



Power Hydrogen Evaluation of Apexification Materials: EndoCal 10, Mineral Trioxide Aggregate and Calasept Plus

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ABSTRACT

Aim: The present study was to evaluate pH values of apexification materials.

Materials and methods: The materials were placed in 1 cm long and 4 mm diameter tubes. After sample immersion, glass flasks were hermetically sealed with rubber caps to attenuate any effects of external environmental factors and maintained at 37°C. The power hydrogen of the pastes was measured 1 hour, 3 hours, 8 hours, 24 hours, 72 hours and 7 days after preparation. pH was calibrated with solutions of known pH (7.0). Mann-Whitney test were used to determine significant differences.

Results: The mean pH of all medications was < 12.0 throughout the experiment. At 24 hours, EndoCal 10 had the highest pH of all the materials ($p < 0.05$).

Conclusion: According to the results obtained, it may be concluded that calcium oxide (EndoCal 10) presented the highest pH compared with mineral trioxide aggregate (MTA) and Calasept Plus [Ca(OH)₂].

Clinical significance: Apexification is an important treatment in immature teeth. For this reason, used materials in apexification should have some properties, such as high pH and stimulating to be hard tissue. Therefore, the material should be chosen carefully in apexification of immature teeth.

Keywords: Apexification, Immature teeth, pH.

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INTRODUCTION

Apexification is a procedure progressing a teeth with immature apex through the formation of osteocementum or other bone like tissue.¹⁻³ Many materials, such as calcium hydroxide [Ca(OH)₂], zinc oxide, calcium phosphate and mineral trioxide aggregate (MTA), have been used to stimulate hard tissue formations during apexification procedure.

Calcium hydroxide has been commonly used for apexification procedures based on its antibacterial, antiresorptive, and tissue-dissolving properties.⁴⁻⁶ The high pH of Ca(OH)₂ destructively affects on cell membranes and protein structure of bacteria.⁷ As is well-known, periapical tissues tolerates and subsequently resorbes Ca(OH)₂.⁸ However, Ca(OH)₂ chemically alters lipopolysaccharide which affects its various biological properties.⁹⁻¹³ High pH, which is bactericidal in Ca(OH)₂ is determined by the liberation of hydroxyl ions, which requires an ideal time for effective destruction of microorganisms, and also inhibits osteoclastic activity.¹⁴ The time required for apical barrier formation in apexification using Ca(OH)₂ may be considerable, often as long as 20 months; and other conditions, such as age and presence of symptoms or periradicular radiolucencies may affect the time needed to form an apical barrier.

Mineral trioxide aggregate (MTA) is also used in the apexification treatment because of its favorable sealing ability and biocompatibility.¹⁵ Mineral trioxide aggregate allows for an immediate apical barrier to be formed and, thus, immediate obturation of the root canal system. Mineral trioxide aggregate may, therefore, solve some of the problems currently experienced in apexification using

Ca(OH)₂. It produces the equivalent amount of apical hard tissue with no more inflammation than Ca(OH)₂. Mineral trioxide aggregate has shown potential outcome in carrying out apexification of immature permanent teeth.¹⁵

A calcium oxide (CaO) product, with the trade name EndoCal 10, a new material used for apexification, is composed of calcium oxide, zinc oxide, ethylene glycol, and purified water. Calcium oxide formerly known as Biocallex 6.9 in the past was redeveloped by manufacturers and renamed EndoCal 10. EndoCal 10 has good physicochemical properties, presents biocompatibility, prolonged release of calcium hydroxide, and stimulates and isolates vital cells of the endodontium and chemical lysis of necrosed cells.^{16,17}

However, at present, limited data is available concerning its properties except the manufacturer introduction. Additionally, there is no information about the pH ability of EndoCal 10 in comparison to other apexification materials.

The purpose of the present study was to assess the pH values of the EndoCal 10 agent, and compare it with two apexification materials; Calasept Plus [Ca(OH)₂] and MTA commonly used in apexification.

