Interfacial Strength of Resilon and Gutta-Percha to Intraradicular Dentin

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Abstract

Strengthening of Resilon-filled roots via an adhesive interface should be reflected by improvement in the interfacial strength and dislocation resistance between the root fillings and intraradicular dentin. This study compared the interfacial strengths of Resilon/Epiphany and gutta-percha/AH Plus using a thin-slice push-out test design. Failure modes of root slices after push-out testing were examined with environmental scanning electron microscopy. The gutta-percha group exhibited significantly higher interfacial strength than the Resilon group, when premature failures that occurred in Resilon root slices were included in the statistical analysis. The gutta-percha root slices failed exclusively along the gutta-percha/sealer interface. The Resilon root slices failed predominantly along the sealer/dentin interface with recognizable, fractured resin tags. Detachment of the Resilon from the Epiphany sealer was also surprisingly observed in some specimens. The similarly low interfacial strengths achieved with both types of root filling challenges the concept of strengthening root-filled teeth with the new endodontic material.

Key Words

Resilon, gutta-percha, push-out test, interfacial strength, environmental SEM

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The use of gutta-percha and root canal sealers for obturating root canals has remained the standard of care in endodontics, despite their inability to routinely achieve an impervious seal along the dentinal walls of the root canal (1, 2). To reduce apical and coronal leakage (3, 4), both total-etch and self-etch adhesives have been employed experimentally for sealing intraradicular dentin before the obturation of root canals with gutta-percha (5–7). However, these techniques were hampered by the lack of copolymerization between the methacrylate-based dentin adhesives, the epoxy resin- or zinc oxide eugenol-based root canals sealers, and gutta-percha (8). The use of resin cements alone for root canal obturation also engenders difficulties in their removal during re-treatment (9). These obstacles have apparently been resolved by the recent launching and intensive promotion of a polyester-based thermoplastic root filling material (Resilon; Resilon Research LLC, Madison, CT) that is claimed to be bondable to methacrylate-based resins (10).

Resilon is thermoplastic because of the incorporation of polycaprolactone (11), a biodegradable aliphatic polyester (12) that has a low glass transition temperature of −62°C (13). It is bondable to methacrylate-based resins as it contains dimethacryate resins (11). This highly filled, radio-opaque root-filling material can couple to a variety of dentin adhesives and resin-type sealers, including Epiphany (Pentron Clinical Technologies, Wallingford, CT), Real Seal (SybronEndo, Orange, CA) and Next (Heraeus-Kulzer, Armonk, NY). The adjunctive use of self-etch adhesives and methacrylate-based resin sealers with Resilon purportedly creates a monobloc between the intraradicular dentin and the root-filling material that is more resistant to both bacterial leakage (10) and root fracture (14) when compared with similar teeth that were root-filled with gutta-percha and conventional sealers.

Strengthening of the Resilon-filled roots with an adhesive joint should be reflected by improvements in interfacial strength and dislocation resistance between the root filling material and intraradicular dentin (15), which may be evaluated using thin-slice push-out tests (16–20). Thus, the aims of this study were to compare the interfacial strengths and failure modes of Resilon/Epiphany-filled root canals with those that were obturated with gutta-percha and a nonbonding endodontic sealer. The null hypothesis tested was that there is no difference in the interfacial strength of Resilon and gutta-percha to intraradicular dentin.

Materials and Methods

Twenty extracted single-rooted human teeth were used in this study. For each tooth, canal patency was established using a #10 Flex-o-file (Dentsply Maillefer, Tulsa, OK). Cleaning and shaping were performed with a crown-down technique, using Profile nickel-titanium rotary instruments (Dentsply Maillefer). Each canal was prepared to ISO size 25, 0.06 taper and 1-mm short of the apex. The canals were irrigated in between instrumentation with 17% ethylene diamine tetra-acetic acid (EDTA) and 3% sodium hypochlorite. EDTA was used as the final rinse before root canal obturation, as required by the manufacturer of Epiphany. This protocol was employed for all teeth to eliminate the introduction of an additional experimental variable. The debrided root canals were dried with multiple paper points and randomly divided into two equal groups.

