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Dislocation resistance of ProRoot Endo Sealer, a calcium silicate-based root canal sealer, from radicular dentine

B. P. Huffman[1], S. Mai[2], L. Pinna[3], R. N. Weller[4], C. M. Primus[5], J. L. Gutmann[6], D. H. Pashley[7] & F. R. Tay[4,7]

[1]School of Dentistry, Medical College of Georgia, Augusta, GA, USA; [2]Guanghua School of Stomatology & Institute of Stomatological Research, Sun Yat-sen University, Guangzhou, China; [3]Universita degli Studi di Cagliari, Reparto di Odontoiatria Conservatrice, Sardinia, Italy; [4]Department of Endodontics, School of Dentistry, Medical College of Georgia, Augusta, GA, USA; [5]Primus Consulting, Bradenton, FL, USA; [6]Department of Endodontics, Baylor College of Dentistry, Texas A&M University System Health Science Center, Dallas, TX, USA; and [7]Department of Oral Biology, School of Dentistry, Medical College of Georgia, Augusta, GA, USA

Abstract

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Aim To examine the dislocation resistance of three root canal sealers from radicular dentine with and without immersion in a simulated body fluid (SBF), using a modified push-out test design that produced simulated canal spaces of uniform dimensions under identical cleaning and shaping conditions.

Methodology Sixty single-rooted caries-free human canine teeth were used. Standardized simulated canal spaces were created using 0.04 taper ProFile instruments along the coronal, middle and apical thirds of longitudinal tooth slabs. Following NaOCl/ethylenediamine tetra-acetic acid cleaning, the cavities were filled with ProRoot Endo Sealer, AH Plus Jet or Pulp Canal Sealer. After setting, half of the cavities were tested with a fibre-optic light-illuminated push-out testing device. The rest were immersed in SBF for 4 weeks before push-out evaluation. Failure modes were examined with stereomicroscopy and field emission (FE)-scanning electron microscopy.

Results Location of the sealer-filled cavities did not affect push-out strengths. ProRoot Endo Sealer exhibited higher push-out strengths than the other two sealers particularly after SBF storage ($P < 0.001$). Failure modes were predominantly adhesive and mixed for Pulp Canal Sealer and AH Plus Jet, and predominantly cohesive for ProRoot Endo Sealer. Spherical amorphous calcium phosphate-like phases that spontaneously transformed into apatite-like phases were seen in the fractured specimens of ProRoot Endo Sealer after SBF storage.

Conclusions When tested in bulk without a main core, both 'sealer type' and 'SBF storage' were significant in affecting push-out results. The ProRoot Endo Sealer demonstrated the presence of spherical amorphous calcium phosphate-like phases and apatite-like phases (i.e. *ex vivo* bioactivity) after SBF storage.

Keywords: calcium silicate-based sealer, dislocation resistance, *in vitro* bioactivity, thin-slice push-out test.

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Introduction

The use of a sealer and a thermoplastic core material for filling root canals is the accepted norm in contemporary root canal procedures. As leakage from the apical or coronal direction is a possible cause of root treatment failure (Madison & Wilcox 1988, De Moor & Hommez 2000), a root canal sealer should exhibit good sealing (Laghios *et al.* 2000) and adhesive properties (Wennberg & Ørstavik 1990, Gettleman *et al.* 1991, Timpawat *et al.* 2001, Lee *et al.* 2002a,b, Saleh *et al.* 2003, Tagger *et al.* 2003). A sealer may be

Correspondence: Dr Franklin R. Tay, Department of Endodontics, School of Dentistry, Medical College of Georgia, Augusta, GA, 30912-1129, USA (Tel.: +1 706 7212033; fax: +1 706 7216252; e-mail: ftay@mcg.edu).

conceptualized as a joint created between the radicular dentine and the filling material. Similar to other prosthetic joints in the body, the ability to resist dislocation during function is crucial to their survival (Scifert *et al.* 1999, Weale *et al.* 2002, He *et al.* 2007). For a root canal sealer, the ability to resist disruption of the established seal via micromechanical retention or friction is highly desirable during intraoral tooth flexure (Panitvisai & Messer 1995) or preparation of cores or postspaces along the coronal- and middle-thirds of canal walls (Muñoz *et al.* 2007).

Predictable clinical results have been reported with the use of gutta-percha in conjunction with zinc oxide eugenol or epoxy resin-based root canal sealers (Salehrabi & Rotstein 2004, Tilashalski *et al.* 2004). Nevertheless, there is a continuous quest for alternative sealers or root filling materials with better seal and dislocation resistance. Although the correlation between the sealing property of a root canal sealer and its adhesive characteristics has not been firmly established, it is essential that the dislocation resistance of a root canal sealer to dentine is not adversely affected by the seepage of body fluids when there is a breach of either the apical or coronal seal.

ProRoot Endo Sealer (Dentsply Tulsa Dental Specialties, Tulsa, OK, USA) is an experimental calcium silicate-based root canal sealer that is designed to be used in conjunction with a root filling material in either the cold lateral, warm vertical or carrier-based filling techniques. The major components of the powder component are tricalcium silicate and dicalcium silicate, with the inclusion of calcium sulphate as a setting retardant, bismuth oxide as a radiopacifier and a small amount of tricalcium aluminate. The liquid component consists of a viscous aqueous solution of a water-soluble polymer. Similar to other tricalcium silicate and dicalcium silicate-containing biomaterials, the sealer produces calcium hydroxide on reaction with water (Gou *et al.* 2005, Camilleri & Pitt Ford 2006, Wang *et al.* 2008). It is also anticipated that release of calcium and hydroxyl ions from the set sealer will result in the formation of apatites as the material comes into contact with phosphate-containing fluids (Sarkar *et al.* 2005), via spontaneous transformation from initial amorphous calcium phosphate phases (Tay *et al.* 2007, Tay & Pashley 2008).

