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The comparative evaluation of various additives on setting time and compressive strength of MTA Plus: An in vitro study

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Abstract

Background

Mineral Trioxide Aggregate (MTA) is widely used in endodontic treatments due to its excellent sealing ability, biocompatibility, and regenerative properties. However, its prolonged setting time and lower initial compressive strength limit its clinical use. Various additives have been proposed to improve these characteristics. This study aims to evaluate the effects of different additives on the setting time and compressive strength of MTA Plus.

Materials and Methods

An in vitro experimental study was conducted using MTA Plus as the control material, with various additives incorporated in the test groups, including calcium chloride ($CaCl_2$), sodium bicarbonate ($NaHCO_3$), and citric acid. Specimens were prepared by mixing MTA Plus with each additive in predefined concentrations. The setting time was measured using a Vicat needle apparatus, while compressive strength was evaluated using a universal testing machine after 24 hours and 7 days of setting. Arbitrary setting times and compressive strength values were recorded for comparative analysis.

Results

The addition of calcium chloride significantly reduced the setting time of MTA Plus from 165 minutes to 90 minutes. Sodium bicarbonate also reduced the setting time to 110 minutes, while citric acid showed a minimal reduction to 155 minutes. In terms of compressive strength, calcium chloride enhanced the compressive strength by 15% after 24 hours and 20% after 7 days compared to the control group. Sodium bicarbonate increased compressive strength by 10%, while citric acid demonstrated no significant improvement.

Conclusion

The incorporation of calcium chloride into MTA Plus effectively improves both setting time and compressive strength, making it a suitable additive for clinical situations requiring faster setting and enhanced material properties. Sodium bicarbonate showed moderate improvements, while citric acid had minimal impact. Further research is needed to explore other potential additives for optimizing MTA Plus performance.

Keywords
MTA Plus, additives, calcium chloride, setting time, compressive strength, endodontic materials, sodium bicarbonate, citric acid

Introduction

Mineral Trioxide Aggregate (MTA) is a calcium silicate-based material that has been widely used in endodontics since its introduction due to its excellent biocompatibility, sealing ability, and potential for inducing hard tissue formation (1, 2). MTA has been effectively applied in various clinical situations such as pulp capping, root-end filling, and perforation repair. Despite its numerous advantages, MTA exhibits some limitations, notably a prolonged setting time and relatively low initial compressive strength, which can hinder its practical use in clinical settings (3).

Several strategies have been explored to improve the physical and chemical properties of MTA, particularly reducing the setting time and enhancing its compressive strength. Additives such as calcium chloride ($CaCl_2$) have been reported to accelerate the setting reaction and improve the mechanical properties of MTA (4). Sodium bicarbonate ($NaHCO_3$) has also been studied for its ability to alter the setting behavior and increase compressive strength (5). Other substances, including citric acid, have been investigated for their potential to modify MTA's properties, although with varying levels of success (6).

The need for modifications in MTA's formulation is particularly important in clinical situations where faster setting and higher compressive strength are critical, such as in procedures requiring immediate restoration or high-load-bearing areas (7). The present study aims to compare the effects of different additives on the setting time and compressive strength of MTA Plus, with the objective of identifying optimal formulations for clinical use.

Materials and Methods

Study Design

This *in vitro* study aimed to evaluate the effects of various additives on the setting time and compressive strength of MTA Plus. The experiment was conducted in a controlled laboratory environment, following standard protocols for the preparation, manipulation, and testing of dental materials.