Warm Vertical Compaction of Resilon

Epiphany self-etching primer was introduced into the root canal with a microbrush, with the excess removed with paper points. A nonstandardized Resilon master

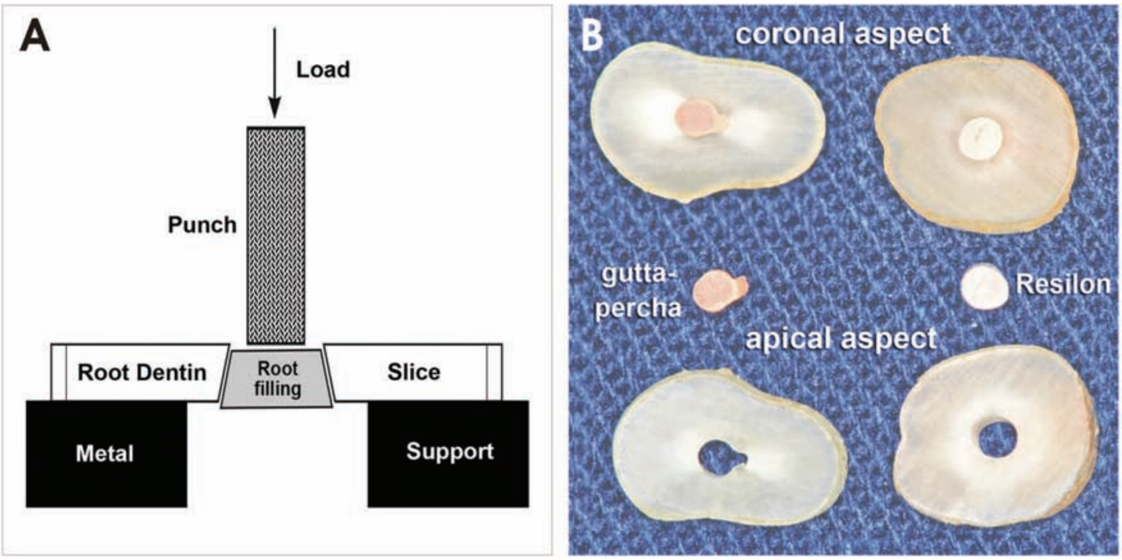

Figure 1. (*A*) Schematic representation of the set-up for testing the interfacial strength of a root canal filling with the thin-slice push-out test. (*B*) Digitized photographs of the coronal and apical portions of root slices prepared from the gutta-percha/AH Plus group and the Resilon/Epiphany group before and after pushing out the root fillings. The latter remained intact after dislodging from the root slices.

cone was tried-in to within 1 mm of working length. Epiphany sealer was then placed into the root canal using a lentulo spiral, and the Resilon was downpacked using the continuous wave condensation technique (System B, SybronEndo, Orange CA) at 150°C. Backfilling was performed with Obtura II (Spartan, Fenton, MO) at 140°C. After backfilling, the coronal surface of the root filling was light-cured for 40 s to achieve an immediate coronal seal.

Warm Vertical Compaction of Gutta-Percha

A nonstandardized gutta-percha master cone (Hygienic, Coltène/Whaledent Inc., Mahwah, NJ) was used with an epoxy resin-based root canal sealer (AH Plus, Dentsply Maillefer). Root canals were obturated using the continuous wave condensation technique at 200°C and backfilled with Obtura II at 185°C.

After root filling, the access cavities were restored with a noneugenol temporary filling (Coltosol, Coltene, Mahwah, NJ) and stored in distilled water at 37°C for 24 hr. Each root was sectioned at 2 mm below the cementoenamel junction into 3 to 4 1-mm thick serial slices with a slow-speed saw (Isomet, Buehler, Lake Bluff, IL) under water cooling. This resulted in 33 slices for the Resilon group and 30 slices for the gutta-percha group. Digitized images of the coronal and apical of each slice were captured at 10× magnification using a CCD camera attached to a stereomicroscope.

Thin-Slice Push-Out Test

The thicknesses of each root slice was measured by means of a digital caliper. After securing with cyanoacrylate glue to a loading fixture, each slice was subjected to compressive loading via a universal testing machine (Controls S.P.A., Milan, Italy) that was equipped with a 1 mm-diameter cylindrical plunger. The plunger was positioned so that it only contacted the root filling on loading, introducing shears stresses along the interfaces. The loading force was applied at a crosshead speed of 0.5 mm/min in an apical-coronal direction, so as to displace the root-filling toward the larger, coronal part of the root slice (Fig. 1*A*). Failure was manifested by the extrusion of the intact cone of root filling from the root slice (Fig. 1*B*), and confirmed by the appearance of a sharp drop along the load/time curve recorded by the testing machine.