Whereas the retentive potential of geosynthetics (Marques 2005), concrete reinforcements (Lee *et al.* 2002a,b) and rigid postsystems within canal spaces (Mitchell *et al.* 1994, Teixeira *et al.* 2006) may be evaluated *en masse* using conventional pull-out test designs, thermoplastic root filling materials and sealers are not amendable to gripping that is a prerequisite for this type of mechanical testing (Goracci *et al.* 2007). Thus, the thin-slice push-out test has been used quite frequently for evaluating the dislocation resistance of root filling materials (Gesi *et al.* 2005, Sousa-Neto *et al.* 2005, Gancedo-Caravia & Garcia-Barbero 2006, Skidmore *et al.* 2006, Ungor *et al.* 2006, Bouillaguet *et al.* 2007, Fisher *et al.* 2007, Jainaen *et al.* 2007, Nagas *et al.* 2007, Sly *et al.* 2007, Ureyen Kaya *et al.* 2008). The strength of that experimental design is that each horizontal root slab being tested is derived from a root filled canal and contains a cross-section of the thermoplastic root filling material and sealer to be investigated. In the present study, a modified thin-slice push-out test was designed to evaluate the dislocation resistance of root canal sealers that were applied in bulk to simulated canal spaces without the use of thermoplastic material cores. The null hypothesis tested was that there are no differences in the dislocation resistance of three root canal sealers from radicular dentine when the set sealers are tested with and without immersion in a simulated body fluid (SBF).

Materials and methods

Preparation of simulated canal spaces

Sixty intact, caries-free human canine teeth were collected after the patients' informed consents were obtained under a protocol reviewed and approved by the Human Assurance Committee of the Medical College of Georgia, Georgia, USA. For each tooth, a 0.90 ± 0.05 mm thick longitudinal slab was prepared by making buccolingual sections parallel to the longitudinal axis of the tooth using a slow-speed diamond saw (Isomet; Buehler Ltd, Lake Bluff, IL, USA) under water-cooling. A Plexiglas platform containing a cylindrical well was affixed to the base of a mini drill press to generate vertically oriented, truncated cavities of uniform dimensions within the tooth slab (Fig. 1a). A 0.6 mm drill bit was first used to prepare pilot holes in the radicular dentine adjacent to the dental pulp. Each pilot hole was carefully drilled so that it was equidistant from the cementum and the canal wall. Two pilot holes each were prepared in the coronal, middle and apical thirds of the root.

Each hole was subsequently enlarged using a size 40, 25 mm long 0.04 taper ProFile nickel titanium rotary instrument (Dentsply Tulsa Dental Specialties). To ensure optimal cutting efficacy, a new instrument

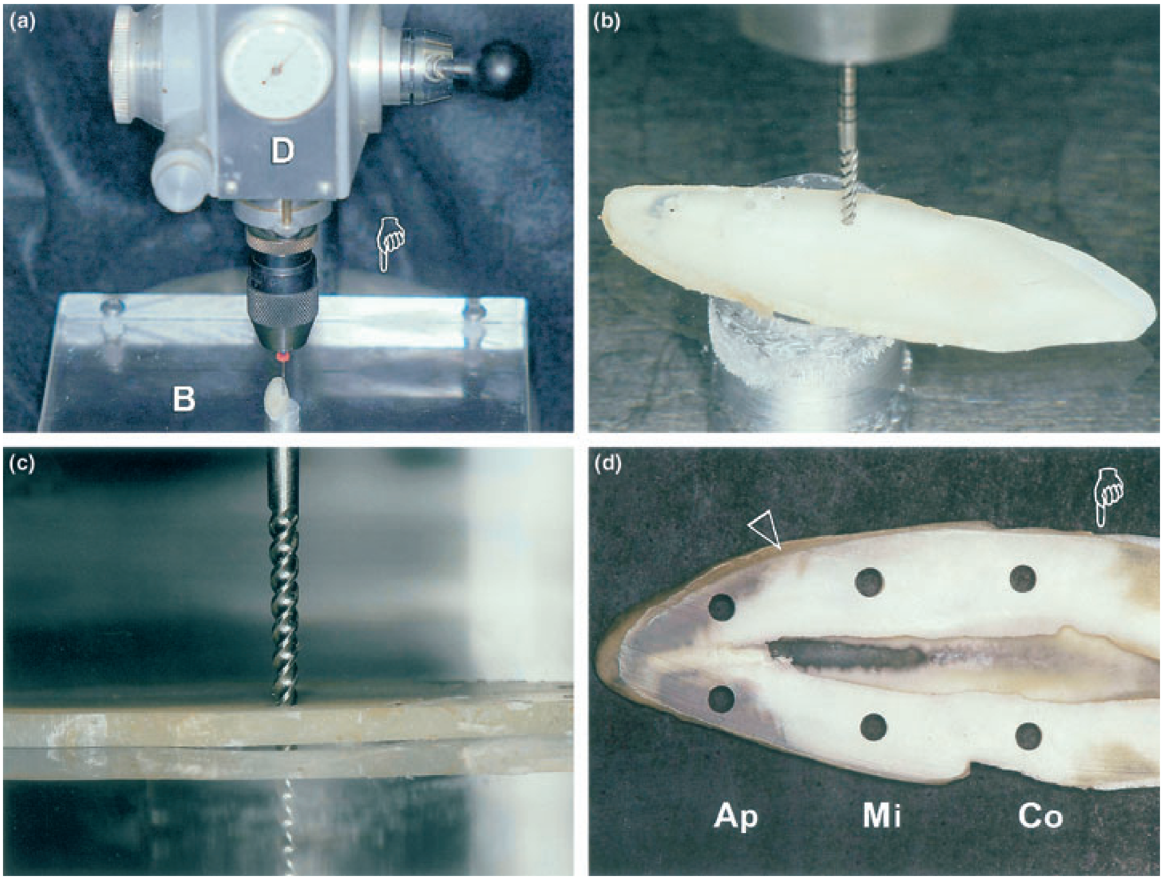

Figure 1 Experimental setup for the preparation of perpendicular truncated cavities of uniform dimensions in different locations of a longitudinal tooth slab. (a) A mini drill press (D) with a 25 mm thick Plexiglas platform (B) affixed to its base (pointer). (b) A tooth slab was placed over a supporting well in the Plexiglas platform. A 0.04 taper size 40 Profile nickel titanium rotary instrument was inserted through a pre-drilled pilot hole in the tooth slab to create a truncated hole with the basal diameter corresponding to the D_{16} diameter (i.e. 1.04 mm) of the rotary instrument. The drill press was set to drill exactly to the same depth every time to ensure that each hole has the same circumference. (c) As slanted preparations are not amendable to push-out testing, the current setup ensured that all cavities were created perpendicular to the tooth slab. (d) Two tapered cavities each were prepared in the apical (Ap), middle (Mi) and coronal (Co) thirds of the root dentine. Pointer: cementoenamel junction; open arrowhead: cementum.

