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The effect of resin-based sealers on fracture properties of dentine

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Abstract

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Aim To determine whether resin-based sealer cements are able to strengthen root dentine, as measured by work of fracture (*Wf*), micro-punch shear strength (MPSS) and resistance to vertical root fracture (VRF).

Methodology One hundred and twenty extracted premolar teeth were randomly assigned amongst four treatments before testing: intact, root canals prepared but unfilled, or root filled using epoxy- or urethane dimethacrylate (UDMA)-based sealer (plus core material). Samples were then prepared for measuring *Wf*, MPSS or VRF using standard test procedures. Data were analyzed using one-way ANOVA with significance set at $P < 0.05$.

Results For all three tests, root canals filled using epoxy resin-based sealer were not statistically significantly different compared with UDMA resin ($P = 1$ for *Wf*, $P = 0.7$ for MPSS and $P = 0.12$ for VRF), or different from both sound and prepared dentine ($P > 0.05$). There was also no significant difference between sound dentine and prepared dentine for both *Wf* ($P = 0.92$) and resistance to VRF ($P = 1$).

Conclusions Neither epoxy nor UDMA resins used as sealer cements enhanced fracture resistance of root dentine when placed within root canals of extracted teeth.

Keywords: micro-punch shear strength, sealer cement, vertical root fracture, work of fracture.

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Introduction

Vertical root fracture is one cause of failure that can occur during or after root canal treatment. The increased susceptibility to fracture results from the cumulative loss of tooth structure from caries, trauma and restorative and endodontic procedures rather than from changes in the properties of dentine after root canal treatment (Sedgley & Messer 1992). Endodontic irrigants (NaOCl, EDTA) may affect the mechanical properties of dentine specimens after prolonged exposure. Many studies have reported a decrease in micro hardness of dentine specimens treated with NaOCl only (Slutzky-Goldberg *et al.* 2004), EDTA only (Cruz-Filho *et al.* 2001, Eldeniz *et al.* 2005), or both (Ari *et al.* 2004). However, an effect on fracture resistance of the root after chemomechanical preparation of the canal space has not been demonstrated.

Conflicting reports have been published as to whether the strength of roots (as measured by resistance to experimental root fracture) could be restored after canal preparation and root filling with a core material and sealer. Neither glass ionomer cement (Apicella *et al.* 1999, Johnson *et al.* 2000) zinc-oxide eugenol based sealer (Apicella *et al.* 1999, Cobankara *et al.* 2002), nor epoxy resin-based sealer (Lertchirakarn *et al.* 2002, Zandbiglari *et al.* 2006) were able to strengthen endodontically treated roots significantly, although contradictory results have been reported for glass ionomer sealer (Cobankara *et al.* 2002, Lertchirakarn *et al.* 2002) and epoxy resin-based sealer (Cobankara *et al.* 2002). Recently, it has been reported that a new resin sealer (Resilon® Pentron, Wallingfort,

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CT, USA) may possibly strengthen the root (Teixeira *et al.* 2004, Hammad *et al.* 2007, Schäfer *et al.* 2007).The Resilon® system consists of a self-etching primer, urethane dimethacrylate (UDMA)-based sealer and polycaprolactone core material, which are claimed to create a 'monoblock' in which the sealer is bonded to both the canal wall and the core material. Contradictory results have also been reported in relation to a strengthening effect of methacrylate based resins (Carvalho *et al.* 2005, Stuart *et al.* 2006, Wilkinson *et al.* 2007).

Several studies have documented the extensive penetration of resin-based sealers into dentinal tubules (Weis *et al.* 2004, Bergmans *et al.* 2005, Mamootil & Messer 2007, Patel *et al.* 2007). The resin penetrates not only the main tubules but also tubule branches (Mamootil & Messer 2007). The aim of this study was to investigate whether the extensive resin infiltration is able to enhance the fracture resistance of dentine and hence to 'reinforce' the root against fracture. The hypothesis tested in the study was that epoxy (AH Plus™, Dentsply DeTrey, Konstanz, Germany) and UDMA (Resilon®) resins used as sealer cements are able to strengthen root dentine, as measured by work of fracture (*Wf*, the work required to form a new surface of unit area), micro-punch shear strength (MPSS) and resistance to vertical root fracture (VRF).

