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An in vitro Comparison of Bond Strength of Different Sealers/Obturation Systems to Root Dentin Using the Push-Out Test at 2 Weeks and 3 Months after Obturation

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Significance of the Study

- In this study, the push-out bond strength of the TotalFill BC™ sealer was comparable to AH Plus® but higher than the EndoREZ® sealer. It increased over time (1.74 ± 0.43 and 3.69 ± 1.20 MPa at 2 weeks and 3 months after obturation, respectively). Hence, a bioceramic sealer could be a good material of choice for obturation.

Keywords

Bioceramic sealer · Bond strength · EndoREZ® · Push-out test · TotalFill BC™

Abstract

Objective: To evaluate the push-out bond strength and failure modes of different sealers/obturation systems to intraradicular dentin at 2 weeks and 3 months after obturation compared to AH Plus®/gutta-percha. ***Materials and Methods:*** A total of 180 root slices from 60 single-canal anterior teeth were prepared and assigned to 5 experimental groups (n = 36 in each group), designated as G1 (AH Plus®/gutta-percha), G2 (TotalFill BC™ sealer/BC-coated gutta-percha), G3 (TotalFill BC™ sealer/gutta-percha), G4 (EndoREZ® sealer/EndoREZ®-coated gutta-percha), and G5 (EndoREZ® sealer/gutta-percha). Push-out bond strengths of 18 root slices in each group were assessed at 2 weeks and the other 18 at 3 months after obturation using a universal testing machine. Data were analyzed using repeated measures ANOVA. An independent t test was used to compare the mean push-out bond strength for each group at 2 weeks and 3 months after obturation. ***Results:*** The mean push-out bond strengths of G4 and G5 were significantly lower than those of G1, G2, and G3 ($p < 0.05$) at both 2 weeks (G1: 1.46 ± 0.29 MPa, G2: 1.74 ± 0.43 MPa, G3: 1.74 ± 0.43 MPa, G4: 0.66 ± 0.31 MPa, G5: 0.74 ± 0.47 MPa) and 3 months after obturation (G1: 1.70 ± 1.05 MPa, G2: 3.69 ± 1.20 MPa, G3: 2.84 ± 0.83 MPa, G4: 0.14 ± 0.05 MPa, G5: 0.24 ± 0.10 MPa). The mean push-out bond strengths of G2 (3.69 ± 1.20 MPa) and G3 (2.84 ± 0.83 MPa) were higher at 3 months compared to 2 weeks after obturation (G2: 1.74 ± 0.43 MPa, G3: 1.33 ± 0.29 MPa). ***Conclusion:*** The TotalFill BC™ obturation system (G2) and the TotalFill BC™ sealer/gutta-percha (G3) showed comparable bond strength to AH Plus®. Their bond strength increased over time, whereas the EndoREZ® obturation system (G4) and EndoREZ sealer (G5) had low push-out bond strength which decreased over time.

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Introduction

Three-dimensional obturation that completely seals the entire root canal system from any bacterial ingress from oral cavity and periradicular tissues is important for successful endodontic treatment [1]. One of the desirable properties of the root canal filling is its ability to adhere to radicular dentin [2]. Good adhesion eliminates percolation of fluids between the filling and the root canal wall [3], and resists dislodgement of fillings during subsequent manipulation, e.g., postspace preparation [4]. In the early 2000s, manufacturers had further incorporated adhesive dentistry into endodontics by introducing obturation systems with emphasis on obtaining a "monoblock" in which a single cohesive unit is formed through bonding of the core material, sealing agent, and root canal dentin [5]. Examples of such systems include Resilon (Pentron Clinical Technologies, Wallingford, UK), Activ GP (Brasseler, Savannah, GA, USA), EndoREZ® (Ultradent, South Jordan, UT, USA) and the latest TotalFill BC™ (FKG Dentaire, La Chaux-de-Fonds, Switzerland).

