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Push-out bond strengths of the dentine–sealer interface with and without a main cone

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Abstract

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Aim To evaluate the push-out bond strength of the dentine–sealer interface with and without main cone for three resin sealers.

Methodology Thirty extracted maxillary premolar teeth with two separate canals were prepared using 0.04 taper Profile instruments to size 35–45. Teeth were divided into three groups for filling using AH Plus™, EndoREZ® or Resilon® sealers. In each tooth, one canal was filled with a matching single-cone technique, and other was filled with sealer alone. A 1 mm slice of mid-root dentine was prepared for the push-out test. Failure modes after push-out were examined under microscopy and field emission-scanning electron microscopy. Data were analysed using two-way ANOVA and paired *t*-tests, with significance set at $P < 0.05$.

Results Overall, the epoxy resin-based sealer provided the highest push-out bond strengths. Push-out bond strengths were significantly higher ($P < 0.001$) when canals were filled with sealer alone than those filled with main cone and sealer (AH Plus™ 6.6 and 2.0 MPa, respectively; Resilon® 3.4 and 0.4 MPa; EndoREZ® 0.9 and 0.4 MPa). Sealers appeared to behave differently as thin films in association with a main cone, compared with bulk material. They failed in cohesive mode within the thin film, leaving a layer of sealer on the canal surface. Bulk sealer showed predominantly adhesive failure at the dentine–sealer interface, with a clean dentine wall and with resin tags either partially pulled out or sheared off at the interface.

Conclusion Push-out bond strengths of resin sealers were much lower when the sealer was present as a thin layer.

Keywords: push-out bond strength, sealer cement.

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Introduction

Successful root canal treatment depends on the thorough debridement of the root canal system, the elimination of pathogenic organisms and finally the complete sealing of the canal space to prevent ingress of bacteria from the oral environment and spread to the periapical tissue (Sundqvist *et al.* 1998). The physical properties necessary for this function include adaptation and adhesion of the filling material to the root canal wall, because gutta-percha does not directly bond to the dentine surface (Skinner & Himel 1987). Ideally, the sealer should be capable of producing a bond between core material and dentine wall.

Different types of sealer have been introduced to endodontics, including those based on zinc oxide eugenol, glass–ionomer cement and a range of resins. Epoxy resin-type sealers have been used for many years. They showed higher bond strength to dentine than zinc oxide eugenol types, calcium hydroxide-based sealer and a glass–ionomer sealer (Wennberg & Ørstavik 1990, Lee *et al.* 2002, Tagger *et al.* 2002). A hydrophilic, urethane dimethacrylate (UDMA) resin-based sealer (EndoREZ®) has been developed for filling root canals in association with a single-cone technique, and has been shown to achieve extensive resin penetration into dentinal tubules similar to epoxy resin

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(Bergmans *et al.* 2005), although not by all authors (Sevimay & Kalayci 2005). Another methacrylate resin-based sealer is the Resilon® (RealSeal®, Sybron-Endo, Glendora, CA, USA) system. The Resilon® system consists of a self-etching primer, 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 (Shipper & Trope 2004, Shipper *et al.* 2004), which resists shrinkage and strengthens the root (Teixeira *et al.* 2004).

Numerous studies have investigated the bond between sealer and the canal wall (Wennberg & Ørstavik 1990, Lee *et al.* 2002, Tagger *et al.* 2002, Saleh *et al.* 2003, Gogos *et al.* 2004, Doyle *et al.* 2006) including the effect of the smear layer on bond strength (Saleh *et al.* 2002, Eldeniz *et al.* 2005, Hayashi *et al.* 2005). More recently, a 'push-out' test has been described to measure the bond between sealer, canal wall and the core material (Thompson *et al.* 1999, Chandra & Ghonem 2001). The test is intended to assess the extent to which the sealer and core material are bonded into a solid mass as well as the strength of the bond to the canal wall. Several authors have reported a higher interfacial strength of gutta-percha/AH Plus sealer than that of Resilon/Epiphany (Gesi *et al.* 2005, Ungor *et al.* 2006, Sly *et al.* 2007). However, one study has found the opposite (Skidmore *et al.* 2006).

