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Enhancing the bond of a resin-based sealer to root dentine

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Abstract

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Aim To evaluate whether application of a total-etch/separate adhesive layer can enhance the bond of a UDMA-based sealer to dentine.

Methodology The root canals of 20 decoronated maxillary premolar teeth with two canals were prepared to size 35–45, 0.04 taper using rotary NiTi instruments. The canals of each tooth were treated with application of either a total-etch/separate adhesive or the manufacturer-recommended primer, before root filling with sealer plus matching master cone using warm vertical compaction. After setting, roots were sectioned perpendicular to the long axis to obtain 1-mm-thick slices, and the root filling was subjected to the push-out test using a plunger closely matched to canal diameter. The roots of another 15 single-rooted premolars were sectioned in a bucco-lingual direction, and the cut surfaces were ground flat for microshear bond strength testing. One-half of the specimens were coated with a separate total-etch/adhesive layer, and then, a cylinder of sealer cement 1 mm diameter × 1.5 mm high was bonded to the prepared surface of all specimens. Microshear bond strength was measured in a universal testing machine after 48 h. Data were analysed using ANOVA and paired *t*-tests, with significance set at $P < 0.05$.

Results Use of a separate total-etch/adhesive markedly increased both microshear bond strength and push-out strengths compared with standard primer ($P < 0.001$).

Conclusion Application of a separate adhesive layer significantly increases bond strength of UDMA-based sealers to root dentine.

Keywords: microshear bond strength, push-out bond strength, Resilon®, total-etch/separate adhesive layer.

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Introduction

The concept of bonding within the root canal and the development of urethane dimethacrylate (UDMA)-based materials as root canal sealers initially came from restorative dentistry, as adhesive dentistry rapidly evolved and gained worldwide acceptance in the 1990s. Laboratory studies of dentine bonding systems have shown that these materials can provide bond strengths to dentine that approach or even exceed bond strengths to enamel (Barkmeier & Erickson 1994). Further development of adhesive materials such as one-step self-etch primer systems meant that the number of steps used was reduced (Vargas *et al.* 1997, Van Meerbeek *et al.* 2003), whilst dual- and self-cured materials eliminated the need for light curing systems in inaccessible locations such as root canals (Teixeira *et al.* 2004a).

With the introduction of the Resilon® UDMA-based root filling system (Pentron, Wallingford, CT, USA), it was claimed that such materials have several advantages over conventional root fillings, because they provide a superior bond allowing for less microleakage (Shipper *et al.* 2004) and increased fracture

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resistance (Teixeira *et al.* 2004b). However, the idea that Resilon® systems can produce a monoblock (the formation of a single interface between sealer, core and dentine) has been challenged (Tay *et al.* 2006, Jainaen *et al.* 2007, Sly *et al.* 2007, Rahimi *et al.* 2009). To create a monoblock, the bond strengths of sealer to both intraradicular dentine and the core material should be high. However, a number of laboratory-based studies using shear (Hiraishi *et al.* 2005, Rahimi *et al.* 2009) and push-out techniques (Gesi *et al.* 2005, Ungor *et al.* 2006, Fisher *et al.* 2007, Jainaen *et al.* 2007, Sly *et al.* 2007, Üreyen Kaya *et al.* 2008) have shown that the bond of Resilon® systems to root dentine is not superior and in some cases even inferior to other conventional sealers.

Newly developed UDMA-based sealers such as MetaSEAL (Parkell, Inc., Farmington, NY, USA) and EndoREZ® (Ultradent, South Jordan, UT, USA) are self-adhesive, which eliminates the use of a separate self-etching primer to create an initial bond to root canal wall dentine. In other UDMA-based filling systems such as Resilon®, following application of Resilon® self-etch primer, the manufacturer recommends direct application of the Resilon® sealer. The use of a total-etch/separate bonding agent may assist with tubule penetration and allow polymerization before the sealer cement is applied, which may help to improve the bond strength of Resilon® systems to dentine.