The EndoREZ® system consists of a urethane dimethacrylate-based self-priming sealer and a polybutadiene-diisocyanate-methacrylate resin-coated gutta-percha (GP) core material. It is hydrophilic and has an affinity for moisture present deep in dentinal tubules and lateral canals [6].

The TotalFill BC™ obturation system contains bioceramic nanoparticles impregnated in TotalFill BC™ points and BC sealer that utilizes the moisture naturally present in dentinal tubules to initiate its setting reaction [7]. The BC sealer is biocompatible and osteogenic, and exhibits zero shrinkage [8]. According to the manufacturer, a gap-free obturation is achieved when the bioceramic particles found in the BC sealer are used in conjunction with the bioceramic particles in BC points.

AH Plus® (Dentsply Maillefer, Ballaigues, Switzerland) is a 2-component paste/paste root canal sealer based on epoxy-amine resin. It has an extensive market history. Furthermore, it has always been used as a reference and standard in many investigations [8–11]. The chemical composition of EndoREZ®, TotalFill BC™, and AH Plus® is summarized in Table 1.

Resistance to dislodging force by root filling materials during function is highly desirable. The push-out test is widely used to assess the bond strength at the root canal filling/dentin interface [12]. The push-out test produces a more homogeneous stress distribution and less variability than the microtensile bond test [13]. It yields repetitive results and does not need highly sophisticated equipment [8].

Table 1. Chemical composition of EndoREZ®, TotalFill BC™, and AH Plus®

Sealers	Type of sealer	Chemical composition
EndoREZ®	Methacrylate resin based	Bismuth compound, urethane dimethacrylate, triethylene glycol dimethacrylate, peroxide initiator
TotalFill BC™	Calcium silicate based	Zirconium oxide, calcium silicates, calcium phosphate, calcium hydroxide, filler, thickening agents
AH Plus®	Epoxy resin based	Epoxy resins, zirconium oxide, iron oxide, calcium tungstate, silicone oil

Degradation of the resin bond in restorative applications has been shown to be due to the functional forces and unpolymerized resin [14]. An interfacial gap is always present in resin-bonded restorations, be it in restorative dentistry or bonded obturating materials, and this gap formation increases with time [14]. Loss of bond strength was first detected in the laboratory at 3 months [14]. To date, no bond strength test has been reported at 3 months after obturation. Therefore, the objective of this study was to evaluate the push-out bond strength of different obturation systems (TotalFill BC™ obturation system: TotalFill BC™ sealer/BC-coated GP, TotalFill BC™ sealer with GP; EndoREZ® obturation system: EndoREZ® sealer/EndoREZ®-coated GP, EndoREZ® sealer with GP) to intraradicular dentin at 2 weeks and 3 months after obturation compared to AH Plus® with GP.

Materials and Methods

Specimen Preparation

Sample size calculation was done using the Power and Sample Size calculation program (Vanderbilt Biostatistics, Nashville, TN, USA). The estimated power was 0.80 and the significance level was set at $\alpha = 0.05$. A minimum of 5 teeth per group were needed for this study to reject the null hypothesis.

Sixty extracted maxillary central incisors were selected. Exclusion criteria were caries, curved roots, open apices, cracks, or previous root canal treatments. A radiograph was taken for each tooth sample to ensure no intracanal abnormality (e.g., sclerosed canal, root resorption) was present. The teeth were disinfected in 0.5% chloramines T trihydrate solution and stored in distilled water until use. The teeth were decoronated to obtain approximately 16-mm-long root segments. Canal patency was achieved using K-file size #10. The first bind file for each root was determined, and those with a first bind file equal or smaller than K-file size #25 were included in the study. Working length was established by placing