Materials and methods

General methods

Teeth

Single canal premolar teeth extracted for orthodontic reasons from patients aged between 14 and 20 years old were kept in 1% chloramine T (pH = 7.8) (Sigma-Aldrich Co., St. Louis, MO, USA) at 4 °C until use. The teeth were obtained under a protocol approved by the Human Research Ethics Committee, University of Melbourne, Australia. All teeth were examined under light microscopy (Leica DML, Leica Microsystems Wetzlar GmbH, Wetzlar, Germany) to rule out cracks or other defects. In the first two experiments, teeth were randomly assigned to groups using a random numbers table; in part 3, teeth were assigned to groups using stratified randomization.

Canal preparation

Canals were prepared at working length 0.5 mm short of the patency length using 0.04 taper ProFile (ProFile® Dentsply Tulsa Dental, Johnson City, TN, USA) to master apical rotary (MAR) size 35–45, which was three sizes larger than the first instrument binding at working length. Sodium hypochlorite (1% NaOCl, 1 mL) was used to irrigate each canal after every instrument, using a 27 gauge irrigating needle. After completion of preparation, the canals were rinsed with 5 mL 15% ethylenediamine tetraacetic acid (EDTA). A final rinse of 5 mL distilled water was used to remove any residual irrigating solution. All irrigants were left in the canal for 5 min. Canals were dried using paper points. The teeth were kept moist at all times by wrapping them in saline-soaked gauze.

Root filling procedures

A 0.04 tapered master cone matched to the final MAR instrument was used. For AH Plus™ (Dentsply DeTrey, Konstanz, Germany), the master cones were 0.04 taper gutta-percha. For Resilon® (RealSeal®, SybronEndo, Glendora, CA, USA), the 0.04 Resilon® core material was used as recommended. Sealers were prepared according to manufacturers' instructions. AH Plus™ was mixed using the AH Plus Jet® mixing system, and then introduced into the root canal orifices with the intraoral tip. For the Resilon® group, after the canal was dried with paper points, the self etch Resilon primer® was placed into the root canal system to the working length with a microbrush, allowed to soak for 30 s, and excess primer was removed with a dry paper point. The Resilon sealer (RealSeal®) was mixed by the auto mix syringe. The sealer was inserted into the canal using a paste filler (FKG Dentaire, La Chaux-de-Fonds, Switzerland); the master cone was also lightly coated with sealer and seated to working length in a slow plunging motion. At the completion of filling, all samples were immediately placed in a nitrogen chamber for 2 h to ensure that the methacrylate-based sealers had set without the presence of inhibiting oxygen. Without this step Resilon® failed to set. All samples were then stored at 37 °C and 100% humidity for 48 h to allow the sealer cements to set completely.

Part 1. Work of fracture

Forty teeth were used in this experiment. Ten teeth were randomly selected as controls, and canals of the remaining 30 teeth were prepared as described above. The prepared teeth were then divided randomly into three groups each of 10 teeth, for root filling using the two different resin sealers, or with canals left unfilled.

All teeth were then prepared for the work of fracture (*Wf*) test. The tooth was decoronated 2 mm below the

