Evaluation of Selected Properties of Mineral Trioxide Aggregate Sealer Cement

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Abstract

Introduction: The addition of a water-soluble polymer to mineral trioxide aggregate (MTA), which to date has been used primarily to seal lateral root perforations and as a root-end filling material, resulted in material that is suitable for use as an endodontic sealer. **Methods:** MTA was mixed with water at powder/liquid ratios of 4 and 3.33 and an addition of 2 μL to 20 μL of water-soluble polymer. The materials were tested for flow and film thickness, and the optimal quantity of polymer required to conform to EN ISO 6876 Section 4.3.1, 4.3.4 (2002) was determined. The resultant MTA sealer was tested for radiopacity using methods suggested by ISO 6876 (2002) using pulp canal sealer (PCS) as control. The effect of polymer addition on the hydration characteristics of the MTA and calcium silicate cement was evaluated by assessing paste microstructure under the scanning electron microscope after 30 days and by collecting 50 quantitative analyses of the hydration products and plotting the data as atomic ratios. Plots of Al/Ca versus Si/Ca and S/Ca and Al/Ca were drawn. **Results:** High additions of polymer were required for the flow and film thickness of MTA to conform to ISO 6876 (2002). The resultant cement sealer had a lower radiopacity than PCS but greater than the 3-mm thickness of Al specified by the international standard. The addition of polymer did not modify the hydration mechanism of MTA. **Conclusions:** The addition of a water-soluble polymer to MTA did not alter the hydration characteristics of the material and resulted in a material with improved properties suitable for use as endodontic sealer cement. *(J Endod 2009;35:1412–1417)*

Key Words

Flow, hydration, mineral trioxide aggregate, radiopacity, root canal sealer

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White mineral trioxide aggregate (MTA) is composed of tricalcium silicate, dicalcium silicate, tricalcium aluminate, calcium sulphate, and bismuth oxide. On hydration, the calcium silicates and bismuth oxide form calcium silicate-bismuth hydrate and calcium hydroxide. The tricalcium aluminate in the presence of calcium sulphate hydrates and forms ettringite, which is converted to monosulphate once the calcium sulphate is depleted (1, 2).

MTA is used primarily to seal lateral root perforations (3, 4) and as a root-end filling material (5–8). The use of MTA as a root-end filling material was identified because the material is hydraulic that sets in the presence of water. The uses of MTA have been mostly limited mostly because of its long setting time, inadequate compressive strength comparable to that of base and lining materials, and its poor workability. Attempts at improving the workability of Portland cement clinker (9) and other fast setting cement formulations (10, 11) have been made by using a polymer added to the mixing liquid. The polymer did not seem to affect the biocompatibility of the material (12, 13).

Newer developments of calcium silicate-based cement include the root canal sealers. Currently, two formulations are available namely Endo-CPM-Sealer and ProRoot Endo Sealer. The formulation of the former has not been reported. ProRoot Endo Sealer has been reported to be based on calcium silicate and is mixed in a liquid to a powder ratio of 1:2. The liquid is composed of water and a viscous water-soluble polymer. No details of the polymer used are given in both publications on this material (14, 15). Endo-CPM-Sealer was shown to be biocompatible and stimulated mineralization (16). Endo-CPM-Sealer caused mild to moderate reactions at 7 days of testing, which decreased with time. The response was similar to Angelus MTA and Sealapex. Mineralization and granulations birefringent to the polarized light were observed with all materials (16). ProRoot Endo Sealer showed similar sealing properties to epoxy resin-based sealer when evaluated using the fluid filtration system. In addition apatite-like crystalline deposits along the apical and middle thirds of the canal walls were observed (14). ProRoot Endo Sealer exhibited higher pushout strengths than AH Plus Jet or Pulp Canal Sealer particularly after storage in simulated body fluid. The mode of failure was mostly cohesive (15).

The aim of this study was to determine the optimal amount of polymer necessary to achieve a flow and film thickness that meet the EN ISO 6876; 2002 (17) criteria. The resultant formulation was assessed for radiopacity, and its hydration characteristics were analyzed by scanning electron microscopy and atomic ratio plots and compared with calcium silicate cement.

Materials and Methods

Preliminary Investigation

A preliminary investigation was performed to determine the quantity of polymer required by MTA to attain the flow and film thickness of a dental root canal sealer as recommended by EN ISO 6876; 2002 (17). The materials used in this study included mineral trioxide aggregate (MTA White; Dentsply, Tulsa Dental Products, Tulsa, OK) mixed with water only and also with additions of 2, 5, 10, 15, and 20 μL of water-soluble polymer (Glenium; Degussa Construction Chemicals, Manchester, UK) for every gram of cement and Pulp Canal Sealer (PCS; Kerr-Hawe S.A., Bioggio, Switzerland) used as control. The powder/liquid ratio was 4 or 3.33 for the MTA, whereas PCS was mixed as recommended by the manufacturer and also at a lower powder to liquid ratio.

Determination of Flow of Material

The flow of the material was tested as recommended by EN ISO 6876; 2002 (17). Using a graduated pipette, 50 μL of the material were dispensed on a glass plate measuring 40 $\times$ 40 mm and 5 mm in thickness. The second glass plate was placed centrally on top of the sealer followed by the 100-g weight after 180 seconds from the start of mixing. The assembly was left in place for 10 minutes from the start of mixing, after which the maximum and minimum diameters of the compressed disc of sealer were measured. The mean diameter was calculated if the diameters agreed to within 1 mm. If not, the test was repeated. Three determinations were made for each material tested.

