Solubility and Disintegration of New Calcium Aluminate Cement (EndoBinder) Containing Different Radiopacifying Agents

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Abstract

Introduction: The aim of this study was to evaluate the solubility and disintegration of EndoBinder (EB) containing 3 different radiopacifying agents, bismuth oxide (Bi_2O_3), zinc oxide (ZnO), or zirconium oxide (ZrO_2), in comparison with gray mineral trioxide aggregate (GMTA) and white MTA (WMTA). **Methods:** Ten specimens of each cement were made in a stainless steel matrix (20 × 1.5 mm) according to Specification no. 57 of American National Standards Institute/American Dental Association: EB + Bi_2O_3, EB + ZrO, EB + ZnO, WMTA, and GMTA. The specimens were weighed on an accurate analytical scale and immersed in 50 mL distilled and deionized water at 37°C for 7 days. Afterwards, specimens were dried and weighed again to determine mass loss (%). Resulting solutions were analyzed in an atomic absorption spectrophotometer for identification and quantification of chemical elements released. **Results:** All cements presented mean values of solubility and disintegration above the American National Standards Institute/American Dental Association Specification no. 57. EB + Bi_2O_3 presented the lowest mass loss (5.08%) and WMTA (6.65%) the highest, with no statistically significant difference ($P > .05$). The release of several chemical elements was observed, mostly metal ions. Only GMTA and EB + Bi_2O_3 showed the presence of Cr, with significant difference ($P < .05$). EB + ZnO presented the highest levels of Pb, followed by WMTA ($P < .05$). For As, the cements presented different release levels, with EB + ZnO showing the highest and GMTA the lowest levels ($P < .05$). However, the amounts of As and Pb released were lower than the safe limit proposed by ISO 9917-1. **Conclusions:** Irrespective of the radiopacifying agents used, EndoBinder presented similar behavior to MTA. *(J Endod 2014;40:261–265)*

Key Words

Arsenic, calcium aluminate cement, lead, MTA, solubility

Certain procedures performed during endodontic therapy require a specific sealing cement to obtain a successful treatment (1). This cement must be biocompatible (2), noncarcinogenic and non-genotoxic (3), must not stain dental structures (4), must be radiopaque (5), and be insoluble in oral fluids (6).

Mineral trioxide aggregate (MTA) was originally developed for retrograde filling and treatment of radicular and furcal perforations (1), and because of its good clinical performance, it has been used in several other applications such as pulpotomy and pulp capping (1).

However, some of the negative features of MTA must be pointed out, such as the long setting time (1), poor handling characteristics (7), low compressive strength (8), low flow capacity (9), high incidence of dental structure staining (4), high solubility (6), and presence and release of arsenic (2, 10), with levels above those recommended by the ISO 9917-1 standard (11).

MTA has undergone a series of modifications in its original formulation because of its poor handling characteristics and long setting time (7). Studies in which these cement compositions have been analyzed have demonstrated that Angelus MTA presents a higher quantity of calcium carbonate, calcium silicate, and zinc-barium phosphate than the conventional types, thus contributing to a significant improvement in its physicochemical properties (1). Although the MTA setting time is lower than that of Portland cement, it is still considered long enough to make the material unstable when in contact with moisture before it has completely hardened (7, 9). Considering effects such as these, the development of new materials with adequate biological and physicochemical properties is justified (6).

Therefore, a new calcium aluminate–based cement called EndoBinder (Binderware, São Carlos, SP, Brazil) was developed by the Federal University of São Carlos (UFSCar-Brazil, patent number PI0704502-6) to preserve the properties and clinical applications of MTA without its negative features. EndoBinder is composed of (% by weight) Al_2O_3 (≥68.0), CaO (≤31.0), SiO_2 (0.3–0.8), MgO (0.4–0.5), and Fe_2O_3 (<0.3), which, according to several studies, presents adequate biological properties (12, 13).

There were 3 main reasons for the development of EndoBinder. (1) The selection of the reagent materials allows control of impurities such as Fe_2O_3, which promotes tooth darkening (4), and free MgO and CaO, which may be responsible for an

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undesirable expansion of the material in contact with moisture (1). (2) Although MTA contributes to making the medium an inhospitable environment for microorganism growth (14), after it is hydrated, an excessive concentration of it might lead to fibrosis of the adjacent tissues. Therefore, the balance between the phases rich in Al_2O_3 and $CaCO_3$, which the EndoBinder synthesis process allows, promotes better results with regard to biocompatibility and physicochemical properties (12), in addition to allowing (3) the formation of autogenous phases capable of helping to control the cement setting time, thereby avoiding the unnecessary inclusion of additives (15).

Hydraulic cements, such as EndoBinder and MTA, depend entirely on the physicochemical interaction with the medium, which is associated with the solubility and disintegration of the material (16). It is known that because of its long setting time (1), MTA is highly unstable when moistened before it has completely hardened (7, 9), which is the prevailing condition in the oral medium where the cement is routinely used (17). This situation promotes greater solubility and disintegration of MTA and, consequently, greater release of components present in the cement (18). Thus, it is necessary to evaluate this physicochemical property of EndoBinder, as well as the released components, to gain better understanding of its biological behavior.

