Original Article

Push-Out Bond Strength of Bioceramic Materials in a Synthetic Tissue Fluid

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Abstract

Objective: This study compared the push-out bond strength of EndoSequence Root Repair Material (ERRM) and Bioaggregate (BA), new bioceramic materials, to that of mineral trioxide aggregate (MTA) after incubation in phosphate-buffered saline (PBS), a synthetic tissue fluid, for either 1 week or 2 months.

Materials and Methods: One-hundred and twenty root sections were filled with ProRoot MTA, BA, or ERRM. Each tested material was then randomly divided into two subgroups (n = 20): root sections were immersed in PBS for 1 week or 2 months. The bond strengths were measured using a universal testing machine. After that, the failure modes were examined with stereomicroscopy and scanning electron microscopy (SEM). The push-out data and failure mode categories were analyzed by two-way ANOVA and chi-square tests, respectively.

Results: The bond strength of ERRM was significantly higher than that of BA and MTA at both incubation periods. No significant difference was found between the bond strength of MTA and BA at either 1 week or 2 months. Increasing the incubation time to 2 months resulted in a significant increase in bond strength of all the materials. The failure mode was mainly mixed for MTA and BA, but cohesive for ERRM at both incubation periods.

Conclusion: ERRM had significantly higher bond strength to root canal walls compared to MTA and BA. Increasing the incubation time significantly improved the bond strength and bioactive reaction products of all materials.

Key Words: BioAggregate; Bond Strength; EndoSequence Root Repair Material; Mineral Trioxide Aggregate

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INTRODUCTION

An ideal endodontic material when used to seal communications between the root canal system and periodontium should be biocompatible, dimensionally stable, adhere to the dentin, and remain in place under dislocating

forces [1]. Mineral trioxide aggregate (MTA) has been shown to be a good biomaterial for root-end filling, perforation repair, pulpotomies, and apexification [2,3]. Recently, new bioceramic materials have been introduced to the market as alternatives to MTA. Bioceramic components or ceramic materials with osteoinductive properties are used in medicine and dentistry as replacements and implants [4]. Alumina, zirconia, hydroxyapatite, bioactive glass, calcium phosphate and some calcium silicate-based materials are considered to be bioceramic materials [5]. ERRM (Brasseler USA, Savannah, GA, USA), a new bioceramic material, is a hydrophilic, insoluble, radiopaque, and aluminum-free material composed of calcium silicates, zirconium oxide, tantalum oxide, calcium phosphate monobasic, and filler agents; it is delivered as a premixed product in both low viscosity paste form dispensed from a syringe and a high viscosity putty form. Moisture is required for the materials to set and harden. The working time is more than 30 minutes, and the setting time is 4 hours under normal conditions. ERRM is of alkaline pH [6], biocompatible [5,7], antibacterial [8], and able to seal root-end cavities [9].

BA (Innovative BioCeramix, Vancouver, BC, Canada) was recently developed and delivered in powder form that is composed of tricalcium silicate, dicalcium silicate, calcium phosphate monobasic, amorphous silicon dioxide, and tantalum pentoxide [10]. It was described as a cytocompatible [11] and antibacterial material [10] that is able to seal root-end cavities [12], induce mineralized tissue formation [13], and differentiation of human periodontal ligament fibroblasts [14]. However, in an ex vivo perforation model, BA has been reported to have lower bond strength compared to MTA-Angelus [15]. Formation of hydroxyapatite crystals as a result of the interaction between MTA and phosphate-containing materials such as tissue fluid and/or dentin (i.e. bioactivity), a common characteristic of calcium silicate-containing biomaterials [16,17], has been demonstrated [18,19]. It has been stated that calcium ions released by MTA interact with phosphate in PBS, a synthetic tissue fluid, to form an apatite layer at the MTA-dentin interface and chemically bond the MTA to dentin via a diffusion-controlled reaction between its apatitic surface and dentin [18]. The biomineralization process promoted by the interaction of MTA with dentin in the presence of a phosphate-containing fluid [18,20-22] increased the push-out bond strength of MTA [21, 23]. Furthermore, the bioactivity of ERRM and BA in the presence of a synthetic tissue fluid has been shown [24]. Little information is available on the bond strength of ERRM. Therefore, this laboratory study was conducted to compare the bond strengths of ERRM putty, BA, and white ProRoot MTA (Dentsply Tulsa Dental, Tulsa, OK, USA) using a push-out force methodology following immersion in PBS incubated at 37ºC for either 1 week or 2 months.

