Effect of Acidic Environment on the Push-out Bond Strength of Mineral Trioxide Aggregate

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Abstract

Introduction: Reduced surface microhardness and decreased sealing ability have been shown after the placement of mineral trioxide aggregate (MTA) in an acidic environment. In this study, the effect of an acidic environment on the push-out strength of MTA was evaluated. **Methods:** Eighty root dentin slices from freshly extracted single-rooted human teeth were sectioned and their lumen instrumented to achieve a diameter of 1.3 mm. One gram of tooth-colored ProRoot MTA (Dentsply Tulsa Dental, Johnson City, TN) was mixed with 0.33 g of distilled water and introduced into the canals of the root-dentin slices and treated with ultrasonic energy. The specimens were then randomly divided into four groups ($n = 20$) and wrapped in pieces of gauze soaked in phosphate buffer saline solution (pH = 7.4) and butyric acid buffered at pH values of 4.4, 5.4, or 6.4, respectively. They were then incubated for 4 days at 37°C. The push-out bond strengths were then measured using a universal testing machine. The slices were examined under a light microscope at ×40 magnification to determine the nature of the bond failure. The data were analyzed using one-way analysis of variance and the Tamhane post hoc test. **Results:** The greatest mean push-out bond strength (7.28 ± 2.28 MPa) was observed after exposure to a pH value of 7.4. The values decreased to 2.47 ± 0.61 MPa after exposure to a pH value of 4.4. There were significant differences between the groups ($p < 0.001$). Inspection of the samples revealed the bond failure to be predominantly adhesive. **Conclusion:** The force needed for displacement of MTA was significantly lower in samples stored at lower pH values. *(J Endod 2010;36:871–874)*

Key Words

Acid, mineral trioxide aggregate, MTA, pH, push-out bond strength

An ideal root-end filling material should be biocompatible, dimensionally stable, adhere to the root-end cavity walls, resist dislocating forces, prevent the passage of the bacteria, and be unaffected by the presence of tissue fluid that may, in an infected area, be acidic (1). Mineral trioxide aggregate (MTA) has most of these essential properties and is recommended as a root-end filling material (2). It is also recommended for use as an apical barrier in the treatment of immature teeth with necrotic pulps (2). Variations in the pH of host tissues as a result of preexisting disease (1) may affect the physical and chemical properties of the material. Reduced hardness (3, 4) in addition to decreased sealing ability (5) has been shown after placement of MTA in an acidic environment. However, the effect of an acidic environment on the bond strength of dentin to MTA has not been studied. The purpose of this study was to evaluate the push-out bond strength between MTA and intraradicular dentin after exposure to a range of acidic pH levels.

Materials and Methods

Freshly extracted human teeth including mandibular single-rooted premolars or maxillary anterior incisors that were either intact or contained only small carious lesions were selected and stored in 0.5% chloramine-T at 4°C for up to 1 month before use. Midroot dentin was sectioned horizontally into slices with a thickness of 1.0 mm. A diamond saw microtome (SP1600 microtome; Leica, Nußloch, Germany) was used to obtain 80 root dentin slices. The lumen of the root dentin disks were instrumented with Gates Glidden burs (Dentsply Maillefer, Ballaigues, Switzerland), sizes 2 to 5, to achieve a standardized diameter of 1.3 mm. A 0.33-g aliquot of distilled water was added to 1.00 g of tooth-colored ProRoot MTA (Dentsply Tulsa Dental, Johnson City, TN). The water was absorbed by the MTA powder before gentle mixing. The mixed material was then introduced incrementally with no pressure into the lumens of the root-dentin slices. Saline-moistened Gelatamp (Roeko-Coltène/Whaledent, Langenau, Germany) was used as a matrix to prevent extrusion of the material below the inferior surface of the specimens. The MTA in each sample was then treated with ultrasonic energy for 30 seconds at scale 5 using a CPR-2D tip (Obtura Spartan, Fenton, MO) attached to a Suprasson P5 ultrasonic booster (Satelec, Merignac, France) without contact with the dentin walls or Gelatamp. The specimens were then divided randomly into four groups ($n = 20$). In group A, the specimens were wrapped in pieces of gauze soaked in phosphate buffer saline solution (pH = 7.4). In groups B, C, and D, specimens were wrapped in pieces of gauze soaked in butyric acid buffered at pH values of 6.4, 5.4, or 4.4, respectively, and then incubated for 4 days at 37°C.