The objective of this study was to evaluate the solubility and disintegration of EndoBinder (EB) containing different radiopacifying agents (Bi_2O_3, ZnO, or ZrO_2), in addition to analysis of the components released by it, in comparison with gray MTA (GMTA) and white MTA (WMTA). The hypothesis tested was that EndoBinder would present better physicochemical properties than MTA.

Materials and Methods

The cements tested in this study are described in Table 1.

Fifty test specimens (n = 10) were made by using a stainless steel matrix (20 × 1.5 mm), according to Specification no. 57 of American National Standards Institute/American Dental Association (ANSI/ADA) (19). The cements were manipulated in accordance with the manufacturers' recommendations, with the proportion of 1 g powder to 0.21 mL distilled water for EndoBinder and 1 dose of powder to 1 drop of distilled water for MTA.

After manipulation, the cements were inserted into the matrix, and an impermeable nylon thread was placed inside the cement. Next, a glass plate covered with cellophane film was placed on the upper side of the matrix, and the entire ensemble was pressed.

After a period corresponding to 3 times the setting time of each cement, test specimens were removed from the matrix and then weighed on an accurate analytical scale (Model AB204; Mettler-Toledo, Barueri, SP, Brazil). Each test specimen was lifted by the nylon thread and individually placed in a plastic receptacle containing 50 mL distilled and deionized water. Care was taken to avoid any contact between the test specimen and the inner surface of the receptacle. After this, the receptacles were sealed and stored in an incubator at 37°C for 7 days.

TABLE 1. Cements Used in the Study

Cements	Manufacturer
EndoBinder + 20% (weight) Bi_2O_3	Binderware, São Carlos, SP, Brazil
EndoBinder + 20% (weight) ZnO	
EndoBinder + 20% (weight) ZrO_2	
GMTA	Ângelus, Londrina, PR, Brazil
WMTA	

On conclusion of this period, the test specimens were removed from the receptacles, rinsed with deionized water, and dried with absorbent paper. At the end of this stage, specimens were placed in a dehumidifier containing silica gel at 37°C for 24 hours. Next, they were weighed again. The weight loss (mg) of each test specimen was determined by the difference between the first and the second weighing. The values obtained in milligrams were converted into percentages.

To analyze the components released by the cements when immersed in distilled and deionized water, 10 mL of the resulting solution from each receptacle after the solubility and disintegration test was analyzed in an atomic absorption spectrophotometer (Perkin-Elmer Analyst 100; Perkin-Elmer Inc, Norwalk, CT). To determine and quantify the elements released, lanthanum oxide was added to the samples to eliminate ionic interferences, especially by phosphate ions. A standard solution of 10 mg/dL was diluted in water to the following concentrations: 0.025 mg/dL, 0.05 mg/dL, 0.1 mg/dL, 0.25 mg/dL, 0.5 mg/dL, and 1.0 mg/dL. These diluted samples were aspirated into a chamber to be burned after being mixed with acetylene (fuel) and nitrous oxide (oxidant). Specific lamps crossing the flames were used to identify and quantify the chemical elements present in the samples. The elements quantified were ruthenium (Ru), lead (Pb), nickel (Ni), chromium (Cr), arsenic (As), cadmium (Cd), and calcium (Ca).

The results (in triplicate) were calculated and expressed in milligrams concentration unit per kilograms (mg/kg - ppm).

The normal distribution of data was tested by the Kolmogorov-Smirnov test, and the values obtained for solubility and disintegration (1-way analysis of variance [ANOVA], Bartlett test, $P < .05$) and quantification of chemical elements released (1-way ANOVA, Tukey test, $P < .05$) were statistically analyzed by using the GraphPad Prism 4.0 software program (GraphPad Software, La Jolla, CA).

Results

The values obtained in the solubility and disintegration test and their comparisons are shown in Table 2.

The results demonstrated that all cements presented solubility and disintegration above the limit proposed by Specification no. 57 of ANSI/ADA (19). EB + Bi_2O_3 presented the lowest mass loss (5.08%) and WMTA (6.65%) the highest, with no statistically significant difference ($P > .05$).

The mean values of different chemical elements released by the cements after the solubility and disintegration test are shown in Table 3.

The release of several chemical elements was observed, mostly metal ions. Only GMTA and EB + Bi_2O_3 showed the presence of Cr, with statistically significant difference ($P < .05$). EB + ZnO presented the highest levels of Pb, followed by WMTA ($P < .05$). For As, the cements presented different levels of release, with the highest shown by EB + ZnO and the lowest by GMTA ($P < .05$). However, the amounts of As and Pb released were below the safe limit proposed by the ISO 9917-1 Specification (11).

With regard to Ca release, WMTA presented the highest levels and GMTA the lowest, with significant difference between them and in comparison with the other cements ($P < .05$). EndoBinder presented intermediate values, with the highest being shown by EB + ZnO, with statistically significant difference from the other cements ($P < .05$).

Discussion

Radiopacity is one of the most important properties of materials used in endodontic therapy (5). Therefore, cements used for this