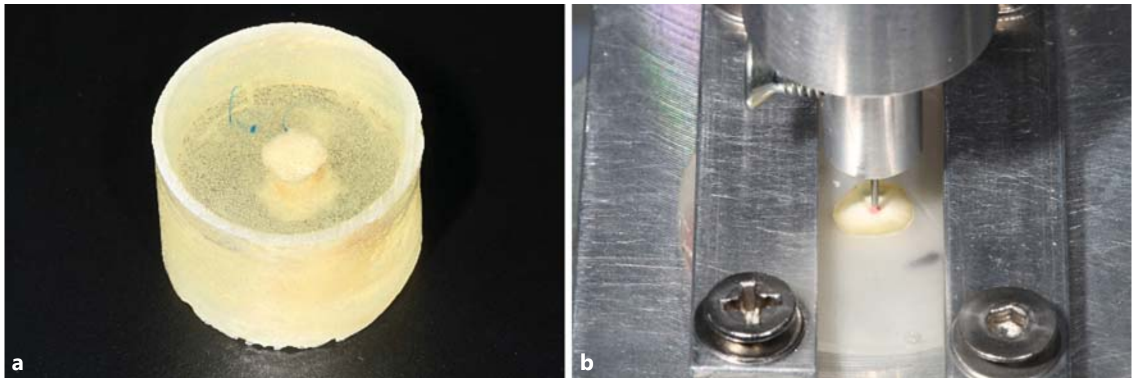

Fig. 1. a Root embedded in epoxy resin. **b** Positioning of the root slice on the jig.

a stainless steel K-file size #10 into the canal until the tip of the file was visible at the apical foramen and then subtracting 1 mm. Cleaning and shaping were done using ProTaper Universal (Dentsply Maillefer) in a modified crown-down manner according to the manufacturer's instructions using a gentle in-and-out motion up to size F4 (#40/0.06).

Canals were passively irrigated with 5 ml of 5.25% sodium hypochlorite solution between each instrument using a 31-G NaviTip (Ultradent, South Jordan, UT, USA) double-side port irrigation needle with a conventional syringe at 1 mm short from the working length. At the end of the procedure, 5 ml of 17% EDTA solution (SmearClear; SynbronEndo, Orange, CA, USA) was used for irrigation for 1 min, followed by 5 ml of 5.25% sodium hypochlorite and a final rinse with copious amounts of distilled water. The roots were then divided randomly into 5 groups with 12 roots per group based on the obturation systems tested.

Obturation

Group 1 (AH Plus®/GP): AH Plus® sealer was mixed on a paper pad and applied to the root canal walls using a lentulo spiral size 3 (Dentsply Maillefer) at 100 rpm. A #40/0.06 taper GP cone (ProTaper GP F4; Dentsply Maillefer) was coated with AH Plus® sealer and pumped a few times in the root canal. The cone was removed, recoated with sealer, and then inserted up to the working length. Group 2 (TotalFill BC™ obturation system – TotalFill BC™ sealer/BC-coated GP): TotalFill BC™ sealer was syringed into the canal and then a #40/0.06 taper TotalFill BC™ cone was placed in the canal up to working length. Group 3 (TotalFill BC™ sealer/GP): TotalFill BC™ sealer was syringed into the canal and then a #40/0.06 taper GP was placed in the canal up to working length. Group 4 (EndoREZ® obturation system – EndoREZ® sealer/EndoREZ®-coated GP): EndoREZ® sealer was syringed into the canal and then a #40/0.06 taper EndoREZ® cone was placed in the canal up to working length. Group 5 (EndoREZ® sealer/GP): EndoREZ® sealer was syringed into the canal and then a #40/0.06 taper GP was placed in the canal up to working length.

After completion of obturation, the coronal access of all specimens was sealed with glass ionomer cement (GC Fuji IX GP; GC Asia), and all 5 groups were stored in an incubator at 37°C and 100% humidity. Each group was further subdivided into 2 subgroups (A and B; 6 in each subgroup) for push-out assessment at 2 weeks and 3 months, respectively.