Resin-based sealers undergo polymerization shrinkage, which may affect the quality of the bond to dentine and to core material (Tay *et al.* 2005b). Also, during filling using a core material, sealer cement is generally present as a thin layer, with two interfaces: between dentine and sealer (D/S) and between sealer and the main cone (S/M). If the entire canal is filled with sealer, only one interface (D/S) is present. The behaviour of a resin-based sealer in a thin film may also be different from its behaviour in bulk. Therefore, the aim of this study was to assess the relative bond strengths between D/S and S/M, by testing canals filled with and without a main cone. The null hypotheses tested were: (i) there is no difference in push-out bond strength due to the presence or absence of main cone; (ii) there is no difference between the types of sealer materials tested.

Materials and methods

Instrumentation and obturation

Thirty extracted maxillary premolar teeth with two separate canals were used. All teeth were extracted for orthodontic reasons from patients aged 14–20 years and were immediately 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, Melbourne, Australia. The teeth were decoronated at the cemento-enamel junction using a slow speed diamond saw (Struers, Ballerup, Denmark) under water coolant. Each root canal was checked for patency using a size 10 K-file (FlexOFiles; Dentsply Maillefer, Ballaigues, Switzerland) until the file was seen at the apical foramen. Sixty canals were prepared at working length 0.5 mm short of the patency length using 0.04 taper Profile instrument (Dentsply Maillefer) to master apical rotary (MAR) size 35–45. Sodium hypochlorite (1% NaOCl, 1 mL) was used to irrigate each canal after every file using a 27-gauge irrigating needle. Apical patency was maintained by passing a size 10 K-file through the apical foramen. After completion of canal preparation, the canals were rinsed with 5 mL 15% ethylenediamine tetraacetic acid (EDTA) as the final rinse to minimize the residual effect of NaOCl on free radical polymerization. A final rinse of 5 mL distilled water was used to remove any remnant of the irrigating solutions. Canals were dried using paper points. The teeth were kept moist at all times by wrapping them in saline-soaked gauze.

The teeth were then randomly divided into three groups (using a random numbers table) for filling with three resin sealers. For each tooth, buccal and lingual canals were randomly allocated (using a random numbers table) to either main cone plus sealer or sealer only. For the canal filled using a main cone, a 0.04 taper master cone was matched to the final MAR file size. For AH Plus™ (Dentsply DeTrey, Konstanz, Germany) and EndoREZ® (Ultradent, South Jordan, UT, USA) sealers, the master cones were 0.04 gutta-percha. For Resilon®, the 0.04 Resilon® core material was used as recommended. The master cone was checked in the canal prior to placement by noting the point where 'tugback' at working length was first achieved.

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. EndoREZ®, with two-part chemical set, was mixed in an Ultra-Mixer® (Ultradent) and dispensed using a narrow diameter syringe (Skini® Syringe (Ultradent)) with a fine-tipped cannula (NaviTip (Ultradent)). 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 removed with a dry paper point. The Resilon sealer (RealSeal®) was mixed by the auto-mix syringe. For filling with a main cone, 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. The second canal was filled with sealer only using a paste filler in a conventional slow speed handpiece (approx. 6000 rpm); sealer was introduced into the canal until it was full.

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, both EndoREZ® and Resilon/RealSeal® 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.

Preparation for push-out bond testing

Teeth were sectioned using a 300 μm-thick sintered diamond wafering blade (Struers) perpendicular to the root canal at low speed with constant water cooling. A 1 mm-thick section of mid-root dentine was prepared, at a level calculated to yield a main cone diameter slightly greater than 0.5 mm (based on main cone size and taper). Both apical and coronal aspects of each sample were photographed and examined before testing to confirm a circular canal shape and that the sealer filled the entire canal space without voids. If the canal was not circular in shape (one sample) or there was any void in the sealer (one sample), it was excluded from the experiment and a replacement tooth prepared in the same way.