Previous work has indicated that the thickness of a sealer cement layer is often of the order of 10–20 μm (Weis *et al.* 2004). The behaviour of this thin layer is different from that of sealer in bulk (Jainaen *et al.* 2007, Rahimi *et al.* 2009). Extensive sealer penetration into tubules leaves the interfacial area depleted of resin, resulting in a weaker bond strength (Jainaen *et al.* 2007). Therefore, this study investigated whether the bond strength between sealer and dentine might be enhanced following application of a separate adhesive layer to seal the tubules before placing the sealer. The null hypothesis tested was that application of total-etch/separate adhesive layer does not increase push-out and microshear bond strengths (MSBS) of a UDMA-based sealer to root canal dentine.

Materials and methods

Push-out testing

Instrumentation and obturation

Twenty maxillary premolar teeth extracted for orthodontic reasons with intact crowns and two separate canals were used. All teeth were stored in 1% chloramine T (pH 7.8) (Sigma-Aldrich Co., St Louis, MO, USA) at 4 °C until use. The experimental protocol was approved by the Human Research Ethics Committee, University of Melbourne, Melbourne, Australia. Each tooth was decoronated at the cementoenamel junction (CEJ) using a slow speed diamond saw (Struers, Ballerup, Denmark). The working length of each canal was visually determined using a size 10 K-file until it reached the apical foramen, then subtracting 1 mm from this measurement. Forty canals were prepared using the crown-down technique and 0.04 taper ProFile instruments (Dentsply Maillefer, Ballaigues, Switzerland) to master apical rotary sizes 35–45. After preparation, the canals were irrigated with 5 mL of 1% sodium hypochlorite (NaOCl) followed by 5 mL of 17% ethylene diamine tetraacetic acid (EDTA) to remove the smear layer. Finally, each canal was irrigated with 10 mL of distilled water according to the manufacturer's instructions.

For each tooth, buccal and lingual canals were randomly allocated (using a random numbers table) to either manufacturer-recommended self-etch primer and obturation or a total-etch/separate adhesive layer application prior to obturation with the Resilon® system.

For the manufacturer-recommended self-etch primer group, canals were dried with paper points, and each canal was lightly coated with self-etch Resilon primer® using a microbrush. Excess primer was removed after 30 s with a dry paper point. The Resilon sealer (RealSeal®, SybronEndo, Glendora, CA, USA) was mixed using the auto-mix syringe. The canal was filled with a 0.04 taper Resilon® master cone plus Resilon® sealer (RealSeal®). The sealer was inserted into the canal using sizes 35–45 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. For the total-etch/separate adhesive group, the canal was etched for 30 s with ScotchbondTM Multi-purpose etchant (3M ESPE, Dental Products, St. Paul, MN, USA) using a microbrush. Following this, the canal was thoroughly irrigated with distilled water and dried with paper points, and a thin layer of AdperTM Single Bond two adhesive (containing 10% nanofiller, 3M ESPE, Dental Products) was applied to the canal wall. This adhesive was applied with a microbrush and light cured for 30 s using a LED curing light (ElipарTM S10 LED, 1200 mW cm^{-2} intensity) at a distance of approximately 1–2 cm from the most coronal aspect of the root. The rest of the canal filling process was carried out as above. Warm vertical compaction was used in the coronal third of the canals, using the System-B heat source (Analytic Technology, Redwood City, CA, USA) and variable size Schilder pluggers (Thompson EndoPlugger, EndoSolutionsTM, York, UK).

Upon completion of canal 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 (Jainaen *et al.* 2007). All samples were then stored at 37 °C and 100% humidity for 48 h to allow the sealer cements to set completely (following manufacturer's recommendation).

Preparation for push-out testing

The push-out apparatus was modified from a previous study (Jainaen *et al.* 2007). Teeth were sectioned perpendicular to the root canal at low speed with constant water cooling. A 1-mm-thick section was taken from the coronal third of both roots. Both apical and coronal aspects of each sample were examined under the microscope (10× magnification) and photographed before testing to confirm an approximately circular canal shape and that the sealer filled the entire canal space without any voids. A microscope at 10× magnification was used to orient the specimens during loading.