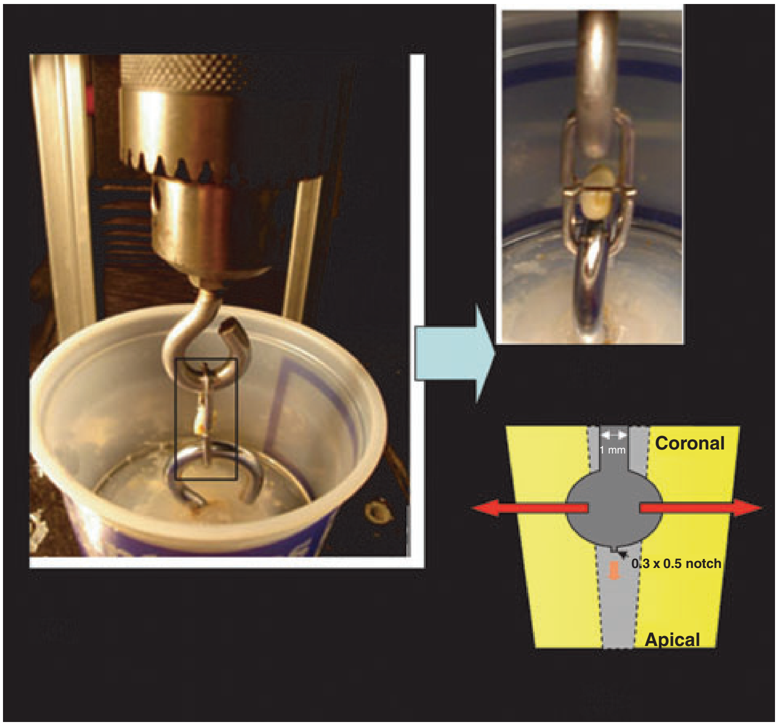

Figure 1 The mounting system for testing root dentine specimens for work of fracture (*Wf*). (a) The water bath and couplings for attaching the samples were mounted on the Instron machine. (b) Stiff hooks were inserted into a groove cut part way into the root sample, which were then connected to the couplings to pull the sample apart. (c) Schematic illustration the direction of fracture of root segment in coronal to apical direction.

cemento-enamel junction (CEJ), and a 5 mm long section from the coronal third was cut using a slow speed diamond saw (Struers, Ballerup, Denmark). A 1 mm wide groove was cut in a mesio-distal direction on the coronal surface to a depth of 2 mm, through the canal space (Fig. 1b). A notch was then created on each site of the groove to accommodate the mounting jig. A further notch 0.3 mm wide and 0.5 mm deep was made at the base of the groove to guide the direction of fracture through the canal space (Fig. 1c).

The sample was mounted in a water bath at 37 °C. The water bath and the mounting for holding the root section were installed on an Instron universal testing machine (Instron model 5544, Instron Corp, Canton, MA, USA) with two stiff hooks inserted into the grooves (Fig. 1a). The test was run at a displacement of 0.2 mm min^{-1} until the sample split. The load-displacement data were recorded and adjusted for compliance of the system. The fracture surface was photographed under light microscopy (Leica DML, Leica Microsystems Wetzlar GmbH, Wetzlar, Germany) and the surface area measured using image analysis software (UTHSCSA Image Tool version 3, San Antonio, TX, USA). The energy required for fracture was calculated by the area under the load-displacement graph as described by Kahler *et al.* (2003). *Wf* was calculated in J m^{-2}.

Representative samples from each group were prepared for scanning electron microscopic examination. The fractured dentine surface was mounted, sputter coated with gold and examined under field emission-scanning electron microscopy [(FE-SEM); Philips XL 30 FEG, Eindhoven, the Netherlands].

Part 2. Micro-punch shear strength

Forty teeth were decoronated at the CEJ using a slow speed diamond saw (Struers) under water coolant. Ten teeth with no canal preparation served as a control (intact tooth). The canals of 30 teeth were instrumented as described before; the teeth were then randomly divided into three groups of 10 teeth each for filling using the two resin sealers or remaining unfilled (prepared canal only).

A 200 μm cross section was cut from the coronal third of the root with a diamond saw. Each sample was lapped and polished with silicon carbide paper in series, using 1200, 2400 and 4000 grit until the thickness was approximately 100 μm as measured with a digital caliper to the nearest 0.001 mm. The tooth slices were then stored in a moist (90% humidity) environment until the micro-punch shear test.

A micro-punch shear apparatus was modified from the micro-punch shear apparatus described by Sedgley & Messer (1992), using a punch 0.3 mm in diameter. The dentine slice was positioned over the central hole in the lower die. The micro-punch test was conducted in buccal and palatal locations, in inner dentine approximately 0.5 mm from the root canal space (measured with a microscope and measuring grid), where resin infiltration of tubules is normally seen (Jainaen *et al.* 2007, Mamootil & Messer 2007). A minimum of four punches were made in each section.

The upper die was placed over the lower die and secured with screws so that the dentine slice was constrained, using appropriate force to protect the tooth slice from damage. The micro-punch apparatus was then mounted on the Instron universal testing machine, and a load was applied to the punch at a constant crosshead speed of 0.1 mm min^{-1} until perforation occurred. MPSS was calculated by the equation:

$$\text{Shear strength} = \text{Force}/\pi \times \text{diameter} \times \text{thickness}$$

Force is in Newtons, diameter of the punch and thickness of specimen are in mm, MPSS is in MPa.