The filling material was loaded with a 0.5 mm-diameter cylindrical stainless steel plunger that provided almost complete coverage over the main cone 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 samples were aligned over a 1 mm diameter circular hole at the centre of a 10 mm-thick perspex plate. The samples were mounted in an apical to coronal direction to avoid any constriction interference due to root canal taper during push-out testing. The tests were conducted at a cross-head speed of 0.5 mm min^{-1} using a 100 N load cell set at 50 N maximum load. The highest value recorded was taken as the push-out bond strength. Photographs of both sides of the samples were again taken to check for anomalies and the thickness of the sample was measured with a digital caliper to within 0.01 mm. The area under load was calculated by ½ × (circumference of coronal aspect + circumference of apical aspect) × thickness. The push-out value in MPa was calculated from force (N) divided by area in mm^2.

After the push-out bond strength test, both sides of the sample including the main cone and sealer plug were examined under light microscopy (Leica DML, Ernst-Leitz-Strasse, Wetzlar, Germany) to determine the mode of failure. Each sample was evaluated at 20× magnification and put into one of the categories: (i) adhesive failure at the D/S interface; (ii) combination adhesive failure at both the D/S and S/M interface, and (iii) mixed failure in both adhesive and cohesive modes.

Representative samples from the different modes of failure categories from each group were split vertically for scanning electron microscope (SEM) examination. The pulpal wall of each sample plus the main cone and sealer plug were mounted on stubs, 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.

Statistical analysis

The data of push-out bond strength were analysed by two-way ANOVA with sealer type and presence of main cone as independent variables, and *post hoc* pair-wise comparisons were performed using Tukey multiple comparisons. The Student paired *t*-test was conducted within the same sealer type with and without main cone. For each outcome, statistical significance was set at $P < 0.05$.

Results

Push-out bond strength

Push-out bond strength varied considerably amongst samples for all sealer types. A $\log_{10}$ transformation of data was performed before performing the ANOVA to ensure the normality of distribution. Highly significant differences were found with respect to both sealer types ($P < 0.001$) and the presence or absence of a main

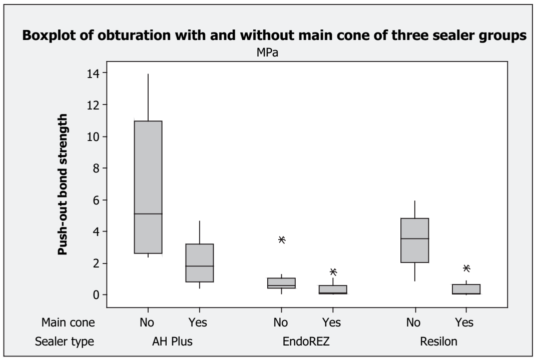

Figure 1 Push-out bond strength of three resin sealer groups obturated with and without main cone. Asterisks indicate outliers (individual values more than 1.5 interquartile range). Overall, the epoxy resin sealer (AH Plus™) showed a higher push-out bond strength than either of the two methacrylate resin-based sealers ($P < 0.001$, two-way ANOVA), which were not significantly different from each other ($P = 0.15$). The Student paired *t*-test comparisons for each sealer showed significant differences with and without main cone for the epoxy resin-based sealer (2.0 ± 1.4 vs. 6.6 ± 4.3 MPa, $P < 0.001$) and for Resilon® (0.4 ± 0.5 vs. 3.4 ± 1.6 MPa, $P < 0.001$), but not for EndoREZ® (0.4 ± 0.5 vs. 0.9 ± 1.0 MPa, $P > 0.05$).

cone ($P < 0.001$, two-way ANOVA) (Fig. 1). Overall, the epoxy resin sealer (AH Plus™) had a higher push-out bond strength than the two methacrylate resin-based sealers (EndoREZ® and Resilon®, $P < 0.001$), which were not significantly different from each other ($P = 0.15$). Push-out bond strength was much greater when the entire canal was filled with sealer rather than sealer plus main cone, by a factor of two- to eightfold. The Student paired *t*-test comparisons for each sealer showed significant differences with and without main cone for the epoxy resin-based sealer (2.0 ± 1.4 vs. 6.6 ± 4.3 MPa, $P < 0.001$) and for Resilon® (0.4 ± 0.5 vs. 3.4 ± 1.6 MPa, $P < 0.001$), but not for EndoREZ® (0.4 ± 0.5 vs. 0.9 ± 1.0 MPa, $P > 0.05$).

