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Comparison of Solubility, Ph Change and Calcium Ion Release from Newly Developed Bioceramic Ceremagnum Plus with Commercially Available MTA Angelus-An Invitro Study

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KEYWORDS

Ceremagnum Plus, MTA Angelus, Calcium Ion Release, pH, Solubility, Bioactivity, Regenerative Endodontics

ABSTRACT:

Background: Bioinductive materials such as Mineral Trioxide Aggregate (MTA) and newly formulated alternatives like Ceremagnum Plus play a pivotal role in regenerative endodontics. This study aimed to compare the calcium ion release, pH, and solubility profiles of Ceremagnum Plus and MTA Angelus to evaluate their potential in clinical applications.

Materials and Methods: Calcium ion release was measured using a calcium ion-selective electrode (ISE) connected to an Orion Star A210 meter, calibrated with 0.01 M and 0.1 M standards. The test solutions were incubated at 37°C, and readings were recorded at 0 hr, 30 min, and 1 hr, with results expressed in ppm. pH was evaluated after immersing set samples in Hank's Balanced Salt Solution (HBSS) for 3 hours using a calibrated pH meter. Solubility was assessed using stainless steel ring molds containing the test materials, and weight loss was calculated at 30 minutes and 1 hour using the standard formula:

Results: Ceremagnum Plus exhibited a higher mean pH (13.83 ± 0.29) compared to MTA Angelus (12.00 ± 0.50), suggesting enhanced alkalinity and potential for stimulating hard tissue formation. On Day 1, MTA Angelus showed greater calcium ion release (0.25 ± 0.0707 ppm), whereas Ceremagnum Plus demonstrated a significantly higher sustained release by Day 7 (5.5 ± 0.71 ppm vs. 2.5 ± 0.71 ppm), indicating prolonged bioactivity. Regarding solubility, Ceremagnum Plus showed lower mean solubility at both 30 minutes (2.10 ± 1.54%) and 1 hour (2.72 ± 0.96%) compared to MTA Angelus (1.06 ± 0.27% at 30 minutes, 2.13 ± 0.53% at 1 hour), reflecting greater dimensional stability and water resistance.

Conclusion: Ceremagnum Plus demonstrated superior long-term calcium ion release, higher alkalinity, and lower solubility compared to MTA Angelus. These characteristics suggest enhanced bioactivity, antibacterial potential, and long-term stability, making Ceremagnum Plus a promising material for endodontic applications requiring sustained healing and sealing capabilities.

1. Introduction

In an effort to function as a barrier, safeguard the dental pulp complex, and maintain its vitality, direct pulp capping entails applying a dental substance to the exposed pulp.Because calcium hydroxide-based materials may release calcium (Ca) and hydroxyl (OH) ions, they are the most often used agents for both direct and indirect pulp capping.[1]

On the contrary, these substances are soluble and cause a necrotic layer to grow at the material–pulp interface, raising the local pH.[2]

Dycal is a radiopaque calcium hydroxide-based substance that self-sets (2.5–3.5 min) and is used as a liner.Its alkaline pH (pH 9–11) stimulates the formation of secondary dentine when the material is in direct contact with the pulp. Its toxicity to pulp cells is well documented. Radiopaque Portland cements, also known as mineral trioxide aggregate (MTA) cements (ProRoot

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MTA, MTA-Angelus, and others), are calcium silicate materials used for endodontic repair that were initially introduced as gray cements.[3]

MTA cements have the potential to be employed as pulp capping materials since they have calcified tissue-conductive activity, aid in the mineralization process in human dental pulp cells, and promote the development of human orofacial mesenchymal stem cells. MTA interacts with tooth pulp tissue more effectively and better than calcium hydroxide materials because it causes less pulp inflammation and has a limited caustic effect on pulp tissue necrosis soon after application.[4]

The high pH makes it difficult for any bacteria that could still be present after root canal therapy or cavity preparation to live and induce or sustain periapical inflammation.[5] Additionally, alkaline pH promotes the release of alkaline phosphatase and bone morphogenetic protein-2, which are involved in the mineralization process, and speeds up the nucleation of apatite. [6]As a result, new cements that resemble calcium silicate MTA have just been released.In addition to having an antibacterial impact, hydroxyl ions induce pulpal necrosis, which starts the healing process. Additionally, they cause the mineralized dentine matrix to release growth factors, metalloproteinases, and proteoglycans.[7]

These chemicals have the ability to tell pulpal undifferentiated cells to move to the site of damage, multiply, differentiate into cells that resemble odontoblasts, release organic extracellular matrix, and start mineralization. [8] It has been demonstrated that calcium ions promote the growth of cultured dental pulp cells and can instruct them to express osteopontin and bone morphogenetic protein mRNA.[9] Compared to calcium hydroxide, reports show that calcium silicate cements accelerate tertiary dentinogenesis and result in higher-quality dentine bridge development.[10]

One theory is that variations in ion release are the cause of the various behaviors seen in vivo.