was used for each tooth slab. The drill press and the thickness of the Plexiglas platform were configured so that the rotary instrument penetrated the cylindrical well to the same depth every time (Fig. 1b). This permitted preparation of all truncated cavities to the D_{16} diameter of the rotary instrument (i.e. 1.04 mm) along the surface of the tooth slab. Inadvertent preparation of cavities with nonvertical extrusion paths was prevented by aligning the rotary instrument perpendicular to the tooth slab (Fig. 1c). The experimental design ensured that all cavities created in the coronal, middle and apical thirds of the roots had comparable dimensions. The artificial canal spaces were also completely devoid of calcospherites that are found along the mineralization front of the noninstrumented portions of natural root canal spaces. This eliminated the issue of unpredictable augmentation in sealer dislocation resistance that is caused by the presence of undercuts and increased contact areas in calcospherite-containing canal walls. The tooth slabs were divided randomly into six groups of 10 slabs each for evaluation of three endodontic sealers with or without immersion in a SBF. Six cavities were created in each tooth slab, with the two apical cavities residing in transparent, sclerotic radicular dentine (Fig. 1d). For each group, 20 simulated canal spaces were available from each of the three respective radicular dentine locations ($n = 20$).

Filling of root canal sealers

The tooth slabs were immersed in 17% ethylenediamine tetra-acetic acid (EDTA) and ultrasonicated for 5 min to dissolve the smear layer created during the hole-shaping procedures. The slabs were further immersed in 6.15% sodium hypochlorite and ultrasonicated for 5 min to remove organic debris and the demineralized collagen matrix created during EDTA application. The rationale for *en masse* cleaning was to further ensure that differences in dislocation resistance of the sealers from different dentine locations were not caused by inadequate cleaning of the apical radicular dentine.

The three sealers investigated in this study were Pulp Canal Sealer (SybronEndo; Sybron Dental Specialties Inc., Orange, CA, USA), AH Plus Jet (Dentsply Caulk, Milford, DE, USA) and the experimental ProRoot Endo Sealer. The former two sealers were mixed according to the manufacturers' instructions. The calcium silicate-based sealer was mixed with a liquid-to-powder ratio of 1 : 2 and covered with moist gauze to avoid evaporation of the water component. All cavities from one tooth slab were filled with one type of sealer. Each tooth slab was placed over a Mylar strip (Angst & Pfister, Geneva, Switzerland), which in turn was placed over a microscope glass slide. For Pulp Canal Sealer and ProRoot Endo Sealer, the sealer was mixed and placed inside a 19-gauge AccuDose Needle Tube (Centrix, Shelton, CT, USA). The sealer was dispensed into the cavities so that each hole was filled with excess sealer. For AH Plus Jet, the sealer was dispensed directly from the double-barrel mixing syringe via an intraoral tip attached to an auto-mixing tip. The surface of the tooth slab was then covered with another Mylar strip and a glass slide. The assembly was secured with binder clips so that excess sealer was expressed laterally from the surface and bottom Mylar strips. The assemblies were transferred to humidors and stored under 100% relative humidity for 1 week until all the sealers had completely set.

The binder clips were released and the Mylar strips were removed from the tooth slab to expose the set sealers. The top and bottom surfaces of each tooth slab were polished with 800-grit silicon carbide papers under running water to remove the excess sealer. For each sealer, one subgroup of 10 tooth slabs was tested immediately after polishing, whilst the other subgroup of 10 tooth slabs was immersed for 4 weeks at 37 °C in a phosphate-containing SBF prior to testing. The SBF contained 136.8 mmol L^{-1} NaCl, 3.0 mmol L^{-1} KCl, 2.5 mmol L^{-1} $CaCl_2 \cdot 6\ H_2O$, 1.5 mmol L^{-1} $MgCl_2 \cdot 6H_2O$, 0.5 mmol L^{-1} $Na_2SO_4 \cdot 10\ H_2O$, 4.2 mmol L^{-1} $NaHCO_3$ and 1.0 mmol L^{-1} $K_2HPO_4 \cdot 3H_2O$ in deionized water (pH 7.4). To prevent bacterial growth, 0.02% sodium azide was also included in the SBF.

Dislocation resistance evaluation

The dislocation resistance of the set root canal sealers was evaluated using a thin-slice push-out test design (Chandra & Ananth 1995, Chandra & Ghonem 2001). Prior to testing, the thickness of each tooth slab was measured to the nearest 0.01 mm using a pair of digital calipers. A 0.7 mm diameter carbon steel cylindrical plunger was used for the push-out test. The plunger had a clearance of about 0.1 mm from either side of the dentinal wall when it is perfectly aligned with the apical part of the truncated hole. The plunger was attached to a 10 kg load cell that was connected to a universal testing machine (Vitrodyne V1000 universal tester; Liveco Inc., Burlington, VT, USA). The push-out device consisted of a clear Plexiglas platform with a vertical cylindrical channel, which served as the support for the tooth slab and provided space for the vertical movement of the plunger through the truncated hole (Fig. 2a). To ensure optimal alignment of the plunger with the sealer-filled hole, a horizontal channel was drilled through the Plexiglas platform into the vertical channel (Fig. 2b). A fibre-optic light guide was inserted into the horizontal channel to provide high intensity illumination of the truncated hole during the alignment procedure. Each root slab was secured with sticky wax in an apical-coronal direction to the supporting Plexiglas platform, so that the smaller diameter apical side of the sealer-filled hole was facing the plunger. Each sealer-filled hole was subjected to compressive loading at a cross-head speed of 10 μm s^{-1} in order to displace the set sealer toward the coronal aspect of the hole. As the plunger contacts the set sealer on loading, shear stresses were introduced along the sealer-dentine interface, causing the set sealer to be dislocated from the walls of the radicular dentine. Failure was confirmed by the appearance of a sharp drop along the load/displacement curve recorded by the testing machine. After performing push-out testing of the first hole, the tooth slab was carefully removed and realigned with the