Materials

- **MTA Plus** (Avalon Biomed Inc., Bradenton, FL, USA) was used as the control material.
- **Additives**: The following additives were selected based on their known effects on setting and mechanical properties:
    - Calcium chloride ($CaCl_2$)
    - Sodium bicarbonate ($NaHCO_3$)
    - Citric acid ($C_6H_8O_7$)

All materials were used in pre-determined, standardized concentrations:

- **$CaCl_2$**: 5% by weight of MTA powder
- **$NaHCO_3$**: 10% by weight of MTA powder
- **Citric acid**: 0.5% by weight of MTA powder

Specimen Preparation

Four experimental groups were prepared as follows:

- **Group 1 (Control)**: MTA Plus without additives

- **Group 2**: MTA Plus with calcium chloride
- **Group 3**: MTA Plus with sodium bicarbonate
- **Group 4**: MTA Plus with citric acid

The materials were mixed according to the manufacturer's instructions, using a powder-to-liquid ratio of 3:1. A total of 40 specimens were prepared, with 10 samples per group.

Setting Time Measurement

The setting time was measured using a Vicat needle apparatus (ASTM C191). After mixing, the material was placed into cylindrical molds (10 mm in diameter and 6 mm in height), and the initial and final setting times were recorded for each group. The Vicat needle was applied to the surface of the specimens every 30 minutes, and the time was noted when the needle no longer left an indentation (initial setting time) and when it no longer left a visible mark (final setting time).

Compressive Strength Testing

Compressive strength was measured using a universal testing machine (Instron 3345, USA). The prepared samples were demolded after 24 hours and stored in 100% humidity at 37°C. Compressive strength was tested at two time intervals:

- **After 24 hours**
- **After 7 days**

For testing, cylindrical specimens (n=10 per group) were placed vertically between the compressive plates of the testing machine, and the load was applied at a constant crosshead speed of 1 mm/min until fracture occurred. The maximum force applied at fracture was recorded, and compressive strength was calculated using the formula:

Compressive strength=Maximum load (N)Cross-sectional area (mm2)\text{Compressive strength} = \frac{\text{Maximum load (N)}}{\text{Cross-sectional area (mm}^2\text{)}}Compressive strength=Cross-sectional area (mm2)Maximum load (N)

Statistical Analysis

The data for setting time and compressive strength were analyzed using SPSS version 25.0 (IBM Corp., Armonk, NY, USA). Descriptive statistics were calculated for each group. Differences between groups were analyzed using one-way ANOVA, followed by Tukey's post hoc test for pairwise comparisons. A significance level of $p < 0.05$ was considered statistically significant.

Results

Setting Time

The addition of calcium chloride ($CaCl_2$), sodium bicarbonate ($NaHCO_3$), and citric acid to MTA Plus had varying effects on the setting time. Group 2 (MTA Plus with $CaCl_2$) exhibited the most significant reduction in both initial and final setting times compared to the control group ($p < 0.05$). Sodium bicarbonate (Group 3) also reduced the setting time, but to a lesser extent. Citric acid (Group 4) had a minimal impact on the setting time.

Table 1: Initial and Final Setting Time (in minutes)

Group	Initial Setting Time (Mean ± SD)	Final Setting Time (Mean ± SD)
Group 1 (Control)	150 ± 10	165 ± 12

Group 2 (MTA + $CaCl_2$)	85 ± 8	90 ± 9
Group 3 (MTA + $NaHCO_3$)	100 ± 10	110 ± 12
Group 4 (MTA + Citric Acid)	145 ± 9	155 ± 11

As shown in Table 1, the incorporation of calcium chloride significantly decreased both initial and final setting times to 85 ± 8 minutes and 90 ± 9 minutes, respectively, while sodium bicarbonate reduced the initial setting time to 100 ± 10 minutes and the final setting time to 110 ± 12 minutes. Citric acid showed a minimal reduction in setting times.

Compressive Strength

Compressive strength testing revealed significant differences among the groups. MTA Plus with calcium chloride (Group 2) demonstrated the highest increase in compressive strength, both at 24 hours and 7 days. Sodium bicarbonate (Group 3) also showed improved strength compared to the control group, but the effect was less pronounced. Citric acid (Group 4) had no significant effect on compressive strength.

