Assessment of Color Stability of White Mineral Trioxide Aggregate Angelus and Bismuth Oxide in Contact with Tooth Structure

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Abstract

Introduction: Dental discoloration with use of materials containing bismuth oxide has been reported. It is postulated that the discoloration is a result of chemical interaction of bismuth oxide with dentin. The aim of the study was to analyze dental color alteration and the chemical interaction of bismuth oxide with the main components present in composite (methacrylate) and in dentin (collagen). **Methods:** Fifty bovine teeth were prepared and filled with white mineral trioxide aggregate (MTA) Angelus, Portland cement (PC) with 20% zirconium oxide, or PC with 20% calcium tungstate and then sealed with composite. Triple antibiotic paste and unfilled samples were the positive and negative controls, respectively. The specimens were stored in separate flasks immersed in tap water at 37°C with ambient light blocked out. The color assessment was performed with a spectrophotometer at different intervals, namely before filling and 24 hours, 15 days, and 30 days after filling. The color change and the luminosity were calculated. The statistical analysis was performed by using nonparametric Kruskal-Wallis and Dunn tests ($P < .05$). The interaction of the bismuth oxide, zirconium oxide, and calcium tungstate with collagen and methacrylate was assessed by placing the materials in contact, followed by color assessment. **Results:** The analysis of color change values showed that all the materials presented color alteration after the evaluated periods. Statistically higher luminosity was verified for PC/20% zirconium oxide in comparison with white MTA Angelus ($P < .05$). The teeth filled with white MTA Angelus demonstrated a grayish discoloration with evident dentin staining. Bismuth oxide exhibited a color change when in contact with collagen. **Conclusions:** The color of white MTA Angelus was altered in contact with dental structures. Collagen, which is present in dentin matrix, reacted with bismuth oxide, resulting in a grayish discoloration. The use of an alternative radiopacifier to replace bismuth in white MTA is indicated. *(J Endod 2014;40:1235–1240)*

Key Words

Bismuth oxide, spectrophotometry, tooth discoloration, white MTA

White mineral trioxide aggregate (MTA) was developed as a root-end filling material and is also used for pulp capping, perforation, and apexification (1, 2). Some of these procedures require maintenance of the cement in the coronal portion for long period (3). It is critical that the interaction of the cement with the dentinal components does not result in dental staining, principally when esthetics is directly involved.

The first formulation of MTA presented a gray color, which prejudiced its use in esthetics areas (2, 4). The dental staining encouraged manufacturers to develop a tooth-colored formulation. White MTA (WMTA) was introduced to solve the problem related to dental color alteration, with a reduction of Al_2O_3, MgO, and FeO (4). Reduction of FeO resulted in reduction of the aluminoferrite phase, which is responsible for the gray color of gray MTA (5). However, various studies have verified *in vitro* discoloration even with the white formula (4, 6–9). A marked gray-colored alteration was observed in contact with the dentin (7, 8). Clinically, similar dental staining with WMTA was also widely reported (10, 11).

MTA is composed mainly of tricalcium and dicalcium silicates (12). The radiopacity is provided by the addition of approximately 20% bismuth oxide (13). Bismuth has been hypothesized as the component responsible for the color alteration of WMTA (9, 14). The literature reporting color alterations of the dental tissues in contact with the bismuth compounds only mentions the discoloration, but no reason is given as to what causes the bismuth to discolor the tooth structure. It is hypothesized that bismuth oxide interacts with the collagen present in dentin, resulting in a color change. The aim of this research was to investigate the color change induced by bismuth oxide and WMTA Angelus in contact with the tooth structure and also with collagen, which is the main constituent of the dentin. Furthermore, the interaction of bismuth oxide with methacrylate, which is the precursor of dental composites, was also investigated.

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Materials and Methods

Sample Preparation

A total of 50 bovine teeth were obtained. They were cleaned, and the crowns were sectioned with a 0.3-mm diamond disk (Isomet; Buehler, Lake Bluff, IL) to obtain 10 × 10 mm enamel-dentin blocks. The thickness of each block was standardized at 3.5 ± 0.1 mm and checked with a vernier. A cavity with a diameter corresponding to 5.0-mm diameter and 1.5-mm depth was prepared in the center of dentinal surface with a high-speed diamond bur 4054 (Medical Burs Sorensen, São Paulo, SP, Brazil). The tooth samples and preparation are shown in Figure 1. The specimens were then immersed in 1% sodium hypochlorite for 30 minutes, washed with distilled water, and placed in 20% EDTA (pH 7.7) for 2 minutes (6). A final flush of distilled water was performed, and the samples were dried with gauze. The specimens were randomly divided into 3 groups (n = 10) as follows: WMTA (WMTA Angelus; Angelus, Londrina, PR, Brazil), Portland cement (PC) with 20% zirconium oxide (ZO) (Sigma-Aldrich, São Paulo, SP, Brazil), and PC with 20% calcium tungstate (CT) (Sigma-Aldrich).