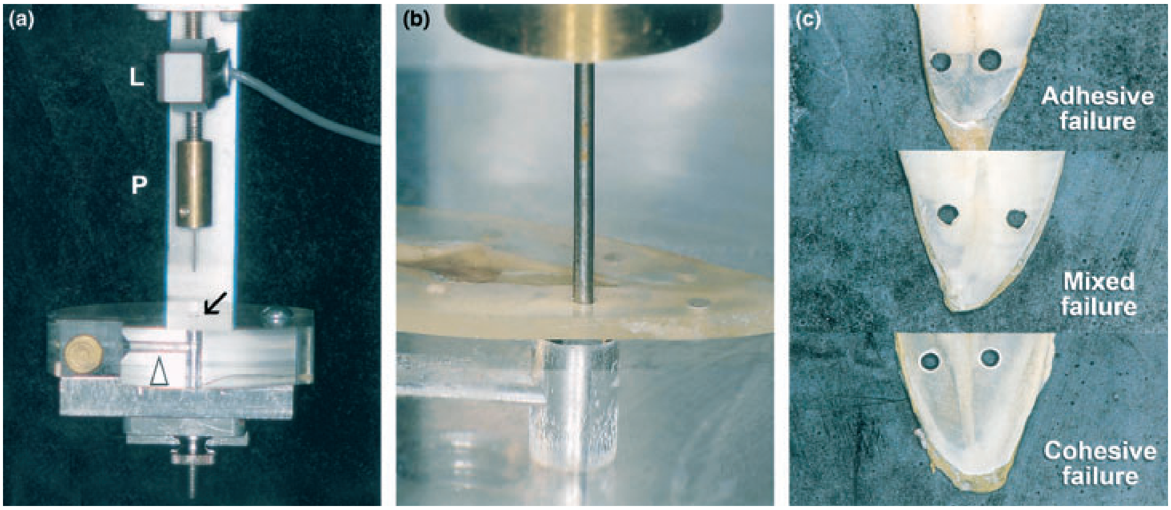

Figure 2 Experimental setup for the thin-slice push-out test. (a) The plunger (P) was connected to a 10 kg load cell (L). The plunger was aligned with the cylindrical well (arrow) of a clear Plexiglas stage. The latter had a side channel (open arrowhead) for the fitting of a fibre-optic light guide. (b) Each tooth slice was placed on top of the cylindrical well. The plunger had a diameter of 0.7 mm whilst the truncated hole had diameters of about 0.94 and 1.04 mm along its top and base. The use of light illumination ensured that the plunger was aligned with the centre of the hole so that the sealer was pushed out without the plunger contacting the wall of the hole. (c) Examples of adhesive failure, mixed failure and cohesive failure of the sealers, as observed through a stereomicroscope after the push-out test.

second hole. The procedures were repeated until the set sealers were dislodged from all the six cavities within a tooth slab. After the push-out test, each root slab was examined with a stereomicroscope at 30× magnification to determine the mode of failure. Failure modes were classified as: adhesive failure along the sealer-dentine interface; cohesive failure within the sealer, and mixed failure that consisted of partial adhesive failure along the dentinal walls and partial cohesive failure within the sealer (Fig. 2c).

Digitized photographs of each tested hole were taken from the coronal and apical aspects of the tooth slab together with a millimetre scale for calibration purpose. Such a procedure was performed after completion of the push-out test as this permitted better contrast of the circumference of the cavities. The circumferences of the coronal (C) and apical aspects (A) of each cavity were measured from the digitized images using image analysis software (Image 4.01; Scion Corp., Frederick, MA, USA). The area of the sealer-dentine interface was approximated by $0.5 \times (C + A) \times h$, where h represents the thickness of the tooth slab. Dislocation resistance of the sealer, as represented by the push-out strength, was computed by dividing the maximum load (N) derived from the load displacement curve with the sealer-dentine interfacial area (mm^2) and expressed in mega-Pascals (MPa). The same procedures were applied to those tooth slabs that had been immersed in SBF for 4 weeks.

Statistical analysis

Each sealer-filled hole was treated as a statistical unit. For each of the six subgroups, data ($n = 20$) from the three radicular dentine locations (i.e. coronal, middle and apical thirds) were analysed using one-way ANOVA to determine if dislocation resistance of a particular sealer was affected by the location of the sealer. As there were no differences in the dislocation resistance amongst dentine locations in all the six subgroups, data from the coronal, middle and apical aspects of each subgroup were pooled together for further analysis ($n = 60$). As the pooled data were not normally distributed, $\log_{10}$-transformation of the data was performed to normalize the data before statistical evaluation. The transformed pooled data were evaluated using a two-way ANOVA design, with sealer type and SBF storage as independent variables. *Post hoc* pair-wise comparisons were performed using Tukey multiple comparisons. The Student paired t-test was conducted within the same sealer type to examine if there was difference between the subgroup that was tested without SBF immersion and the other that was tested after SBF immersion. Statistical significance was set at $\alpha = 0.05$.

Scanning electron microscopy

After push-out testing, two slabs from each of the six subgroups were air-dried, sputter-coated with gold/palladium, and examined using a field emission scanning electron microscope (Model XL-30 FEG; Philips, Eindhoven, The Netherlands) at 15 KeV. The objective of the morphologic examination was not to reiterate the assessment of failure modes that had been performed using stereomicroscopy. Rather, the higher resolution of a field emission microscope was utilized to substantiate whether calcium phosphate-like phases and their phase transformation could be identified after the calcium silicate-based sealer was immersed in the phosphate-containing SBF.