Failure mode

Under light microscopy at 20× magnification, all sealers used with a main cone appeared to show adhesive failure either at the D/S interface or a combination of D/S and failure at the S/M interface. No instances were found of adhesive failure at the S/M interface alone or of pure cohesive failure within the sealer. AH Plus™ with a main cone and sealer failed 40% in adhesion between D/S and 60% in combination

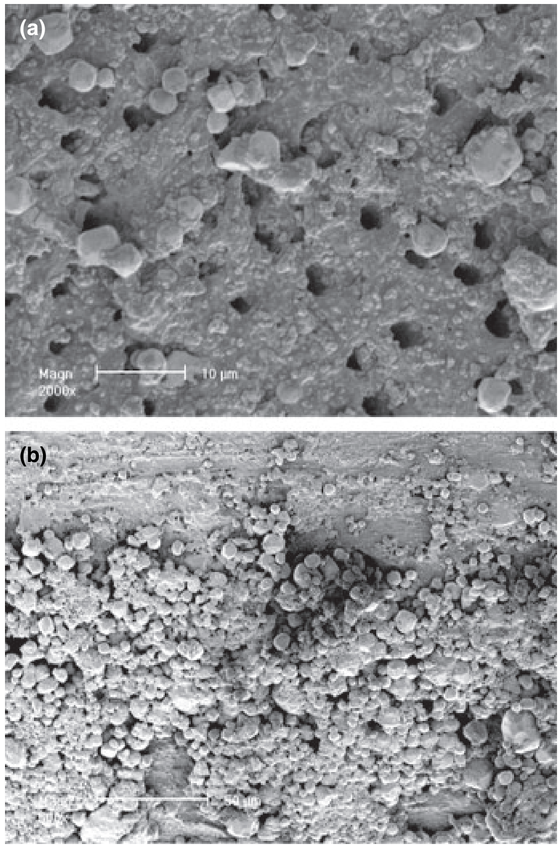

Figure 2 (a) Field emission-scanning electron micrograph of the canal wall after push-out shows the dentine covered by a gritty layer of sealer of canal obturated (canal obturated with main cone and AH Plus™). The filler particles varied from approximately 0.2 to 20 μm in size. No area of the dentine wall devoid of particles was seen. (b) FE-SEM micrograph of the surface of the main cone plug shows filler particles with little surrounding matrix (canal obturated with AH Plus™ and main cone).

adhesion between D/S and S/M, where as EndoREZ® and Resilon® had 70% adhesive failure between D/S and 30% combination failures. When filled with sealer only, adhesive failure between D/S was found in 80% of samples for AH Plus™ and EndoREZ® and 90% for Resilon®; the remainder failed in mixed mode between adhesive and cohesive (within the sealer).

When samples were examined under FE-SEM at higher magnification (×400–4000), a different pattern emerged (Figs 2–7). The apparent adhesive failure between D/S or combination of D/S and S/M failure modes was observed as predominantly cohesive failure within the sealer itself. The surface of the canal wall was coated with a layer of sealer cement with filler

