Bonding of Resin-based Sealers to Root Dentin

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Abstract

This study compared the microshear bond strength of three resin-based sealers to root dentin and assessed whether sealer cements behave differently in thin and thick films. Extracted maxillary premolars were sectioned buccolingually, and 45 root halves were randomly allocated for microshear bond testing with the three resin sealers in thin and thick films. The microshear bond strength was then calculated in MPa. Failure modes were examined under light and scanning electron microscopy. Data were analyzed by using analysis of variance, with significance set at $p < 0.05$. Overall, the epoxy resin-based sealers had the highest microshear bond strength to root dentin compared with urethane dimethacrylate–based sealers ($p < 0.001$). Bond strengths for the thick sealer group were significantly higher than the thin sealer group ($p < 0.001$) and may reflect different patterns of behavior when the sealer is present as a thin layer. *(J Endod 2009;35:121–124)*

Key Words

AH Plus, EndoRez, microshear bond strength, Resilon, resin-based sealer

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Sealer cements are an essential component of root-filling materials to fill any voids and gaps between the main root-filling material and root dentin. Good adhesion to tooth material within the root canal is one of the ideal properties of a sealer cement (1), which potentially influences both leakage and root strength (2-5). Bond strength testing is the best measure of adhesion. Adhesion of the root canal filling to the dentinal walls is advantageous for two main purposes. In a static situation, it should eliminate any space that may allow percolation of fluids between the filling and the canal wall (6). In a dynamic situation, it is needed to resist dislodgement of the filling during subsequent manipulation (7).

Gutta-percha does not bond to root dentin and therefore must be used in association with sealer cement to provide a bond between the core material and the root canal wall. Epoxy-based sealers (AH Plus and AH 26; Dentsply Maillefer, Johnson City, TN) have shown a higher bond strength to dentin than zinc oxide–eugenol, glass ionomer, and calcium hydroxide–based sealer (8-11). More recently, the newly developed resin-based sealers (RealSeal System; Pentron, Wallingfort, CT) have been claimed to create a "monoblock," meaning the creation of a solid, bonded, continuous material from one dentin wall of the canal to the other with a superior seal (3, 4).

Both microshear bond testing (12) and push-out tests (13-17) have shown that the bonding of RealSeal plus a urethane dimethacrylate (UDMA)-based sealer to root dentin is not superior to other sealer systems. Jainaen et al (15) have also shown that the behavior of resin-based sealers is influenced by film thickness. They showed that when the entire root canal system was obturated with sealer only (thick film) there was a two- to eight-fold increase in push-out bond strength compared with sealer cement plus core material. This finding suggests that the "ideal" of a thin layer of sealer needs to be reconsidered. Thus, the purpose of this study was to assess (1) if there is a difference in microshear bond strength (MSBS) among resin-based sealer cements and (2) whether sealer cements behave differently in thin and thick films. The MSBS test was used because it allows the measurement of the sealer-dentin bond rather than the overall bond between dentin, sealer, and core material.

Materials and Methods

Tooth Specimen Selection and Preparation

The study protocol was approved by the Human Research Ethics Committee, University of Melbourne, Australia. Human single-rooted premolars extracted for orthodontic reasons were sectioned below the cementoenamel junction with a diamond wafering blade (Struers, Ballerup, Denmark). The root was then split longitudinally in a buccolingual direction, and the inner surface of each root was ground until it was smooth and flat using 1,000-grit silicon carbide paper. The cut surface was pretreated with 1% NaOCl and 17% EDTA solutions for 5 minutes each to remove the smear layer followed by 15 minutes of ultrasonication in distilled water. A total of 45 root halves were randomly allocated into three groups (using a random numbers table) for MSBS testing. Each prepared root half was placed face down onto a glass cover slip and carefully stabilized with sticky wax. A plastic cylinder with a diameter of 1.5 cm and a height of 1.5 cm was placed over the tooth specimen, and a thick mix of dental stone was poured into the cylinder to cover the tooth specimen, leaving the flat face exposed (Fig. 1). After the stone was set, the entire unit was placed in a 37°C incubator with 95% humidity for 24 hours before testing.