Results

Representative load–displacement curves of the three root canal sealers are shown in Fig. 3a. Despite the differences in the magnitude of the maximum load achieved in the three sealers, their load–displacement curves demonstrated were characterized by four regions. There was an initial linear increase in load (zone I) that corresponded with the increase in shear stresses along the sealer-dentine interfaces as the compressive load was applied from the base of the inverted truncated sealer core. Prior to reaching the maximum compressive load, the shear stresses reached a critical value whereupon delamination was initiated from the top of the inverted core. The increase in Poisson's ratio along the nondelaminated part of the core (i.e. expansion) resulted in increased work to continue the delamination and hence a change in the slope of the load–displacement curve (zone II). Upon reaching the maximum load, propagation of shear stresses toward the bottom of the interface resulted in complete interfacial delamination and a sudden sharp drop in the recorded load (zone III). During the final push-out phase (zone IV), resistance to displacement by sliding friction and surface roughness of the delaminated sealer core resulted in a progressive, less abrupt decline in the recorded load as the delaminated core was displaced out of the truncated hole.

For each sealer with or without SBF immersion, no significant differences were observed amongst the push-out strengths obtained from different dentine locations (Fig. 3b). Thus, data from the apical, middle and coronal thirds of the roots were pooled to provide a more robust analysis of the effects of sealer type and SBF immersion on push-out strengths (Fig. 3c). When the specimens were tested without SBF immersion, significant differences ($P < 0.001$) were observed amongst the three sealers, with the calcium silicate-based sealer producing the highest push-out strength (16.2 ± 6.5 MPa) followed by AH Plus Jet (3.5 ± 1.7 MPa) and Pulp Canal Sealer (0.7 ± 0.6 MPa) in decreasing order. Significant differences in push-out strength was also observed for specimens that were tested after they were immersed in SBF for 4 weeks ($P < 0.001$), following the same order as previously described (calcium silicate-based sealer 22.4 ±5.0 MPa; AH Plus Jet 6.6 ± 1.7 MPa; Pulp Canal Sealer 0.4 ± 0.3 MPa). Interaction of these two factors were also significant ($P < 0.001$). For the AH Plus Jet and the calcium silicate-base sealer, Student paired t-tests revealed significant differences ($P < 0.05$) between the push-out strengths generated from specimens that were tested without SBF immersion and those that were tested after immersion in SBF.

The per cent distribution of failure modes amongst the six subgroups is presented in Fig. 4. No cohesive failure was observed for Pulp Canal Sealer. This sealer also exhibited an increase in the percentage of adhesive failure after storage in SBF. A preponderance of mixed failures was seen in AH Plus under the two storage conditions, whilst cohesive failures within the sealer were predominantly identified for the calcium silicate-based sealer.

Under scanning electron microscopy, failures classified as adhesive failures in the Pulp Canal Sealer groups invariably contained some sealer remnants along the dentinal walls (not shown). However, the overall impressions of those dentinal walls were still relatively smooth when compared with the mixed failures observed in the other sealer groups. A cohesive failure in AH Plus Jet after SBF immersion is shown in Fig. 5a. A high magnification view of the fractured sealer surface revealed characteristic multi-faceted fillers that were partially embedded, amongst other smaller fillers, within a resinous matrix (Fig. 5b). A mixed failure mode in the calcium silicate-based sealer after SBF immersion is depicted in Fig. 6a. Spherical bodies were identified along the sealer–dentin interface as well as the surface of the fractured sealer (Fig. 6b). These spherical phases were not observed from fractured specimens of the same sealer that had not been immersed in SBF (not shown). Very high magnification views of the specimens that had been immersed in SBF

