Physical and Chemical Properties and Subcutaneous Implantation of Mineral Trioxide Aggregate Mixed with Propylene Glycol

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Abstract

Introduction: The aim of this study was to evaluate the physical, chemical, and biological properties of mineral trioxide aggregate (MTA) mixed with 80% distilled water and 20% propylene glycol (PG) compared with MTA mixed with distilled water only. **Methods:** Flowability, film thickness, and solubility were analyzed according to American National Standards Institute/American Dental Association specification 57/2000. Initial and final setting times were assessed according to American Society for Testing and Materials specification C266/08. Porosity was assessed by using mercury intrusion porosimetry after 1 and 28 days of hydration, and the pH and calcium ion release were assessed after 3, 24, 72, and 168 hours. For the tissue reaction, the cements were implanted in 24 albino rats (2 groups, n = 12). An analysis of the inflammatory infiltrate was performed after 15, 30, and 60 days. **Results:** MTA + PG exhibited lower film thickness and higher final setting time. No differences were verified for flowability ($P > .05$). MTA + PG showed high porosity at 1 day of hydration ($P < .05$). All the test cements demonstrated an alkaline pH. Microscopic analysis of the specimens revealed neoformation of connective tissue in contact with the cements. **Conclusions:** The introduction of PG as a mixing vehicle alters the physical and chemical properties of MTA and is biologically acceptable. *(J Endod 2016;42:474–479)*

Key Words

Biocompatibility, mineral trioxide aggregate, physical properties, propylene glycol

Mineral trioxide aggregate (MTA) is applied directly in contact with the pulpal and periodontal tissues. The direct contact of this cement with the tissues results in stimulation of repair and induction of mineralization with no toxicologic effects (1–5). Despite these satisfactory properties, the consistency of MTA has been questioned in the literature (6–8). The manufacturers recommend mixing the powder with distilled water (DW), which results in a sandy, dry material that is difficult to place at the surgical site (7). To improve the handling characteristics of MTA, some authors have evaluated different vehicles, one of which is propylene glycol (PG) (7, 9, 10).

PG (1,2-propanediol) is a water-miscible organic solvent frequently used as a vehicle for calcium hydroxide in dentistry and as a solvent in cosmetic and pharmaceutical industries (6, 7, 11). This substance is also added in topical medicaments to enhance the permeability of tissue barriers (12). In dentistry, PG increases the permeability of calcium hydroxide into the dentinal tubules (13). The water-miscible and viscosity characteristics of PG encouraged its use in reducing MTA grittiness (6–8, 14). Studies have shown that the addition of PG to MTA + DW results in similar periapical tissue response (6) and improvement of sealing ability and adhesion (8, 14). PG is best added to DW with which MTA is mixed. Proportions of 100% PG did not allow MTA setting (7); however, a ratio of 20% PG to 80% DW significantly improved the flowability and adhesion, increased the setting time, and did not interfere with either the pH or calcium ion release (7, 8).

The effect of the addition of 20% PG to 80% DW for MTA mixing requires further investigation before clinical application. The aim of the present study was to evaluate the physicochemical properties of flowability, setting time, film thickness, solubility, porosity, pH, and calcium ion release and the biological tissue response. The null hypothesis was that MTA + PG present high flowability, high setting time, and low film thickness and do not interfere with solubility, porosity, pH, calcium ion release, and biological response.

Materials and Methods

The materials used in the study were MTA Angelus (Londrina, Paraná, Brazil) mixed with 80% DW and 20% PG and MTA mixed with DW with a water/powder ratio of 0.3 as recommended by the manufacturer. The water/powder ratio was kept standard even for the group with the PG addition.

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Flowability

According to American National Standards Institute/American Dental Association (ANSI/ADA) specification 57, a volume of 0.5 mL cement was placed on a glass plate (15). Three minutes after the start of mixing, another plate with a mass of 120 ± 2 g was applied centrally over the cement. Ten minutes after the start of mixing, the load was removed, and the average major and minor diameters of the compressed materials were measured in millimeters by using a digital caliper (Mitutoyo MTI Corporation, Tokyo, Japan). The test was repeated 3 times for each group.

Film Thickness

A total of 0.5 mL test material was placed in the center of a 5-mm-thick glass plate, and a second plate was positioned centrally over the cement according to ANSI/ADA specification 57 (15). Three minutes after the start of mixing, a load of 150 N was applied vertically over the glass plates. Ten minutes after the start of mixing, the thickness of the 2 glass plates and the interposed material was measured in millimeters by using a digital caliper (Mitutoyo MTI Corporation). The film thickness was measured by subtracting the measurements of the plate thickness after material placement from the combined plate thickness without the material. The test was repeated 3 times for each group.