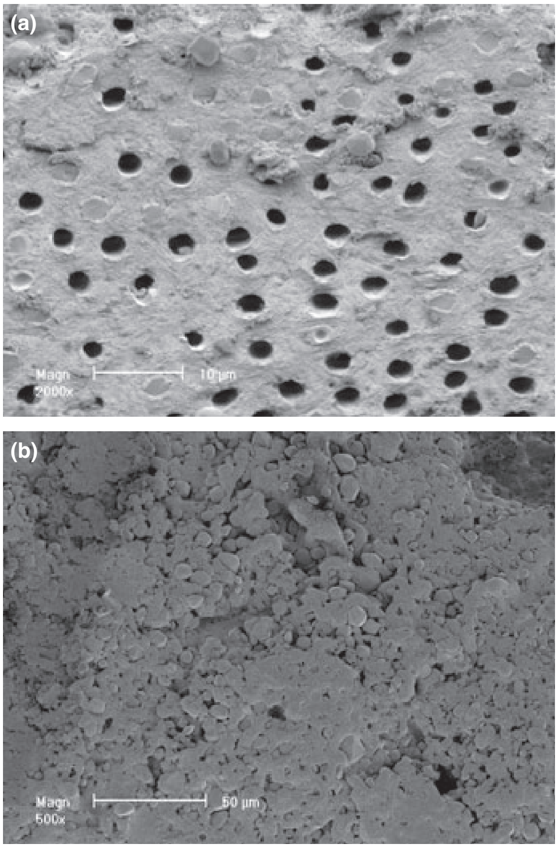

Figure 3 (a) Field emission-scanning electron micrograph of the fractured surface of the canal wall of a canal obturated with AH Plus™ only shows clear dentine with some resin tags partly or completely torn away from the surface whilst some still completely occlude dentinal tubules. (b) The surface of the pushed-out AH Plus™ sealer plug shows matrix surrounding filler particles with some voids.

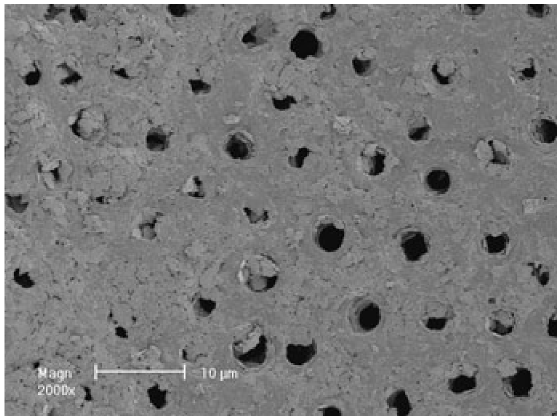

Figure 4 Field emission-scanning electron micrograph of the dentine surface of the canal wall (canal obturated with main cone and EndoREZ®) shows plate-like filler particles covering the dentine surface. Some finer particles clogged or partially occluded dentinal tubules, but no resin tags or clean dentine surface were found.

particles embedded in the resin matrix and projecting above its surface (Figs 2a, 4 and 6). Resin particles often obscured tubule orifices, and there was no evidence of resin tags pulling out of dentinal tubules. Especially with the epoxy resin sealer, the sealer layer appeared depleted of resin matrix, with filler particles prominent on the fractured surface of both the canal wall and the pushed-out main cone (Fig. 2a,b).

In canals filled with sealer only, failure was predominantly adhesive at the D/S interface, as evidenced by the presence of resin tag formation and resin tags that had been pulled out from the dentine surface. Clear dentine surfaces were found where some tags had sheared off but remained in the dentinal tubules (Figs 3a, 5a and 7). The pushed-out sealer plug surface had matrix surrounding filler particles (Fig. 3b) and some resin tags left on the sealer plug (Fig. 5b). Many resin tags of Resilon® sealer were hollow, with only a thin layer of resin formed around the periphery of the tubule (Fig. 7), whereas the tags formed by AH Plus™ and EndoREZ® appeared solid in cross-section (Figs 3a and 5a).

Penetration of sealer into dentinal tubules was observed with all sealer types, to a depth of at least 200 µm. The appearance of sealers penetrating into dentinal tubules is shown in Fig. 8.

Discussion

The ideal for root canal filling is that the entire root canal space is filled with no gaps or voids. Sealer and core materials should form a uniform, chemically bonded mass that is also bonded to dentine to minimize leakage. Ørstavik *et al.* (1983) found no correlation between leakage (using dye penetration) and bond strength of sealer to dentine. They argued that if the low bond strength of the D/S interface did not affect apical leakage, then bonding between root filling material and sealer does not seem to be an essential property of a sealer (Wennberg & Ørstavik 1990). With the development of resin-based sealers, the strength of the bond has received greater attention; the possibility of creating a 'monoblock' of sealer and core material that also bonds to the canal wall has introduced the prospect of strengthening the root-filled tooth against fracture (Teixeira *et al.* 2004, Schäfer *et al.* 2007).