The circumferences of the coronal (Cc) and apical aspects (Ca) of each slice were measured from the digitized images, using an image analysis software (Image 4.01, Scion Corp., Frederick, MA). The interfacial area of the root filling was approximated by $0.5(Cc+Ca)h$, where h is the root slice thickness. Interfacial strength was computed from the load obtained at detachment and the estimated interfacial area. As the data were not normally distributed, they were analyzed using the Mann-Whitney rank sum test, with statistical significance set at $\alpha = 0.05$. Root fillings that were dislodged prematurely during slicing were assigned null values and included in the statistical analysis.

Environmental Scanning Electron Microscopy (ESEM)

After the push-out tests, five "empty" slices from each group were randomly selected for morphologic examination. They were kept in water to prevent them from dehydration. The coronal surfaces were brought into relief by etching with 10% phosphoric acid for 15 s. After thorough rinsing with distilled water, the wet specimens were secured with carbon tape to aluminum stubs, placed on the Peltier (cooling) stage of a field emission-ESEM (Philips XL-30 ESEM-FEG; Eindhoven, The Netherlands) and examined without coating at 15 kV using the gaseous secondary electron mode at 4°C and 5.9 Torr to achieve a 95% relative humidity (21).

Results

All root slices from the gutta-percha group remained intact, while 6 out of 33 slices from the Resilon group dislodged during slicing. When these premature failures were included in the statistical analysis, the interfacial strength of the Resilon group (0.50 ± 0.41 MPa) was significantly lower ($p = 0.025$) than that of the gutta-percha group (0.94 ± 0.77 MPa).

Interfacial failure in the Resilon group predominantly occurred along the surface of the intraradicular dentin (Fig. 2*A*), from which fractured resin tags could be identified from the dentinal tubular orifices (Fig. 2*B*). Detachment of the Resilon from the resin sealer could be seen in two specimens that exhibited premature failure during slicing (Fig. 2*C*). For the gutta-percha group, the intraradicular dentin was covered with the AH Plus sealer (Fig. 3*A*), with remnant blebs of the