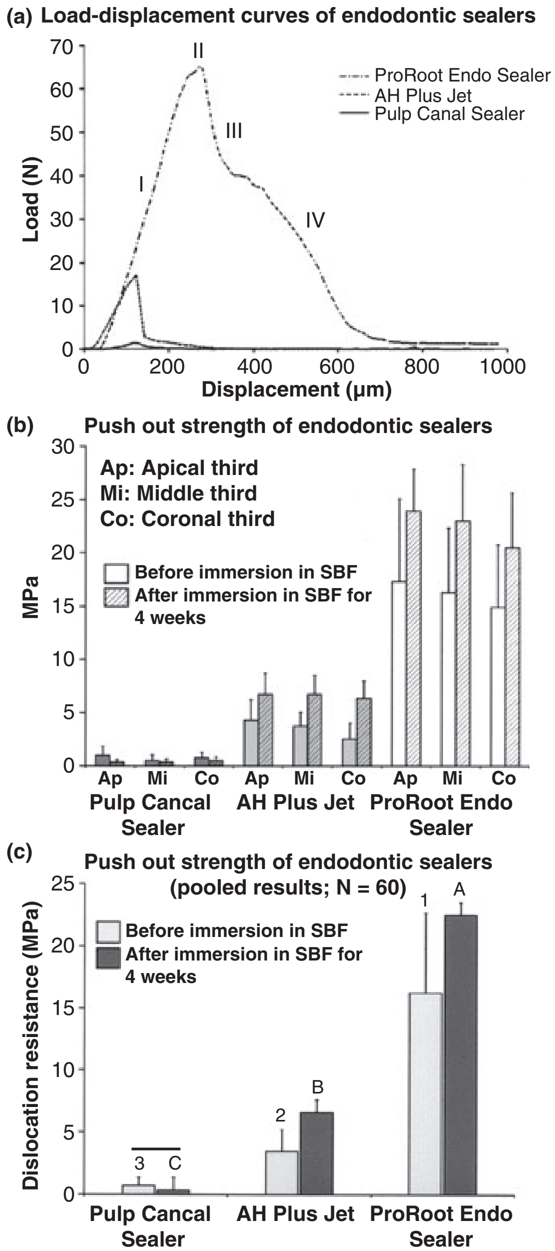

Figure 3 Push-out strength results. (a) Representative load-displacement curves of the three sealers that were tested in bulk without an accompanying gutta-percha core. Load is expressed as Newtons (N) and displacement is expressed as microns (μm). Zone I: initial linear increase in load; zone II: change in slope of the load–displacement curve before reaching maximum load; zone III: initial sudden sharp drop in recorded load upon interfacial delamination; zone IV: final push-out phase. When magnified, the four regions described for ProRoot Endo Sealer could also be seen in the load–displacement curves of AH Plus Jet and Pulp Canal Sealer. (b) dislocation resistance (expressed as MPa) of the three sealers in the apical third (Ap), middle third (Mi) and coronal third (Co) of the root dentine with and without storage in a simulated body fluid (SBF) (n = 20/location/storage subgroup). As there were no statistical differences in the push-out strengths of each sealer amongst different locations at each time period, data from the three locations were pooled (n = 60) for subsequent statistical comparisons. (c) The pooled data was analysed using a two-way ANOVA design with sealer type and SBF storage as independent variables. For specimens tested without SBF immersion, sealers with different numerals above their corresponding data columns represent significant differences ($P < 0.001$). For specimens that were tested after they were immersed in SBF, sealers with different upper case letters above their corresponding data columns represent significant differences ($P < 0.001$). For each sealer type, a horizontal bar above the respective columns for the two immersion protocols indicates no statistical difference ($P > 0.05$).

before testing revealed phase transformation of the spherical bodies to spherules with clustered polycrystalline surfaces (Fig. 6c). Individual crystallites that protruded from the surface of these spherules were about 40–70 nm in diameter (Fig. 6d).

Discussion

This study utilized a modified push-out protocol that was designed specifically to examine the retentive potential of sealers in radicular dentine. Although the study design is far removed from clinical practice, the results indicate that under identical cleaning and shaping conditions that may not be easily achieved under a clinical setting, the dislocation resistance of a particular sealer is independent of the location of the radicular dentine. Moreover, the dislocation resistance of the three sealers were significantly different from each other and that two of the three sealers exhibited higher dislocation resistance after immersion in SBF. Thus, the null hypothesis has to be rejected. Although a modified push-out test design was used in this study, it is interesting to note that the relatively low push-out strengths for AH Plus and Pulp Canal Sealer were similar to the range reported for similar sealers (2.00 ± 0.65 MPa for AH Plus and 0.79 ± 0.52 MPa for Kerr EWT sealer) in a previous study (Fisher *et al.* 2007).

Although testing designs that involve the use of natural canal spaces have obvious pragmatic appeal to clinicians, there are severe limitations from a materials science perspective. First, the application of a compressive load on top of a thermoplastic material, which has

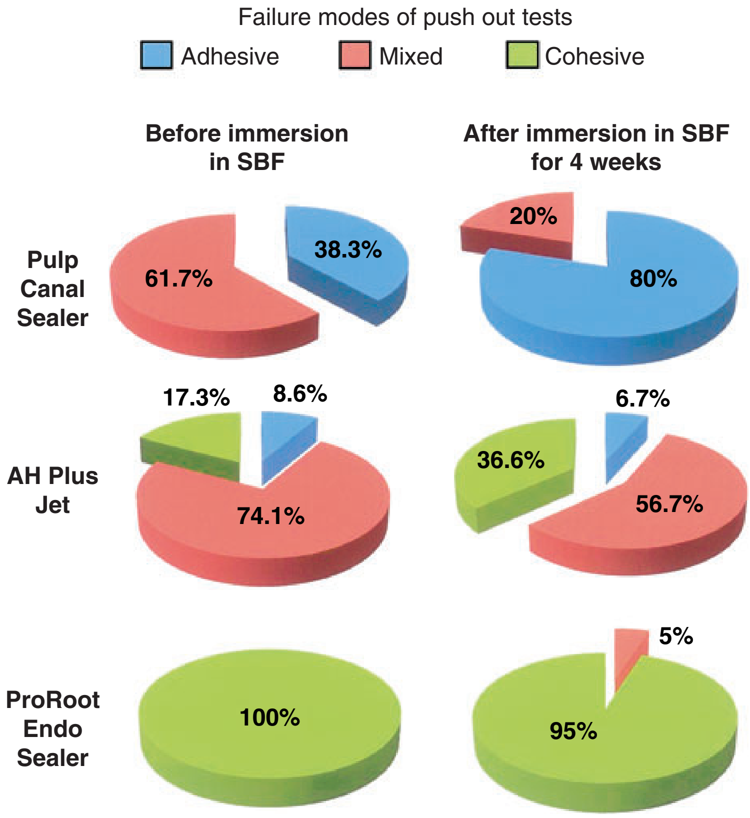

Figure 4 Distribution of adhesive, mixed and cohesive failures of the three sealers in specimens that were tested without immersion in simulated body fluid (SBF) and specimens that were tested after immersion in SBF for 4 weeks.

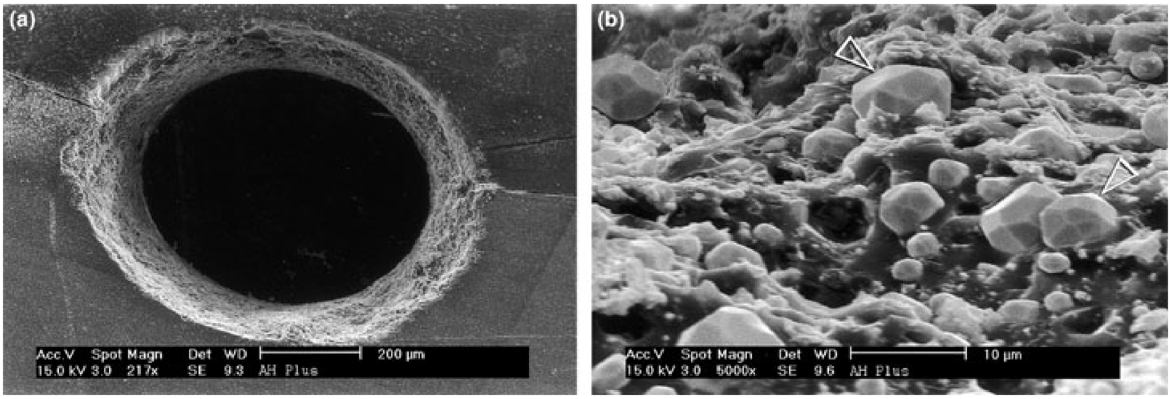

Figure 5 Scanning electron microscopy (SEM) of AH Plus after immersion in simulated body fluid (SBF) and push-out testing. (a) Low magnification SEM of a cohesive failure mode exhibited by a specimen from the AH Plus Jet group after SBF immersion. (b) A higher magnification view showing the presence of large, multi-facet fillers (open arrowheads) that are characteristic of the AH Plus sealer. These fillers were embedded in a resinous matrix together with other fine filler particles.