Part 3. Resistance to vertical root fracture

Forty single canal premolars were selected. The crown was resected at the CEJ, and bucco-lingual width at the coronal end was measured with a caliper. From bigger to smaller, all roots were then sequentially assigned to four groups. The first group, with no preparation, served as intact dentine. The remaining 30 roots were then prepared as described above to master apical file size 40–45. The second group (no root filling) served as prepared dentine. The third group was root filled with AH Plus™ and gutta-percha. The fourth group was root filled with RealSeal® and Resilon® core material.

Each root was cut to a standard length of 10 mm. The root segment was then embedded vertically in polysiloxane putty (Coltene, Whaledent Inc, Akron, OH, USA). A cylindrical hardened steel rod (3.2 mm diameter) with a sharpened conical tip was attached to the upper part of the Instron universal testing machine, with the tip centred in the canal orifice. A vertical load was applied at a crosshead speed of 1 mm min^{-1} until the root split. The maximum load at the time of fracture was recorded in Newtons.

Statistical analysis

The data for *Wf*, MPSS and resistance to VRF were analysed by one-way ANOVA (Minitab Inc, State College, PA, USA), and *post hoc* pair-wise comparisons were performed using Tukey multiple comparisons. For each outcome statistical significance was set at $P < 0.05$.

Part 4. Properties of sealer materials

Compressive strength, tensile strength and work of fracture of the two set sealer materials were measured. Five samples each for compressive and tensile tests were prepared and tested according to an International Standard test (ISO (9917-1:2003(E)) (2003). Five samples for work of fracture were prepared with the same dimensions as dentine samples (5 × 3 mm), and then tested as for the tooth samples. The modulus of elasticity was derived from compressive strength graphs.

Table 1 The effect of resin-based sealers on work of fracture (*Wf*), MPSS and resistance to VRF of all groups are shown in mean ± SD, $n = 10$. AH Plus™/gutta-percha had no statistically significant effect on *Wf* compared to Resilon®/RealSeal® ($P = 1$), or intact and prepared dentine ($P < 0.05$). The MPSS of roots filled with epoxy resin (AH Plus™) was not different from that of roots filled with UDMA-based resin (Resilon®), or sound dentine, $P = 0.7$. No significant differences were found amongst resistance to VRF of four groups ($P = 0.12$)

Groups	Work of fracture (J m^{-2})	MPSS (MPa)	Resistance to VRF (*n*)
Intact dentine	351.8 ± 192.2	67.9 ± 21.6	442.8 ± 108.7
Prepared dentine	301.2 ± 120.0	65.7 ± 24.0	410.4 ± 79.7
AH Plus™/gutta-percha	255.8 ± 109.5	63.3 ± 11.9	450.6 ± 170.3
Resilon®/RealSeal®	280.8 ± 150.3	73.9 ± 17.9	437.2 ± 71.9

Results

The mean values for *Wf*, MPSS and resistance to fracture for all AH Plus™ and Resilon®-filled roots, prepared dentine and sound dentine are shown in Table 1. A square root transformation of data was performed before the ANOVA to ensure normality of distribution. Neither epoxy resin-based nor UDMA-based sealer had a statistically significant effect on *Wf*, compared with both sound and prepared dentine ($P > 0.05$). There was no significant difference between sound dentine and prepared dentine ($P = 0.92$) (Table 1). The MPSS of dentine of roots filled with epoxy resin (AH Plus™) was not different from that of roots filled with Resilon®, or from sound or prepared dentine, $P = 0.7$ (Table 1).

The mean values of the loads required to split roots vertically for the different groups ranged from 410 to 451N. Despite attempts to standardize root size as much as possible, the large variation within each group precluded the demonstration of any statistically significant differences amongst groups ($P = 0.12$) (Table 1). Canal preparation did not significantly reduce fracture strength and use of resin sealers did not increase it ($P = 1$).

Under FE-SEM, fractographs of canals filled with AH Plus™ and Resilon® showed an irregular pattern of the fracture surface, starting from inner dentine along the dentinal tubules. It left step formations with different levels parallel to dentinal tubule direction. The fracture surface showed clear dentinal tubules parallel to the fracture surface (Figs 2 and 3). Variable numbers of resin tags were present in the Resilon® -filled specimen (Fig. 2), but were abundant in the AH Plus™ group (Fig. 3a). At higher magnification in the AH Plus™ group, collagen fibrils of the dentine matrix remained attached to the resin in the tubule after the root was split, suggesting the strong bond between resin and dentine (Fig. 3b).