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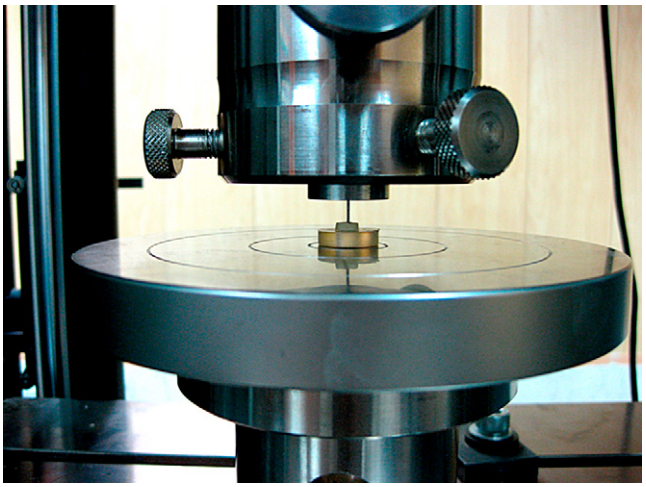

Figure 1. A cylindrical stainless steel plunger attached to the load cell of the universal testing machine loading on MTA inside a root section.

The push-out bond strengths were measured using a universal testing machine (Z050; Zwick/Roell Group, Ulm, Germany). The samples were placed on a metal slab with a central hole to allow the free motion of the plunger. The compressive load was applied by exerting a downward pressure on the surface of the MTA using a 1.00-mm diameter cylindrical stainless steel plunger at a speed of 1 mm/min (Fig. 1). The plunger had a clearance of approximately 0.2 mm from the margin of the dentinal wall to insure contact with MTA only. The maximum load applied to MTA at the time of dislodgement was recorded in newtons. In order to express the bond strength in MPa, the recorded value was divided by the adhesion area of root canal filling calculated by the following formula: $2\phi r \times h$, where r is the root canal radius and h is the thickness of the root-dentin slice in millimeters. The slices were then examined under a light microscope at $\times 40$ magnification to determine the nature of the bond failure. Each sample was categorized into one of three failure modes (Fig. 2): adhesive failure at the MTA and dentin interface, cohesive failure within MTA, or mixed failure. The data were analyzed using one-way analysis of variance followed by the Tamhane post hoc test.

Results

The results are summarized in Figure 3. The greatest mean push-out bond strength (7.28 ± 2.28 MPa) was observed after exposure to a pH level of 7.4. The values decreased to 2.47 ± 0.61 MPa after exposure to a pH level of 4.4. There were significant differences between the groups ($p < 0.001$). The Tamhane post hoc test revealed that the mean bond strength values of specimens exposed to pH 4.4 and 5.4 were significantly different from the others ($p < 0.001$). No significant difference was found between the values of specimens exposed to pH levels of 6.4 and 7.4. Inspection of the samples revealed the bond failure to be predominantly adhesive for all groups.

Discussion

It has been suggested that two exceptional properties of MTA, biocompatibility and sealing ability, originate from the physicochemical reactions between MTA and dentin (6). Investigation of the bond strength between MTA and dentin will reveal the value of adhesion between them. There are various methods for evaluating the adhesion of a dental material to dentin including tensile, shear, and push-out strength tests. Loxley et al (7) evaluated the effect of various intracanal

