Plus, with its higher amorphous content, may exhibit faster setting, enhanced bioactivity, and improved handling properties. These findings suggest that Ceremagnum Plus could be a promising alternative to MTA-Angelus, with potential clinical advantages that warrant further investigation through mechanical and biological studies.

1. Introduction

Since its first introduction in the 1990s, mineral trioxide aggregate (MTA) has shown itself to be an exceptional material due to its superior sealing ability, biocompatibility, and capability to create hard tissue. Additionally, it has perfect qualities like radiopacity, dimensional stability, antibacterial activity, and moisture tolerance. [1]MTA is currently being used clinically for a number of endodontic and restorative dental applications, including apexification, vital pulp therapy, root perforation repair, and root-end fillings.[2]

Despite being thought to having perfect qualities, MTA's use has been restricted because of its high cost, challenging handling qualities, lengthy setting time, and discolouration possibility.[3]Due to these MTA flaws, ongoing attempts are made to create updated versions of MTA.A novel material alternative to MTA has claimed to have finer particle size than the currently available MTA products named Ceremagnum plus.X-ray diffraction (XRD) is a potent technique for identifying and characterizing the materials' crystalline phase composition.The aim of this research was to examine and contrast the crystal structures of Ceremagnum plus and MTA-Angelus.[4]

2. Methodology

Materials used in this study included Ceremagnum plus and white MTA-A (Angelus, Londrina, PR, Brazil). In the current study, water was used for mixing. For the formulation of novel bioceramic Ceremagnum plus: KH2PO4 and Mg0 were taken in molar ratio of 1:1 then sintered at 900 deg. for 3hrs.1000mg of KMgPO4,1000mg of CaSiO3,32.4mg of cerium oxide and 65.1mg of zirconium oxide as radio opacifiers ,39.45 mg NaF was weighed separately for the test group. Whereas 700mg for MTA Angelus was used as control group .Every weighed sample was put into an Epstein-Rosenberg tube. [5]

Mentioned proportions grinded into homogeneous powder for 10mins —100ul of CaCl2 liquid solution triturated by micropipette is added to 300mg of prepared powder. Each combination was made for the set form in accordance with the manufacturer's directions, then it was poured into a mold and the specimen's top surface was scraped with a spatula. Phase analysis was carried out for set forms of tested materials. The set samples were in the incubator for three days at 37°C and 100% humidity. After that, the set materials were mounted for XRD examination.[6] The current investigation used an X-ray diffractometer equipped with a Ni filter and CuKa radiation, operating at 45 kV voltage and 40 mA current. The 2–70°2θ scan range and 2°2θ scan speed per minute were set.[7]

Every phase, or crystalline material, in a compound has a distinct diffraction pattern made up of many X-ray peaks. The standard data recorded in the Powder Diffraction Files (PDF) located in the International Centre for Diffraction Data (ICDD) database matched the peaks at the specified intensity that represented the diffraction patterns of the tested materials.[8]The crystallinity percentage was determined using the peak intensity method, where the relative proportion of crystalline and amorphous phases was calculated by integrating the crystalline peak areas and comparing them with the total diffraction area.[6]

3. Results

The XRD results of the samples are shown in

Figures 1-2

XRD results as shown and explained below:

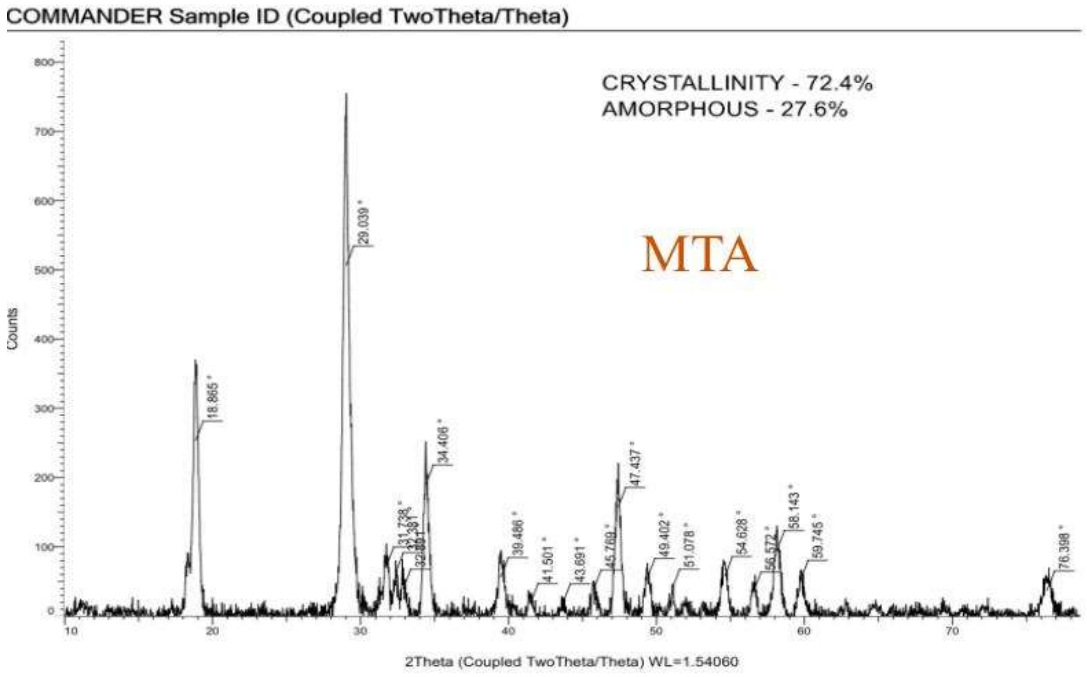

Figure 1

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1. Crystallinity and Amorphous Content:

- The material has 72.4% crystallinity, indicating a highly ordered structure.
- The remaining 27.6% amorphous phase suggests some degree of disordered or glassy material, which may contribute to its bioactivity and hydration properties.

2. Peak Identification:

- The strong peak around $2\theta \approx 29.0^\circ$ is a characteristic diffraction peak, likely corresponding to calcium silicate phases such as tricalcium silicate (C_3S) or dicalcium silicate (C_2S), which are key components of MTA.[9]
- Additional peaks at 18.8°, 34.4°, 39.4°, 47.4°, 54.6°, and others suggest the presence of various crystalline phases, likely including:
- Calcium hydroxide ($Ca(OH)_2$), which forms as a hydration product.
- Bismuth oxide (Bi_2O_3), a radiopacifier commonly added to MTA.

