The Properties of a New Endodontic Material

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Abstract
The purpose of this study was to analyze the physical properties and chemical compositions of a new experimental cement (NEC) and compare them with mineral trioxide aggregate (MTA); pH, working time, setting time, dimensional changes following setting, flow, film thickness, and chemical composition of NEC and MTA were assessed. For chemical compositions, all specimens were imaged and analyzed by scanning electron microscopy and electron probe microanalysis (EPMA). The physical properties were performed according to ISO 6876:2001. Working time, pH, and dimensional changes of NEC and MTA showed similar results. Shorter setting time was obtained with the NEC compared with MTA (p < 0.05). The NEC showed more flow than MTA. In addition, the film thickness of the NEC was considerably less than the MTA (p < 0.01 and p < 0.001, respectively). EPMA investigations indicated that lime (CaO) was the dominant compound in NEC and MTA; however, other compounds were significantly different. It was concluded that the chemical composition of NEC is different compare with MTA; it can be concluded that the NEC exhibits acceptable physical properties. (J Endod 2008;34:990–993)

Key Words
Composition, electron probe microanalysis, mineral trioxide aggregate, new material, physical properties, root-end fillings

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oot-end fillings are able to seal the content of a root canal system. This sealing prevents egress of microorganisms or byproducts into periradicular tissues (1). An ideal root-end filling material should be biocompatible, antibacterial, nontoxic, and radiopaque, and it should not be resorbable or soluble in an oral environment. Besides these characteristics, it is also expected to be cost-effective, easy to handle, and adaptable to the cavity walls as closely as possible (1, 2). Some examples of the existing root-end–filling materials are gutta-percha, zinc oxide eugenol-based cements (ie, Super-EBA and IRM), composite resin, glass ionomer cement, Gavi, gold foil, polycarboxylate cement, polyvinyl cement, amalgam, Vitremer, and mineral trioxide aggregate (MTA) (1–3). Unfortunately, most of them have shown different levels of weakness in biocompatibility, leakage, solubility, handling properties, moisture incompatibility, and price (3, 4). MTA was introduced to the market in 1998 in order to overcome the mentioned disadvantages. The patent of this material was applied in 1995 (5, 6). MTA is a powder consisting of fine hydrophilic particles set in the presence of moisture. This material has a setting time of about 4 hours, and 3 hours after mixing, its pH value is 12.5 (7). White MTA replaced the gray one in 2002. MTA is a mechanical mixture of three powder ingredients: Portland cement, bismuth oxide, and gypsum (8). Portland cement as the major component of MTA is composed of dicalcium silicate, tricalcium silicate, tricalcium aluminate, and tetracalcium aluminoferrite (5). The color changes between gray and white MTA are because of a lesser amount of iron in white MTA (9). ProRoot MTA is used for both surgical and nonsurgical purposes such as root-end filling, perforation repairs, direct pulp capping, and apexification (10, 11).

Recently, a new experimental cement (NEC) consisting of different calcium compounds (ie, calcium oxide, calcium phosphate, calcium carbonate, calcium silicate, calcium sulfate, calcium hydroxide, and calcium chloride) was developed. Clinical uses of NEC are similar to MTA. This material can be handled and set in an aqueous environment. It has good handling characteristics and forms an effective seal when used as root-end–filling material (12). NEC is also able to produce hydroxyapatite with its endogenous and exogenous ion sources (13). The results of an in vivo study on dogs showed that as pulp capping materials, MTA and NEC showed similar favorable results. These results were better than calcium hydroxide (14).

The aim of this in vitro study was to analyze the physical and chemical properties of NEC as a new root-end–filling material and compare it with white MTA as a gold standard.

Materials and Methods

In this experiment, MTA (tooth-colored formula, ProRoot MTA; Dentsply, Tulsa, OK) and NEC were used. For each material, working time, setting time, dimensional changes, film thickness, and flow were measured three times according to International Organization for Standardization (ISO) 6876:2001. The mean values and standard deviation were calculated and recorded. A Metrohm 744 pH meter (Metrohm Ltd, Herisa, Switzerland) was applied for pH measurement. This pH meter was previously calibrated with standard solutions at pH 4.0 and 7.0. The cements were inserted into plastic tubes (length = 10 mm, diameter = 1.5 mm), and the tubes were weighted before and after filling to define and standardize the mass of cement in each tube. Five samples were used for each material. Then, each tube was sealed in a flask containing 10 mL of deionized distilled water, sealed, and stored at 37°C. The tubes were removed after an hour, and the electrode was inserted into the flask and readings were taken at 24°C. The data were statistically analyzed by one-way analysis of variance with α = 0.05 as the level for statistical significance.
For electron probe microanalysis (EPMA), the samples were mounted in individual discs with three holes in each to ensure compatibility with the electron probe microanalysis. After insertion of fresh mixed cements in cavities, the samples were immersed in saline solution and allowed to set in an incubator at 37°C for 48 hours. All samples were polished and carbon coated by using a Dynavac coating unit (CS300; Dynavac, Sydney, Australia). A JEOL SEM (JSM6400; JEOL, Tokyo, Japan) equipped with an Oxford Instruments light element dispersive spectrometer detector (Oxford Instruments, Abingdon, England) was used for imaging and determining the composition. All analyses were carried out at 15 kV and 1 nA of probe current. Atomic number, absorption, and fluorescence corrections were applied throughout (15). Each analysis was performed by using an area scan taken at 200× magnification to provide an average value for the overall composition. The final result for each sample was based on the average of 12 analyses (four analyses for each of the three cavities in the disc) with the overall error provided by the standard deviation. Carbon concentrations were not measured, and the amounts of carbon and water were therefore assumed by difference.