The apical aspect of each slice was marked with an indelible pen and the specimens aligned from an apical to coronal direction over a 1.5-mm-diameter circular hole at the centre of a 10-mm-thick rigid plate. To reduce the possibility of any constriction interference, variable size steel plungers (0.35, 0.5 and 0.8 mm in diameter) were used that provided almost complete coverage over the canal without touching the canal wall.

The plunger was mounted in the upper part of a universal testing machine (MTS Corporation, Eden Prairie, MN, USA). The punch moved downward at a crosshead speed of 0.5 mm min^{-1} until the shear stresses caused the root filling material to debond and be pushed out. The highest value in Newtons (N) was recorded and converted to megapascals (MPa) by taking the average of the two perimeters (coronal and apical) and the thickness of the specimen into account, according to the following formula (Patierno *et al.* 1996):

$$\text{Push-out bond strength (MPa)} = \text{Maximum load (N)} / \text{Adhesion area of root filling (mm}^2\text{)}$$

Microshear bond testing

Tooth specimen selection and preparation

Fifteen single-rooted premolars were used. The crown of each tooth was resected at the CEJ with a sintered diamond wafering blade (Struers). The root was then split longitudinally in a bucco-lingual direction, and the cut surface was ground flat using 1000-grit silicon carbide paper. The specimens were pre-treated with NaOCl and EDTA solutions for 5 min to remove the smear layer and expose the tubules. This was followed by 15 min of ultrasonication in distilled water. A total of 30 root halves were randomly allocated into two groups (using a random numbers table) for specimen bonding.

Preparation for microshear bond testing

Each prepared root half was placed face down onto a glass cover slip (Menzel-Glaser®, Braunschweig, Germany) and carefully stabilized with sticky wax (Kemdent, Swindon, UK). A polyvinylchloride cylinder (PVC) 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 (Die Stone Whip-mix, Henry Schein, Sydney, NSW, Australia) was carefully poured into the PVC tubing to cover the tooth specimen. Resilon sealer (RealSeal®) was prepared according to manufacturer's instructions and introduced into polyethylene (PE) tubes with a diameter of 1.0 mm and a height of 1.5 mm. The MSBS could not be conducted using core material (Resilon®) bonded via sealer cement (RealSeal®) to the dentine surface, because the core material is too weak to withstand the applied stress. To simulate the thin film of sealer present clinically and in the push-out test, preset bulk sealer material was bonded to the dentine surface with a thin layer of fresh sealer. Therefore, the PE tubes were filled with approximately 1.4 mm of sealer and immediately placed into a nitrogen chamber for 2 h to ensure that the UDMA-based sealers set without the presence of inhibiting oxygen. Samples were allowed to pre-set at 37 °C in 100% humidity for 48 h. On one root half, the root dentine was lightly coated with the manufacturer-recommended self-etch Resilon primer® using a microbrush. Excess primer was removed after 30 s with a dry paper point. The Resilon sealer (RealSeal®) was mixed using the auto-mix syringe and a thin sealer layer of approximately 0.1 mm was applied to the 1.4-mm pre-set sealer and specimens were randomly bonded to either mid-root or coronal aspects of the root dentine blocks. On the other root half, the dentine specimen was etched for 30 s with ScotchbondTM Multi-purpose etchant (3M ESPE, Dental Products) using a microbrush, washed with distilled water, and dried with a triplex syringe. A thin layer of AdperTM Single Bond two adhesive (3M ESPE, Dental Products) was applied to the dentine specimen and cured for 30 s using a LED curing light. RealSeal® was then bonded following the same process as above. For all samples, a light load of 40 g was applied to each PE tube to allow for initial adhesion. Specimens were stored at 37 °C in 100% humidity for 48 h.