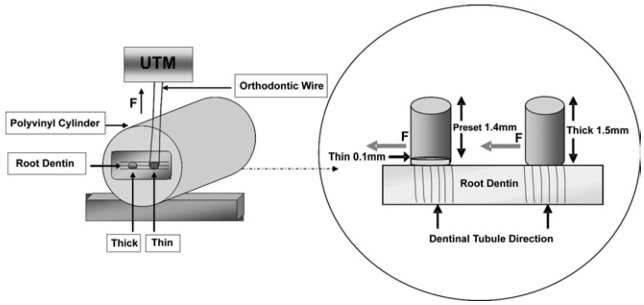

Figure 1. Schematic illustration of the microshear bond test apparatus. Thick and thin samples were bonded onto the same root dentin perpendicular to the dentinal tubules. An orthodontic wire loop engaged around each sample and shearing forces were produced by the universal testing machine (UTM).

Sealer Cements and Bonding

Three resin-based sealers (AH Plus, RealSeal, and EndoREZ; Ultradent, South Jordan, UT) were mixed according to manufacturers' instructions and introduced into polyvinyl chloride (PVC) tubings with an internal diameter of 1.8 mm (2.54 mm^2 bonded surface area) and a height of 1.5 mm. For the thin specimen preparation, the tubing was filled with approximately 1.4 mm of sealer to allow for subsequent bonding to dentin. The UDMA-based sealer groups were immediately placed in a nitrogen chamber for 2 hours to ensure they set without the presence of inhibiting oxygen. All resin-based sealer samples were then placed in a 37°C incubator with 95% humidity and allowed to set for 48 hours. After this, a thin sealer layer of approximately 100 μm was applied to the preset sealer, and the specimens were randomly bonded (using a random numbers table) to either midroot or coronal aspects of the root dentin blocks (Fig. 1). A pilot study showed no regional effect on bond strengths between the coronal and middle third of root dentin. A 40-g load was applied to each PVC tube to allow initial adhesion. For the thick samples, the empty tube was placed on the dentin surface and then entirely filled with resin sealer (Fig. 1). The UDMA-based thin and thick sealer samples were placed in a nitrogen chamber for 2 hours. All samples were then placed in a 37°C incubator with 100% humidity and allowed to set for 1 week.

Adhesive Strength Testing

Specimens were mounted for MSBS testing using a universal testing machine (Mecmesin; Imperial 1000, West Sussex, UK). A wire loop prepared from orthodontic stainless steel ligature wire (260 μm diameter) was placed around the sample adjacent to the dentin surface. The test was run at a crosshead speed of 1 mm/min, generating a shear stress at the sealer-dentin surface (Fig. 1). The maximum load (N) for specimen debonding was recorded and converted to MSBS (MPa). Data were analyzed using analysis of variance with sealer type and thickness as independent variables. Outliers were excluded from the analysis. Post hoc pair-wise comparisons were performed using Tukey multiple comparisons. A $\log_{10}$ transformation of data was performed before the analysis of variance to ensure normality of distribution. Statistical significance was set at $p < 0.05$.

Microscopic and Scanning Electron Microscopy Observation

After microshear bond testing, all specimens were examined under light microscopy (Leica DML; Ernst-Leitz-Strasse, Wetzler, Germany) at 20× magnification. The failure mode was recorded as adhesive, mixed (cohesive and adhesive), or cohesive. Representative samples of debonded specimens (dentin surface) were mounted on stubs, sputter coated with gold, and examined by using a scanning electron microscope (SEM; Philips XL 30 FEG, Eindhoven, The Netherlands).

Results

Highly significant differences were found with respect to both sealer type ($p < 0.001$) and film thickness ($p < 0.001$) (Fig. 2). Overall, the epoxy resin sealer (AH Plus) showed a higher MSBS than either of the two UDMA resin-based sealers (EndoREZ, $p < 0.001$ and RealSeal, $p < 0.01$), and RealSeal showed a significantly higher MSBS than EndoREZ ($p < 0.01$). MSBS was significantly greater for the thick film rather than the thin film specimens ($p < 0.001$). Comparisons for each sealer showed significant differences between thick and thin film MSBS (AH Plus, 6.05 ± 2.24 vs 2.04 ± 1.23 MPa, $p < 0.001$; RealSeal, 3.70 ± 1.82 vs 1.39 ± 0.95 MPa, $p < 0.001$; EndoREZ, 1.77 ± 1.14 vs 0.69 ± 0.37 MPa, $p < 0.01$).

