

Color Stability, Radiopacity, and Chemical Characteristics of White Mineral Trioxide Aggregate Associated with 2 Different Vehicles in Contact with Blood

Bruno Martini Guimarães, DDS, MSc,* Talita Tartari, DDS, MSc,*
Marina Angélica Marciano, DDS, PhD,* Rodrigo Ricci Vivan, DDS, PhD,*
Rafael Francisco Lia Mondeli, DDS, PhD,* Josette Camilleri, DDS, PhD,[†]
and Marco Antonio Hungaro Duarte, DDS, PhD*

Abstract

Introduction: Discoloration of mineral trioxide aggregate (MTA) can be exacerbated by the interaction of the cement with body fluids such as blood. This study aimed to analyze the color alteration, chemical characteristics, and radiopacity of MTA manipulated with 2 different vehicles after immersion in blood or distilled water (DW). **Methods:** MTA mixed with 100% DW or 80% DW/20% propylene glycol (PG) as vehicles were placed into rubber rings and incubated at 37°C and 100% relative humidity until set. Color assessment and scanning electron microscopy/energy-dispersive spectroscopy analysis were performed after setting and repeated after 7, 15, and 30 days after immersion in blood and DW. Statistical analysis for color alteration and radiopacity was performed using nonparametric Kruskal-Wallis and Dunn tests ($P < .05$). **Results:** When 80% DW/20% PG was used as the vehicle, significantly lower color alterations were observed for all time periods compared with 100% DW when immersed in blood ($P < .05$). All surfaces displayed morphologic changes after immersion in both media because of loss of bismuth. A decrease in radiopacity was observed over time in all groups, with a statistically significant difference after 30 days for groups DW immersed in blood and 80% DW/20% immersed in both media ($P < .05$). **Conclusions:** The ratio of 80% DW/20% PG as a vehicle for MTA results in a lower color alteration when in contact with blood. (*J Endod* 2015;41:947–952)

Key Words

Blood, characterization, color stability, tooth discoloration, white mineral trioxide aggregate

Mineral trioxide aggregate (MTA) is a calcium silicate–based cement containing bismuth oxide as a radiopacifier (1). It was developed as a root-end filling material but is also used for vital pulp therapies, treatment of root fractures, perforations, and apexifications (2–4). Dental discoloration has been reported after the application of MTA (5, 6). The interaction of MTA with sodium hypochlorite, which is used regularly for irrigation in endodontics resulted in a black MTA surface. The bismuth oxide reacted with sodium hypochlorite forming bismuth carbonate, which is light sensitive (7). The influence of light on the color stability of MTA has been reported (8). Dental staining resulting from the interaction of the cement with dental hard tissues and tissue fluids is detrimental, especially in esthetic regions (3).

The contamination of MTA by blood has been investigated in a number of laboratory studies in terms of the effect on its physical properties (9), leakage (10), displacement (11), and marginal adaptation (12). Blood contamination has a detrimental effect on the chemical properties of MTA, resulting in a lack of formation of the crystalline calcium hydroxide in the early stage of the hydration process (9). The interaction of blood with MTA when used for vital pulp therapy revascularization or perforation repair has also been implicated with discoloration (6). The mechanisms of how blood contamination exacerbates the discoloration process are currently unknown (6).

Propylene glycol (PG) is frequently used in dentistry as a vehicle for calcium hydroxide and has also been tested as an additive to improve MTA mixing (13–16). The ratio of 80% distilled water (DW)/20% PG increased the setting time, improved flowability, pH, and calcium ion release in the initial periods of the experiment (15). The effect of PG on the color alteration of MTA is unknown.

The aim of this investigation was to analyze color alteration, chemical characteristics, and radiopacity of MTA Angelus (Londrina, PR, Brazil) manipulated with different vehicles after immersion in blood or DW for different periods of time.

Materials and Methods

White MTA (Angelus) was mixed with 100% DW or 80% DW/20% PG as vehicles (15). The powder/liquid ratio was the same for all groups (1 g powder to 0.3 mL liquid).

From the *Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Bauru, São Paulo, Brazil; and [†]Department of Restorative Dentistry, Faculty of Dental Surgery, University of Malta, Msida, Malta.

Address requests for reprints to Dr Bruno Martini Guimarães, Al Octávio Pinheiro Brisolla, 9-75-CEP 17012-901, Bauru School of Dentistry, University of São Paulo, Bauru, São Paulo, Brazil. E-mail address: brunomgui@usp.br
0099-2399/\$ - see front matter

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<http://dx.doi.org/10.1016/j.joen.2015.02.008>

After setting, the materials were distributed according to the immersion solution, which was manual defibrinated sheep blood (to avoid coagulation) as described by Yeh et al (17) and DW, both allocated inside culture plates in a quantity of 3 mL (Corning Inc, New York, NY) according to the following groups:

1. MTA + 100% DW immersed in blood
2. MTA + 100% DW immersed in DW
3. MTA + 80% DW/20% PG immersed in blood
4. MTA + 80% DW/20% PG immersed in DW

Color Assessment

The cements were mixed and placed into rubber rings, with a 10-mm internal diameter and 1-mm height. The rings filled with the materials were stored in an incubator to completely set at 37°C and 100% relative humidity for 2 hours (15).