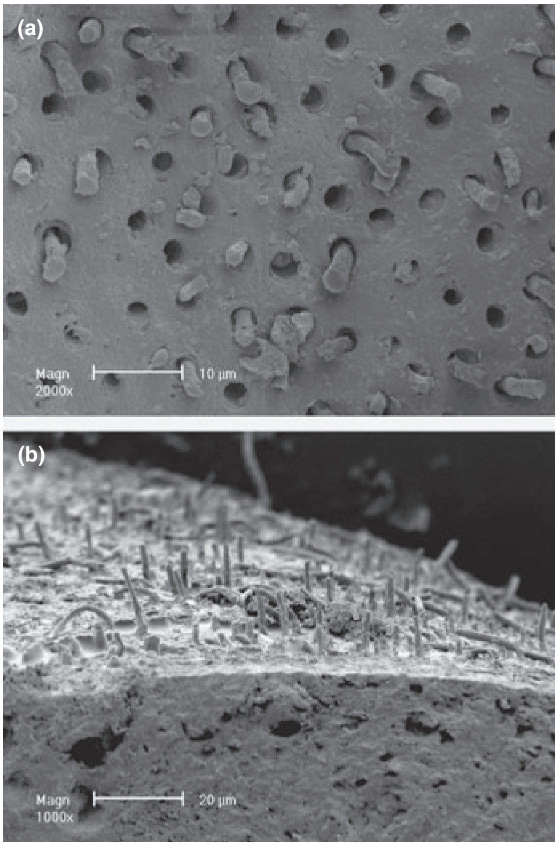

Figure 5 (a) Field emission-scanning electron micrograph of the dentine surface of the pulpal wall of a canal obturated with EndoREZ® sealer only shows a clean dentine wall with resin tags partially or completely pulled out. (b) The surface of the EndoREZ® resin plug shows resin tags pulled out from dentinal tubules.

The purpose of this study was to measure separately the bond strength of the D/S interface and the S/M interface by using premolars with two canals, filling one with sealer only (to measure the D/S bond strength), and the other with sealer plus main cone (which will include both D/S and S/M interfaces). Use of sealer alone is not recommended clinically for canal filling, and was included only to permit the separate measurement of the D/S interface bond strength. For evaluating the mechanical properties of the interfaces, the thin slice push-out test is an important experimental tool (Chandra & Ghonem 2001). The advantages of this method over tensile and shear strength tests are that it is less sensitive to small variations amongst specimens and to variations in stress distribution during load application, and that it is easy to align samples for testing (Ungor *et al.* 2006). It has

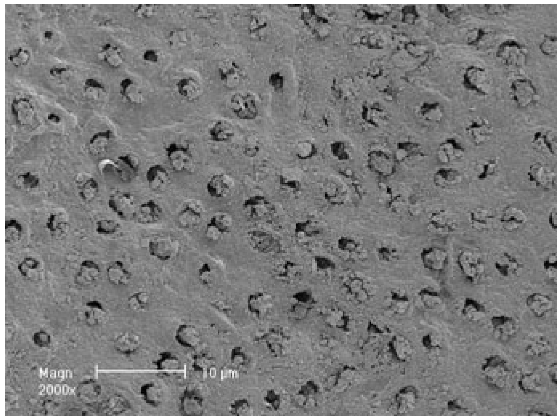

Figure 6 Field emission-scanning electron micrograph of the canal wall of a canal obturated with main cone and Resilon® shows residual sealer material covering the surface. Fine filler particles occlude or partly occlude dentinal tubules.

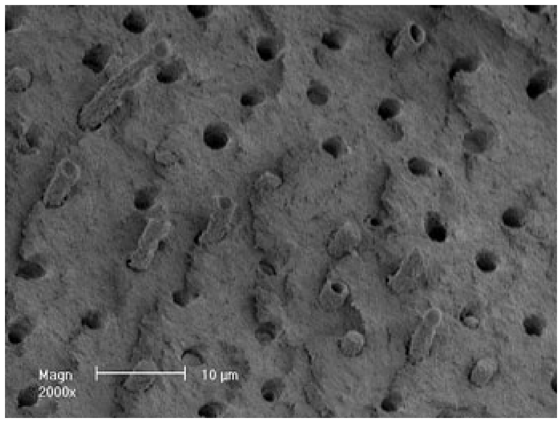

Figure 7 Field emission-scanning electron micrograph of the pulpal wall of a canal obturated with Resilon® sealer only shows a clean dentine surface with some resin tags partly or completely torn away from the tubules. Note the hollow appearance of some tags.

been found reliable in bond strength evaluation in 1 mm-thick samples (Skidmore *et al.* 2006).