The specimens were mounted for testing with the universal testing machine (MTS Corporation). A wire loop prepared from an orthodontic stainless steel ligature wire (0.26 mm in diameter) was placed around the sample. The test was run at a crosshead speed of 1 mm min^{-1}, to produce shearing stresses that would debond the sealer from the dentine surface. The maximum load (N) for specimen debonding was recorded and then converted into microshear bond strength (MPa) based on the bonded surface area.

SEM observation

Representative samples of debonded surface from both microshear strength and push-out test were split vertically for SEM examination (method previously described by (Mamootil & Messer 2007, Jainaen *et al.* 2007, Rahimi *et al.* 2009). Samples were mounted on stubs with the canal wall upwards, sputter coated with gold and examined under a field emission-scanning electron microscope (FE-SEM; Philips XL 30 FEG, Eindhoven, The Netherlands). Penetration of sealer into dentinal tubules was also observed under SEM, after the dentine surface was demineralized with a 10 min application of 15% EDTA, followed by 10 min of 5% NaOCl to remove any organic debris (Mamootil & Messer 2007).

Statistical analysis

Both microshear bond and push-out bond strength were analysed using the student paired t-test to compare between Resilon® primer and total-etch/separate adhesive layer. For each outcome, statistical significance was set at $P < 0.05$.

Results

The mean push-out and MSBS values for canals treated with Resilon® primer and total-etch/separate adhesive layer groups are shown in Table 1. The total-etch/adhesive group had a significantly higher push-out strength and MSBS compared to Resilon® primer ($P < 0.001$, student paired t-test), by a factor of four- to sixfold.

Table 1 Comparison of bond strength tests between total-etch/adhesive technique and Resilon® self-etch primer

Group	Microshear bond strength (MPa)	Push-out strength (MPa)
Self-etch[a]	0.7 ± 0.4 n = 15	0.5 ± 0.4 n = 20
Total etch/ adhesive	2.6 ± 0.8[b] n = 15	3.2 ± 2.5[b] n = 20

[a]As recommended by the manufacturer.
[b]Significantly higher than the self-etch group (Student's paired t-test, $P < 0.001$).

SEM observations

Push-out testing

A common finding using FE-SEM was that for the canals filled with Resilon® core material and Resilon® primer, adhesive failure consistently occurred between sealer and the dentine interface (Fig. 1), despite evidence of resin tag formation.

For the canals treated with a total-etch/separate adhesive layer and filled with RealSeal®/Resilon® core material, extensive resin penetration occurred at the sealer–dentine interface (Fig. 2). Failure occurred more commonly between core material and sealer (15 out of 20 sections tested) and there was close contact between sealer and the adhesive layer (Fig. 2).

MSBS testing

The dentine surface after microshear testing showed that some tubules contained resin whilst others were empty, presumably from tags pulling out of the tubules during shear failure (Fig. 3).The size of the filler particles meant that these particles could not enter the tubules and hence a filler-enriched surface was left behind at the interface (Fig. 3). Furthermore, the dentine surface was covered by a gritty layer of resin with nonuniform distribution of large flat plate-like filler particles, many >10 μm across, which tended to lie flat against the dentine surface (Fig. 3).

Discussion

The use of a sealer cement between the core root filling material and the dentine of the canal wall results

Table 2 Composition of materials used in the study

Materials	Components	Batch no.	Manufacturer
ScotchbondTM Multipurpose Etchant	35% phosphoric acid	5FE 0610132	3M ESPE, St. Paul, MN, USA
AdperTM Single Bond 2	Bisphenol-A diglycidyl ether dimethacrylate, HEMA, dimethacrylate, colloidal nanofiller 10%, solvent, water	6JR 184141	3M ESPE, St. Paul, MN, USA
RealSeal®	UDMA, PEGDMA, EBPADMA, Bis-GMA resins, silane-treated barium borosilicate glass, barium sulphate, silica, calcium hydroxide, bismuth oxychloride, peroxide, photo initiator, stabilizer and pigment	9722010	SybronEndo, Glendora, CA, USA
Self-Etch RealSeal® primer	Sulphonic acid, HEMA, water, polymerization initiator	179661	SybronEndo, Glendora, CA, USA
RealSeal® Core	Thermoplastic synthetic polymer (polyester)	117741A	SybronEndo, Glendora, CA, USA

