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Surface Morphological Alterations of An Indigenously Developed Bioceramic Phosphate-Based Cement: Ceremagnum Plus as An Indirect Pulp Capping Agent: An Invitro Study

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ABSTRACT

The present study investigates the amorphicity and crystallinity of a newly developed phosphate-based bioceramic cement, Ceramagnum Plus, formulated to enhance bioactivity and mechanical properties. The composition includes potassium magnesium phosphate ($KMgPO_4$), tricalcium silicate ($CaSiO_3$), cerium oxide (CeO_2), zirconium oxide (ZrO_2), and sodium fluoride (NaF), with controlled particle size to optimize setting behavior. The addition of $CaSiO_3$ promotes the formation of an amorphous silico-phosphate phase, balancing the crystalline $MgKPO_4 \cdot 6H_2O$ structure, as described in previous studies (Tay et al., 2007). The powders were finely ground to 400 mesh for improved homogeneity, consistency, and reaction kinetics.

The scanning electron microscopy (SEM) analysis revealed a heterogeneous microstructure, characterized by fine granular and needle-like structures, suggesting a well-integrated amorphous and crystalline phase. Comparative SEM analysis with MTA Angelus White, a widely used endodontic material, demonstrated finer particle size (0.5–2 µm) in Ceramagnum Plus, compared to larger granules (1–5 µm) and aggregates (~20 µm) in MTA, which may contribute to enhanced handling properties and controlled ion release. The presence of fluoride and cerium oxide in Ceramagnum Plus is expected to improve antibacterial activity, remineralization potential, and radiopacity.

This study highlights the structural advantages of Ceramagnum Plus over conventional MTA, providing a promising alternative for dental and orthopedic applications. Future investigations will focus on hydration kinetics, mechanical properties, and in vitro bioactivity to further validate its clinical potential.

Keywords: *Bioceramic cement, phosphate-based cement, Ceramagnum Plus, MTA Angelus, amorphicity, crystallinity, SEM analysis, bioactivity.*

1. INTRODUCTION

Vital pulp therapy aims to preserve the vitality of a tooth's pulp when the pulp is damaged by injury,decay,or infection but is capable of healing itself.The bioactivity of a restorative material usually denotes that it has a biological effect or is biologically active. This characteristic refers to the potential to induce specific and intentional mineral attachment to the dentine substrate and induce dentin bridge formation.[1]

The U.S. Food and Drug Administration authorized the use of mineral trioxide aggregate (MTA) in 1998 after it had been studied for endodontic uses since the early 1990s.For endodontic applications, MTA was regarded as the first hydraulic calcium silicate-based cement (CSC) to be patented.[2]

It has been used to treat pulpotomy, apexification, various root canal procedures, and pulp capping, which is the process of treating the essential pulp.MTA has a 92.5% success rate when applied to cure exposed pulp via direct pulp capping, according to a 10-year follow-up study conducted by Daniele. [3]In the treatment of dentine hypersensitivity, CSCs have more recently been employed as root canal sealants.

Furthermore, CSC can be used to treat teeth with necrotic pulp and an open apex to create an apical barrier (apexification).[4] When CSC comes into contact with synthetic tissue fluids, hydroxyapatite crystal formation starts, leading to the formation of calcified structures. [5]In fact, a chemical analysis of the elements present in MTA and Portland cement (PC) revealed that

PC has chemical elements similar to MTA, with the exception of bismuth, which is only found in MTA. As a result, PC has been thoroughly researched as a substitute for MTA.[6] However, it is crucial to emphasize that PC and MTA are not the same materials.The sluggish setup, high cost, and difficulty of manipulation are some of the drawbacks of MTA, which is used in Biodentine.

The sealing capacity of MTA cement was enhanced by the addition of calcium chloride. [7]Nevertheless, the hydration process will be impacted and the cement itself may suffer if a chemical addition is put to it before it has had time to hydrate.[8]According to reports, the material's physicochemical characteristics are significantly influenced by the size and surface area of the particles.[9]Hence an indigenously developed bioceramic named Ceremagnum plus with faster setting time has been tested for surface morphological alterations in this study

2. MATERIALS AND METHODS

Ceremagnum plus and white MTA-A (Angelus, Londrina, PR, Brazil) were among the materials used in this study. To create the novel bioceramic Ceremagnum plus: KH_2PO_4 and Mg0 were taken in a molar ratio of 1:1 and sintered at 900 degrees for three hours. As radio opacifiers, 1000 mg of $KMgPO_4$, 1000 mg of $CaSiO_3$, 32.4 mg of cerium oxide, and 65.1 mg of zirconium oxide along with 39.45 mg NaF were used. The test group was weighed separately, while the control group was given 700 mg of MTA Angelus. Each weighed sample was placed into an Epstein-Rosenberg tube.