Determination of Film Thickness

The film thickness of the materials was determined by using the method described in EN ISO 6876; 2002 (17). The combined thickness of two glass plates each measuring 5 mm in thickness and having a surface area of 200 mm2 was measured by using a micrometer to an accuracy of 1 μm. The materials were mixed and placed in between the glass plates. After 3 minutes from the start of mixing, the plates were loaded in a loading device (Triaxial; ELE International, Leighton Buzzard, United Kingdom), and a load of 150 N was applied until the sealer filled the area in between the glass plates. After 10 minutes from the start of mixing, the thickness of the combined glass plates and sealer was measured by using a micrometer. Three determinations were made for each material under test.

Further Studies

The adequate polymer proportioning that produced a material that satisfied the recommendations of EN ISO 6876; 2002 (17) for flow and film thickness was selected. The resultant material, MTA sealer (MTAS), was tested for radiopacity using pulp canal sealer as control. The hydration mechanisms of MTAS compared with calcium silicate-based cement with added polymer were determined.

Evaluation of Radiopacity

The experimental protocol was based on EN ISO 6876 Section 7.8; 2002 (17) for dental root canal sealing materials. The MTAS and pulp canal sealer (PCS) were mixed and compacted into stainless steel ring moulds 10 mm in diameter and 1-mm high and pressed against 2 glass cover slips to make the specimen 1-mm thick. Three specimens of each material were prepared. The cements were allowed to cure for 24 hours at 37°C and 100% relative humidity covered by a plastic sheet to avoid cement desiccation and after they were removed from the moulds and stored in distilled water at 37°C for 7 days.

The cements were placed directly on a cassette loaded with a cephalostat-type film with an intensifying screen (Kodak, Rochester, NY) adjacent to a 10-step wedge made of aluminium in which each step measured 1 mm in height (Agfa Mamoray; Agfa Gevaert, Mortsel, Belgium) and x-ray irradiated using a standard x-ray machine (GEC Medical Equipment Ltd, Middlesex, United Kingdom) at tube voltage of 50 kV, current of 50 mA, and exposure time of 0.05 seconds. The target to film distance was set at 100 cm. Three specimens per material under test were arranged on the cassette, and two x-rays were taken of the specimens. Eight layers of lead foil covered a small area of each film to obtain a small area of nonexposure. The radiographs were processed in an automatic processing machine (Clarimat 300; Gendex Dental Systems, Medivance Instruments Ltd, London, United Kingdom). A photographic densitometer (PTWdensix, Freiburg, Germany) was used to measure the density of the radiographic images of the specimens, each aluminium step, and the unexposed part of the film. Three density values of each material were obtained for each radiograph of each specimen, and the mean density was calculated. The net radiographic density was calculated by subtracting the base and fog value from the gross radiographic density. The base and fog value is the inherent optical transmission density (lowest density) of a film base plus the nonimage density contributed by the developed emulsion. Graphs were plotted for net radiographic density of the aluminium steps (NRDAL) versus the logarithm of the thickness of aluminium (log d) for each radiograph. From the resultant plots, the gradient and the intercept were calculated for each film. Linear regression of the data was obtained using the following formula:

$$\mathrm{NRD_{AL}} = m.\log d + \mathrm{I}$$

where NRDAL was the net radiographic density of the aluminium step wedge, m was the gradient, log d was the logarithm of the step height, and I was the intercept.

By rearranging this equation into the following:

$$\log \frac{d = (\mathrm{I} - \mathrm{NRD})}{-m}$$

the logarithm of the relevant thickness of aluminium for each material could be calculated from its net radiographic density for each film taking into consideration that specimen thickness was 1 mm. Logarithms of step height were then converted to thicknesses of aluminium (18). The data were evaluated using SPSS software (SPSS Inc, Chicago, IL). Parametric tests were performed as the data were normally distributed. Analysis of variance with p = 0.05 and Tukey post hoc test was used to perform multiple comparison tests.

Microscopy of Cured Cements

The MTA was mixed with water and 20 μL of polymer at a powder to liquid ratio of 3.33. This formulation was selected because it provided material flow and film thickness that met the EN ISO 6876, 2002 (17) criteria. The same dosage of polymer was used and mixed with calcium silicate cement (Aalborg White, Aalborg, Denmark). The pastes were compacted in a circular mould 30 mm in diameter using a stainless steel plugger. The materials were cured in sealed plastic (polythene) containers at 37°C for 30 days using a thermostatically controlled water bath (MGW Lauda M 20; Leica Microsystems SA, Rueil-Malmaison, France), after which they were immersed in acetone for 4 days to remove any remaining water and then dried in a vacuum desiccator for 8 t hours. The dried paste pieces were set in epoxy resin using vacuum impregnation. The hardened resin block was sawed (Labcut 1010; Agar Scientific, Stansted, United Kingdom) and ground under copious water irrigation using progressively finer grits of abrasive paper to produce a flat surface. A thin conductive coating of evaporated carbon was applied to the sections before examination in the scanning electron microscope (SEM). The SEM used was an ISI SS40 (ISI, Tokyo, Japan), with an energy-dispersive x-ray system (SAMx Numerix, Levens, France) and a standard beryllium window x-ray detector. The beryllium window was used to absorb x-rays emitted from light elements. Quantitative analyses were performed using x-ray standards obtained from minerals for each element, with the exception of bismuth. A bismuth standard was obtained using particles of bismuth oxide in the uncured MTA. Oxygen was calculated by stoichiometry. The sections were examined by using backscattered electron imaging. The analysis of hydration products was performed by the following:

1. Identifying and labeling of hydration products viewed under the SEM in the backscatter mode.
2. Examining the sections in more detail by collecting a series of 50 quantitative analyses of the hydration products and plotting

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