the tendency to flow during testing generates results that are susceptible to erroneous interpretation. Unless the rheological properties of the materials being compressed are equivalent (Kohyama *et al.* 2003, Törnqvist *et al.* 2004), statistical comparison of the results derived from two thermoplastic root filling materials is virtually meaningless. This could also have been responsible, in part, for the recent report that sealers tested in thin films using the thin-slice push-out test were considerably weaker than when the same sealer

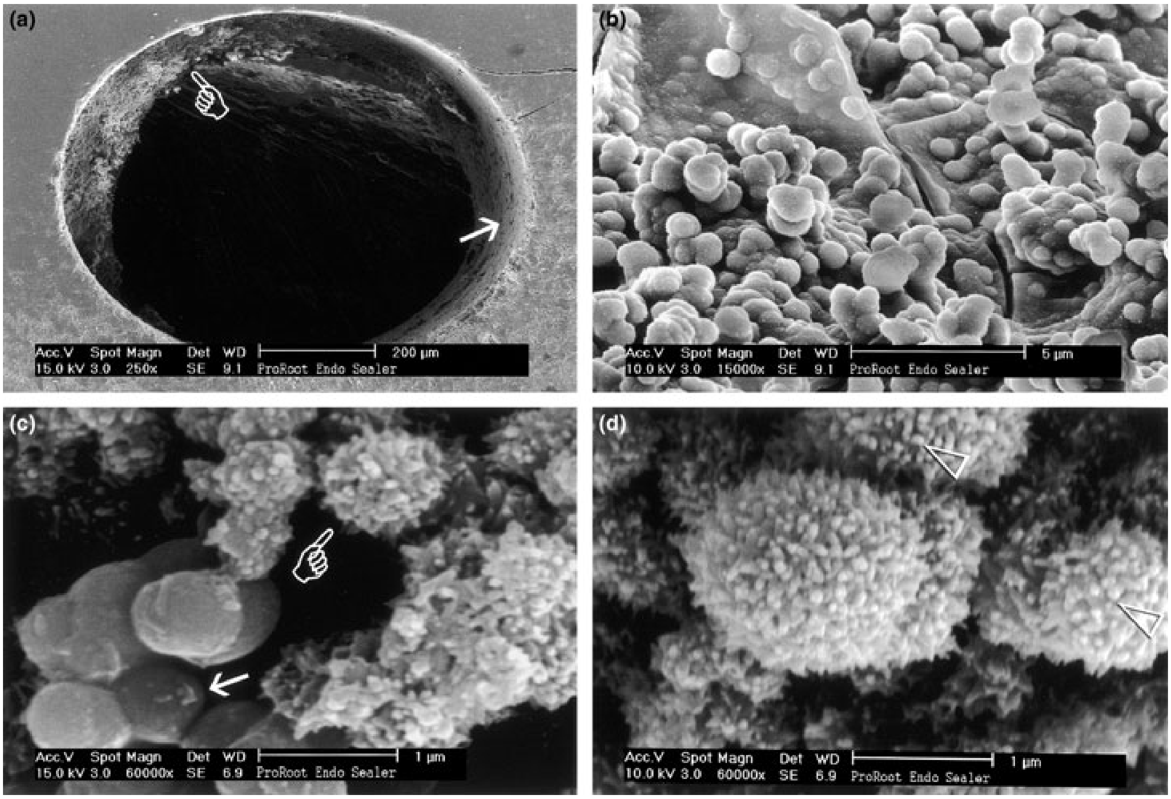

Figure 6 Scanning electron microscopy (SEM) of ProRoot Endo Sealer after immersion in simulated body fluid (SBF) and push-out testing. (a) Low magnification SEM of a mixed failure mode exhibited by a specimen from the ProRoot Endo Sealer group after SBF immersion. Sealer remnants (pointer) could be seen on part of the wall. The remaining part of the walls was devoid of sealer remnants and appeared comparatively smooth (arrow). (b) A higher magnification view of the sealer remnants, showing the presence of spherical bodies on sealer surface. These spherical bodies were previously shown to be amorphous calcium phosphate-like spheres that were formed by the reaction of calcium hydroxide released by the calcium silicate with the phosphate ions present in the SBF. (c) A very high magnification view showing that some of the amorphous calcium phosphate-like spheres (arrow) were spontaneously transformed into apatite-like clusters along the surface of those spheres (pointer). (d) A close-up view of a spherical apatite-like cluster showing the presence of individual apatite-like crystallites (open arrowheads).

was tested in bulk by eliminating the thermoplastic core material from the canal space (Jainaen *et al.* 2007). The important results generated by those authors provided incontestable substantiation that the so-called 'push-out bond strength' produced by the conventional thin-slice push-out test is not a material property. It is prudent to emphasize that the mechanical and physical properties of engineering and biomaterials such as flexural strength, fracture toughness or melting point should exhibit a consistent range of values under identical testing conditions (Callister 1994).

To minimize the shortcoming of applying a compression stress over a compliant material, the largest plunger that corresponds to the size of the thermoplastic root filling material is usually selected for the thin-slice push-out test (Gesi *et al.* 2005, Bouillaguet *et al.* 2007) Whilst this is a legitimate compromise, the procedure succinctly requires the use of different diameter plungers for different depths of a tapered root canal. As the contact surface areas of the plungers are different, data generated from different parts of the canal walls are nonstandardized. Thus, it is futile to statistically compare the results generated by a conventional thin-slice push-out test from the coronal third, versus those generated from the middle and apical thirds of the canal walls.