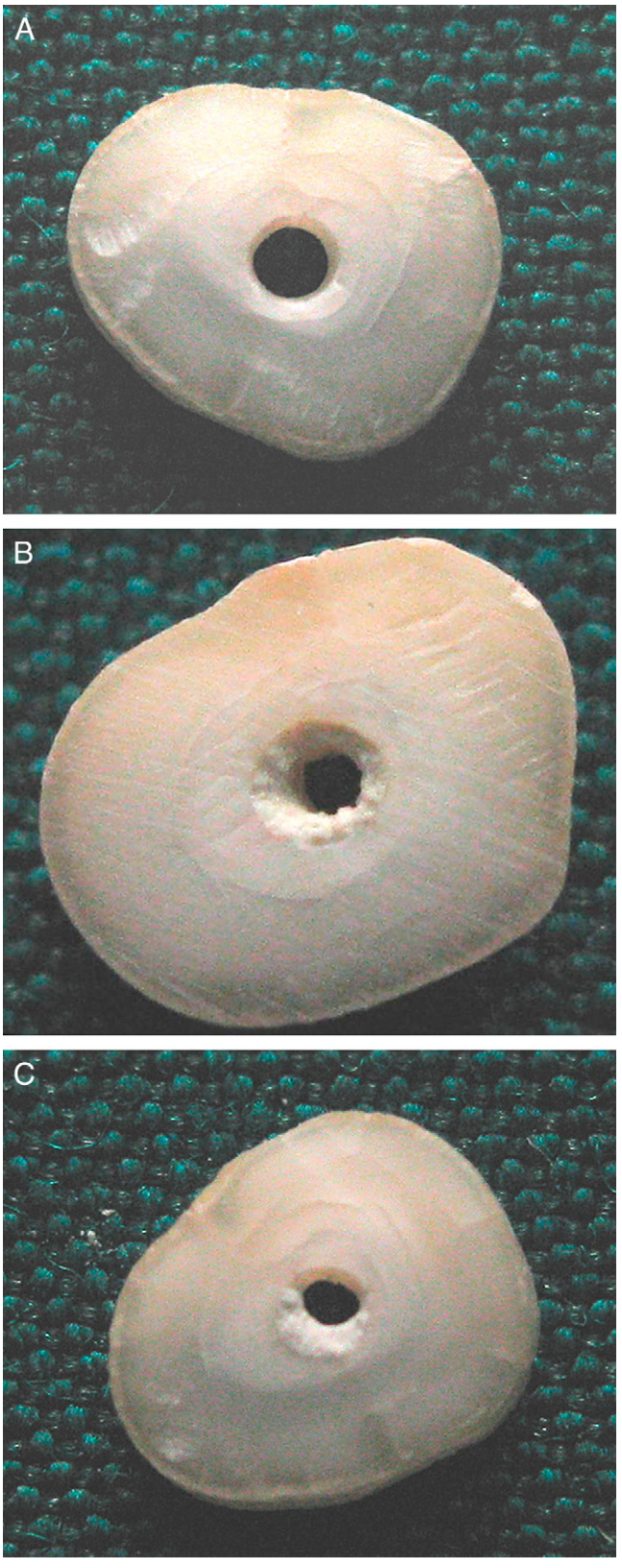

Figure 2. Various failure modes. (*A*) Adhesive failure; note the clean canal wall. (*B*) Cohesive failure within MTA. (*C*) Mixed failure; note the MTA residual inside the canal.

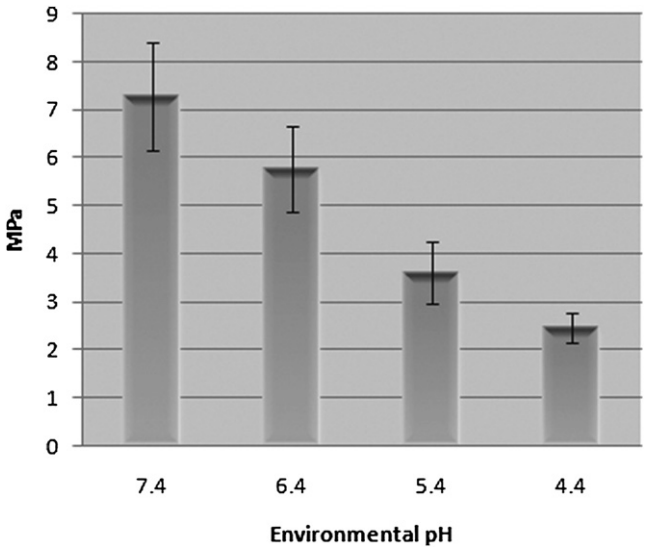

Figure 3. The effect of pH on the bond strength between MTA and dentin .

oxidizing agents on the push-out strengths of MTA, Super EBA (Harry J. Bosworth Co., Skokie, IL), and IRM (Caulk-Dentsply, Milford, DE). MTA was significantly less resistant to displacement than Super EBA or IRM. In our study, the push-out test method was used to test the bond strength between MTA and dentin while exposed to butyric acid solutions with several pH values.