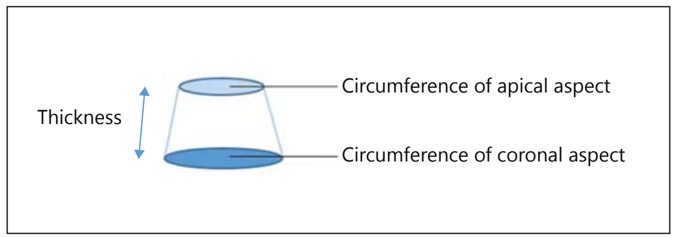

Fig. 2. Diagramatic representation of debonded area.

Push-Out Assessment

Sixty roots were embedded in a rubber mold individually using epoxy resin (Mirapox; Miracon, Malaysia) (Fig. 1a). After the setting of the mold, the apical 4 mm of each root was sliced and discarded as the size of the filling material at this level was very small. Subsequently, 3 successive root slices 2 mm in thickness and a total of 180 root slices were obtained from the remaining portion of each root using a water-cooled precision diamond saw (Micracut 125 low-speed precision cutter; Metkon, Turkey). The thickness of each root slice was verified using a digital caliper (Mitutoyo/Digimatic, Tokyo, Japan). The circumference of the apical and coronal aspect and the diameter of the apical aspect of each slice were measured using a stereomicroscope (Olympus SZ X7; Leica, Tokyo, Japan) at 25× magnification before subjecting the specimens to the push-out test.

Each specimen was carefully positioned on the jig with the apical surface facing the plunger (Fig. 1b) of a universal testing machine (Shimadzu, Japan) equipped with a 0.6-mm-diameter cylindrical stainless steel plunger. To facilitate the correct positioning, a 2.5× dental surgical loupe (EyeMag® Smart; Carl Zeiss Meditec AG, Germany) with light illumination was used. The plunger was in contact with the filling material only to avoid misreading by fracturing the root dentin, thus avoiding inconsistent readings. The parameter for the universal testing machine was the compressive load applied to the filling material at a speed of 0.5 mm/min until bond failure occurred.

The bond strength was measured in megapascals (MPa) and calculated by dividing the debonded force (N) over the debonded area of the root canal filling (mm^2). The debonded area was calcu-

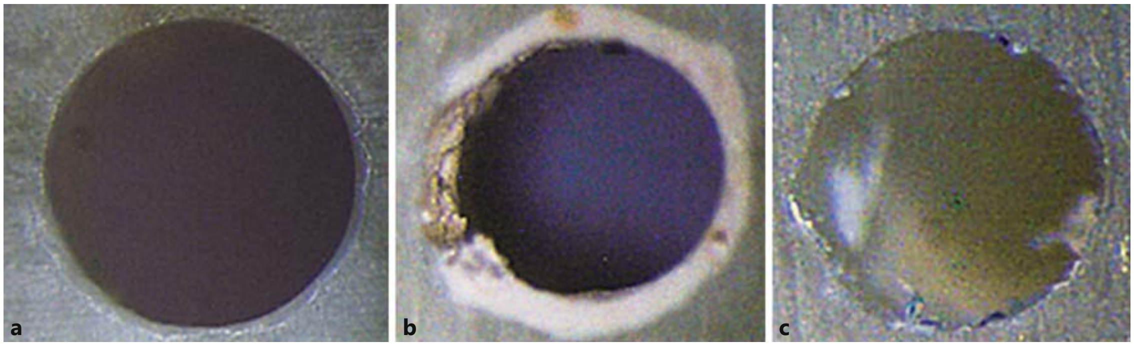

Fig. 3. Stereomicroscopic examination of the samples at 56× magnification and various failure modes. **a** Adhesive failure at the dentin-sealer interface. **b** Cohesive failure within the filling material. **c** Mixed failure in both adhesive and cohesive modes.