Setting Time

The setting time was determined according to American Society for Testing and Materials specification C266-08 (16), with modifications based on ISO specification 6876 (2012) (17). The cements were mixed and placed into stainless steel rings (10 mm in internal diameter and 2 mm in height). Three stainless steel rings were filled with each material and stored in an incubator at 37°C and 95% ± 5% relative humidity. A 113.4-g Gilmore needle was used to determine the initial setting time. The final setting time was determined with a 453.6-g Gilmore needle. This procedure was repeated at 60-second intervals, and the time was measured by using a digital chronometer. The setting times were measured from the start of mixing to the time at which no indentations could be seen on the surface of the specimen.

Solubility

Three cylindrical polytetrafluoroethylene molds (20 mm in internal diameter and 1.5 mm in thickness) were filled with freshly mixed cement paste. A nylon thread was placed inside the material. The assembly was placed in an incubator (37°C, relative humidity of 95%) for a period corresponding to 3 times that of the setting time. The cements were removed from the mold and weighed 3 times each with an accuracy of 0.0001 g (UMark 210; Bel Engineering, Monza, Italy). The specimens were suspended by the nylon thread and placed inside a plastic vessel containing 50 mL deionized DW. The containers were stored for 24 hours in an incubator. Then the specimens were rinsed with deionized water, blotted dry with absorbent paper, placed in desiccators for 24 hours, and then reweighed. The experiment was repeated 3 times for each material. The solubility was considered as the weight loss of each specimen (initial mass minus final mass) expressed in percentage (17).

Porosity

The porosity of the cements after 1-day and 28-day hydration was assessed by using mercury intrusion porosimetry. Three cube specimens (7 × 7 × 7 mm) were prepared for each material, allowed to set for 24 hours, and subsequently immersed in Hank's balanced salt solution (H6648; Sigma Aldrich, St Louis, MO) for 1 or 28 days. The materials were taken out of the solution and dried for 4 days in an incubator at 60°C. The porosity was measured in a 2-stage process by using mercury intrusion porosimeter (PoreMaster; Quantachrome Instruments, New York, NY). Calibrated mercury displacement pycnometry was performed, followed by low-pressure and high-pressure mercury intrusion porosimetry. The volume of mercury that intruded the pores of the specimen was measured. Porosimetry data were processed by using PoreMaster.

A gas pycnometer (Quantachrome Instruments) with helium was used to determine the average absolute density (g/cm^3). The pycnometer operates on the Archimedes principle of gas displacement to determine the volume. Density of the specimen can be calculated from the specimen weight and volume.

pH and Calcium Ion Release

Eighty artificial acrylic teeth (n = 10) each with a cavity of 3-mm depth and 1.4-mm diameter were filled with the cements and immersed individually in 10 mL deionized water. After 3, 24, 72, and 168 hours, the teeth were placed in new flasks containing equal volume of new deionized water. The pH of the water in which the teeth had been kept was measured with a pH meter (model 371; Micronal, São Paulo, SP, Brazil) previously calibrated by using buffer solutions of pH 4, 7, and 14. Room temperature was maintained at 25°C during the assessment.

The water used for pH assessment was the same as the water used to measure the calcium ion release. For determination of calcium ion release, an atomic absorption spectrophotometer (AA6800; Schimadzu, Tokyo, Japan) equipped with a calcium-specific hollow cathode lamp was used at the following operating conditions: lamp current, 3 mA; fuel, acetylene; support, oxygen; stoichiometry, reducing; wavelength, 422.7 nm; and slit, 0.2 nm.

Standard solutions containing 10, 20, 40, and 80 mg L^{-1} calcium were prepared. The readings of the calcium ion release were compared with a standard curve obtained from readings of the standard solutions. This reading was performed in the same periods used for the pH level measurement.

Subcutaneous Implantation

Twenty-four adult male albino rats (*Rattus norvegicus*), weighing approximately 300 g, were selected (Ethical approval CEP 006-2009). The mixed cements were inserted in sterile polyethylene tubes (10 mm in length and 1 mm in internal diameter), and an immediate subcutaneous implantation was performed in the dorsal region of the rats. The animals were divided into 2 groups (n = 12) according to the cement type. Each animal received 1 implant. For the surgical procedures, the rats were anesthetized with a combination of ketamine and xylazine (Vet Brands Int, Miramar, Florida) (0.05 mL/100 g).

TABLE 1. Mean and Standard Deviation of Flowability, Film Thickness, Solubility, Initial/Final Setting Time, and Porosity at 1 and 28 Days

	Flowability (mm)	Film thickness (μm)	Initial setting time (min)	Final setting time (min)	Solubility (%)	Porosity 1 day (%)	Porosity 28 days (%)
MTA + DW	$13.60^a \pm 0.13$	$101.0^a \pm 6.24$	$13.60^a \pm 1.30$	68.33 ± 1.53	$1.82^a \pm 0.11$	24.60^a	22.56^a
MTA + PG	$17.31^a \pm 1.44$	$68.67^b \pm 13.87$	$17.31^a \pm 1.40$	$103.00^b \pm 3.35$	$-0.25^b \pm 0.25$	44.34^b	26.08^a

Different superscript lowercase letter in each column indicates statistical differences between groups.

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