Due to their poor mechanical qualities, calcium hydroxide cements should only be applied thinly to the cavity's deepest regions. It appears that MTA is no different.[10]

2. Materials and methods

Ceremagnum plus and white MTA-A (Angelus, Londrina, PR, Brazil) were among the materials used in this study. Water was used for mixing. To create the novel bioceramic Ceremagnum plus: KH2PO4 and MgOwere taken in a molar ratio of 1:1 and sintered at 900 degrees for three hours. As radio opacifiers, 1000 mg of KMgPO4, 1000 mg of CaSiO3, 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 CaCl2 liquid solution that has been triturated using a micropipette.

Fig. 1. Novel bioceramic powder

Calcium Ion Release Test:

Sample preparation

Turn on the Orion Star A210 meter.

Connect the calcium ISE and reference electrode (if separate) to the meter.

Calibrate the electrode using at least two standard calcium solutions (e.g., 0.01 M and 0.1 M). Add ISA to all standards(Calcium Ionic Strength Adjuster (ISA), e.g., Orion 910011) and samples in a 1:50 ratio (e.g., 2 mL ISA in 100 mL solution) to maintain constant ionic strength and adjust pH.

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Calibration

Rinse the electrode with deionized water and blot dry between measurements.Place the electrode in the lowest concentration standard with ISA.

Stir gently and wait for a stable reading (check meter's stability indicator).

Record or accept the value when stable. Repeat with higher concentration standard (s). The meter will generate a calibration curve (mV vs. log[Ca^{2+}]).

To evaluate calcium ion release, a standardized solution preparation protocol was followed. One milliliter (1 mL) of the test solution—either Ceremagnum Plus or MTA extract—was mixed with 9 mL of distilled water in a clean beaker. To ensure accurate ion measurement, 0.2 mL of Calcium Ionic Strength Adjuster (ISA) was added to the mixture. This prepared solution was then incubated at 37°C to replicate physiological oral conditions. Measurements were subsequently taken at various time intervals, specifically at 0 hours, 30 minutes, and 1 hour, to observe ion release kinetics over time.

For the measurement of calcium ion concentration, a calcium ion-selective electrode was used. The electrode was inserted into the incubated test solution, which was continuously stirred using a magnetic stirrer to maintain uniform distribution and prevent settling of any particulates. The electrode provided millivolt (mV) readings,

which corresponded to calcium ion concentration in the solution. These mV values were then converted into actual concentrations in parts per million (ppm or mg/L) using a pre-established calibration curve derived from standard calcium solutions.

Finally, the results obtained at different time points were compared to assess the rate and extent of calcium ion release from each material. A higher concentration of released calcium ions was interpreted as indicative of greater bioactivity and mineralization potential, which are critical attributes for materials used in regenerative endodontic procedures.

pH analysis

To analyze the pH variation of the soaking solution and calcium ion release, each MTA material was prepared and filled into a mold with an internal diameter of 10 mm and a height of 1 mm. After filling, the specimen was stored at 37 ± 1 ◦C for 24 h. The specimen was separated from the mold and was then immersed in 10 mL of Hank's balanced salt solution (HBSS; H6648, Sigma Aldrich, St. Louis, MO, USA) . A pH meter (Orion 4 Star,Thermo Fisher Scientific Inc., Singapore) calibrated using buffer solutions of pH 4.01, 7.00 and 10.01 was used. The pH variation of the HBSS-immersed specimen was measured at 3hr.

Solubility test

Six ring molds of stainless steel having inner diameter of 20 mm ± 0.1 mm and a height of 1.5 mm ± 0.1 mm were manufactured. With acetone, the molds were washed for 15 min in an ultrasound bath and then were dehydrated in air for 30 min. Mixing of MTA and Ceremagum plus was done accordingly. The molds were positioned on a glass slab and mixing of materials was done, ultimately molds were filled to excess with the mixed test materials corresponding to two groups. Then, with a Mylar strip, the molds were roofed, and thereafter, the glass plate was sited on top of the molds to eliminate any extra material and then the molds were placed inside an incubator cabinet at 37°C.

After 24 h, molds were removed from the incubator, and the exposure of molds to air for 15 min was done. Each mold was assessed with balance, three times to record the average reading and this weight was noted as initial dry weight (IDW) of the samples. Six amber-colored glass bottles were labeled and dried. The mass of these labeled and dried bottles was noted as dry bottle weight. Five milliliters of deionized water was poured into these bottles and the molds were shifted into them. The bottles were kept in an incubator at 37°C for 24 h. After 24 h, bottles were eliminated from the incubator, and with the help of a syringe, washing of each mold in bottles with 15 mL of distilled water was done. Extra residual water in the bottles was vaporized in an oven by keeping a temperature slightly below boiling point and then the bottles were dried in the oven at 105°C and cooled down in the same oven.