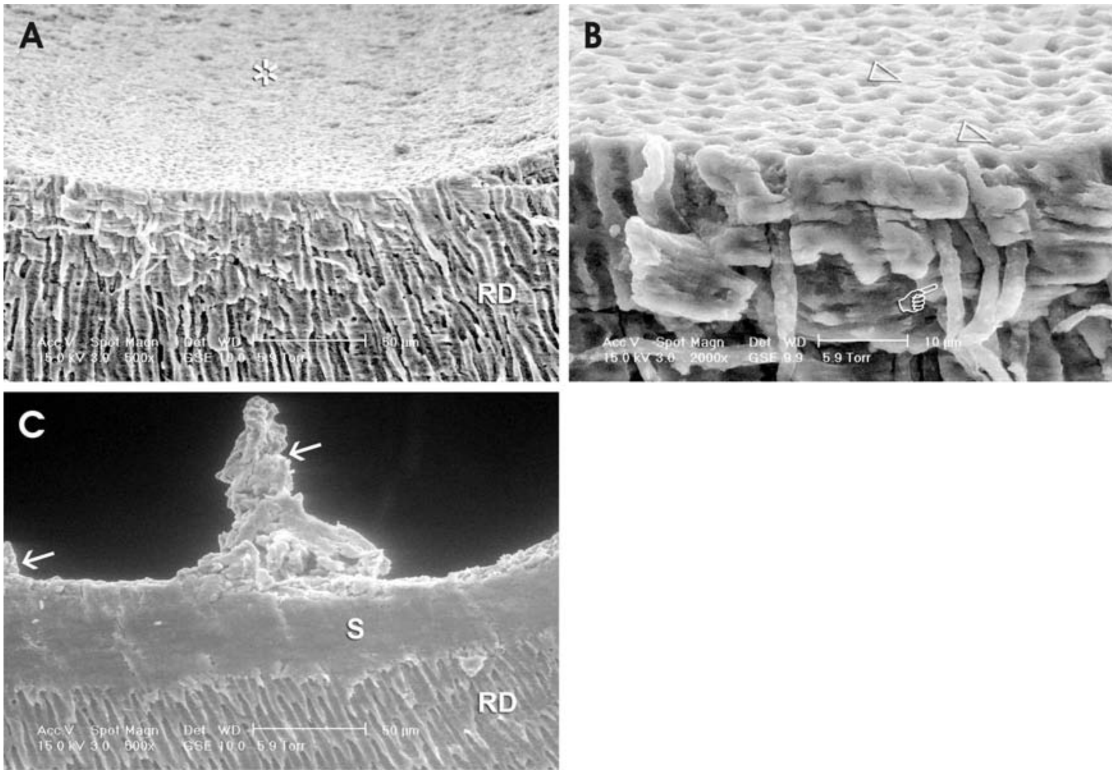

Figure 2. ESEM images illustrating the types of interfacial failure in the Resilon/Epiphany group. They represented résumés of the events that accounted for the dislodging of the root fillings. (*A*) A low magnification micrograph showing the most commonly identified type of interfacial failure—along the surface of the bonded intraradicular dentin (asterisk). RD: sectioned surface of the root dentin slice. (*B*) A higher magnification view of the previous micrograph, showing remnant resin tags (pointer) on the cut surface of the root slice, and fractured resin tags (open arrows) on the surface of the debonded intraradicular dentin. (*C*). A type of interfacial failure that was identified from specimens in which the root fillings dislodged prematurely during root slice preparation. The Resilon material, which was supposed to be bondable to methacrylate resins, was detached from a thick resin-sealer layer (S). The latter remained bonded to the underlying root dentin (RD). Blebs of resin sealer (pointers) extended into the space previously occupied by the Resilon, probably provided some form of mechanical retention for the polyester root filling material.

sealer extending into the space previously occupied by the gutta-percha (Fig. 3*B*). Large angular crystallite fillers from the sealer could be seen along the detached surface (Fig. 3*C*).

Discussion

In light of the push-out test results, the null hypothesis that there is no difference in the interfacial strength of Resilon and gutta-percha to intraradicular dentin has to be rejected. In gutta-percha-filled canals, the polyisoprene root filling material detached from the sealer, with the latter retained on the dentin surface. As there is no bonding between the gutta-percha, root sealer and dentin, the low interfacial strength in this group is anticipated, as resistance to dislocation, or fracture, is derived directly from Coulomb's friction (22–24). This is probably enhanced by the presence of surface asperities (25, 26) such as the protrusion of sealer blebs (Fig. 3*B*) and large crystalline fillers (Fig. 3*C*) into the heat-softened gutta-percha.

In the presence of an adhesive interface, the even lower interfacial strength of the Resilon-filled root slices was unexpected. Even when premature failures were excluded, the remaining Resilon slices demonstrated only similar strength (0.60 ± 0.38 MPa; $p = 0.173$) as the gutta-percha group. As the adhesive was applied after EDTA was employed as the final rinse, the compromising oxidizing effect of NaOCl on sealer polymerization (27, 28) should be negligible. Unlike gutta-percha specimens, the weak link in Resilon-filled root canals resided predominantly along the sealer-dentin interface. This should not be caused by the retention of the smear layer or the inability of the adhesive to penetrate dentinal tubules, as fractured resin tags could be identified at least from some tubular orifices (Fig. 2*B*).

The pattern in which these resin tags were fractured suggested that they were split along a propagating crack front, via the presence of stress raisers that pre-existed along the surface of the adhesive-bonded dentin (29–31). These stress raisers could have been formed by direct contact of the Resilon with dentin that resulted from the nonuniform distribution of the resin sealer, or the presence of gaps that were initiated by polymerization shrinkage of the resin sealer. Although the Epiphany sealer polymerizes relatively slowly (approximately 25 min) when it is allowed to self-cure, which is the likely scenario in the middle and apical thirds of the root canals, its rate of polymerization may be accelerated by heat generated during warm vertical compaction and Obtura back filling (32). It is also notoriously known that postspaces created in root canals exhibit highly unfavorable cavity configuration factors that are detrimental to the relief of polymerization shrinkage stresses (33, 34). Considering that whole root canals are much longer than postspaces, the conditions for stress relief by resin flow would be substantially worse, particularly when the manufacturer's instructions on light-curing of the coronal portion of the Epiphany sealer is followed to establish an immediate coronal seal.