Failure Mode and SEM Observation

In the thin film groups, no failures were observed at the preset sealer-sealer interface; all failures were at the sealer-root dentin interface. Under light microscopy, 70% of all specimens examined had undergone mixed (both adhesive and cohesive) failure. However, on SEM observation of the dentin surface, more detail of the failure mode was visible. In the thin-layer specimens, there was evidence of a residual layer of resin and filler particles over the dentin surface. In the AH Plus thin-layer group, the dentin surface was covered by a very thin layer of resin with filler particles embedded in it; many of the larger filler particles had no visible resin attached (Figs. 3*A* and *B*). Some tubules contained resin, whereas others were empty. RealSeal contained large flat plate-like filler particles, many greater than 10 μm across, overlying the dentin surface and with only a small quantity of irregular resin matrix covering the plates (Figs. 3*C* and *D*). Where the dentin surface was exposed, tubules often contained resin tags sheared off at the dentin surface.

Discussion

Root canal filling typically involves the use of a core material plus sealer cement, and it is widely considered that the sealer layer should be as thin as possible to minimize dissolution when in contact with tissue

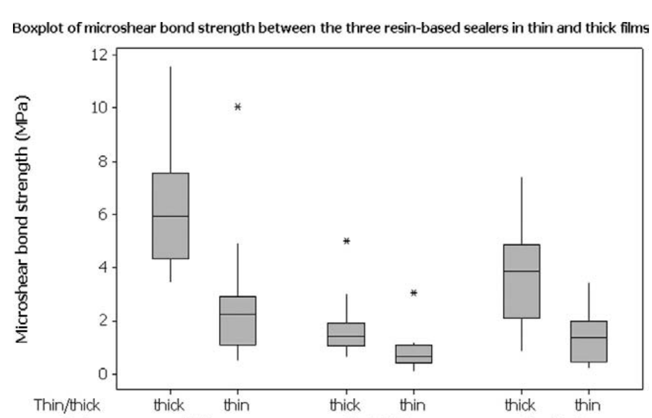

Figure 2. Shear bond strength of the three resin-based sealer groups in thin and thick films. Asterisks indicate outliers (individual values more than 1.5× interquartile range). Overall, the epoxy resin sealer (AH Plus) showed a higher shear bond strength than either of the two UDMA resin-based sealers (EndoREZ, $p < 0.001$ and RealSeal, $p < 0.01$). RealSeal showed a statistically higher shear bond strength than EndoREZ ($p < 0.01$). The thick film specimens showed a statistically higher shear bond strength to root dentine when compared with the thin film specimens ($p < 0.001$).