Results

Scanning electron microscopy of white MTA showed two specific phases throughout the material. The samples contained a complex mixture of mineral phases with highly visible randomly distributed 5 to 40 μm particles of bismuth oxide, which were added to the composition as the opacifier. The mineral phases of white MTA and NEC specimens revealed the presence of coarse crystalline particles in a general groundmass of finer amorphous material (Fig. 1).

The results of EPMA are presented in Table 1. This analysis revealed that lime (CaO), silica (SiO₂), and bismuth oxide (Bi₂O₃) formed 81.57% of the total weight of the white MTA. Oxides of iron, sulfur, phosphorus, titanium, and sodium as well as elemental chlorine were found at trace (<0.99 wt %) levels in white MTA. According to Table 1, the results of the EPMA of NEC cements revealed that lime is the predominant component of this material, and the concentration of other constituents, except some trace elements, is considerably different from the white MTA.

The mean and standard deviation of working time, setting time, dimensional changes, film thickness, and flow measured for the test materials are presented in Table 2. A considerable significant difference was found between the results of setting times, film thickness, and flow of test materials (p < 0.01). There was no significant difference in pH, working times, and dimensional changes between white MTA and NEC.

Discussion

MTA acted as a gold standard in this in vitro study. A number of investigations reported MTA as a root-end–filling material presenting excellent apical sealing and showed its superiority in comparison with other commonly used materials (16–18). Using MTA as a root-end filling in periapical surgery causes the healing of periapical lesions to a nearly normal condition (19, 20).

| TABLE 1. Electron Probe Microanalysis Results (Concentration wt% and Error) for White MTA and NEC. |
|-------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
|       | CaO    | SiO₂   | Bi₂O₃  | Al₂O₃  | MgO    | FeO    | SO₃    | P₂O₅   | TiO₂   | Na₂O   | CI     | H₂O+C  | SUM    |
|-------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| MTA (n = 2) | 44.27 (3.28) | 21.19 (1.60) | 16.11 (4.91) | 1.89 (0.10) | 1.32 (0.13) | 0.42 (0.17) | 0.51 (0.13) | 0.22 (0.10) | 0.12 (0.09) | 0.02 (0.04) | 0.40 (0.13) | 13.53 | 100 |
| NEC (n = 2) | 51.81 (1.26) | 6.28 (0.79) | ND     | 0.95 (0.31) | 0.23 (0.12) | ND     | 9.48 (1.46) | 8.52 (0.41) | ND     | 0.35 (0.07) | 0.18 (0.04) | 22.2 | 100 |

ND, not detected; H₂O+C, water, carbon dioxide, and other not mentioned elements.
MTA showed slight expansion upon setting, which was not significantly different from NEC. However, the new material exhibited reasonable film thickness and flow, which was statistically different from MTA. The slight expansion and reasonable flow and film thickness of MTA can ensure an effective seal after setting and reduce the subsequent leakage. These physical properties may account for the superior sealing ability of MTA over other root-end-filling materials (21–24). Also, NEC showed acceptable film thickness, flow, and comparable sealing ability compared with MTA (12).

The fineness of cement is a major factor influencing its rate of hydration and, consequently, its strength and setting characteristics. The United States patent for MTA states that the Portland cement used in the manufacture of MTA had a Blaine number in the range of 450 to 460 m²/kg (5, 6). Although NEC is finer, the NEC tested in this study appeared to fulfill the physical requirements of a root-end-filling material from the point of consistency, workability, adaptability, and setting time.

The EPMA results showed that the predominant elements in white MTA were calcium, silicon, and bismuth (presented as oxides), respectively. These were in agreement with our previous findings (9, 25). The predominant elements in NEC were calcium, sulfur, phosphorus, and silicon, respectively, which are dissimilar with MTA.

It was reported that calcium and phosphorous were the main components of MTA (7). This study, however, showed that the observed concentrations of phosphorus in MTA in contrast with NEC were close to the limit of detection (Table 1). These results appear to be entirely in agreement with previous findings (9, 25, 26) and the information supplied by the Dentsply Tulsa dental company, in which the phosphorus is not reported as a significant element in MTA (27).

The cementogenic activity of MTA is because of its release of an abundance of calcium ions, which interact with phosphate groups in the surrounding tissue fluid to form hydroxyapatite on the surface of white and gray MTA (28). Therefore, the clinical success of MTA can be attributed to its biocompatibility. On the other hand, EPMA revealed endogenous phosphorus in NEC (Table 1). Therefore, it seems reasonable to suspect that the presence of significant calcium and phosphate ions in NEC is most likely to form the hydroxyapatite compared with white MTA (13).

Calcium in its hydroxide form is the main chemical compound released by MTA when mixed with water (29). This may be explained with the similarities in the behavior of MTA and CH when used in direct pulp-capping procedures (30, 31). The CH release, along with the sealing ability, may account for the greater success obtained with MTA in direct contact with vital tissues. NEC consists of CH, so the same reaction may occur for NEC.

Our result showed that high alkalinity of NEC is comparable with white MTA, a favorable property related to its bactericidal properties (7), providing an excellent tight seal (16), and biocompatibility (32). The important constituents of NEC are alkaline earth metal oxides and hydroxides (eg, calcium oxide and CH), calcium phosphate, and calcium silicate. During and after mixing with its liquid, hydration reactions take place, producing CH. This production is mostly because of the reactions involving calcium silicates, calcium phosphate, and calcium oxide in addition to the presence of CH. CH dissociated into calcium and hydroxyl ions, increasing the pH and calcium concentration.

Based on the results obtained from this in vitro study, it was concluded that NEC as an experimental root-end-filling material differs chemically from MTA. NEC also exhibited acceptable physical properties. Further research is needed to establish clinical use.

**References**