The physical properties of the two materials are shown in Table 2. Resilon® showed a significantly higher *Wf* than AH Plus™ ($P < 0.001$); however, there was no difference between the two materials in

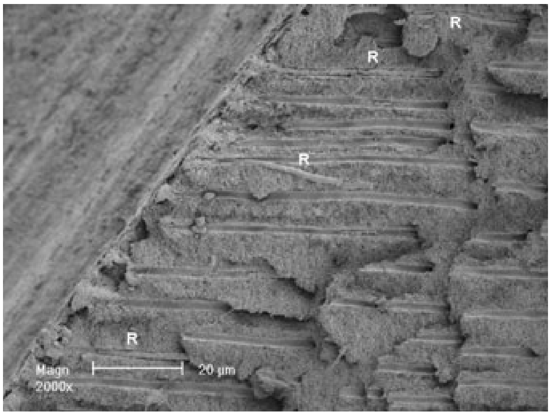

Figure 2 Field emission-scanning electron microscopy (FE-SEM) micrograph of the fracture surface of a sample obturated with Resilon®. An irregular pattern of fracture surfaces were observed. Few dentinal tubules were seen with resin tags left in. R shows resin tags.

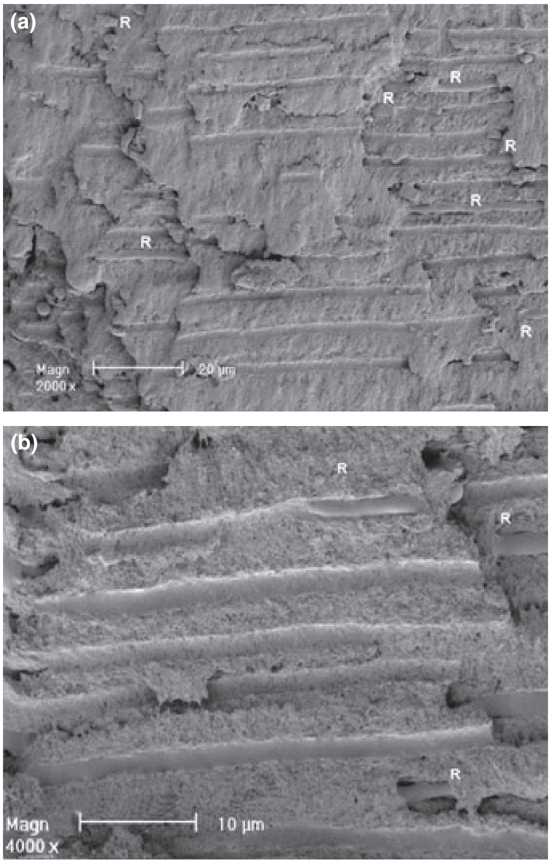

Figure 3 (a) Field emission-scanning electron microscopy (FE-SEM) micrograph of the fractured surface of a sample obturated with AH Plus™, after fracture parallel to tubule direction. An irregular pattern of fracture surfaces was observed. The canal wall is to the left of the specimen. Most tubules are filled with resin. (b) FE-SEM micrograph at higher magnification of AH Plus™ group. Several of the tubules have a layer of dentinal matrix overlying the tubules, indicating that the fracture occurred within orthodentine rather than through the tubule.

Table 2 The physical properties of AH Plus^{TM} and Resilon$^{®}$ are shown. The work of fracture of Resilon$^{®}$ was significantly higher than that of AH Plus^{TM} ($P < 0.001$)

Physical property	AH Plus^{TM}	RealSeal$^{®}$	Dentine
Compressive strength (MPa)	66.40 ± 17.66	72.45 ± 15.10	297**
Tensile strength (MPa)	16.67 ± 3.13	18.32 ± 1.74	51.7**
Work of fracture (J m^{-2})	27.82 ± 15.60*	147.62 ± 32.02*	351.8 ± 192.2***
Modulus of elasticity (GPa)	0.31 ± 0.15	0.24 ± 0.01	10–20**

*Statistically significant difference.
**Data from (Craig 1989) and (Kinney *et al.* 1999).
***From Table 1.

compressive and tensile strengths. Data for dentine are also presented from the literature (Craig 1989, Kinney *et al.* 1999) and Table 1, and show that both sealers are substantially weaker than dentine.