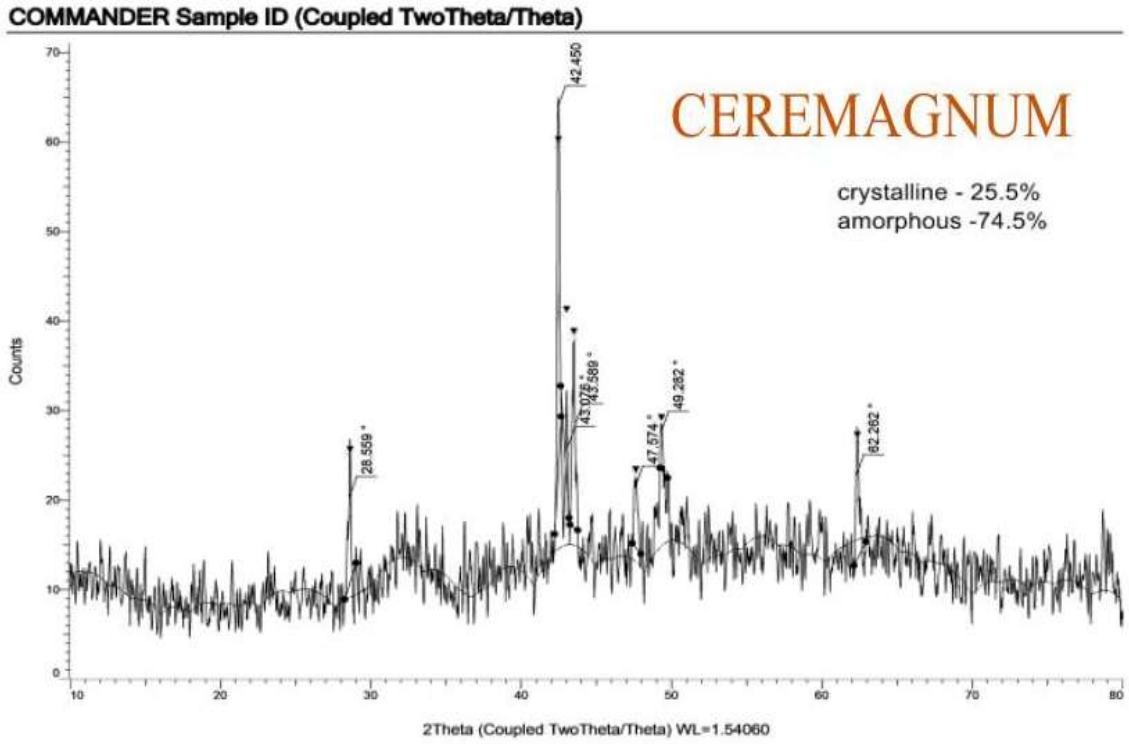

Figure 2

1. Crystallinity and Amorphous Content:

- The material has 25.5% crystallinity, which is relatively low.
- The 74.5% amorphous phase suggests a predominantly disordered structure, typical of glassy or cementitious materials with high reactivity.

2. Peak Identification and Material Composition:

- The most intense peak appears at $2\theta \approx 42.45^\circ$, indicating a dominant crystalline phase.
- Additional peaks are observed at 23.55°, 29.88°, 49.28°, 62.23°, among others.
- The radio-opacifiers cerium oxide showed peak at 62.26 whereas ZrO2 showed 28.55 deg 2θ phases.

The lower intensity and broad nature of peaks suggest the presence of calcium silicate or phosphate-based glass-ceramics, which are commonly used in bioactive dental materials.

- The broad amorphous hump in the background reinforces the idea that the material has a significant glassy or hydrated phase.

4.Discussion

A material's physical, chemical, and mechanical qualities can be better understood by identifying the main constituents or compounds that make up the material.MTA is now the most widely used and compelling substance in restorative endodontic procedures, such as vital pulp therapy, apical plug application, retrograde filling, and root perforation repair.[10] But MTA takes a long time to set, which could result in complications later on or possibly therapeutic failure. Several attempts have been made recently to shorten the setting time of MTA by utilizing various additives, such as Na2CO3 and Na2HPO4, without fully comprehending MTA's hydration behaviors. [11]However, MTA has a number of handling issues, including a tendency to wash away and a lack of cohesive property when combined with water.[12] One of the major challenges when applying the MTA to extremely small root canal areas is sandy property.[13]

The main components or compounds included in a material can be identified by the use of XRD.Finding the diffraction pattern of each crystalline phase, which is defined by a distinct collection of peaks (referred to as Bragg's peaks), with a certain diffracted intensity (y-axis) and diffracted angle at a given place, is the technique's fundamental idea. By comparing the data of the tested specimens using peaks and relative intensities with a sizable collection of "standard" data supplied by the ICDD, phase identification is achieved.[14]The X-ray diffraction (XRD) analysis revealed significant differences in the crystallinity and phase composition of the tested materials, highlighting their distinct structural characteristics and potential clinical implications.[15]The calcium silicate hydrate is the solitary amorphous phase in MTA, which is mostly composed of crystalline phases.[16] Tricalcium silicate was a component shared by all of the materials investigated in this investigation. The primary ingredient in the creation of calcium silicate hydrate, which gives cement its initial strength, is tricalcium silicate. [17]The tricalcium aluminate phase is one of the primary phases of MTA-A. Although tricalcium aluminate is the most reactive component and interacts with water quickly, it makes up a very small portion of the strength.[18]