Table 2. Mean push-out bond strength (MPa) in the experimental groups at 2 weeks and 3 months after obturation

Groups	Bond strength		p value	Mean difference (I – J)
	2 weeks (I)	3 months (J)		
G1	1.46 ± 0.29^a	1.70 ± 1.05^d	0.37	–0.24
G2	1.74 ± 0.43^a	3.69 ± 1.20^c	<0.001	–1.95
G3	1.33 ± 0.29^a	2.84 ± 0.83^c	<0.001	–1.51
G4	0.66 ± 0.31^b	0.14 ± 0.05^e	<0.001	0.52
G5	0.74 ± 0.47^b	0.24 ± 0.10^e	<0.001	0.50

Values are mean ± SD. G1, AH Plus®/gutta-percha (GP); G2, TotalFill BC obturation system – TotalFill BC sealer/BC-coated GP; G3, TotalFill BC sealer/GP; G4, EndoREZ obturation system –EndoREZ sealer/EndoREZ-coated GP; G5, EndoREZ sealer/GP. Different superscript letters indicate statistically significant differences among groups (p < 0.05). a > b; c > d > e.

lated using the following formula [15]: debonded area (mm^2) = [circumference of apical aspect (mm) + circumference of coronal aspect (mm)]/2 × thickness (mm) (Fig. 2).

Mode of Failure Assessment

After the push-out test, both sides of the sample including the main cone and sealer plug were examined under stereomicroscope (Olympus SZ X7; Leica) at 56× magnification to determine the mode of failure. The failures were subsequently categorized into (a) adhesive failure at the dentin-sealer interface (Fig. 3a), (b) cohesive failure within the filling material (Fig. 3b), and (c) mixed failure in both adhesive and cohesive modes (Fig. 3c).

Data Analysis

The Statistical Package for the Social Sciences (SPSS), version 21 (SPSS Inc., Chicago, IL, USA) was used to analyze the data. Repeated measures ANOVA was used to analyze the variance between different filling materials. The significance value was set at $\alpha = 0.05$. Multiple post hoc comparisons were selected to reveal the existence of significant differences among the groups. An independent t test ($\alpha = 0.05$) was used to compare the mean push-out bond strength for each group at the 2-week and 3-month intervals.

Results

The mean values and standard deviations of the push-out bond strength (MPa) at 2 weeks and 3 months after obturation are shown in Table 2. The difference in the mean values among groups at 2 weeks and 3 months after obturation regardless of location was significant ($p <$ 0.05). The mean push-out bond strengths of G4 and G5 were significantly lower than those of G1, G2, and G3 ($p < 0.05$) at both 2 weeks and 3 months after obturation. However, no statistical difference was detected between G4 and G5.

The G2 and G3 groups showed higher mean push-out bond strength at 3 months (3.69 ± 1.20 and 2.84 ± 0.83 MPa, respectively) compared to 2 weeks after obturation (1.74 ± 0.43 and 1.33 ± 0.29 MPa) ($p < 0.01$) (Table 2). For both G4 and G5, the mean push-out bond strength 3 months after obturation (0.14 ± 0.05 and 0.24 ± 0.10 MPa, respectively) was significantly lower compared to 2 weeks after obturation (0.66 ± 0.31 and 0.74 ± 0.47 MPa) ($p < 0.01$). For G1, no statistically significant difference in mean push-out bond strength was seen at 3 months after obturation compared to 2 weeks after obturation. Stereomicroscopic examination of 180 samples revealed that most of the samples showed mixed failure mode regardless of postobturation interval (Table 3).