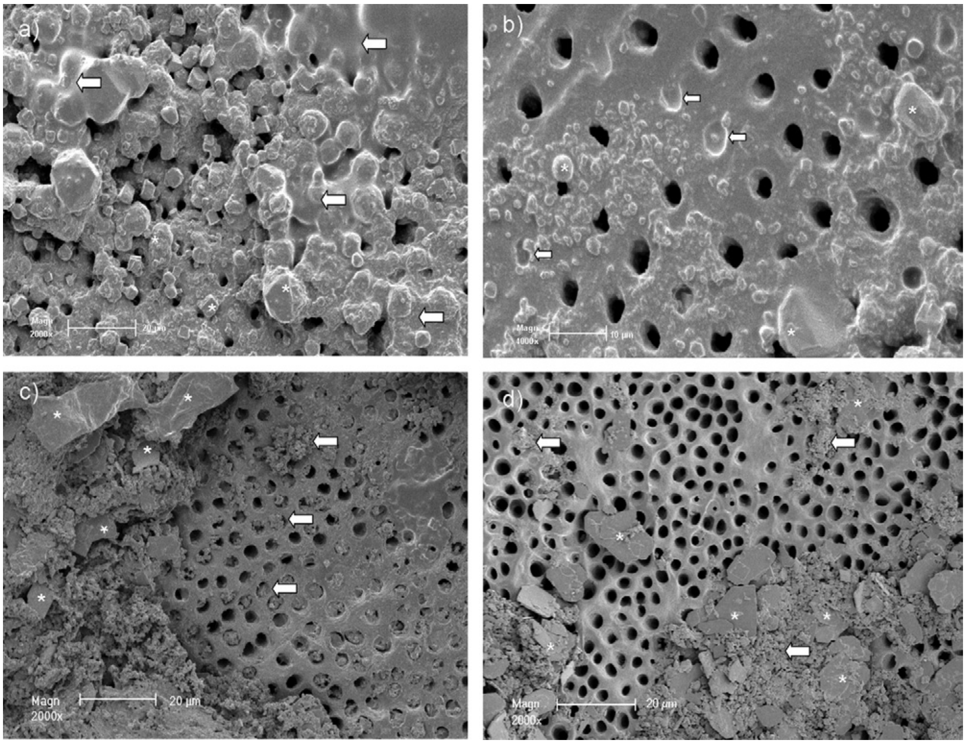

Figure 3. (*a*) SEM image of AH Plus in thin film showed a resin depleted layer with various sizes of spherical shaped filler particles. *Indicates filler particle, arrow is for resin. There appears to be an uneven distribution of resin overlying the filler particles. The sealer layer is depleted of resin (2,000× magnification). (*b*) SEM image of AH Plus in thin film showing the dentinal tubules partially filled with resin and the presence of resin tags (4,000× magnification). (*c*) SEM image of RealSeal in thin film showing the gritty irregular resin layer covering the plate-like filler particles over the dentin surface. Notice the filler particles are aligned parallel to the dentin surface, predisposing to shear failure (2,000× magnification). (*d*) SEM image showing large plate-like filler particles at the interface when RealSeal was applied as a thin layer. Some tubules are filled with resin, whereas most have either lost the resin tags during testing or were not filled when the sealer was applied (2,000× magnification).

fluids (18, 19). With resin-based sealers, sealer penetration into tubules is very extensive (20-23); more precisely, the resin component penetrates tubules, whereas filler particles, which are mostly too large to enter tubules, remain at the interface (15). This depletion of resin from the interfacial layer was proposed as a reason for the low bond strength as measured by the push-out test (15). Filling the entire canal space with sealer is not recommended clinically and was used strictly for experimental purposes. It provides a large reservoir of resin at the interface. As a result, bond strength increased substantially (15).

A conventional root filling has two interfaces: between dentin of the canal wall and the sealer and between the sealer and core material. Experimentally, it is difficult to measure the bond strength of the two interfaces separately (24). To investigate further the effect of resin depletion on bond strength of sealer to dentin, the MSBS test was used in this study. To achieve the equivalent of a thick sealer layer with adequate material to prevent depletion of the resin at the interface, sealer was added in bulk (1.5 mm thick) to the PVC cylinder placed on the dentin surface before polymerization. To create the equivalent of a thin sealer layer, a 1.4-mm thickness of sealer (preset) was first polymerized separately within the tubing and then cemented to the dentin surface by placing a thin layer of fresh material on the preset surface. During subsequent MSBS testing, no instances of failure of the sealer-sealer bond were observed.

As in the previous study (15), the resin-based sealers showed significantly higher shear bond strengths when bonded in thick rather than in thin films. This result occurred regardless of whether the resin was epoxy based (AH Plus) or UDMA based (EndoREZ and RealSeal). Despite the absence of a hybrid layer with epoxy resin, MSBS was highest in this group for both thick and thin layers. Shear and push-out bond strengths of UDMA-based sealers have been variously reported to be higher (25) or lower (9, 12-16, 24, 26, 27) than epoxy-based sealers.

The type, size, and shape of filler particles may also influence the bond strength of the different sealer types (17, 27). The large plate-like structure of RealSeal filler particles appeared to align in layers that were parallel to each other and the dentin surface, possibly creating cleavage planes that readily fail in shear mode (Fig. 3*C*). Also, when the filler particle size is larger than the dentinal tubule diameter, only the unfilled resin component is able to penetrate the tubules. Incorporating nanofillers into future sealers may help enhance the bonding between the sealer and dentin (28).

Conclusions

Overall, the epoxy resin-based sealer had the highest shear bond strength when compared with UDMA-based sealers. The bond strengths with a thick sealer layer were significantly higher than when the sealer was present as a thin film. Further development of resin-based sealer cements should take into account the penetration by resin into dentinal tubules.

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