MATERIALS AND METHODS

Sample Preparation

All the tested apexification materials [EndoCal 10 (Albuca Inc, Pointeclaire, Canada), MTA (Dentsply, Tulsa, OK), and Calasept Plus (Nordiska Dental, Sweden)] were mixed according to the manufacturers' instructions and then placed in 1 cm long glass tubes (Difco Lab, Detroit, Michigan, USA) with open ends that were 4 mm in diameter containing 10 ml deionized water. Empty PVC tubes (Deutsch and Neumann, Berlin, Germany) comprised the control group (n = 5) 10 samples were used for each experiment group. After sample immersion, glass flasks were hermetically sealed with rubber caps to attenuate any effects of external environmental factors and maintained at 37°C during all experimental periods; 1, 3, 8, 24, 72 hours, and 7 days.

pH Measurement

The pH of the pastes were measured at 1, 3, 8, 24, 72 hours and 7 days after preparation using a pH meter (Procyon, digital pH meter model AS 720, electrode A 11489, Procy Instrumental Científica, São Paulo, SP, Brazil). The pH was calibrated with solutions of known pH (7.0) before and after measurements at each period. Between each measurement, the pH meter electrode was washed with ultrapure water (Millipore, Milli-Q Plus) and blotdried. Each measurement was carried out triplicate, and the mean value was recorded.

STATISTICAL ANALYSIS

The data was analyzed statistically by the Kruskal-Wallis non-parametric test using SPSS. The Mann-Whitney test was used to determine significant differences in susceptibility to intracanal medication among experiment species. Significance level was set at $p < 0.05$.

RESULTS

The mean pH values measured for the apexification materials at different time periods are shown in Table 1. The controls showed no noticeable change over the experimental period.

The mean pH of all medications was < 12.0 throughout the experiment. At 24 hours, EndoCal 10 had the highest pH of all the materials ($p < 0.001$).

At the end of first hour, the mean pH value of MTA was observed as higher than the others and it was statistically different than the other materials ($p < 0.05$). There was no difference among EndoCal 10, Ca(OH)₂, and the Control groups ($p > 0.05$).

At the end of the third hour, EndoCal 10 showed a statistical difference ($p < 0.001$), but there was no difference between MTA and Ca(OH)₂ ($p > 0.05$). This difference was continued from this time until the 72nd hour. Finally, at the end of the 72nd hour, there was no difference between EndoCal 10 and Ca(OH)₂ ($p > 0.05$). However, these materials were different than MTA

Table 1: The mean pH values of the materials depending on the time

Time period	MTA	EndoCal 10	Ca(OH) ₂	Control
1 hour	7.503 ± 0.03 ^a	7.208 ± 0.05 ^b	7.27 ± 0.12 ^b	7.184 ± 0.04 ^b
3 hours	8.084 ± 0.13 ^A	8.406 ± 0.18 ^B	8.071 ± 0.09 ^A	7.226 ± 0.03 ^C
8 hours	8.39 ± 0.18 ^d	9.045 ± 0.16 ^f	8.766 ± 0.05 ^g	7.268 ± 0.03 ^h
24 hours	9.036 ± 0.19 ^D	11.782 ± 0.08 ^F	10.112 ± 0.09 ^G	7.306 ± 0.03 ^H
72 hours	8.925 ± 0.13 ^k	11.046 ± 0.08 ^m	10.619 ± 0.29 ^m	7.41 ± 0.02 ⁿ
7 days	8.287 ± 0.28 ^K	10.831 ± 0.24 ^L	9.742 ± 0.31 ^M	7.392 ± 0.01 ^N

There were significant differences among the different characters statistically



statistically ($p < 0.05$). At the end of the experiment period (7th day), there was a significant difference among the groups ($p < 0.001$).

DISCUSSION

Common clinical signs, such as exudation, hemorrhage, perforation, root resorption, trauma, or incomplete root formation, may indicate the need for intracanal dressing.¹⁸ In these situations, it is beneficial to clean the canals and to fill them with a medication.

There are several apexification materials available in the dental market. Increased pH has been reported to have a beneficial effect. The pH from root canal sealers are the most important chemical characteristics for the promotion of mineralization.^{19,20} Calcium hydroxide is widely used in apexification. However, apexification using MTA provides an alternative treatment modality in immature pulpless teeth. Apexification with MTA and also calcium oxide agents require significantly less time than $\text{Ca}(\text{OH})_2$. Calcium oxide is a self-regulating system, a material that is both an active disinfectant and an obturator, as required by the surrounding conditions. EndoCal 10 and MTA include CaO in its composition. During its setting, hydration reactions take place, resulting in production of $\text{Ca}(\text{OH})_2$. When the set material is placed in a solution, it dissociates into OH^- and Ca^{+2} ions, increasing the pH concentrations in the medium.²¹ The high pH of the medications may neutralize the acids secreted by osteoclasts and this may help preventing further destruction of mineralized tissue that plays an important role in hard tissue formation.^{22,23}

In this respect, few studies have been reported related to the pH of apexification materials, except for Endocal 10.^{14,23-25}

Duarte²⁴ reported that MTA immersed in deionized water produced a pH of 9.07, 24 hours after immersion. This result is very similar in the present study (pH mean for MTA, 9.06, 24 hours after immersion). Mineral trioxide aggregate contains calcium oxide, which is converted to a calcium hydroxide ion upon contact with tissue, fluid or water, thus, raising the pH.