The previously mentioned proportions ground into a uniform powder for ten minutes — 300 mg of the produced powder is mixed with 100 ul of $CaCl_2$ liquid solution that has been triturated using a micropipette.One operator mixed and handled each tested substance in accordance with the manufacturer's instructions. For each material, five disc-shaped samples measuring 5 mm in diameter and 2 mm in height were created by packing cement into a Perspex mold.[10]After that, the assembly (for each specimen) was wrapped in gauze soaked in deionized water and placed in an incubator set at 37°C and 100% humidity for three days, or until the material had completely cured before the previously mentioned examination.The specimens were then placed directly on aluminum stubs and viewed using a scanning electron microscope (SEM). [11]The specimens' surfaces were inspected, and their centers were further examined after they were fractured on a glass slab with a spatula. Imaging was carried out using secondary electron signals at 1000 magnification using 20kV accelerating voltage and a 9–14mm working distance. Each sample underwent two surface morphological examination and EDAX chemical analysis in distinct regions.

3. RESULTS

For the experimental group :Ceremagnum plus

FIGURE 1

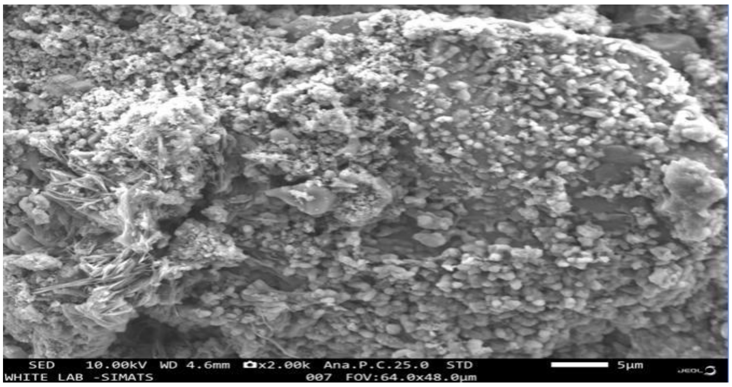
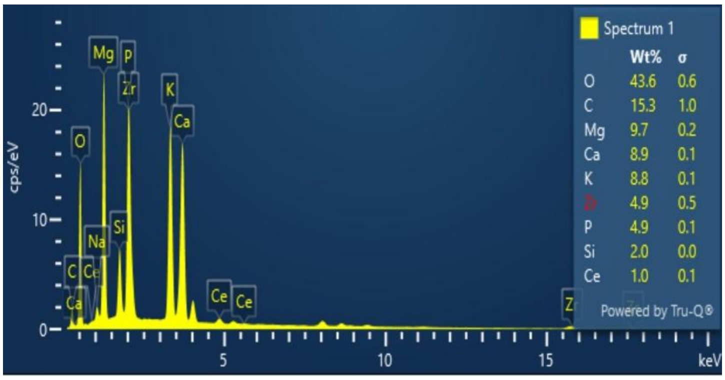