A notable advantage of Resilon, according to the manufacturer, is its ability to bond to methacrylate-based resin sealers via the incorporation of dimethacrylates in the polyester-based material. Thus, it is rather surprising that debonding occurred between the Resilon and the Epiphany sealer (Fig. 2*C*), as resin composites normally couple well to

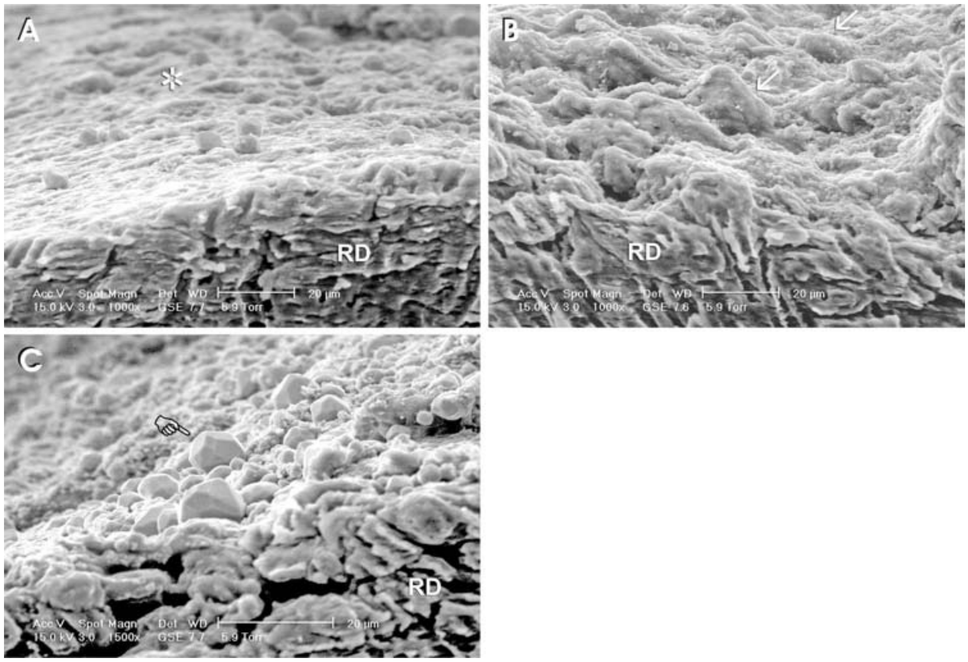

Figure 3. Representative ESEM images taken from the gutta-percha/AH Plus group following dislodging of the root fillings with the thin-slice push-out test. (*A*) An overall view of a characteristic type of interfacial failure that occurred by the detachment of the gutta-percha, with remnant epoxy resin sealer (asterisk) over the surface of the intraradicular dentin. RD: sectioned surface of the root dentin slice. (*B*) A higher magnification view, showing blebs of remnant resin sealer (arrows) that projected into the space previously occupied by the unbonded gutta-percha. (*C*) Crystalline structures (pointer) that represent large, remnant fillers (probably zirconium oxide or calcium tungstate—AH Plus, MSDS data) derived from the AH Plus resin sealer. RD: root dentin slice surface.

dentin adhesives or resin cements. One possible reason could be the low concentration of dimethacrylates that is present in matrix component of Resilon. Another possible reason could be the absence of free radicals within the well-polymerized Resilon material for effective coupling with the Epiphany sealer (35). These issues should be investigated in future studies, by testing the bond strengths of flat, smooth surfaces of Resilon to the resin sealer, to eliminate the contribution of surface asperities for mechanical retention.

Within the limits of this study, it may be concluded that the interfacial strength achieved with Resilon/Epiphany to intraradicular dentin is not superior to that of gutta-percha and a conventional epoxy-resin sealer. The results challenge the concept of strengthening root canals with the new root filling system.

Acknowledgments

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