MATERIALS AND METHODS

Sample preparation

Sixty freshly extracted single-rooted human teeth were selected and stored in 0.5% chloramine-T at 4ºC. The middle portion of each root was sectioned perpendicular to the long axis to produce two discs of 2.00 ± 0.05 mm thickness using a water-cooled diamond blade on a precision cut-off machine (Mecatome, Presi, France). Finally, 120 root slices were produced. The canal lumen of each specimen was widened using size 2 to 5 Gates Glidden burs (Dentsply Maillefer, Ballaigues, Switzerland), to produce a standardized internal diameter of 1.3 mm. The specimens were immersed in 17% EDTA for 3 minutes followed by 5.25% sodium hypochlorite for the same period of time and then washed with distilled water and dried. The root specimens were divided randomly into the following groups:

Group M (MTA): One gram of white ProRoot MTA powder was mixed with an aliquot of 0.33 mL distilled water.

The resultant slurry material was then placed into the canal space with minimal pressure.

Group B (BA): One gram of BA powder was mixed with an aliquot of 0.33 mL deionized water included in the package. The resultant slurry material was then introduced into the canal space with minimal pressure.

Group E (ERRM):

The canal spaces were filled with ERRM putty, which is premixed by the manufacturer. A PBS solution containing 1.7 g of KH2PO4, 11.8 g of Na2HPO4, 80.0 g of NaCl, and 2.0 g of KCl in 10 L of H2O (pH=7.2) was prepared.

Specimens were wrapped in pieces of gauze soaked in PBS for 1 hour.

Each group was then randomly divided into two subgroups each of 20. In subgroups M1, B1, and E1, the root sections were immersed individually in sterile Eppendorf plastic tubes (Eppendorf-Elkay, Shrewsbury, MA, USA) containing 2 mL of PBS and stored in PBS for 1 week; and in subgroups M2, B2, and E2, for 2 months. The PBS solution was replaced every 5 days to replenish the buffering capacity of the PBS [19]. All specimens were incubated at 37ºC.

Push-out test

After the experimental periods, the specimens were submitted to the push-out test. The filling material was loaded with a 1-mm diameter cylindrical stainless steel plunger. Loading was performed on a universal testing machine (Z050, Zwick/Roell, Ulm, Germany) at a speed of 0.5 mm/min.

The maximum load applied to the filling material before debonding was recorded in Newton. To express the bond strength in megapascals (MPa), the load at failure recorded in Newton (N) was divided by the canal wall area in mm^2. After the push-out testing, the root sections were examined under a stereomicroscope at 40× magnification to determine the failure mode. Modes of failure were defined as follows:

(1) Cohesive: failure was entirely within the cements;

(2) Adhesive: failure was at the cement/dentin interface;

(3) Mixed failure.

Scanning electron microscopy analysis

Two specimens, representative of the failure modes from each group, were then split longitudinally along the center of the canal. The pulpal walls of the specimens were mounted on aluminium stubs, sputter coated with gold, and examined under a SEM (Vega II XMU, Tescan, Czech Republic) at 15 kV to evaluate the failure modes and cement remnants on dentin walls. The push-out data were analyzed by two-way ANOVA and Holm-Sidak post hoc tests. Significance level was set at $p < 0.05$. The chi-square test was used to analyze the data for failure modes and the significance level was set at $p < 0.025$ using Bonferroni correction.

RESULTS

Bond strength

The mean bond strength values for each test group are presented in Table 1.

Table 1. Bond Strength Values in MPa and Failure Modes for the Experimental Groups

Group (*n* = 20)	Bond Strength	Failure Mode (%)		
		Adhesive	Cohesive	Mixed
M1: MTA/1W	2.61 (1.1)	25	10	65
M2: MTA/2M	8.4 (2.86)	10	20	70
B1: BA/1W	2.1 (0.96)	30	0	70
B2: BA/2M	7.25 (2.35)	5	5	90
E1: ERRM/1W	11.7 (1.66)	0	70	30
E2: ERRM/2M	17.79 (3.78)	0	80	20

MTA, Mineral trioxide aggregate; BA, Bioaggregate; ERRM, EndoSequence Root Repair Material; 1W, 1-week incubation time in PBS; 2M, 2-month incubation time in PBS