In the presence of tissue fluid, hydration of MTA powder results in the development of hydroxyapatite crystals and formation of a hybrid layer between dentin and MTA (6). This reaction can be simulated by mixing MTA powder with disodium hydrogen phosphate, a phosphate-containing solution (8). The composition and morphology of the hydroxyapatite crystals is related to various factors including the environmental pH (9). The ideal pH for this reaction is 7.00 (6). The ensuing hydroxyapatite crystals cover MTA, fill the microscopic gap between MTA and dentin, and create a chemical bonding, and, subsequently, because of the precipitation of calcium phosphate, the environmental pH rises to 11.00 (6). Torabinejad et al (10) reported the pH value of MTA itself to be between 10.5 and 12.9. On hydration, MTA can release calcium hydroxide (11). The formation of calcium hydroxide and the precipitation of calcium phosphate can explain the ability of MTA to maintain the pH of the surrounding environment at a high level (11, 12). It may also explain some of its biological properties such as the ability to increase osteoblast activity and the induction of hard-tissue formation (13).

In some clinical situations, MTA might be exposed to an inflamed environment with a low pH level (1). The application of MTA in a low pH situation may influence its physical and chemical properties (3). Namazikhah et al (4) showed that the lowest and greatest surface hardness values of MTA were found after exposure to pH levels of 4.4 and 7.4, respectively. Scanning electron microscopy evidence also suggests the development of a porous surface and lack of needle-like crystals in more acidic solution. Furthermore, Saghiri et al (5) reported that the time needed for leakage to occur was significantly shorter in samples stored at lower pH values. In these studies (4, 5), specimens were exposed to butyric acid with pH values of 4.4, 5.4, 6.4, and 7.4. Lee et al (3) also revealed that an acidic environment of pH 5 adversely affected both the physical properties and the hydration behavior of MTA. Watts et al (14) also reported that compressive strength of both white and gray MTA significantly decreased when mixed with local anesthetic solution and exposed to an environment of pH of 5.0. However, there was no significant difference in compressive strength of both white and gray MTA when mixed with water and exposed to a pH of 5.0 or 7.4. They suggested the use of sterile water as the mixing liquid rather than local anesthetic solution. In addition, various types of acid may have different effects on the physical and chemical properties of MTA. The type of acid was not stated by Lee et al (3) or Watts et al (14), and this lack of information may be one of the reasons for the different findings. In our study, to simulate the clinical conditions associated with periradicular infections (4), butyric acid was selected because it has been reported to be one of the byproducts of anaerobic bacterial metabolism (15). The results showed that the mean push-out bond strength of MTA to intraradicular dentin decreased significantly after exposure to pH levels of 4.4 and 5.4 compared with pH levels of 6.4 and 7.4. The lowest and greatest bond strength values were at pH levels of 4.4 and 7.4, respectively. These results could be caused by the alterations in the physical and chemical properties of MTA in such a low pH environment (3, 4). Moreover, the formation of hydroxyapatite crystals and subsequently the formation of a hybrid layer at the MTA-dentin interfacial gap are likely to be disturbed in an acidic environment. In the present study, the bond failures observed in all experimental groups were predominantly at the MTA-dentin gap (adhesive type). This result is in accordance with Vanderweele et al (16) who reported that MTA-dentin bond failures were usually adhesive. The adhesive mode of failure may have occurred as a result of the short storage time before evaluation of the bond strength, which was 4 days in the present study and 7 days in the study by Vanderweele et al (16). Hachmeister et al (17) suggested that the formation of chemical bonding leads to enhanced attachment of dentin to MTA over time. Sarkar et al (6) showed that teeth filled with MTA and stored in synthetic tissue fluid for 2 months produced an adherent interfacial layer at the dentin wall that resembled hydroxyapatite in composition. They also reported that in the presence of humidity the tensile strength of the bond between dentin and MTA substantially increased at 3 days with a further moderate increase at 21 days (6). Further long-term studies are suggested to evaluate the effect of aging on the MTA-dentin bond strength.

Hachmeister et al (17) evaluated the retention characteristics of MTA when placed as an apical barrier with and without prior use of nonsetting calcium hydroxide. They revealed that by increasing the thickness of the MTA plug and thus increasing the contact area between MTA and dentin, resistant to dislodgement regardless of the use of calcium hydroxide was increased significantly. Therefore, according to the findings of the present study and Hachmeister et al (17) when exposure to an acidic environment is unavoidable, an application of a thicker layer of MTA may be beneficial. In addition, before the placement of MTA in an infected and/or inflamed low pH environment, the application of nonsetting calcium hydroxide to neutralize the pH is suggested. In conclusion, under the conditions of this study, the force needed for the displacement of MTA from root dentin to occur was significantly lower in samples stored at lower pH values.

Acknowledgments

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