TABLE 1

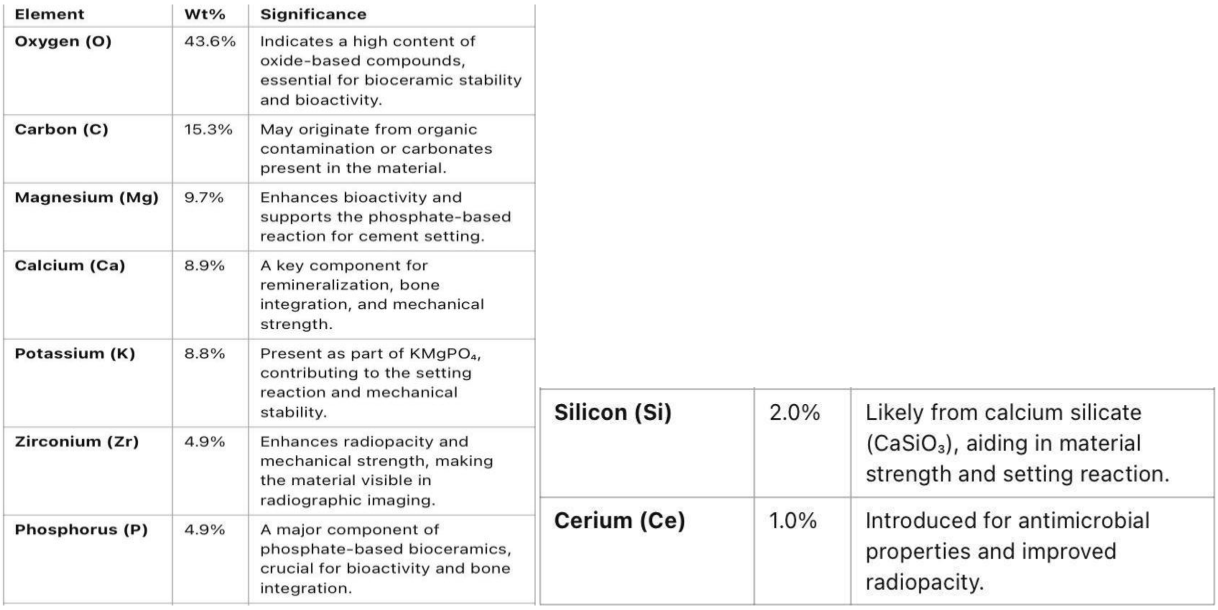


The SEM image with a scale of 5 µm of Ceremagnum plus reveals the microstructure of a bioceramic containing tricalcium silicate, $KMgPO_4$, cerium oxide, zirconium oxide, and NaF_2, suggesting a multi-phase material suitable for biomedical applications. The microstructure exhibits a highly irregular and porous morphology, essential for bioactivity and cell interaction. A combination of granular, agglomerated, and needle-like structures is observed, with fine granular particles likely attributed to tricalcium silicate, zirconium oxide, and cerium oxide, while needle-like formations could correspond to $KMgPO_4$ and sodium fluoride (NaF_2). The porous texture enhances ion exchange, a crucial feature for bone regeneration and dental applications. [12]

Elemental and phase distribution analysis suggests a heterogeneous composition, with cerium and zirconium oxides improving mechanical stability and radiopacity. [13]Functional properties include hydration potential from tricalcium silicate, structural reinforcement by $KMgPO_4$ and zirconium oxide, and controlled fluoride release from NaF_2, promoting remineralization. The porous structure aids biological integration, facilitating nutrient exchange and cell attachment. [14]The SEM and EDAX analyses highlight high oxygen and phosphorus content, indicative of phosphate phases crucial for bioactivity. The significant magnesium and potassium levels suggest a magnesium potassium phosphate cement (MKPC) base, known for rapid setting and biocompatibility. Calcium and silicon presence support a calcium silicate-phosphate hybrid structure, contributing to mechanical properties and hydration reactions.[15] Zirconium enhances radiopacity and strength, while cerium offers antimicrobial benefits.

The bioceramic material exhibits a faster setting time due to phosphate-based chemistry, higher radiopacity from zirconium and cerium, superior bioactivity through increased calcium, magnesium, and phosphate content, and improved mechanical strength from a balanced Ca-Si-P-Mg phase structure.

Elemental Composition (Wt%):



Element	Wt%	Significance
Oxygen (O)	43.6%	Indicates a high content of oxide-based compounds, essential for bioceramic stability and bioactivity.
Carbon (C)	15.3%	May originate from organic contamination or carbonates present in the material.
Magnesium (Mg)	9.7%	Enhances bioactivity and supports the phosphate-based reaction for cement setting.
Calcium (Ca)	8.9%	A key component for remineralization, bone integration, and mechanical strength.
Potassium (K)	8.8%	Present as part of $KMgPO_4$, contributing to the setting reaction and mechanical stability.
Zirconium (Zr)	4.9%	Enhances radiopacity and mechanical strength, making the material visible in radiographic imaging.
Phosphorus (P)	4.9%	A major component of phosphate-based bioceramics, crucial for bioactivity and bone integration.
Silicon (Si)	2.0%	Likely from calcium silicate ($CaSiO_3$), aiding in material strength and setting reaction.
Cerium (Ce)	1.0%	Introduced for antimicrobial properties and improved radiopacity.