The third limitation involves the testing of root fillings that comprise multiple, nonuniform interfaces. Whilst the uneven distribution of stress fields around interfaces with variable circumferential thickness cannot be over-emphasized (Shirazi-Adl & Forcione 1992, Mequid & Zhu 1995), the uncertainty with respect to which interface was consistently dislodged imposes rigorous challenges when specific hypotheses such as the dislocation resistance of sealers from radicular dentine are to be tested.

The fourth limitation is that one is almost certain to find noninstrumented areas that co-exist with instrumented areas in an oval-shaped canal that has been cleaned and shaped (Peters 2004). For the noninstrumented areas that are treated with sodium hypochlorite as an irrigant, one should expect increases in both undercut retention and surface contact areas within the calcospherite-containing regions (Wakabayashi *et al.* 1993, Tatsuta *et al.* 1999) that inadvertently augments the dislocation resistance of the sealer being investigated. For example, comparing the results generated from a natural canal space with 50% noninstrumented canal walls versus one that has 20% noninstrumented canal walls may result in erroneous conclusions on the dislocation resistance of various sealers from radicular dentine. It is unrealistic to quantify the extent of noninstrumented natural canal walls from a root slab either before or after a push-out test. Because of these limitations, a modified push-out strength testing design was utilized in the present study.

Even without SBF immersion, the calcium silicate-based sealer was approximately 16 times as difficult to be dislodged from the radicular dentine walls as Pulp Canal Sealer, and almost four times as resistant to dislodging as AH Plus Jet. This may be due, in part, to the hardness of the calcium silicate-based sealer after setting in the presence of 100% relative humidity. As natural root canals cannot be completely dehydrated (Amyra *et al.* 2000, Hosoya *et al.* 2000) due to the retention of moisture within the dentinal tubules, similar hardness should be expected of the set sealer when it is used for filling natural canals. The tenacity of this sealer to radicular dentine cannot be solely attributed to sealer penetration into the dentinal tubules following depletion of the smear layer, as the dentine from the apical third of the roots is often highly sclerotic. It is beyond the scope of this study to provide definitive annotations on whether the increased dislocation resistance is caused by the frictional resistance or micromechanical/chemical adhesion of the sealer to dentine (Shirazi-Adl 1992, Goracci *et al.* 2005). This issue should be further investigated in the future using more advanced transmission electron microscopy and chemoanalytical techniques. Nevertheless, the increased dislocation resistance of the calcium silicate-based sealer to radicular dentine should be advantageous in maintaining the integrity of the sealer–dentine interface during tooth flexure, as well as during the preparation of postholes within the filled canal spaces.

The concern on whether the dislocation resistance of root canal sealers is adversely affected by the contamination of body fluids was simulated in the present study by immersing the specimens in a SBF. This is an exaggerated simulation as the entire tooth slab was immersed in the SBF after the cavities were filled with sealers. The increase in dislocation resistance of the AH Plus Jet is probably caused by swelling of the epoxy resin component after water sorption (Fernández-García & Chiang 2002, Dömötör & Hentschke 2004). For the calcium silicate-based sealer, continuous maturation of the material (Andriamanantsilavo & Amziane 2004) may also have increased the material's dislocation resistance. However, the occurrence of spherical phases along the sealer–dentine interface and within the remnant fractured sealer after the specimens were immersed in the phosphate-containing SBF is notable. These spherical phases have previously been identified as amorphous calcium phosphate when Portland cement was immersed in a phosphate-containing fluid (Tay *et al.* 2007). Amorphous calcium phosphate phases undergo spontaneous transformation to carbonated apatites (Gadaleta *et al.* 1996), producing hollow spherules of apatite clusters (Eanes 2001, Tay & Pashley 2008) that contributed to the *ex vivo* bioactivity of calcium silicate-containing materials when they interact with phosphate ions. Similar apatite-containing clusters had been observed when Mineral Trioxide Aggregate was immersed in phosphate-containing fluids (Sarkar *et al.* 2005). The apatitic composition in these spherules has also been established using x-ray diffraction (XRD) and Fourier transform-infrared spectroscopy (FT-IR) (Tay *et al.* 2007). No attempt was made to analyse the comparatively smooth spherical phases and the crystallite-containing spherules in this study, as these phases were present adjacent to calcium-phosphate rich dentine and on the surface of the fractured sealer. The use of energy dispersive X-ray analysis to analyse these surface phases would have yielded information that includes the subsurface elemental composition of the dentine and sealer components. Likewise, these phases were not amendable for collection and purification for XRD and FT-IR analyses. Thus, they are only referred to as amorphous calcium phosphate-like and apatite-like in the present study. Generation of these reaction phases only in specimens that were immersed in the SBF could also have resulted in the increase in frictional resistance of the sealer–dentin interface. Although it is presumptuous to correlate the '*in vitro* bioactivity' (i.e. the ability to form carbonate hydroxyapatite on the surface of a biomaterial when it is exposed to SBF) (LeGeros 2002, Zhao *et al.* 2005, Panzavolta *et al.* 2008) observed in the present study with 'clinical bioactivity' (i.e. the property of the material to develop a direct, adherent and strong bonding with the bone tissue) (Hench *et al.* 1978, Hench 1994), the issue of 'clinical bioactivity' associated with the use of endodontic sealers in general is of practical clinical interest and should be duly investigated.

Conclusion

Within the limits of the modified push-out testing design utilized in the present *ex vivo* study, it may be concluded that:

- Under identical cleaning and shaping conditions, the dislocation resistance of ProRoot Endo Sealer, AH Plus Jet and Pulp Canal Sealer are independent of the location of the radicular dentine.
- The dislocation resistance of the three sealers are in descending order: ProRoot Endo Sealer, AH Plus Jet and Pulp Canal Sealer.
- Both ProRoot Endo Sealer and AH Plus Jet exhibited higher dislocation resistance after immersion in a SBF.
- ProRoot Endo Sealer exhibited amorphous calcium phosphate-like phases that spontaneously transformed into apatite-like phases after immersion in the phosphate-containing SBF. This phenomenon probably accounts for the *in vitro* bioactivity of this calcium silicate-based sealer.

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