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Micropush-out bond strengths of gutta-percha versus thermoplastic synthetic polymer-based systems – an *ex vivo* study

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Abstract

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Aim To compare the interfacial strength and failure mode of root fillings consisting of different technique–material combinations.

Methodology Human mandibular premolars ($n = 144$) instrumented to apical size 40 and .06 taper were divided into 12 experimental groups. The root canals were filled with either gutta-percha (groups 1–6) or Resilon (groups 7–12) core materials combined with AH Plus (groups 1, 4, 7, 10), Ketac-Endo (groups 2, 5, 8, 11) or Epiphany (groups 3, 6, 9, 12) using cold lateral compaction (groups 1–3, groups 7–9) or System B with Obtura II (groups 4–6, groups 10–12).Three serial 1.00 ± 0.05-mm-thick root slices were prepared and push-out tests on the filling material were performed. Interactions amongst the compaction techniques, core materials and sealers were analysed using a three-way analysis of variance (ANOVA) ($P < 0.05$). One-way ANOVA and Duncan's Multiple Range tests were used to compare the bond strengths of the 12 groups. Fracture modes of all root slices were evaluated stereomicroscopically at ×40 magnification.

Results All the parameters except compaction techniques had significant interactions ($P < 0.05$). A significant difference was found amongst the groups ($P < 0.05$). Gutta-percha/Ketac-Endo/cold lateral compaction and gutta-percha/AH Plus/cold lateral compaction groups had the highest micropush-out bond strength values ($P < 0.05$). The number of overall cohesive failures was significantly more than that of adhesive failures ($P < 0.05$).

Conclusions The push-out bond strengths of Resilon/Epiphany combinations were lower than those of gutta-percha/conventional root canal sealer combinations. Core materials and sealers may affect the push-out bond strengths of root canal filling materials.

Keywords: AH Plus, Epiphany, gutta-percha, Ketac-Endo, push-out bond strength, Resilon.

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Introduction

Using gutta-percha and root canal sealers for filling of 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 (Venturi & Breschi 2004, Vizgirda *et al.* 2004). Gutta-percha does not bond to the dentine, resulting in the absence of a complete seal (Saunders & Saunders 1990). Many different materials have been proposed as root canal fillings, but none have replaced gutta-percha, which is universally accepted as the 'gold standard' filling material.

Improvements in adhesive technology have fostered attempts to reduce apical and coronal leakage by

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bonding to root canal walls. Total-etch adhesives have been tested with resin cements as alternative root filling materials (Leonard *et al.* 1996, Mannocci & Ferrari 1998) and these laboratory studies reported that dentine adhesives significantly reduced apical leakage. Self-etching primers have also been used for bonding to root canal dentine (Lee *et al.* 2002, Economides *et al.* 2004). However, these techniques were hampered by the lack of copolymerization between the methacrylate-based dentine adhesives, the epoxy resin- or zinc oxide eugenol-based root canal sealers, and gutta-percha (Lee *et al.* 2002). The use of resin cements alone for root canal filling also results in difficulties during re-treatment (Ruddle 2004). Difficult application and lack of radiopacity are the other problems during the use of bonding agents and resins in root canals (Leonard *et al.* 1996, Imai & Komabayashi 2003).

A so-called endodontic monoblock between dental gutta-percha and sealers, such as zinc oxide-eugenol, epoxy resin, calcium hydroxide or glass–ionomer, cannot be created due to lack of chemical union of these materials (Lee *et al.* 2002, Saleh *et al.* 2003). The recent introduction of Resilon (Resilon Research LLC, Madison, CT, USA) as an alternative root filling material offers the promise of adhesion to root dentine (Shipper *et al.* 2004, 2005, Teixeira *et al.* 2004). This thermoplastic synthetic polymer is presented as a root canal filling material having bonding ability to resin sealers through the inclusion of resin with methacryloxy groups. This core material is used in conjunction with a dual-cured resin type sealer such as Epiphany Root Canal Sealant (Pentron Clinical Technologies, Wallingford, CT, USA).

