

# Effect of Acidic Environment on Dislocation Resistance of Endosequence Root Repair Material and Mineral Trioxide Aggregate

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## Abstract

**Objective:** The aim of this study was to compare the effect of an acidic environment on dislocation resistance (push-out bond strength) of EndoSequence Root Repair Material (ERRM putty and ERRM paste), a new bioceramic-based material, to that of mineral trioxide aggregate (MTA).

**Materials and Methods:** One-hundred twenty root dentin slices with standardized canal spaces were divided into 6 groups (n = 20 each) and filled with tooth-colored ProRoot MTA (groups 1 and 2), ERRM putty (groups 3 and 4), or ERRM paste (groups 5 and 6). The specimens of groups 1, 3, and 5 were exposed to phosphate buffered saline (PBS) solution (pH=7.4) and those of groups 2, 4, and 6 were exposed to butyric acid (pH= 4.4). The specimens were then incubated for 4 days at 37°C. The push-out bond strength was then measured using a universal testing machine. Failure modes after the push-out test were examined under a light microscope at ×40 magnification. The data for dislocation resistance were analyzed using the *t*-test and one-way analysis of variance.

**Results:** In PBS environment (pH=7.4), there were no significant differences among materials (P=0.30); but the mean push-out bond strength of ERRM putty was significantly higher than that of other materials in an acidic environment (P<0.001). Push-out bond strength of MTA and ERRM paste decreased after exposure to an acidic environment; whereas ERRM putty was not affected by acidic pH. The bond failure mode was predominantly cohesive for all groups except for MTA in an acidic environment; which showed mixed bond failure in most of the specimens.

**Conclusion:** The force needed for dislocation of MTA and ERRM paste was significantly lower in samples stored in acidic pH; however, push-out bond strength of ERRM putty was not influenced by acidity.

**Key Words:** Acid; Endosequence Root Repair Material; Mineral Trioxide Aggregate; pH, Dislocation

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## INTRODUCTION

An ideal endodontic material should be bio-compatible, radiopaque, antibacterial, dimen-

sionally stable and easy to manipulate. It should be able to adhere to the root-end cavity walls and remain in place under dislocating

forces (i.e. mechanical stresses caused by tooth function or operative procedures) [1-3]. Mineral trioxide aggregate (MTA) demonstrates many of the ideal properties [4]. Although MTA as a root-end filling material and root repair material has gained widespread use since its introduction to the endodontic market, it has some drawbacks such as poor handling properties [5] and difficulty to obtain a consistent mixture when used according to the manufacturer [6].

In an attempt to modify the properties of MTA and overcome its shortcomings, EndoSequence Root Repair Material (ERRM) (Brasseler USA, Savannah, GA, USA) has been recently introduced. According to the manufacturer, ERRM is a bioceramic material composed of calcium silicates, zirconium oxide, tantalum oxide, monobasic calcium phosphate and fillers. It is manufactured as a premixed product in both moldable putty and preloaded syringe paste to provide the clinician with a consistent material. The bioceramic material is produced with nanosphere particles that allow the material to enter into the dentinal tubules and interact with the moisture present in the dentin [7]; this creates a mechanical bond upon setting. Previous studies showed that ERRM was biocompatible [8, 9], able to seal root-end cavities [10], and bioactive [11].

In some clinical situations, endodontic materials may be exposed to an inflamed environment with a low pH level [12]. It has been reported that physical and chemical properties of MTA are affected in an acidic environment [13]. Furthermore, reduced tensile strength [14], push-out bond strength to dentin [15, 16], surface hardness [17] as well as decreased sealing ability [18] have been seen after exposure of MTA to an acidic environment.

To the best of our knowledge, the effect of acidity on dislocation resistance of ERRM has not been investigated. The aim of this study was to compare dislocation resistance of ERRM (putty and paste) after exposure to phosphate buffered saline (PBS) (pH=7.4) or

butyric acid (pH = 4.4) with that of white ProRoot MTA (Dentsply Tulsa Dental, Johnson City, TN).