Table 3. Failure mode (%) for each group at 2 weeks and 3 months after obturation

Groups	2 weeks			3 months		
	adhesive	cohesive	mixed	adhesive	cohesive	mixed
G1	0	33.3	66.7	0	38.9	61.1
G2	0	38.9	61.1	0	44.4	55.6
G3	0	61.1	38.9	5.6	50.0	44.4
G4	0	72.2	27.8	0	55.6	44.4
G5	16.7	22.2	61.1	5.6	22.2	72.2

G1, AH Plus®/gutta-percha (GP); G2, TotalFill BC obturation system – TotalFill BC sealer/BC-coated GP; G3, TotalFill BC sealer/GP; G4, EndoREZ obturation system – EndoREZ sealer/EndoREZ-coated GP; G5, EndoREZ sealer/GP.

Discussion

In the current study, both TotalFill BC™ obturation systems performed equally in push-out bond strength compared to AH Plus® at 2 weeks after obturation, and the bond strengths showed an increase at 3 months after obturation. This could be attributed to the high flowability of the BC sealer [16] which leads to deeper penetration of the sealer into the dentinal tubules for micromechanical interlocking, thus resulting in greater adhesion. Moreover, incorporated calcium phosphates in the sealer facilitate a reaction with calcium hydroxide upon activation by the moisture that remains within the dentinal tubules, producing hydroxyapatite. Hydroxyapatite is coprecipitated within the calcium silicate hydrate phase [17] to produce a composite-like structure, reinforcing the set cement [18].

In this study, the bond strength of TotalFill BC™ was comparable to AH Plus®/GP and higher than EndoREZ® at 2 weeks, and this is consistent with a previous study [10]. Also, the push-out bond strength increased significantly over time from 2 weeks to 3 months after obturation for G2 (BC sealer/BC-coated GP) and G3 (BC sealer/GP). G1 (AH Plus®/GP) showed an increase in bond strength with time, but this was statistically not significant. This result is similar to that of a study by Elbatouty et al. [19], where the push-out bond strength of a bioceramic sealer was compared to AH Plus® and eugenol-based sealer KERR EWT at 1 week, 2 weeks, and 1 month. Their results showed an increasing trend of bond strength from 1 week to 1 month for both TotalFill BC™ and AH Plus®. This finding may be attributed to the increased setting and hardening of the material over time.

In the current study, G4 (EndoREZ® sealer/EndoREZ®-coated GP) and G5 (EndoREZ® sealer/GP) had significantly lower bond strengths regardless of the time interval. A probable explanation could be the high c-factor (ratio of bonded to unbounded resin surfaces) seen in root canals, which concentrates high polymerization stresses between resin-based materials and the canal walls; these stresses may exceed the bond strength of adhesives to dentin, resulting in gap formation along the surfaces [20]. This finding confirmed those of previous studies [9–11, 21] which showed that EndoREZ® has low push-out bond strength. The push-out bond strength of G4 and G5 decreased further at 3 months compared to 2 weeks after obturation and remained the lowest in bond strength among all groups. A possible explanation for this could be the percolation of fluid through the gaps along the surface, which in turn leads to plasticization and hydrolysis of the resin [22]. In addition, collagen degradation may occur due to host-derived matrix metalloproteinases in dentin that are slowly released over time when self-etching adhesives are used. During push-out testing, the specimen should be positioned vertically, with a plunger size slightly smaller than the canal diameter to minimize interfacial sliding friction [23]. The diameter of the apical side of each slice was measured and a push-out plunger of 0.6 mm was chosen as it was within the range of 70–90% of canal diameter. A plunger size 70–90% of the canal diameter has been shown not to affect the bond strength [24].

Conclusions

In this study, the TotalFill BC™ obturation system (G2) and the TotalFill BC™ sealer (G3) showed comparable bond strengths to AH Plus®. The bond strength also exhibited an increase over a 3-month postobturation period. On the other hand, the EndoREZ® obturation system (G4)/EndoREZ® sealer (G5) had significantly low push-out bond strengths which decreased over the same period. Thus, the push-out bond strength of the sealer to intraradicular dentin is influenced by the time after obturation and also the composition of the sealer.

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Disclosure Statement

The authors have no conflicts of interest to declare.

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