Even though only intact maxillary premolar teeth from patients aged no more than 20 years were used, to minimize the effect of continued dentine deposition in older teeth (Torneck 1998), the push-out bond strength varied considerably within groups for all sealer types. This variability reflects the clinical situation, where large differences may be encountered between teeth. All resin sealers showed a two- to eightfold higher push-out bond strength of the D/S

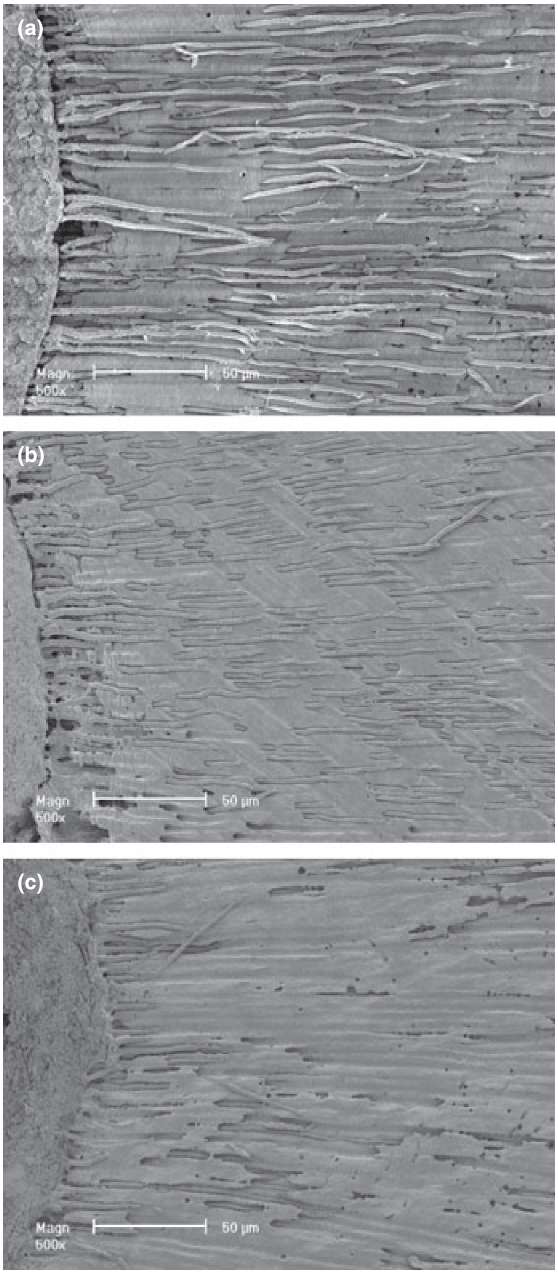

Figure 8 Field emission-scanning electron showing resin penetration into the dentinal tubules of canals obturated with main cone and three resin sealers: (a) AH Plus™, (b) EndoREZ® and (c) Resilon®.

interface (canal filled with sealer alone) than of the combined D/S and S/M interfaces. It is reasonable to speculate that all resin sealers bond much more strongly to dentine than to main cone. Nevertheless, the differences cannot be explained simply in terms of failure at the S/M interface.

Amongst the three resin sealers, AH Plus™ (an epoxy resin sealer) showed the highest push-out bond strength of the D/S interface, compared with two UDMA-based root canal sealers. This result is similar to previous studies (Gesi *et al.* 2005, Ungor *et al.* 2006, Sly *et al.* 2007) which also used the push-out test, although only in association with a core material. However, it is in disagreement with Skidmore *et al.* (2006) who compared Resilon® with a zinc oxide eugenol-based sealer, which has been reported to have lower bond strength compared with epoxy resin (Wennberg & Ørstavik 1990, Lee *et al.* 2002, Tagger *et al.* 2002).

