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Effects of smear layer removal agents on the physical properties and microstructure of mineral trioxide aggregate cement

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ABSTRACT

Objective: To compare the effect of QMix (Dentsply Sirona), 7% maleic acid (MA), and 17% ethylenediaminetetraacetic acid (EDTA) on the microhardness, flexural strength and microstructure of mineral trioxide aggregate (MTA; ProRoot MTA, Dentsply Sirona).

Methods: Forty MTA specimens were divided into four groups: [I] QMix [II] 7% MA [III] 17% EDTA and [IV] distilled water (control). After treatment with 5 mL of the respective solution for 1 min, the specimens were tested for microhardness using a Knoop hardness tester. Forty additional specimens were similarly treated and evaluated for the flexural strength using a universal testing machine. For microstructure evaluation, MTA specimens were treated in a similar manner and examined by X-ray diffractometry and scanning electron microscopy (SEM).

Results: For microhardness, there were no differences between distilled water, QMix and EDTA groups. However, MTA exposed to distilled water had higher microhardness than MA. When compared with QMix and EDTA, MA had lower microhardness; there was no difference between EDTA and QMix. For flexural strength, distilled water group had higher flexural strength than the other agents. There were no differences between EDTA *vs* MA and EDTA *vs* QMix. Specimens treated with QMix had higher flexural strength than MA. X-ray diffraction indicated that EDTA inhibited hydration of MTA. For SEM, all the tested agents altered the microstructure of MTA when compared to distilled water.

Conclusion: MA had more detrimental effect on the physical properties of MTA and EDTA was more detrimental to the hydration of MTA.

Clinical significance: The present study highlights the effect of newer chelating agents on the physical properties and microstructure of MTA. Preventing the deterioration of MTA is important for its long term success in endodontic procedures.

1. Introduction

Mineral trioxide aggregate (MTA) is a biocompatible tricalcium silicate hydraulic cement with numerous applications in endodontics [1,2]. Its clinical applications include repair of resorption, pulp capping, perforation repair, root end filling, apexification, regenerative endodontics and as a root canal sealer [1,2].When mixed with an aqueous solution, ProRoot (white) MTA (Dentsply Sirona, Tulsa Division, Tulsa, OK, USA) sets in approximately 3–4 h to form a hard cement composing mainly of calcium oxide, silicon oxide and bismuth oxide [3]. It has been reported that MTA must be allowed to set in the presence of moisture to optimise the material's physical and chemical properties [4,5]. Factors such as particle size, powder-to-liquid ratio, environmental temperature and presence of air in the mixture may all affect the physical properties of MTA [4,6]. Torabinejad et al. reported that MTA remains soft when it is used in the vicinity of an acidic environment, such as that present in severe inflammation [1]. Nilforoushan et al. reported that the setting behaviour of MTA is adversely affected by the presence of alkaline-earth metal chlorides, including sodium chloride [7]. Lee et al. reported that hydration of MTA is adversely affected by an acidic environment, which results in weakening of the material's microstructure [5].

Ideally, MTA should possess adequate strength especially when used as a furcation perforation repair material, where it will be subjected to forces by occlusal loading. The strength of a brittle material such as MTA may be evaluated by measuring its compressive or flexural

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strength. Flexural strength testing provides a collective measure of both compressive stress on the upper surface and tensile stress on the lower surface of a specimen. It closely simulates the clinical situation because fatigue failure via continuous flexion is the principal contributor to the failure of tooth restorations [8,9]. Microhardness testing of MTA exposed to various root canal irrigants may be used as an indicator of its setting process and its overall strength or resistance to deformation under various setting conditions [10].

The goal of root canal treatment is to completely disinfect the root canal system [11]. This may be achieved by mechanical instrumentation, irrigation with disinfecting solutions and the use of inter-appointment intracanal medicaments. Mechanical instrumentation of the root canal produces a smear layer which covers the dentinal tubules in the canal wall [12]. Removal of the smear layer in bacteria-infected root canals enables more efficient penetration of intracanal medications and irrigants into the dentinal tubules [13,14]. Various agents have been used for the removal of smear layer. Ethylenediaminetetraacetic acid (EDTA) is a commonly used chelating agent for removing canal wall smear layers [15,16]. When EDTA is used as a final irrigant after the placement of MTA, the chelating agent may adversely affect the physical and chemical properties of the set tricalcium silicate cement. 17% EDTA interferes with the hydration of MTA, resulting in a set cement with decreased microhardness, flexural strength and reduced biocompatibility [17,18].

QMix® 2in1 (QMix; Dentsply Sirona, Tulsa Division) is a root canal irrigant which contains EDTA, chlorhexidine, a detergent and water. It has been reported that QMix removes canal wall smear layers as effectively as 17% EDTA [19]. Seven percent maleic acid (MA) is a root canal irrigant that possesses better smear layer removal capability than 17% EDTA or QMix [20,21]. It is also less cytotoxic compared to17% EDTA [22]. To date, no information is available on the effect of QMix or 7% MA on the microstructure and physical properties of MTA. Accordingly, the objectives of the present *in-vitro* study were to compare the effect of QMix, 7% MA and 17% EDTA on the microhardness, flexural strength and microcrystalline structure of MTA cement. The null hypotheses tested were: (1) there are no differences in the potential of QMix, 7% MA and 17% EDTA in reducing the microhardness and flexural strength of set MTA cement; and (2) there is no difference in the microstructure of set MTA cement after it is treated with QMix, 7% MA or 17% EDTA.