For the control group :MTA

FIGURE 2

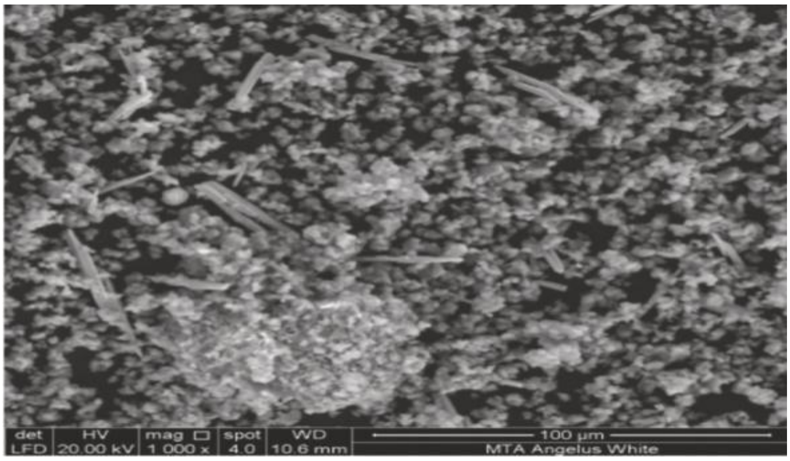
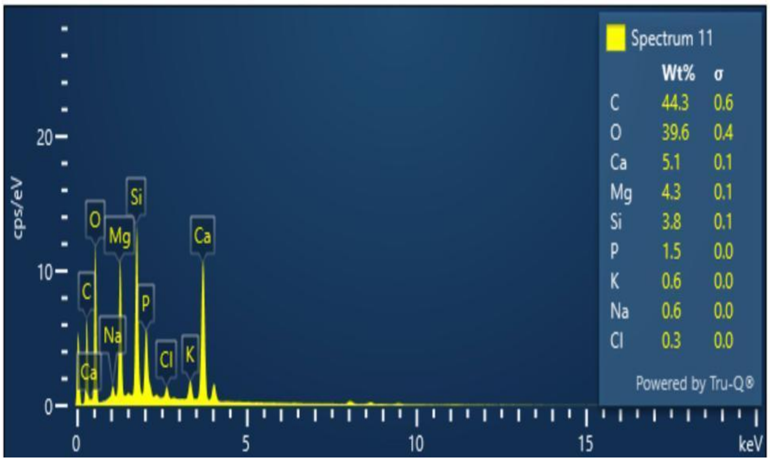


TABLE 2

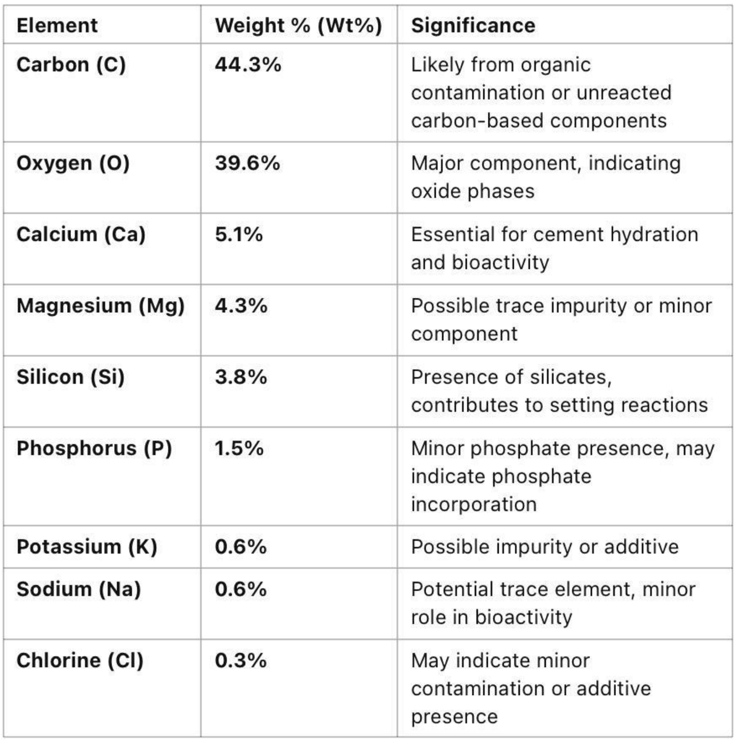


	Wt%	σ
C	44.3	0.6
O	39.6	0.4
Ca	5.1	0.1
Mg	4.3	0.1
Si	3.8	0.1
P	1.5	0.0
K	0.6	0.0
Na	0.6	0.0
Cl	0.3	0.0