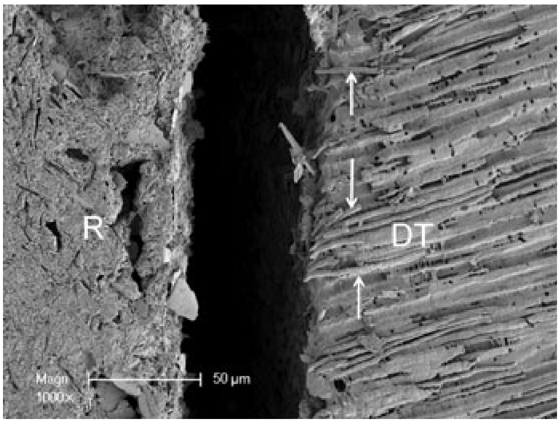

Figure 1 FE-SEM of canal filled with the Resilon® system. Following the push-out test, adhesive failure occurred between sealer and dentine interface, with resin tags infiltrating some dentinal tubules. Arrow, resin tag; R, RealSeal® sealer; DT, dentinal tubules.

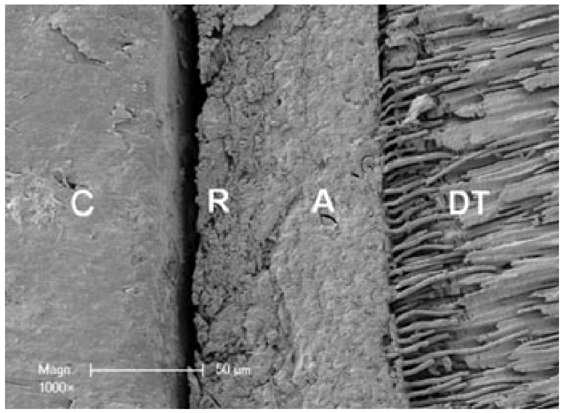

Figure 2 FE-SEM of canals obturated with the total-etch/separate adhesive layer together with Resilon® core and sealer (RealSeal®). Extensive penetration of adhesive occurred at the resin: dentine interface. Failure occurred between Resilon® core material and RealSeal®, with close contact between the sealer layer and adhesive. C, Resilon® core material; R, RealSeal® sealer; A, adhesive, and DT, dentinal tubules.

in two separate interfaces that between the core material and sealer and that between sealer and dentine. This study focussed on the bond between sealer and dentine. Whilst the bond between sealer and core material is also critical to the total bond strength, methods to improve the bond are likely to involve chemical modification of the materials, which is beyond the scope of this study. A potential technique for enhancing the bond strength between sealer and dentine was investigated, using both microshear bond testing and the push-out test. Whilst the push-out test provides a closer simulation of clinical conditions of bonding to the canal wall than microshear bond testing, it does not specifically measure the bond between sealer and dentine. The data from this study show that there was a higher standard deviation of push-out values compared with shear bond values. Previous studies have also indicated that the spread of values and variability in results was high with the push-out test (Gesi *et al.* 2005, Skidmore *et al.* 2006, Fisher *et al.* 2007, Jainaen *et al.* 2007). Although an attempt was made to control the age range of the teeth used (i.e. all teeth were extracted for orthodontic reasons and the age range was from 14 to 20), other variables such as type of dentine, number of tubules and presence or absence of sclerotic dentine could have contributed to the high spread in push-out values. Furthermore, there was great variability in canal shape and size, and thus, it is often difficult to create