Endocal 10 produced higher pH from the beginning and continued to do so throughout the experimental period. The CaO (67%) component of Endocal 10 might play an important role in this pH increasing. Regarding the pH of the cements evaluated, during the experiment periods, EndoCal 10 exhibited the highest values among all materials studied. This may be due to the presence of 37% $\text{Ca}(\text{OH})_2$ in the composition of EndoCal 10.

The higher pH may lead this sealer to exhibit more potent antibacterial effect after some time and may

support more rapid healing of apical periodontitis.²⁶ Probably by eliminating the bacteria with macrophage activation and cell differentiation and inducing biologic sealing of the root apex by the formation of mineralized tissue.²⁵

The two important reasons for using $\text{Ca}(\text{OH})_2$ as an apexification material are stimulation of the periapical tissues in order to maintain health or promote healing and its antimicrobial effects.²³

The high pH of the medications may also neutralize the acids secreted by osteoclasts and this may help in preventing further destruction of mineralized tissue. Concerning pH, if the material releases hydroxyl ion, thus favoring alkalinity, it will also favor repair and promote an antimicrobial action.²³

Considering all the reasoning on pH processes and isolated activities at essential enzymatic sites, it is enlightening to associate $\text{Ca}(\text{OH})_2$, a substance with a high pH, with harmful biological effects on bacterial cells in order to explain mechanism of action. Most bacteria present in the root canal system grow best at a pH around 6.5 to 7.5, and most microorganisms are destroyed at pH 9.5, though a few can survive at pH 11 or higher.²⁷ The effect of the high pH $\text{Ca}(\text{OH})_2$, influenced by the release of hydroxyl ions, is able to alter the integrity of the cytoplasmic membrane by means of chemical injuries to organic components and transport of nutrients; or by means of the destruction of phospholipids or unsaturated fatty acids of the cytoplasmic membrane, observed in the lipidic peroxidation process, which is a saponification reaction.²³

Despite the higher success rate of apical formation using $\text{Ca}(\text{OH})_2$, long-term follow is essential. However, apexification using MTA provides an alternative treatment modality in immature pulpless teeth. Apexification with MTA and also CaO agents require significantly less time than $\text{Ca}(\text{OH})_2$.²⁸ Mineral trioxide aggregate as an apexification material represents a contemporary version of the primary monoblock.²⁹ Apatite-like interfacial deposits form during maturation of MTA, which results in the filling up of gaps induced during the material shrinkage phase and improve the frictional resistance of MTA to the root canal walls. However, CaO stimulates and isolates vital cells of the endothelium.¹⁷ Further studies are necessary for a more detailed evaluation of the properties of this material, including its biological effects.

The methodology of this study consisted of filling standardized tubes with the materials to be tested and immersing in distilled water. The pH was then determined in the resulting solution.³⁰ Numerous authors have utilized similar methodology, immersing the plastic

tubes containing cements in distilled, deionized, or Milli-Q water.³⁰⁻³² Distilled water was used due to its purity and neutral pH.³⁰

The materials could be placed in the root canals and it may be evaluated as more clinically relevant substrate. However, there would be some challenges due to potential differences in size of the apical foramina and anatomic variations.³² Although the method of placing sealers in plastic tubes and immersing them in glass flasks does not imitate clinical conditions, it attempts to closely do so have resulted in complicated models that are difficult to reproduce and sometimes even to interpret. The tubes were open at both ends, which helped to condense the sealers without any voids and later helped in pH measurements. This method offers the advantages of simplicity, replication of the results, and time economy, so that *in vitro* comparisons between different materials can be easily achieved.

CONCLUSIONS AND CLINICAL SIGNIFICANCES

- According to the results obtained, it may be concluded that calcium oxide (EndoCal 10) presented the highest pH compared with MTA and Calasept Plus during all experiment periods.
- However, additional studies are necessary to evaluate further biological effects.
- Apexification is an important treatment in immature teeth. For this reason, used materials in apexification should have some properties, such as high pH and stimulating to be hard tissue.
- Therefore, the material should be chosen carefully in apexification of immature teeth.

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