For the control group, SEM analysis of MTA at 25-micron magnification showed grainy spherical particles with a cotton wool-like appearance. EDAX analysis of the indigenously prepared bioceramic material identified elemental peaks for Ca, PO_4, and Si. The microstructure displayed a heterogeneous particle distribution, including spherical or granular formations likely corresponding to tricalcium silicate (C_3S) and dicalcium silicate (C_2S), which are key to MTA's hydration and setting reactions. Needle-like structures could indicate ettringite or calcium hydroxide ($Ca(OH)_2$) crystals, while some areas exhibited dense agglomerations of partially reacted phases. Upon hydration, MTA forms calcium silicate hydrate (CSH) gel and calcium hydroxide, which support bioactivity by facilitating hydroxyapatite formation, maintaining an alkaline pH for antibacterial effects, and ensuring strong marginal adaptation. The material's moderate to high porosity enhances ion release and bioactivity but may compromise mechanical strength. [16]

Key EDAX observations include a high carbon content (~44.3%), potentially due to organic contamination or preparation artifacts, and a dominance of oxygen (~39.6%), reflecting an oxide-rich composition from calcium silicates. A lower-than-expected calcium content (5.1%) suggests hydration-related variations. The presence of silicon (3.8%) confirms calcium silicate phases' role in hydration and bioactivity, while phosphorus (1.5%) indicates lower phosphate incorporation than the new bioceramic (Ceramagnum Plus). Trace elements like magnesium (4.3%), potassium, sodium, and chlorine may originate from residual reactants, additives, or contamination during processing.

Elemental Composition (Wt%):



Element	Weight % (Wt%)	Significance
Carbon (C)	**44.3%**	Likely from organic contamination or unreacted carbon-based components
Oxygen (O)	**39.6%**	Major component, indicating oxide phases
Calcium (Ca)	**5.1%**	Essential for cement hydration and bioactivity
Magnesium (Mg)	**4.3%**	Possible trace impurity or minor component
Silicon (Si)	**3.8%**	Presence of silicates, contributes to setting reactions
Phosphorus (P)	**1.5%**	Minor phosphate presence, may indicate phosphate incorporation
Potassium (K)	**0.6%**	Possible impurity or additive
Sodium (Na)	**0.6%**	Potential trace element, minor role in bioactivity
Chlorine (Cl)	**0.3%**	May indicate minor contamination or additive presence

Comparison of SEM results:

Feature	**Modified Bioceramic (First Image)**	**MTA Angelus White (Second Image)**
Magnification & Scale	2000x, 5 µm scale (finer details visible)	1000x, 100 µm scale (broader overview)
Microstructure	Fine granular and needle-like structures	Granular and needle-like formations with larger particle agglomerations
Needle-Like Crystals	More defined and abundant, possibly related to $KMgPO_4$ and NaF_2	Present but less prominent, likely ettringite or calcium hydroxide
Particle Size	Smaller, more compact grains, likely due to additional components (cerium oxide, zirconium oxide)	Larger, loosely packed particles, contributing to high porosity
Porosity	Moderate porosity, likely tailored for controlled ion release and mechanical properties	High porosity, enhances ion exchange and bioactivity but may lower strength
Material Composition	Multi-phase ceramic system with added radiopacifiers and fluoride for improved properties	Primarily tricalcium silicate-based, with calcium hydroxide formation for bioactivity

Expected Bioactivity	Enhanced due to fluoride and $KMgPO_4$	High, due to calcium silicate hydration and hydroxyl ion release
Mechanical Strength	Potentially higher, as zirconium and cerium oxides improve stability	May be lower, as high porosity can affect mechanical integrity