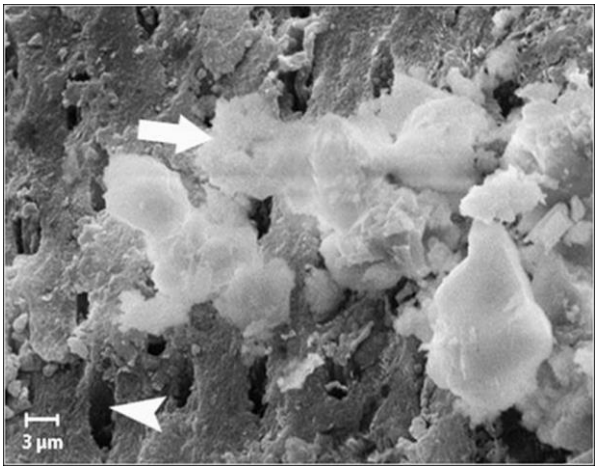

Fig 1. Photomicrograph of a mixed failure mode shows cement remnants attached to dentine (arrow). Open dentine tubules are seen in some areas (arrowhead).

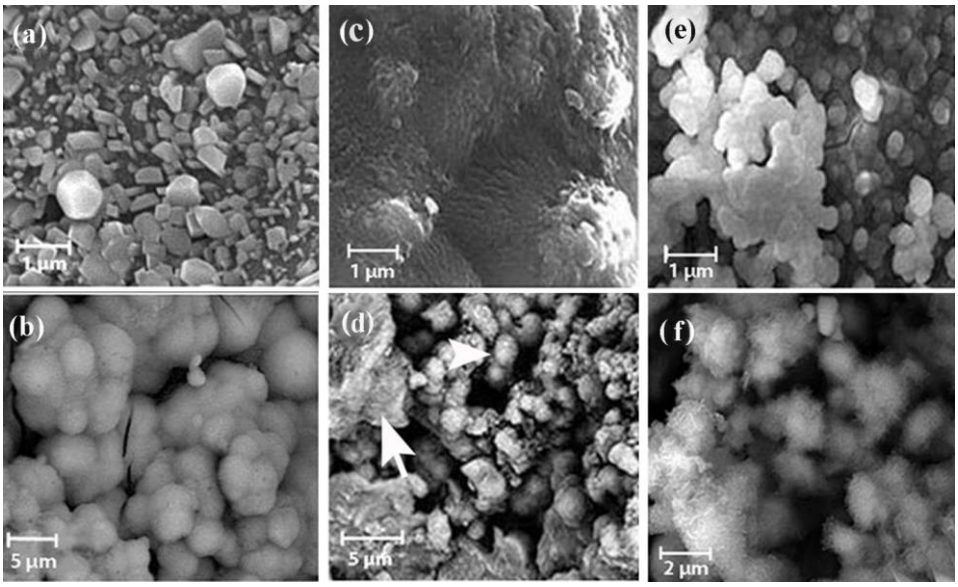

Fig 2. SEM images of material remnants on canal walls. An M1 specimen showing various sharp-edged crystals (a). Formation of spherical aggregates in an M2 specimen (b). A view of cement remnants in a B1 specimen showing relatively homogeneous layer with less crystallinity (c). A photomicrograph of cement remnants on dentinal wall in B2 group showed spherical (arrowhead) mixed with coral-like aggregates (arrow) (d). In E1 specimens, presence of smooth aggregates was notable on the dentinal wall (e). A specimen of E2 group showing spherical crystals with rough surfaces (f).

The interaction between the two factors of material type and incubation period was not significant ($p = 0.33$). The bond strength of ERRM was significantly higher than those of MTA and BA at both 1-week and 2-month incubation periods ($p < 0.001$).

No significant difference was observed between the bond strength of MTA and BA at both incubation times ($p = 0.24$).

By increasing the incubation time, the bond strength of all materials increased significantly ($p < 0.001$).

Inspection of the specimens revealed that the bond failure mode was mainly mixed for MTA and BA, but cohesive for ERRM at both incubation times (Table 1). The failure modes for ERRM were significantly different from those seen for MTA and BA at both incubation times ($p = 0.00$).

Scanning electron microscopy

A photomicrograph of a specimen representative of mixed failure mode is shown in Fig. 1. Pulpal walls of the specimens under SEM exhibited various morphologies of material remnants on the canal walls (Figs 2a-f).

After 2 months substantial crystal growth and formation of apatite-like aggregates were observed (Figs 2b, d and f). M1 specimens revealed various sharp-edged crystals (Fig 2a). In M2 specimens (Fig. 2b), spherical aggregates were prominent, which were not observed in the M1 specimens. B1 specimens had a relatively homogeneous layer with less crystallinity (Fig 2c). In contrast, after 2 months micrographs of material remnants on dentinal walls in the B2 group had spherical crystals mixed with coral-like aggregates (Fig. 2d). In E1 specimens, the presence of smooth aggregates was notable (Fig. 2e). However, in the E2 group after 2 months, spherical crystals with rough surfaces indicating maturation were seen (Fig 2f).