The mass of separate bottle was noted individually as final dry weight (FDW). Solubility of each tester was determined by the following formula and was expressed as a percentage value of IDW.

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$$\text{Solubility} = \frac{\text{FDW} - \text{DBW}}{\text{IDW}} \times 100$$

After recording the FDW at 24 h, deionized water was to the bottles. Then, these bottles were transferred to the incubator for further evaluation at intervals of 30 mins and 1hr using the same method.

3. Results

PH test

Both MTA Angelus and Ceremagnum Plus demonstrated alkaline pH values, consistent with their expected bioactive behavior. However, Ceremagnum Plus exhibited slightly higher mean pH levels (mean = 13.83 ± 0.29) compared to MTA Angelus (mean = 12 ± 0.5).

This higher alkalinity may enhance the antibacterial properties and bioactivity of Ceremagnum Plus.The elevated pH supports hard tissue formation by stimulating alkaline phosphatase activity and encourages a favorable healing response. [11]

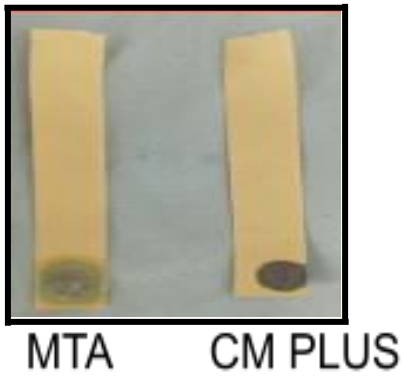

Fig. 2. PH analysis

Table 1: Representing mean and standard deviation

Group	Mean ± SD
MTA Angelus	13.83 ± 0.29
Ceremagnum plus	12.00 ± 0.50

Calcium Ion Release Test

Table 2: Representing mean and standard deviation

Group	Day 1 (Mean ± SD)	Day 7 (Mean ± SD)
Ceremagnum Plus	0.045 ± 0.0071	5.5 ± 0.71
MTA Angelus	0.25 ± 0.0707	2.5 ± 0.71

MTA Angelus showed a higher calcium ion release on Day 1, while Ceremagnum Plus demonstrated a more sustained and significantly higher release by Day 7.

Fig. 3. Calcium ion release calibration

This suggests that although MTA initiates a quicker calcium release, Ceremagnum Plus may offer prolonged bioactivity, supporting continued mineralization and healing over time. The extended ion release of Ceremagnum Plus could contribute to better stimulation of periapical tissue regeneration,

enhancing its biological performance in clinical settings.[12]

Solubility

Ceremagnum plus

Table 3: Representing weight at 0mins, 30mins and 1hr

Group	Initial Weight (g)	Weight at 30 mins (g)	Weight at 1 hr (g)
T1	0.054	0.054	0.053
T2	0.073	0.071	0.071
T3	0.056	0.054	0.054

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Fig. 4. Solubility determination

Table 4: Representing solubility at 30 mins and 1hr(%)

Group	30 mins Solubility (%)	1 hr Solubility (%)
T1	0%	1.85%
T2	2.74%	2.74%
T3	3.57%	3.57%

Table 5: Representing mean solubility and standard deviation

Time Point	Mean Solubility(%)	Standard Deviation
30mins	2.10	1.54
1hour	2.72	0.96

MTA Angelus

Table 6: Representing weight at 0mins, 30mins and 1hr

Group	Initial Weight (g)	Weight at 30 mins (g)	Weight at 1 hr (g)
T1	0.054	0.0535	0.053
T2	0.073	0.072	0.071
T3	0.056	0.055	0.055

Table 7: Representing solubility at 30 mins and 1hr(%)

Group	30 mins Solubility (%)	1 hr Solubility (%)
T1	0.93%	1.85%
T2	1.37%	2.74%
T3	0.89%	1.79%

Table 8: Representing mean solubility and standard deviation

Time Point	Mean solubility(%)	Standard Deviation
30mins	1.06	0.27
1hour	2.13	0.53

Ceremagnum Plus exhibited lower solubility values at both 30 minutes and 1 hour compared to MTA Angelus. This indicates better dimensional stability and resistance to dissolution in aqueous environments. Such behavior is critical in maintaining the integrity of the material over time, especially in moist clinical conditions.[13] The lower solubility of Ceremagnum Plus enhances its long-term sealing ability, making it favorable for applications like root-end fillings or perforation repairs.