With a matching-taper single-cone filling technique, this study showed similar mean push-out bond strength to previous studies that filled the root canal using a warm vertical condensation technique (Gesi *et al.* 2005, Sly *et al.* 2007). EndoREZ® has been recommended for use either with a conventional gutta-percha cone or with specific EndoREZ® points (resin-coated gutta-percha). With conventional uncoated gutta-percha, the present study found low bond strength. A greater adhesive strength to dentine using EndoREZ® may be obtained if a two-step self-etch adhesive is used as reported by Doyle *et al.* (2006). However, gap formation and microleakage were still found along the sealer–dentine and/or the core–sealer interface of EndoREZ® sealer and resin-coated gutta-percha cones (Tay *et al.* 2005b). The RealSeal® sealer and Resilon® cone also showed low bond strength. This suggested that the monoblock concept of Resilon® should be reconsidered. It is possible that the amount of dimethacrylate incorporated in the polycaprolactone-based thermoplastic composite may not be optimized for effective chemical coupling to methacrylate resins (Hiraishi *et al.* 2005). Under the conditions of this study, it was not possible to determine whether a hybrid layer was present in the Resilon/RealSeal® group.

Previous studies have shown that thin layers of sealer are preferred in modern endodontics, because the sealer may shrink during setting and dissolve over time, producing leakage. Tay *et al.* proposed that the configuration factor (C-factor, the ratio of total bonded to unbonded surface area) in bonded root canals exhibited a negative correlation with sealer thickness. As sealer thickness decreases, the unbonded flowable amount of sealer decreases, causing the C-factor to increase rapidly (Tay *et al.* 2005a). Kontakiotis *et al.* (1997) reported that thinner layers had less leakage for a zinc oxide eugenol-based sealer, but no difference was found between thin and thick layers of an epoxy-based sealer (AH 26® [Dentsply]).

The present study found a difference in the mode of failure between thin film and bulk sealer of the three resin sealers. With a thin film, failure was cohesive within the sealer itself, whereas bulk sealer showed adhesive failure between dentine and sealer, leaving partially pulled out resin tags in the dentinal tubules. The distinctive thin-film failure might be explained by an insufficient amount of resin in a thin setting layer. Resin-based sealers (both epoxy and methacrylate resins) have been shown to penetrate dentinal tubules extensively (Weis *et al.* 2004, Bergmans *et al.* 2005). It is possible that the resin matrix material preferentially penetrated the dentinal tubules, leaving a sealer layer that is enriched with filler particles that are larger than the dentinal tubule diameter (Fig. 8). This leaves a sealer with a resin-depleted layer and a filler particle-enriched interface. If the sealer layer does not have sufficient bulk or thickness, the loss of resin into the dentinal tubules may not be compensated for. A weak bond would result due to the excessively high particle ratio in the sealer layer. In contrast, the setting of bulk sealer has a much greater supply of resin available to penetrate into dentinal tubules, without depleting the presence of resin around filler particles, thus holding the sealer together and maintaining cohesive strength.

An unusual feature of the resin tags penetrating dentinal tubules in the Resilon/RealSeal® group was the apparent hollow appearance of some tags (Fig. 7). This feature was not studied systematically and requires further investigation. It may result from polymerization contraction of the resin in association with a high affinity of the resin for the moist tubule surface, or from dilution of the resin by residual water in the tubules.

In the present study, epoxy resin (AH Plus™) had the highest bond strength, which correlates with dye penetration (Sevimay & Kalayci 2005) and fluid filtration tests (Kardon *et al.* 2003). Comparisons of AH Plus™ and Resilon® found either no difference in leakage (Biggs *et al.* 2006, Baumgartner *et al.* 2007) or less with Resilon® than with AH Plus™ sealer (Shipper & Trope 2004, Stratton *et al.* 2006). Therefore, to confirm any correlation between bond strength and leakage, more studies need to be undertaken.

Conclusions

The epoxy resin-based sealer had the highest push-out bond strength compared with UDMA-based sealers when used with a main cone and sealer. The bond strengths after filling with sealer alone were higher than those with main cone and sealer, and may reflect different patterns of behaviour when the sealer is present as a thin layer.

Acknowledgements

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