Adhesion of the root filling to the dentinal walls seems advantageous for two main reasons. In a static situation, it should eliminate any space that allows percolation of fluids between the filling and the wall (Ørstavik *et al.* 1983). In a dynamic situation, it is needed to resist dislodgment of the filling during subsequent manipulation (Saleh *et al.* 2003).

The quality of adhesion on root canal dentine may be influenced by root canal irrigants and conditioners (Morris *et al.* 2001), root canal dentine hydration as a result of pulp removal, and the polymerization stress of resin cement in root canals due to unfavourable cavity configuration factors (Bouillaguet *et al.* 2003).

Bond strength has been measured using conventional tensile tests on external root dentine (Nikaido *et al.* 1999), or on the root canal dentine surface with the pull-out (Varela *et al.* 2003) and the push-out methods (Gesi *et al.* 2005, Skidmore *et al.* 2006, Ungor *et al.* 2006). The push-out test provides a better evaluation of the bonding strength than the conventional shear test because with the push-out test fracture occurs parallel to the dentine-bonding interface, which makes it a true shear test (Drummond *et al.*1996) for parallel-sided samples. It has the benefit of more closely simulating the clinical condition (Sudsangiam & Van Noort 1999). Interfacial strength and dislocation resistance between the root filling material and intraradicular dentine were evaluated using thin-slice push-out tests (Gesi *et al.* 2005, Skidmore *et al.* 2006, Ungor *et al.* 2006).

The aim of this *ex vivo* study was to compare the interfacial strengths and failure modes of root fillings in canals that were filled with different filling technique–material combinations. The null hypothesis tested was that Resilon/Epiphany provides higher interfacial strengths than gutta-percha/conventional sealer combinations.

Materials and methods

Sample selection

One hundred and forty-four freshly extracted human mandibular premolar teeth with straight root canals and anatomically similar dimensions, fully developed apices and free of cracks, caries or fractures, were selected. The teeth were cleaned of soft tissue and calculus.

Access cavities were prepared. Canal patency was determined by passing a file (size 10 K file; Mani K files, Japan) through the apical foramen.

Root canal preparation

The pulp tissue was removed with barbed broach (Dentsply Maillefer, Ballaigues, Switzerland). Canal working lengths were established 1.0 mm short of the apical foramina. The root canals were prepared with the crown-down technique according to the manufacturer's instructions for large canals. The coronal third was prepared to a size 6 Orifice Shaper and the apical third to size 40, .06 taper ProFile NiTi rotary instruments (ProFile; Dentsply Maillefer) to achieve a round canal shape and appropriate dimension for push-out test. Irrigation was performed using 3 mL of 2.5% NaOCl after every change of instrument. A lubricant was used (Glyde File Prep.; Dentsply Maillefer) throughout the cleaning and shaping of the root canal. Following biomechanical preparation and final rinse with 3 mL of NaOCl, 17% EDTA was used for 1 min, followed by distilled water for 1 min. Each canal was dried using size 40 paper points. Samples were randomly divided into 12 ($n = 12$) experimental groups.

Root filling

The root canals were filled with either gutta-percha (groups 1–6) or Resilon (groups 7–12) core materials using one of the three types of sealers: Epoxy resin-based sealer AH Plus (De-Trey-Dentsply, Konstanz, Germany) was used in groups 1, 4, 7, 10; glass–ionomer-based Ketac-Endo (ESPE Gmbh, Seefeld/Oberbay, Germany) was used in groups 2, 5, 8, 11 and groups 3, 6, 9, 12 were filled with composite resin-based Epiphany (Pentron Clinical Technologies). Filling methods were cold lateral compaction (groups 1–3, groups 7–9) or combined warm vertical compaction techniques using System B (Analytic Technology, Redmond, VA, USA) with Obtura II (Obtura Spartan, Fenton, MO, USA) (groups 4–6, groups 10–12).