The cavities were filled with the test cements; a positive control (n = 10) was filled with triple antibiotic paste, and a negative control (n = 10) was left unfilled. WMTA was mixed according to the manufacturer's instructions. Radiopacified PCs were prepared by mixing 1 g powder to 0.3 mL distilled water. Triple antibiotic paste composed of metronidazole, minocycline, and ciprofloxacin was prepared according to Trope (15). The external limit of the cavities was conditioned with 37% phosphoric acid for 30 seconds, washed with distilled water for 1 minute, and dried with an air syringe. A layer of adhesive (Adper Single Bond 2; 3M ESPE, Sumaré, SP, Brazil) was applied to the conditioned external limit of the cavity and light-cured (Optilight LD Max; Gnatus, Ribeirão Preto, SP, Brazil) for 20 seconds to allow the sealing of the interface with resin. The cements were compacted into the prepared cavities at a depth of 1.5 mm. After the cements set, the cavities were sealed with a natural flow resin B2 (Nova DFL, Rio de Janeiro, RJ, Brazil). The polymerization was performed with an LED curing light (Optilight LD Max) for 60 seconds. The specimens were stored separated in dark flasks and were immersed in tap water at 37°C throughout the period of analysis.

Color Assessment of Tooth Structure in Contact with Cements

The color assessments were performed before filling and 24 hours, 15 days, and 30 days after filling. A spectrophotometer (Vita Easyshade; VITA Zahnfabrik, Bad Sackingen, Germany) was used for assessments. The measurement was performed once for each sample in each period. The excess water of the specimen was removed with gauze, and then the specimens were adapted into a wooden box with a 5-mm-diameter hole. The sample was placed in the hole with the enamel side positioned in direction of the outside of the box. The box was closed, and the tip of spectrophotometer was positioned in the hole to measure the color in the same position for all teeth.

The color was established on the basis of the CIELAB color system, as defined by the International Commission on Illumination (16). The value of the luminosity (L*) represents the lightness values, a* represents the values of red-green, and b* represents the values of yellow-blue. These values are numeric and use the formula $\Delta E = [(L_1-L_0)^2 + (a_1-a_0)^2 + (b_1-b_0)^2]^{1/2}$ to achieve an estimated degree of color change (ΔE). The lightness values (L) were obtained for all periods directly in the spectrophotometer and were used to evaluate the darkening of the groups (Fig. 2).

Stereomicroscopic Analysis of Representative Samples

A representative sample of cements was selected and sectioned in the center by using a 0.3-mm diamond disk (Buehler) (Fig. 3). The samples were photographed by using a stereomicroscope (Stemi 2000C; Carl Zeiss, Jena, Germany) and the Axiovision software (Carl Zeiss).

Color Assessment of Tooth and Resin Components in Contact with Radiopacifiers

The main components of dentin (collagen) and resin (methacrylate) were tested in contact with the radiopacifiers present in WMTA Angelus and test cements to verify the consequent color (Fig. 4). Collagen (Sigma-Aldrich) and methacrylate (Sigma-Aldrich) samples were prepared by dissolving 1 g of each powder in 1 mL distilled water. One gram of radiopacifier (bismuth oxide, ZO, and CT) was placed into the containers. The experimental setup was kept incubated at 37°C for 72 hours. The containers were then photographed, and color of the components was compared.

Statistical Analysis

Statistical analysis was performed by using nonparametric Kruskal-Wallis and Dunn tests ($P < .05$) as a result of the absence of normal distribution confirmed in D'Agostino and Pearson analysis.

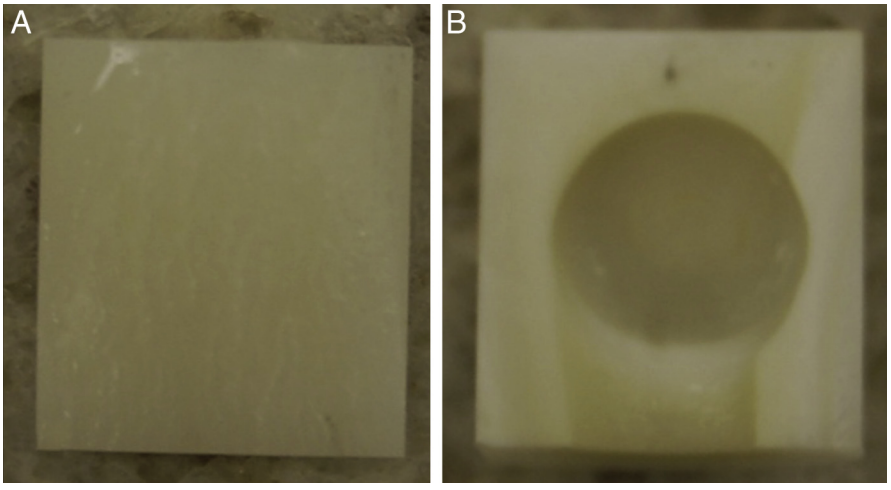

Figure 1. Sample bovine tooth showing buccal surface (*A*) and palatal surface with cavity preparation (*B*).