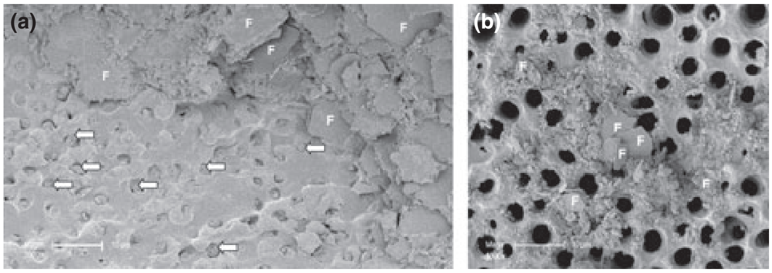

Figure 3 (a) Appearance of the dentine surface after shear bond testing (Resilon® without separate adhesive layer), with exposed tubules containing resin tags and some areas covered with flat plate-like filler particles (2000× magnification). (b) The size of the plate-like filler particles, many >10 μm across, meant that these particles could not enter the tubules and hence a filler-enriched surface was left behind. Some tubules were empty, presumably some tags pulling out of the tubules during shear failure (4000× magnification). F, filler particle; arrow, resin tag.

a well-matching plunger system (with uneven distribution of loading stresses) and to avoid creation of frictional stresses on the canal walls (Goracci *et al.* 2005, Üreyen Kaya *et al.* 2008). In contrast, the MSBS involves more precise sample preparation and carefully controlled testing conditions.

The manufacturer of Resilon® recommends use of a self-etching primer before placement of the highly filled UDMA-based sealer and core material. As seen in the restorative dentistry literature, conventional three step (etch, primer, adhesive) systems generally (though not always) result in much higher bond strengths than the adhesive systems that combine these steps (Castelnuovo *et al.* 1996, Tjan *et al.* 1996, Wilder *et al.* 1998, Bouillaguet *et al.* 2001, Fabianelli *et al.* 2003, Van Meerbeek *et al.* 2003, Hayashi *et al.* 2008). A similar result was noted in this study. Resin tags and bonding to dentine were inconsistently seen on the canal wall when the recommended self-etch resin primer was used. Even though the total-etch/adhesive system used in this study is marketed for use in restorative dentistry and may not be ideal for endodontic applications, it resulted in both a large increase in bond strength to dentine and good bonding to the sealer cement. First, there appeared to be a more uniform and predictable resin infiltration into tubules, which may be a reason for the increased bond strength and decreased failure at the resin/dentine interface. Secondly, the tubules were separately sealed before the sealer cement was applied, so that the thin layer of sealer was not depleted of resin by selective penetration into dentinal tubules (Jainaen *et al.* 2007, Hayashi *et al.* 2008, Rahimi *et al.* 2009). The contribution of a hybrid layer to bond strength, which was not evaluated in this study, has not received a great deal of attention in studies of endodontic materials. Clinically, a light-cured adhesive material is not feasible with currently available technologies. However, in a recent study, Hayashi *et al.* (2008) have shown that a chemical-cured total-etch adhesive material showed more consistent and extensive resin tags when compared with the self-etch dual cure resin cements (for cementing posts). Therefore, as shown in this study, the potential for increasing bond strength is readily apparent. Another problem with use of a separate adhesive material is that the material cannot be predictably applied to the apical and hard to reach areas of the long and narrow root canal system, and further work is needed prior to its clinical adoption.

Interestingly, in the push-out test group, debonding was seen only at the sealer-core interface in the adhesive group (Fig. 3), with much higher bond strengths. It appears that the sealer bonds to the adhesive more strongly than to the core and this maybe another weak link in the Resilon core to sealer bonding mechanism.

It is clear that to date the creation of a monoblock has not been achieved, leading Schwartz (2006) to comment that 'although adhesive obturating materials have greater potential than traditional materials, at this point in their development there is no clear benefit to their use'. The potential for substantially enhancing bond strength to dentine has been demonstrated in this study. Further development is necessary before these techniques can be used clinically.

Conclusions

Application of a total-etch/separate adhesive layer increases both microshear and push-out bond strength of UDMA-based sealers to root dentine, but clinical adoption of this approach will require a reformulation of materials. Although this approach is currently not feasible for clinical use, the potential for a large increase in bond strengths has been demonstrated.

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