2. Materials and methods

2.1. Microhardness

Forty ProRoot MTA cylindrical shaped specimens (6 mm high, 4 mm diameter) were prepared using a split mould. The MTA cement was mixed according to the manufacturer's instructions and packed into the mould using MTA carrier (Dentsply Sirona, Tulsa Division). The specimens were stored in a 100% relative humidity chamber at room temperature for 24 h. After the specimens were completely set, they were separated from the mould and were grounded flat using a polishing machine with a series of ascending grades of silicon carbide abrasive papers (500, 800, 1000, 1200 grit) under distilled water. Final polishing was achieved with 0.1 μm alumina suspension (Ultra-Sol R, Eminess Technologies. Scottsdale, Arizona, USA) on a rotary felt disk.

The polished specimens were randomly divided into four groups (n = 10) based on the irrigant used: [I] QMix:5 mL of QMix (pH 8.0) for 1 min; [II] MA: 5 mL of 7% MA solution (pH 1.3; MilliporeSigma, St. Louis, MO, USA) for 1 min; [III] EDTA: 5 mL of 17% EDTA solution (pH 8.5; Vista Dental Products, Racine, WI, USA) for 1 min; and [IV] control: 5 mL of distilled water for 1 min. For all groups, the specimens were immersed in a beaker containing the respective irrigant. A magnetic stirrer was placed inside the beaker to ensure complete wetting of the specimens with the irrigating solutions. After 1 min of treatment, all the specimens were rinsed with distilled water and air-dried.

Microhardness was determined using a Knoop hardness tester (Matsuzawa Seiki Co. Ltd, Tokyo, Japan). Indentations were made with a Knoop diamond indenter on each specimen using 100 g force and 15 s dwell time. The diamond-shaped indentations were examined with an optical microscope equipped with an image analysis software, to enable accurate measurement of their diagonals. The average length of the two diagonals recorded for each indentation was used to calculate the microhardness value. Three measurements were made on the surface of each specimen, and the mean value was taken as the hardness of that particular specimen.

2.2. Flexural strength

Forty ProRoot MTA beams (25 mm long and 2 mm thick) were prepared using a split mould. The MTA was mixed and placed in the mould in the manner previously described. The set specimens were randomly divided into four groups(n = 10) and treated with the test irrigants in the manner described for microhardness testing. All the specimens were subjected to three-point bending to analyze the flexural strength. The testing procedure consisted of placing the MTA beams on two cylindrical rods mounted parallel to each other at a 20 mm distance. The MTA beams were then loaded at the centre with the indenter. Testing was performed using a universal testing machine (Model 3366, Instron Corp., Norwood, MA, USA). Flexural strength was determined using the equation $S = 3PL/2bh2$, where P = maximum load (N), L = support span (mm), b = width of the specimen (mm), and h = height of the specimen (mm). Data was expressed in megaPascals (MPa).

2.3. Microstructure

2.3.1. X-ray diffraction

The crystalline phases of MTA after treatment with the test irrigants was determined by X-ray diffraction (XRD). Twelve rectangular-shaped ProRoot MTA specimens (5 mm long, 3 mm wide, 2 mm thick) were prepared and randomly divided into four groups (n = 3). The specimens were prepared and treated with the test solutions in the same manner described for microhardness testing. After treatment with the respective solution, the specimens were examined with an X-ray diffractometer (JDX-8P-XRD, JEOL, Tokyo, Japan) using Ni filter and Cu Kα radiation; the latter was generated at 30 kV and 20 mA. The specimens were scanned from 10° to 60° 2θ and the data was collected in a continuous scan mode at a scanning rate of 4°/min. Crystalline changes was identified by a computer-automated system and compared with Joint Committee on Powder Diffraction Standards (JCPDS) files.

2.3.2. Scanning electron microscopy

Twelve cylindrical-shaped ProRoot MTA specimens (6 mm high, 4 mm diameter) were prepared using a split mould. The specimens were randomly divided into four groups (n = 3) and treated with the test irrigants in the same manner described for microhardness testing. The specimens were rinsed and dehydrated subsequently using ascending grades of ethanol (25%, 50%, 75% and 100%) for 15 min each, mounted on metal stubs, coated with gold using an ion-sputtering machine and examined with a scanning electron microscope (SEM; JSM-6010, JEOL). Images were taken to identify the surface characteristics of the MTA specimens at 3500× magnification and 10 kV.

2.4. Statistical analysis

Because the microhardness and flexural strength data appeared to have violated the normality and equal variance assumptions, the data were analyzed separately using Kruskal-Wallis analysis of variance and Dunn's multiple comparison procedures. For all analyses, the significance level was pre-set at $\alpha = 0.05$.