Discussion

Even though many studies have reported a decrease of micro hardness of root dentine after experimental exposure to root canal irrigants (Cruz-Filho *et al.* 2001, Ari *et al.* 2004, Slutzky-Goldberg *et al.* 2004, Eldeniz *et al.* 2005), this study, on the other hand, found no difference in fracture properties of prepared root dentine compared with sound dentine. Although NaOCl and EDTA can soften exposed dentine surfaces after prolonged exposure, the effect may be superficial and limited to the canal wall when irrigants are used under clinically realistic conditions. The length of time that dentine is left exposed to NaOCl as well as the concentration is crucial for breakdown of protein structure caused by alkalinity of NaOCl (White *et al.* 2002). The lack of an effect of cleaning and shaping procedures on *Wf* indicates that the dentine is not significantly weakened by these procedures.

Several studies have reported that the force required to split roots is higher when the canal is obturated with Resilon$^{®}$ than when AH Plus™ is used (Teixeira *et al.* 2004, Hammad *et al.* 2007, Schäfer *et al.* 2007). This study, on the other hand, found no difference in resistance to VRF in canals obturated with AH Plus™ or Resilon$^{®}$ and no difference compared with intact and prepared dentine. This result agrees in part with that of Sagsen *et al.* (2007) who reported no difference between roots filled with either Resilon$^{®}$ and AH 26$^{®}$, although both were higher than prepared dentine. This result corroborates previous studies which reported that neither Resilon nor gutta-percha reinforced root dentine against horizontal fracture (Stuart *et al.* 2006, Wilkinson *et al.* 2007).

The present study also measured work of fracture (*Wf*), which is defined as the work required to form a new surface of unit area (Tattersall & Tappin 1966), as a property of the dentine itself. Only sound dentine has been investigated previously (Rasmussen & Patchin 1984). The epoxy resin-based sealer had no statistically significant effect on *Wf* compared with both sound and prepared dentine ($P > 0.05$), or with canals filled using UDMA-based resin. Similarly, no difference in MPSS was found between the two resins, when inner dentine was tested as the location with the greatest volume of resin within the tubules.

Despite the absence of any observable reinforcing effects of resin infiltration on the fracture properties of dentine in this study, and the variable results on simulated vertical root fracture, the potential for a beneficial effect should not be underestimated. In a previous study, it was concluded that tubule penetration was limited to the resin component of the sealer cement, whilst most filler particles were too large to enter the tubules (Jainaen *et al.* 2007). Thus, the strength of unfilled resins is likely to be much lower than that of filled resins. The use of a nano-filled resin with greater potential for tubule penetration could be a productive approach to increasing strength and fracture toughness of root dentine. To create a stronger bond, more predictable tubule penetration by thorough removal of the smear layer, optimization of resin flow and penetration, and modification of the tubule wall to enhance bonding may also result in improved fracture resistance. It is clear (Fig. 3b) that epoxy resin is capable of strong binding to the tubule wall such that fracture occurs through adjacent intertubular dentine. Thus, the potential for reinforcing dentine is already apparent.

For a root filling material to reinforce roots, the strength and modulus of elasticity of both core material and sealer would need to approximate that of dentine (as well as bond strength). Cohesive strength (the tensile stress when a material begins to flow or break) and modulus of elasticity (or stiffness) of the core materials gutta-percha and Resilon are relatively low (Williams *et al.* 2006, Grande *et al.* 2007). Resilon and gutta-percha core material both demonstrated physical properties of elastomeric polymers, which will not resist stress but will undergo flow or elongate. Both materials have cohesive strength and modulus of elasticity values that are too low to reinforce the roots of root filled teeth. As sealer is needed to bond core material to root dentine, the physical properties of sealers should also be similar to those of root dentine. Compressive strength, tensile strength, elastic modulus and work of fracture were measured for the two resin sealers AH Plus™ and RealSeal® (Table 2). The work of fracture, compressive and tensile strengths of both materials are not different and are very low in comparison with dentine. The modulus of elasticity is also low in comparison with dentine, but similar to that of core materials. Thus, alternative strategies to reinforce root dentine should be considered, as currently available materials do not have the necessary physical properties to achieve a strengthening effect.

Conclusion

Neither epoxy- nor UDMA-based resins used as sealer cements had the effect of enhancing fracture properties of root dentine compared with intact dentine.

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