The crystallinity of MTA-Angelus (72.4%) was substantially higher than that of Ceremagnum Plus (25.5%), suggesting that MTA possesses a more ordered and structured crystalline phase. This higher crystallinity may contribute to MTA’s known stability, mechanical strength, and slower degradation over time. In contrast, the high amorphous content in Ceremagnum Plus (74.5%) indicates a material with a more disordered, glassy structure, which may enhance its bioactivity and reactivity upon hydration. The higher amorphous phase content of Ceremagnum Plus suggests a potentially faster setting reaction and increased ion release, which could influence its biological properties.[19]

The XRD patterns identified characteristic peaks of key components in both materials. MTA exhibited strong peaks at 2θ ≈ 29.0°, 34.4°, and 39.4°, indicative of calcium silicate phases, primarily tricalcium silicate (C_3S) and dicalcium silicate (C_2S), which play a crucial role in the setting and bioactivity of MTA. The presence of calcium hydroxide ($Ca(OH)_2$) as a hydration product and bismuth oxide (Bi_2O_3) as a radiopacifier further confirmed the composition of MTA-Angelus.[20]

For Ceremagnum Plus, the dominant peak at 2θ ≈ 42.45° and additional peaks at 23.55°, 29.88°, 49.28°, and 62.23° suggest a different phase composition, likely consisting of phosphate-based glass-ceramics, calcium silicate, and magnesium phosphate phases. The presence of cerium oxide (CeO_2) at 62.26° and zirconium oxide (ZrO_2) at 28.55° confirms the incorporation of these radio-opacifiers, which enhance the material's radiopacity while potentially influencing its setting and mechanical properties.

The differences in crystallinity and phase composition between MTA and Ceremagnum Plus suggest that these materials may behave differently in clinical applications. MTA's high crystallinity and calcium silicate content contribute to its well-documented long-term stability, controlled hydration, and formation of calcium hydroxide, which is essential for stimulating hard tissue formation.[21] However, its drawbacks, including long setting time and difficult handling properties, remain a concern.On the other hand, Ceremagnum Plus, with its higher amorphous content and phosphate-based composition, may offer advantages such as a faster setting reaction, improved flowability, and increased bioactivity due to higher ion release. The presence of cerium oxide and zirconium oxide could enhance its mechanical properties and radiopacity while influencing its interaction with biological tissues.[22]

Limitations and Future Perspectives

While XRD analysis provided valuable insights into the structural and phase composition of both materials, further studies are necessary to evaluate their mechanical strength, solubility, and in vivo bioactivity. [23]Additional characterization techniques, such as scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS), could provide further confirmation of elemental composition and microstructural features. Moreover, in vitro and in vivo biocompatibility studies are required to determine the long-term clinical performance of Ceremagnum Plus compared to MTA-Angelus.Hence,all the tested materials were similarly composed of tricalcium silicate structures.In addition ceremagnum showed stronger peaks of tricalcium silicate which being the amorphous phase contributes to early strength of cement.Hence the indigenously prepared bioceramic material proved to be more amorphous than MTA with reduced setting time leading to its property to be used as a better pulp capping agent .

5.Conclusion

The findings from this study highlight the fundamental differences between MTA-Angelus and Ceremagnum Plus in terms of crystallinity, amorphous content, and phase composition. MTA exhibits a highly crystalline structure with dominant calcium silicate phases, while Ceremagnum Plus is predominantly amorphous with significant phosphate and magnesium-based phases. [24]This composition is likely intended to enhance biocompatibility, reactivity, and ion exchange, making it suitable for applications like pulp capping, dentin repair, or endodontic sealing. However, its lower crystallinity might mean slightly reduced mechanical strength compared to MTA, though it could

compensate with enhanced biological performance.

These differences suggest potential variations in clinical handling, setting behavior, and bioactivity, which should be further explored through additional studies to establish their suitability for specific endodontic and restorative applications.[25]

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