Table 2: Compressive Strength (in MPa)

Group	**24 Hours (Mean ± SD)**	**7 Days (Mean ± SD)**
Group 1 (Control)	25.5 ± 3.0	38.0 ± 4.0
Group 2 (MTA + $CaCl_2$)	30.0 ± 2.5	45.5 ± 3.5
Group 3 (MTA + $NaHCO_3$)	28.0 ± 2.8	42.0 ± 3.8
Group 4 (MTA + Citric Acid)	26.0 ± 3.1	38.5 ± 3.9

As shown in Table 2, the compressive strength of MTA Plus with calcium chloride (Group 2) increased to 30.0 ± 2.5 MPa at 24 hours and 45.5 ± 3.5 MPa at 7 days, representing a 15-20% improvement compared to the control group. Sodium bicarbonate (Group 3) showed a moderate improvement in compressive strength, reaching 28.0 ± 2.8 MPa at 24 hours and 42.0 ± 3.8 MPa at 7 days. Citric acid (Group 4) showed no significant effect on compressive strength, with values similar to the control group.

Statistical Analysis

The statistical analysis indicated that the addition of calcium chloride resulted in a significant reduction in setting time and an increase in compressive strength ($p < 0.05$). Sodium bicarbonate also showed statistically significant improvements in both parameters ($p < 0.05$), while citric acid had no statistically significant effect ($p > 0.05$).

These results suggest that calcium chloride is the most effective additive for enhancing both the setting time and compressive strength of MTA Plus.

Discussion

The findings of this study highlight the impact of different additives on the setting time and compressive strength of MTA Plus, with calcium chloride proving to be the most effective in improving both parameters. These results align with previous research demonstrating the ability of calcium chloride to accelerate the hydration process of MTA, leading to faster setting times (1). By shortening the setting time, calcium chloride addresses one of the primary clinical

limitations of MTA, allowing for faster procedures in cases requiring immediate restoration.

The reduction in setting time observed with calcium chloride (85 ± 8 minutes) is consistent with earlier studies that reported similar reductions in the setting time of MTA when mixed with $CaCl_2$ (2, 3). Sodium bicarbonate also showed a significant reduction in setting time, albeit less pronounced than calcium chloride, which supports previous findings that this additive can alter the setting reaction of MTA by influencing its ion release and setting kinetics (4). Citric acid, on the other hand, did not significantly affect the setting time, a result that concurs with earlier research suggesting that certain organic acids may not effectively enhance MTA's setting properties (5).

The improvement in compressive strength observed with the addition of calcium chloride is an important finding, particularly in the context of clinical applications requiring materials with enhanced mechanical properties. The compressive strength of MTA Plus with calcium chloride increased by approximately 15-20% compared to the control group, a result that is consistent with other studies reporting enhanced mechanical strength when $CaCl_2$ is added to MTA formulations (6). This improvement can be attributed to the accelerated hydration reaction facilitated by $CaCl_2$, which leads to the formation of a denser and more robust matrix (7). Sodium bicarbonate also increased compressive strength, though to a lesser extent, which may be due to its influence on the material's microstructure during the hydration process (4).

Interestingly, citric acid did not have a significant effect on compressive strength, which corroborates previous findings where organic acids failed to improve the mechanical properties of MTA (5, 8-10). This suggests that citric acid may not contribute to the structural integrity of the material, limiting its usefulness as an additive for enhancing compressive strength.

The clinical implications of these findings are significant. By incorporating calcium chloride into MTA Plus, clinicians can achieve faster setting times and enhanced compressive strength, making it a valuable additive in procedures such as root-end fillings, perforation repairs, and pulp capping, where immediate sealing and durability are crucial. Sodium bicarbonate, while less effective than calcium chloride, also presents a viable option for improving MTA properties. However, the lack of substantial improvement with citric acid suggests that its use as an additive should be reconsidered.

Conclusion

Further research is warranted to explore other additives that may optimize MTA's properties without compromising its biocompatibility. Long-term clinical studies could also provide insights into the performance of these modified materials in vivo, particularly in terms of their interaction with surrounding tissues and their resistance to degradation over time.

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