4. DISCUSSION

Calcium phosphate crystal formation is a common characteristic of calcium silicate-based biomaterials, playing a crucial role in their bioactivity and integration with biological tissues. [17]Research by W. Zhao and Emran et al. has demonstrated that the microstructure and elemental composition of mineral trioxide aggregate (MTA) change over time, with scanning electron microscopy (SEM) revealing surfaces ranging from crystalline formations with depressions and microchannels to needle-like projections. Energy Dispersive X-ray Analysis (EDAX) of MTA confirms its composition as primarily calcium silicate, a factor influencing its setting reaction and biological interactions. To enhance these properties, the optimization of biomaterials requires extensive evaluation of their biocompatibility before they can be recommended for clinical applications. Phosphate-based cements with appropriate three-dimensional structures can effectively bind and concentrate endogenous bone morphogenetic proteins, potentially achieving osteoinductive properties.[18] Bioceramic-based cements, in particular, demonstrate unique bioactivity by setting and hardening in moist conditions, ultimately forming hydroxyapatite at the interface and creating a stable bond with dentin.[19] In such an environment, phosphate reacts partially with calcium silicate hydrogel and calcium hydroxide, leading to hydroxyapatite formation.

The present study compares the elemental composition of a newly formulated bioceramic, Ceramagnum Plus, with MTA Angelus using EDAX. The findings highlight notable differences in composition that influence their setting mechanisms, bioactivity, radiopacity, and mechanical properties. Ceramagnum Plus exhibits higher concentrations of calcium (Ca), magnesium (Mg), potassium (K), and phosphorus (P), indicating an enhanced bioactive formulation compared to MTA. Calcium and phosphorus are essential for hydroxyapatite formation, which promotes osteoconductivity and biointegration. Additionally, the inclusion of potassium magnesium phosphate ($KMgPO_4$) in Ceramagnum Plus supports a phosphate-driven setting reaction, distinguishing it from the calcium silicate hydration mechanism seen in MTA. Conversely, MTA contains a higher proportion of silicon (Si) and carbon (C), reinforcing its identity as a calcium silicate-based cement. The lower phosphorus content (1.5%) in MTA suggests reduced phosphate-driven bioactivity, potentially leading to a slower setting reaction and limited biointegration compared to Ceramagnum Plus.

The setting reaction and working time of these materials are also influenced by their elemental composition. The increased potassium and magnesium levels in Ceramagnum Plus contribute to a faster setting reaction, as $KMgPO_4$ acts as a setting accelerator, expediting chemical bonding and material hardening. [20]In contrast, MTA undergoes a slower hydration reaction due to its calcium silicate nature, leading to longer setting times, which could impact its handling during clinical applications.

One of the key advantages of Ceramagnum Plus over MTA is its superior radiopacity, achieved through the addition of zirconium (Zr) and cerium (Ce). These elements enhance visibility under radiographic imaging, a crucial feature in endodontic and orthopedic procedures. MTA, lacking zirconium and cerium, relies on bismuth oxide for radiopacity, a compound associated with potential biocompatibility concerns. Additionally, the balanced Ca-Si-P-Mg-Zr phase structure of Ceramagnum Plus is expected to enhance its mechanical strength and durability.[21] The increased calcium and phosphate content improve bonding with surrounding tissues, while zirconium contributes to enhanced fracture resistance.[22]

Purity and contamination control are also notable differentiating factors between these materials. The lower carbon (C) content in Ceramagnum Plus (15.3%) compared to MTA (44.3%) suggests a more refined and pure composition. The high carbon content in MTA may indicate the presence of carbonates, hydration by-products, or contaminants that could impact its long-term stability.From a clinical perspective, Ceramagnum Plus demonstrates several advantages over MTA, including improved setting time, bioactivity, radiopacity, and mechanical strength. These properties make it a promising alternative for various biomedical applications, such as root repair in endodontics, bioactive bone cements in orthopedic procedures, and dental restorations requiring superior biocompatibility and radiopacity. [23]However, MTA remains a widely used and clinically validated material, despite its longer setting time, lower phosphate content, and lack of zirconium and cerium, which may limit its handling efficiency and performance in certain applications.[24] The comparative analysis underscores the potential of Ceramagnum Plus as a next-generation biomaterial, offering enhanced functional properties that could improve clinical outcomes across multiple fields of medicine and dentistry.

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