Hydration Characteristics of Calcium Silicate Cements with Alternative Radiopacifiers Used as Root-end Filling Materials

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Abstract

Introduction: Mineral trioxide aggregate (MTA) is composed of calcium silicate cement and bismuth oxide added for radiopacity. The bismuth oxide in MTA has been reported to have a deleterious effect on the physical and chemical properties of the hydrated material. This study aimed to investigate the hydration mechanism of calcium silicate cement loaded with different radiopacifiers for use as a root-end filling material. Methods: Calcium silicate cement loaded with barium sulfate, gold, or silver/tin alloy was hydrated, and paste microstructure was assessed after 30 days. In addition, atomic ratio plots of Al/Ca versus Si/Ca and S/Ca and Al/Ca were drawn, and X-ray energy dispersive analysis of the hydration products was performed to assess for inclusion of heavy metals. The leachate produced from the cements after storage of the cements in water for 28 days and the leaching of the radiopacifiers in an alkaline solution was assessed by using inductively coupled plasma. Results: The hydrated calcium silicate cement was composed of calcium silicate hydrate, calcium hydroxide, ettringite, and monosulfate. Unhydrated cement particles were few. No heavy metals were detected in the calcium silicate hydrate except for the bismuth in MTA. Calcium was leached out early in large quantities that reduced with time. The barium and bismuth were leached in increasing amounts. Copper was the most soluble in alkaline solution followed by bismuth and barium in smaller amounts. Conclusions: The bismuth oxide can be replaced by other radiopacifiers that do not affect the hydration mechanism of the resultant material. (J Endod 2010;36:502-508)

Key Words

Calcium silicate cements, hydration, radiopacifiers, rootend filling materials

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White mineral trioxide aggregate (MTA) was developed for use as a root-end filling material after apicectomy. It is composed primarily of tricalcium and dicalcium silicates (1, 2), which are the main constituent elements of Portland cement used in the building industry (3). ProRoot MTA is composed mainly of 53.1% tricalcium silicate, 22.5% dicalcium silicate, 21.6% bismuth oxide, and small proportions of tricalcium aluminate and calcium sulfate (4, 5). The bismuth oxide is added to enhance the radiopacity of the material. The radiopacity is a very important property for all root-end filling materials because the cement has to be detected radiographically and thus distinguished from surrounding anatomic structures (6).

The radiopacity of MTA varied between different reports for the same brand where ProRoot MTA (Dentsply, Tulsa Dental Products, Tulsa, OK) was reported to have radiopacity values ranging between 5.34 mm Al and 8.26 mm Al (6–12) and also between different brands. MTA-Angelus (Angelus Soluções Odontológicas, Londrina, Brazil) has been reported to have a radiopacity equivalent to 3-3.3 mm Al (13). All other materials used in dentistry as root-end filling materials have higher radiopacity values than ProRoot MTA and MTA Angelus. Both MTAs demonstrated radiopacity values lower than Super-EBA (9.9 mm Al), intermediate restorative material (IRM) (9.3 mm Al), gutta-percha (11.0 mm Al), and amalgam (15.6 mm Al) (7). All radiopacity values reported for root-end filling materials are higher than the 3 mm Al recommended by the International Standards (ISO 6876, Section 7.8; 2002) (14), thus questioning the need for such a high radiopacifier loading in these materials.

Hydration of MTA results in the production of calcium-silicate-bismuth-hydrate, calcium hydroxide, ettringite, and monosulfate (2). The unbound bismuth is leached out to the surrounding tissues (4), and the leaching increases with time. The use of bismuth oxide with Portland cement reduces the material strength (15, 16). Bismuth oxide induces cytotoxicity in dental pulp cells (17). Conversely, most research performed on the biocompatibility of MTA has proved that this material is biocompatible and induces cell growth and activity. This might demonstrate that other factors might be involved in the biocompatibility of MTA, and the bismuth oxide added to the calcium silicate cement might not play a major role in the biocompatibility of the material (11, 18).

Other materials can be used to enhance the radiopacity of calcium silicate-based cements. Addition of gold powder, silver/tin alloy, and barium sulfate results in a radiopacity value of more than 3 mm Al, which is similar to the value reported for MTA Angelus (13). Loading with 25% gold powder and 20% silver/tin alloy renders the calcium silicate cement as radiopaque as ProRoot MTA (12).

The aim of this study was to investigate the hydration mechanism of calcium silicate cement loaded with different radiopacifiers for use as root-end filling materials. In addition, the effect of radiopacifiers on the paste microstructure and whether the radiopacifiers were inert in the alkaline pore fluid were also assessed.

Materials and Methods

The materials used in this study included calcium silicate cement (Aalborg White, Aalborg, Denmark manufactured to BS EN 197-1: 2000, (19) type CEM I), calcium silicate cement replaced with 25% barium sulfate (CSC-Ba) (Sigma-Aldrich, Gillingham, UK), calcium silicate cement replaced with 25% atomized gold (CSC-Au) (Sigma-Aldrich), calcium silicate cement replaced with 20% powdered silver/tin alloy (CSC-AgSn) (Degussa Dental GmbH, Hanau, Germany), and MTA (ProRoot MTA).

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A2 191 Un-hydrated Ca(OH) 250 200 1/31 108 511 50u 0.50 C.60 A3 A4 0.53 0.50 11.43 5/Ca C.40 A/Da 0.3 C.80 0.20 0.25 4En 44 m0.12 C.10 0.00 0.00 Si/Ca u ce Cement-barium sulphate 400<u>–</u> В B2 - I I Ca(OH) <u>90</u>-Barium sulphate 51 8 50µn c.e **B**4 **B**3 0.8 0.40 S/Ce 0.45 = G.10

Calcium silicate cement

Figure 1. Microscopy and analysis of (*A*) calcium silicate cement, and cement replaced with different radiopacifiers (*B*) barium sulfate, (*C*) gold, (*D*) silver/tin, (*E*) ProRoot MTA. (1) Scanning electron micrographs in backscatter mode of transverse sections showing the paste microstructure after 30 days of hydration (original magnification, \times 50); (2) X-ray energy dispersive analysis showing the lack of inclusion of heavy metals in the hydration products of the alternative radiopacifiers; (3) atomic ratio plots of Al/Ca vs Si/Ca; and (4) atomic ratio plots of S/Ca vs Al/Ca showing the various products of hydration (C-S-H, calcium silicate hydrate; CH, calcium hydroxide; Aft, ettringite; AFm, monosulfate).

C.05

c.20

0.30

0.50

3.5

0.0

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