Lateral compaction technique (groups 1–3 and 7–9)

All sealers were mixed according to manufacturers' instructions and placed into the root canal in the same volume (approximately 0.075 mL) using a lentulo spiral. The master points (either gutta-percha or Resilon) were placed into the root canal to full working length. Lateral compaction was performed using a size 30 finger spreader (Mani Spreaders, Tochigi, Japan) to a level approximately 1 mm short of the working length and ISO size 25, 30 accessory cones (Sure-endo, Kyeonggi-do, Korea) coated with sealer were inserted into the canal until the spreader could penetrate no more than 2–3 mm. The excess point was removed with a heated instrument and then compacted vertically.

Combined warm vertical compaction technique (groups 4–6 and 10–12)

Continuous wave of condensation (System B, Analytic Technology) was used after the application of one of the three sealers and two core materials mentioned above. In groups 4–6, a 0.08 taper System B plugger (Analytic Technology) set at 200 °C was introduced into the canal. The tip of the plugger was activated and condensation was terminated within 3 mm of working length. The plugger was held in position for 10 s before the System B was activated for 1 s and withdrawn from the tooth. Backfilling was performed with Obtura II (Obtura Spartan) using 23 gauge needle tips at a temperature of 185 °C. In the Resilon groups (groups 10–12) the System B and Obtura II were used at reduced temperatures (140 and 150 °C respectively). After the filling procedure, all teeth were decoronated using a slow-speed diamond saw (Minitom, Struers, Germany) at 200 rpm to equalize root lengths as 14 mm.

The coronal surface of the root fillings was temporized with a composite resin (Valux Plus, 3M, Espe, St Paul, MN, USA) restoration. The roots were stored in gauze dampened with aqueous solution containing 0.1% sodium azide (NaN_3) for 3 weeks at 37 °C to allow the sealer to set.

Preparation of root slices for micropush-out test

Each root was sectioned perpendicular to the long axis into three serial slices with 1.00 ± 0.05 mm thickness using a low-speed saw running at 200 rpm under water cooling. Thirty-six slices per group were achieved. The apical portion of each root was preserved for measurement of microleakage in another investigation.

Apical and coronal aspects of each slice were then digitally photographed using a stereomicroscope (Olympus SZ6045TR Zoom Streomicroscope, Olympus Optical Co., Ltd, Seoul, Korea) at ×4 magnification. The images were transferred to a computer and an Autocad software program (AutoCAD 2000, San Rafael, CA, USA) was used to measure the circumferences of the root canal from the coronal (C_c) and apical aspects (C_a) of each slice.

Micropush-out test

Each slice was marked on its coronal side with an indelible marker and the thickness of the slices was measured by using digital caliper to within 0.001 mm. After securing to a loading fixture, the filling material was loaded with a 1.0-mm-diameter cylindrical stainless steel plunger. The plunger tip was sized and positioned to touch only the filling material, without contacting the canal walls. The micropush-out tests were performed at a cross-head speed of 0.5 mm min^{-1} using a universal testing machine (Testometric, Rochdale, UK) (Fig. 1). The load was applied on the apical aspect of the root slice to avoid any limitation to filling material movement because of the canal taper. The peak force, at the point of extrusion of the root filling from the slice, was taken

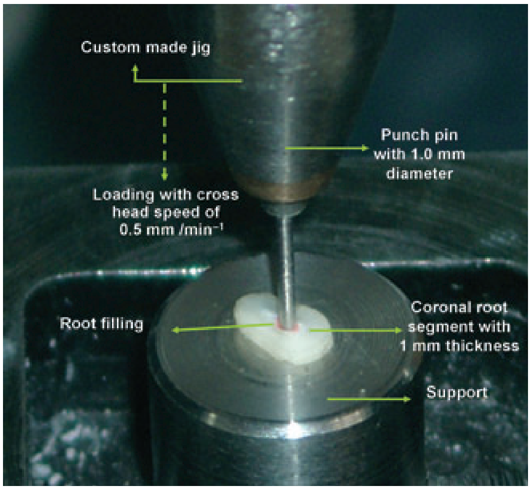

Figure 1 Micropush-out test design.

as point of bond failure and recorded in newtons (N). To express the bond strength in MPa, the load value was divided by the area of the bonded interface. The interfacial area of the root filling was approximated by the following formula:

$$A = 0.5(C_c + C_a) \times h,$$

where h was the root slice thickness (Gesi *et al.* 2005).