DISCUSSION

In this study, bond strengths of ERRM putty were significantly higher than those of MTA and BA at both 1 week and 2 months. In addition, the failure mode of ERRM at both incubation times was mainly cohesive. Therefore, the higher bond strength values obtained for ERRM is most likely due to its greater adherence to dentinal walls. The higher bond strength of ERRM compared to MTA and BA might be attributed to the delivery system of these materials, which is premixed putty for ERRM and separate powder and liquid for both MTA and BA.

The thickening and filler agents added to ERRM to make it putty form might be associated with higher bond strength. It has been reported that the presence of zirconium oxide improved certain physical properties of composite bioceramics [25]. The presence of zirconium oxide in the composition of ERRM might also result in higher bond strength of ERRM. Since this material has been developed only recently, further studies are necessary to investigate its other physicochemical properties. BA has a similar composition to MTA, differing mostly by being aluminium-free [11] and having tantalum oxide as a radiopacifier in place of the bismuth oxide in MTA [10,26]. This is claimed to be associated with improved biological properties [11]. The results of the present study revealed that the bond strength of BA was similar to that of MTA at both incubation times. Furthermore, the bond failure mode for MTA and BA was similar. In contrast, Hashem and Wanees Amin [15] found a higher dislocation resistance value for MTA-Angelus than for BA when placed in furcation perforations after exposure to PBS at 4 and 34 days. Since the methodology and materials of the present study were different from those of Hashem and Wanees Amin [15] a direct comparison is not possible. In the present study, MTA, BA, and ERRM had higher bond strength values at 2-months compared with those observed at 1-week. This could be attributed to the bioactivity of calcium-silicate based materials [18-20,27].

Physicochemical interaction between MTA and root canal walls in the presence of a phosphate-containing fluid resulted in a chemical bond between the apatitic surface of MTA and dentin [18] that may modify the retention and friction of cements on dentin walls [23]. It has been found that samples of MTA immersed in PBS for 2 months had a significantly greater resistance to displacement than that observed for the samples in contact with a wet cotton pellet for 72 hours [21].

In addition, Huffman et al. [28] reported that ProRoot Endo Sealer (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA) had higher push-out strength values after immersion in a phosphate-containing fluid for 4 weeks. Therefore, in the present study the root sections were stored in PBS to partially simulate the in vivo conditions. In the present study, SEM analysis of dentin walls in the M1 specimens revealed the formation of various sharp-edged crystalline structures. However, a homogeneous layer with less crystallinity was seen in B1 specimens that may have been caused by hydration of the nano-sized BA particles [11].

These findings are in accordance with the study conducted by Hashem and Wanees Amin [15] who identified irregular and hexagonal crystals in MTA following a 4-day exposure to PBS, but not with BA. In the present study, all specimens incubated for 2 months had significantly more crystal growth and apatite-like aggregates on dentin walls. Spherical aggregates that were observed following 2 months in all tested materials, are consistent with the findings of Sarkar et al. [18] and Reyes-Carmona et al. [21], who observed the formation of spherical precipitates after immersing MTA in PBS for 2 months. Apatitic composition of these aggregates has been revealed [18,29]. Higher bond strengths following 2-months of incubation can be explained by the bioactivity and interaction of MTA, BA, and ERRM with dentin in a phosphate-containing fluid. Inspection of root sections filled with MTA revealed most failures to be a mixture of cohesive and adhesive at the cement/dentin interface and within the cement at both incubation time periods. This result is in contrast with the findings of Vanderweele et al. [30] and Shokouhinejad et al. [31] who reported that MTA-dentin bond failures were mainly adhesive. The adhesive failure mode found in the study by Shokouhinejad et al. [31] might be attributed to the short incubation time prior to evaluation of the bond strength that was 4 days.

The predominant adhesive failure mode in the study by Vanderweele et al. [30] after 1 week might be explained by not storing the specimens in PBS. In the present study, although an adhesive failure mode was seen in both MTA and BA groups, this mode of failure did not occur with ERRM specimens, possibly due to its putty form.

CONCLUSION

ERRM had greater bond strengths than BA and MTA to the canal walls following incubation times of 1 week and 2 months in PBS. Increasing the incubation time significantly improved the bond strength of all materials. ERRM, BA, and MTA revealed formation of apatite-like crystalline structures following longer incubation time.

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