Cement samples (*n* = 10) were demolded, and the baseline color of the specimens was determined using a spectrophotometer (Vita Easyshade; Vita Zahnfabrik H. Rauter, Bad Säckingen, Germany) against a white Teflon (DuPont, Habia, Knivsta, Sweden) background to obtain the values of L* (lightness, from 0 = black up to 100 = white), a* (from a = green up to +a = red), and b* (from b = blue up to + b = yellow). Three measurements were performed for each sample. The measurements for each group were repeated after 7, 15, and 30 days of immersion in blood and DW allocated inside culture plates.

CIELab color changes (ΔE) were determined according to the Commission Internationale l'Eclairage (International Commission on Illumination) by calculating the distance between 2 points by using the following formula:

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

Delta values were calculated based on the difference between the final and baseline values for each coordinate (L*, a*, and b*), and values ≥ 3.3 were considered clinically unacceptable (18).

Scanning Electron Microscopy/Energy-dispersive X-ray Spectroscopy

All disk samples were also examined by scanning electron microscopy (SEM) (Aspex Express; Fei Europe, Eindhoven, Netherlands) at an accelerating voltage of 15–20 kV after setting and after 7, 15, and 30 days of immersion in blood or DW. At each time point, cement disks were placed on aluminum stubs using adhesive carbon discs and analyzed uncoated. Scanning electron micrographs of the sample surfaces at different magnifications were captured. Elemental analysis by energy-dispersive X-ray spectroscopy (EDX), which is fully integrated to the Aspex Express SEM (Fei Europe, Eindhoven, The Netherlands), was conducted over the entire area to determine the elemental composition of the samples. The specimens were then returned to their respective soaking solutions until further testing. A fresh blood drop was also examined by SEM/EDX.

Radiopacity

Six samples were selected for each group having a diameter and thickness according to ISO 6876:2012 and confirmed using a digital caliper (Mitutoyo Corp, Tokyo, Japan). Radiographs were acquired after setting and after 7, 15, and 30 days of immersion in blood or DW by directly placing the specimens of each group on a 57 × 76-mm Ultra-speed occlusal radiographic film (Eastman Kodak

TABLE 1. Score Values for Color Alteration and Luminosity in the Different Groups Evaluated after Immersion in Blood and Distilled Water

Groups	Immersion substances	ΔE						Luminosity					
		T07, median (min-max)	T15, median (min-max)	T30, median (min-max)	T0, median (min-max)	T07, median (min-max)	T15, median (min-max)	T30, median (min-max)	T0, median (min-max)	T07, median (min-max)	T15, median (min-max)	T30, median (min-max)	
MTA/DW	Blood	72612.88 (31600.39–208049.62) ^{Aa}	68419.58 (38124.22–206688.62) ^{Aa}	374.85 (265.00–626.50) ^{Ab}	17.00 (16.40–19.90) ^{Aa}	4.20 (2.80–7.60) ^{Bb}	3.9 (3.20–7.30) ^{Bb}	4.55 (3.30–7.20) ^{Bb}	17.00 (16.40–19.90) ^{Aa}	16.95 (16.40–17.20) ^{Ac}	18.40 (17.90–18.70) ^{Aa}	17.90 (16.30–19.80) ^{Aab}	
	DW	0.35 (0.25–0.63) ^{Bb}	1.27 (0.50–2.85) ^{Ba}	1.95 (0.50–6.73) ^{Aa}	17.00 (16.40–17.30) ^{Abc}	10.30 (9.90–11.90) ^{Ab}	10.40 (10.00–11.30) ^{Ab}	10.65 (9.90–11.10) ^{Ab}	10.30 (9.90–11.90) ^{Ba}	10.30 (9.90–11.90) ^{Ab}	10.40 (10.00–11.30) ^{Ab}	10.65 (9.90–11.10) ^{Ab}	
MTA/80% DW and 20% PG	Blood	15.62 (9.32–19.08) ^{Ba}	15.80 (13.23–19.03) ^{Ba}	1.65 (0.30–2.20) ^{Bb}	15.70 (15.10–15.90) ^{Ba}	16.30 (16.00–16.90) ^{Bbc}	17.90 (17.80–19.10) ^{Aab}	15.75 (15.10–15.90) ^{Bc}	16.30 (16.00–16.90) ^{Bbc}	16.30 (16.00–16.90) ^{Bbc}	16.30 (16.00–16.90) ^{Bbc}	18.15 (17.40–20.60) ^{Aa}	
	DW	0.88 (0.40–1.82) ^{Ab}	2.59 (2.01–8.12) ^{Aa}	4.02 (2.86–5.94) ^{Aa}	15.75 (15.10–15.90) ^{Bc}	16.30 (16.00–16.90) ^{Bbc}	16.30 (16.00–16.90) ^{Bbc}	17.90 (17.80–19.10) ^{Aab}	15.75 (15.10–15.90) ^{Bc}	16.30 (16.00–16.90) ^{Bbc}	16.30 (16.00–16.90) ^{Bbc}	18.15 (17.40–20.60) ^{Aa}	

ΔE , color alteration; DW, distilled water; L, luminosity; Max, maximum; Min, minimum; MTA, mineral trioxide aggregate; T0, final setting time; T07, 7 days; T15, 15 days; T30, 30 days. Kruskal-Wallis test, *P* < .05; different capital letters in columns indicate statistically significant differences between MTA prepared using different vehicles in the same time interval and in the same immersion substance. Friedman test, *P* < .05; different lowercase letters in rows indicate statistically significant intragroup differences between the periods analyzed.

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