## MATERIALS AND METHODS

### *Tooth Selection and Preparation*

Sixty extracted single-rooted teeth were selected for this study. The crowns were removed, and the middle thirds of the roots were sectioned perpendicular to the long axis by using water-cooled diamond saw cutting machine (Mecatome, Presi, France) in order to obtain  $2.00 \pm 0.05$  mm thick root slices. The lumens of the root slices were enlarged with #2 to #5 Gates-Glidden burs (Dentsply, Maillefer, Ballaigues, Switzerland) to achieve a standardized diameter of 1.3 mm. The specimens were immersed in 17% EDTA followed by 5.25% NaOCl for 3 minutes and then washed with distilled water. The root sections were randomly divided into six groups (n=20 each) as follows:

**Groups 1 and 2:** One gram of white ProRoot MTA powder was mixed with 0.33 ml distilled water and placed in the canal space of root slices with minimal pressure

**Groups 3 and 4:** The canal spaces were filled with the premixed ERRM putty.

**Groups 5 and 6:** The canal spaces were filled with the premixed ERRM paste.

A PBS solution containing 1.7 g of  $\text{KH}_2\text{PO}_4$ , 11.8 g of  $\text{Na}_2\text{HPO}_4$ , 80.0 g of NaCl, and 2.0 g of KCl in 10 L of  $\text{H}_2\text{O}$  (pH=7.4) was prepared. In groups 1, 3 and 5, the specimens were wrapped in pieces of gauze soaked in PBS solution (pH=7.4) and in groups 2, 4 and 6, the specimens were wrapped in pieces of gauze soaked in butyric acid (pH=4.4).

### *Assessment of Dislocation Resistance*

Dislocation resistance of materials was measured using push-out strength test by a universal testing machine (Z050, Zwick/Roell, Ulm, Germany). The specimens were placed on a metal slab with a central hole to allow free motion of the plunger. The compressive load

was applied on the surface of the filling material with a 1-mm diameter cylindrical stainless steel plunger at a speed of 1 mm/min. The maximum load applied to the filling material before dislodgment was recorded in Newtons. In order to express the bond strength in MPa, the recorded value in Newtons was divided by the adhesion area of the root canal filling calculated by the following formula:  $2\pi r \times h$ , where  $\pi$  is the constant 3.14,  $r$  is the root canal radius and  $h$  is the thickness of the root dentin slice in millimeters.

The root slices were then examined under a light microscope at  $\times 40$  magnification to determine the mode of bond failure. Each sample was categorized into one of three failure modes: adhesive failure which occurred at the filling material and dentin interface, cohesive failure which happened within the filling material and mixed failure mode.

The data were analyzed using two-way analysis of variance.

The interaction between two factors of material and pH was significant ( $p=0.015$ ). Therefore, comparison of dislocation resistances was performed using  $t$ -test and one-way analysis of variance. Significance level was set at  $P<0.05$ .

## RESULTS

The mean dislocation resistance value  $\pm$  standard deviation of materials after exposure to PBS and acidic environment is presented in Table 1.

In PBS environment ( $pH=7.4$ ), there were no significant differences among materials ( $p=0.30$ ). However, the mean push-out bond strength of ERRM putty was significantly higher than that of MTA and ERRM paste in  $pH=4.4$  ( $P<0.001$ ); but the difference between MTA and ERRM paste was not statistically significant ( $P=1.00$ ). The results showed that dislocation resistance of MTA and ERRM paste decreased after exposure to an acidic environment; whereas ERRM putty was not affected by acidity. Inspection of the samples revealed the bond failure to be predominantly cohesive for all groups except for MTA in an acidic environment; which showed mixed failure mode in most of the specimens.

## DISCUSSION

Dislocation resistance of a root filling material is an important factor for the success of the endodontic treatment [19, 20]. Consequently, mechanical testing of bonded interfaces can provide important insights into material selection and outcome prediction [21, 22]. There are different methods for assessment of adhesion of a dental material to dentin including tensile, shear, and push-out strength tests.