4. Discussion

The present study aimed to evaluate and compare the physicochemical behavior of Ceremagnum Plus and MTA Angelus with respect to calcium ion release, pH values, and solubility under simulated physiological conditions. These parameters are critical indicators of a material's bioactivity, sealing ability, and clinical performance in regenerative and reparative endodontic procedures.[14]

Calcium ion release is a direct indicator of a material's ability to induce hard tissue formation and stimulate cellular differentiation, particularly of odontoblast-like and cementoblast-like cells.[15] In our study, MTA Angelus exhibited a higher calcium ion release on Day 1 (0.25 ± 0.0707 ppm) compared to Ceremagnum Plus (0.045 ± 0.0071 ppm), indicating a more immediate ion-leaching effect. This aligns with previous findings that MTA rapidly initiates calcium release, contributing to early bioactivity and inflammatory modulation in periapical tissues.[16]

However, by Day 7, Ceremagnum Plus showed a significantly higher calcium ion release (5.5 ± 0.71 ppm) than MTA Angelus (2.5 ± 0.71 ppm). This suggests that Ceremagnum Plus offers a more sustained release profile, which may provide prolonged stimulation of mineralization and hard tissue formation. Sustained calcium ion availability is crucial for long-term signaling in regenerative processes, including osteoinduction and cementogenesis.[17] The extended release profile of Ceremagnum Plus could be attributed to its unique

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formulation, possibly involving modified calcium silicate phases or bioactive additives that prolong ion liberation.

An alkaline pH environment is essential for the antibacterial effect of endodontic biomaterials and for promoting healing responses.[18] The results demonstrated that both materials exhibited alkaline pH levels, consistent with their calcium silicate-based chemistry. However, Ceremagnum Plus displayed a significantly higher mean pH (13.83

± 0.29) compared to MTA Angelus (12.00 ± 0.50). This elevated pH is beneficial not only for inhibiting microbial colonization but also for enhancing alkaline phosphatase activity, which is a key enzyme involved in hard tissue mineralization.[19]

The higher alkalinity of Ceremagnum Plus may be indicative of a higher release of hydroxyl ions, a characteristic that enhances its biocompatibility and regenerative

potential.[20] Furthermore, sustained alkalinity can modulate local immune responses, promoting a more favorable healing environment and reducing postoperative inflammation.[21] These findings suggest that Ceremagnum Plus may offer an enhanced biological response in clinical situations that demand strong antimicrobial and regenerative action.

Dimensional stability and low solubility are essential for maintaining the integrity of an endodontic material, especially in moist clinical conditions.[22] Excessive solubility can lead to microleakage, marginal breakdown, and failure of the restoration.[23] In our study, Ceremagnum Plus demonstrated lower solubility than MTA Angelus at both 30 minutes (2.10 ± 1.54%) and 1 hour (2.72 ± 0.96%), whereas MTA Angelus showed slightly

higher values (1.06 ± 0.27% at 30 mins, 2.13 ± 0.53% at 1 hr).

Although both materials showed acceptable solubility within ISO limits, the lower solubility of Ceremagnum Plus may confer better resistance to dissolution in oral fluids and longer-lasting sealing ability.[24] This can be particularly advantageous in procedures such as root-end fillings, furcal perforation repairs, and pulp capping, where maintaining an intact barrier in a moist environment is critical. The slight increase in solubility at the 1-hour interval in both groups is consistent with the initial hydration and setting reaction of calcium silicate-based materials, which can release soluble salts in early stages.[25]

Clinical Implications

Taken together, the findings of this study indicate that Ceremagnum Plus possesses favorable physicochemical properties that support its application in clinical endodontics. The sustained calcium ion release profile suggests enhanced long-term bioactivity, while its higher pH supports antimicrobial efficacy and potential for promoting hard tissue formation.[26] Additionally, its lower solubility ensures structural stability in wet environments, which is crucial for maintaining the longevity of the seal.

Compared to the well-established MTA Angelus, Ceremagnum Plus demonstrates comparable, if not superior, characteristics in key parameters relevant to regenerative therapy. These observations support its potential as a reliable alternative to traditional materials in procedures requiring pulp preservation, apical sealing, and periapical healing.[27]

5. Limitations and Future Directions

While this study provides valuable insights, it is limited to in vitro conditions. The complexity of the oral environment, including the presence of saliva, proteins, enzymes, and dynamic fluid flow, may influence the in vivo behavior of these materials.

[28]Additionally, long-term studies assessing their mechanical properties, radiopacity, setting time, and biocompatibility using in vivo models and histological evaluation would further validate the clinical efficacy of Ceremagnum Plus.

Future research should also explore the interaction of these materials with stem cells and their influence on gene expression related to mineralization, which will help clarify the underlying mechanisms of their bioinductive behavior.[29]

6. Conclusion

Ceremagnum Plus demonstrated superior performance in terms of sustained calcium ion release, higher alkalinity, and lower solubility compared to MTA Angelus. These

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attributes support its use as a bioactive material in endodontic applications requiring regenerative healing and durable sealing.[30] The material shows promising potential as an advanced substitute for MTA in clinical practice.

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