The push-out test has been shown to be efficient and reliable to assess bond strength of materials [23]. After exposure to tissue fluid, hydration of MTA powder results in the development of hydroxyapatite crystals and formation of a hybrid layer between dentin and MTA [24].

**Table 1.** Dislocation resistance values (mean  $\pm$  SD)

Material	Storage Medium	Mean $\pm$ SD (MPa)
MTA	PBS ( $pH = 7.4$ )	11.45 $\pm$ 4.88 *
	Acid ( $pH = 4.4$ )	7.21 $\pm$ 3.78 **
ERRM putty	PBS ( $pH = 7.4$ )	12.33 $\pm$ 4.75 *
	Acid ( $pH = 4.4$ )	11.81 $\pm$ 4.04 *
ERRM paste	PBS ( $pH = 7.4$ )	9.97 $\pm$ 4.72 *
	Acid ( $pH = 4.4$ )	7.28 $\pm$ 3.13 **

The morphology and composition of the hydroxyapatite crystals relate to different factors such as environmental pH [25]. The ideal pH for this reaction is 7.00 [24].

In some clinical situations, endodontic biomaterials may be exposed to an inflamed environment with a low pH level [12]. The application of materials in a low pH environment may affect their physical and chemical properties [13]. In the present study, butyric acid was selected to simulate the clinical conditions associated with periradicular infections [16]. Various types of acid may have different effects on the physical and chemical properties of materials. It has been shown that butyric acid is one of the byproducts of anaerobic bacterial metabolism [26].

This study revealed that the mean push-out bond strength of MTA and ERRM paste decreased significantly after exposure to acidic pH. This result is in accordance with Shokouhinejad et al. [15] and Hashem and Wanees Amin [17] who reported a decrease in push-out bond strength of MTA after placement in an acidic environment. These results could be caused by the alterations in the physical and chemical properties of calcium-silicate based biomaterials in a low pH environment [13]. Furthermore, Namazikhah et al. [16] 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 solutions. In addition, Lee et al. [13] revealed that acidic environment (pH=5) adversely affected both the physical properties and the hydration behavior of MTA.

The results of this study showed that there was no significant difference among push-out bond strength of three materials after exposure to PBS. However, dislocation resistance of ERRM putty was significantly greater than that of ERRM paste and MTA in the presence of acidic pH. Furthermore, there was no sig-

nificant difference between the dislodgement resistance of ERRM putty after exposure to pH=7.4 and pH=4.4. This result may be attributed to the fillers and thickening agents added to ERRM putty to produce the putty form. According to the manufacturer, there is no difference in the chemical composition of ERRM putty and paste. Further studies are suggested to evaluate composition and other physicochemical properties of ERRM putty and ERRM paste. In the acidic environment, the higher bond strength of ERRM putty compared to MTA may be attributed to the delivery form of these materials, which is premixed for ERRM putty and separate powder and liquid for MTA. The thickening and filler agents included in the composition of ERRM putty may be associated with its higher bond strength. It has been reported that the presence of zirconium oxide improved certain physical properties of composite bioceramics [27]. The presence of zirconium oxide in the composition of ERRM may also result in higher bond strength of ERRM. However, zirconium oxide is also one of the components of ERRM paste; which showed lower dislocation resistance than ERRM Putty after exposure to acidic pH. Since these bioceramic materials have been recently developed, further studies are necessary to investigate their other physicochemical properties. In the present study, bond failures observed in all experimental groups were predominantly within the filling material (cohesive type) except for MTA exposed to acidic environment; which showed mixed failure mode in most of the specimens. This finding is in contrast to Shokouhinejad et al. [15] who reported that MTA-dentin bond failures were usually at the MTA-dentin interface (adhesive type) in both pH=4.4 and 7.4. The difference between thickness of root slices in this study and the previous study [15] may explain the different modes of bond failure. The delivery form of ERRM may result in greater adherence to dentinal walls and cohesive failure mode for this premixed material.

**CONCLUSION**

Under the conditions of this study, the force needed for displacement of MTA and ERRM paste from root dentin was significantly lower in samples stored in acidic pH, while ERRM putty was not affected by acidity.

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