The bond strengths in MPa of the three slices were averaged to obtain a single value for each tooth. After the measurement of bond strength, both sides of the slices were examined under a stereomicroscope at ×40 magnification to determine modes of failure: adhesive at the filling–dentine interface or cohesive within the filling material.

Statistical analysis

The differences between the groups with regard to compaction techniques, core materials and sealers were statistically analysed with the three-way analysis of variance (ANOVA) and the mean values were separated using the Duncan's Multiple Range test. All two-way interactions between the main effects and the three-way interaction were added to the models. Nonsignificant three-way interaction was dropped from the model in the order of decreasing P-value. All remaining interactions were significant ($P = 0.05$). Duncan's Multiple Range test was used for the assessment of the pairwise comparisons.

Results

Ten specimens were dislodged during slicing; 1, 4, 1, 1 and 3 of 36 slices from the groups 5, 6, 9, 10 and 11 respectively. These premature failures were added to the statistical analysis as null push-out strengths. The root canal diameter measured on the digital images of the slices ranged between 1.50 and 3.00 mm due to tapered canal form and it was wider than that of the plugger tip.

The data for all 12 combinations of sealer, core material and compaction technique are provided in Table 1. The results of three-way ANOVA statistical

Table 1 Micropush-out bond strengths (MPa) and failure modes (n) determined by stereomicroscopy

Groups (n = 12)	Micropush-out bond strengths in MPa[a]	Failure mode, n (%)		Number of premature failures[b]
		Adhesive	Cohesive	
Group 1 GP/AP/CLC	3.86 ± 0.64d,e	3 (8.3)	33 (91.7)	0
Group 2 GP/KE/CLC	4.43 ± 1.03e	7 (19.4)	29 (80.6)	0
Group 3 GP/E/CLC	2.22 ± 0.94b,c	5 (13.9)	31 (86.1)	0
Group 4 GP/AP/CWVC	2.75 ± 0.88c	22 (61.1)	14 (38.9)	0
Group 5 GP/KE/CWVC	3.49 ± 0.94d	15 (41.7)	21 (58.3)	0
Group 6 GP/E/CWVC	2.25 ± 1.03b,c	27 (75.0)	9 (25.0)	4
Group 7 R/AP/CLC	2.21 ± 0.91b,c	9 (25.0)	27 (75.0)	0
Group 8 R/KE/CLC	1.74 ± 0.84a,b	9 (25.0)	27 (75.0)	0
Group 9 R/E/CLC	1.49 ± 1.06a	11 (30.6)	25 (69.4)	1
Group 10 R/AP/CWVC	1.99 ± 0.79a,b	22 (61.1)	14 (38.9)	1
Group 11 R/KE/CWVC	1.54 ± 0.97a	24 (66.7)	12 (33.3)	3
Group 12 R/E/CWVC	2.84 ± 1.82c	19 (52.8)	17 (47.2)	0

GP, gutta-percha; R, Resilon; AP, AH Plus; KE, Ketac-Endo; E, Epiphany; CLC, cold lateral compaction; CWVC, combined warm vertical compaction.

[a] Values are mean ± SD. Groups followed by same letters within the column are not statistically significant ($P > 0.05$).

[b] Premature failures were assigned null bond strength values and were included in the statistical analysis.

Table 2 Results of three-way ANOVA statistical analysis

Source of variation	d.f.	Mean square	*F*	*P*
Compaction techniques	1	1.201	1.16	0.284
Core materials	1	51.708	49.79	0.000
Sealers	2	4.992	4.81	0.010
Core materials × compaction techniques	1	8.776	8.45	0.004
Core materials × sealers	2	15.334	14.76	0.000
Compaction techniques × sealers	2	6.850	6.60	0.002
Error	134	1.039		

General mean 2.57 ± 1.34.

analysis with binary interactions and significance levels are shown in Table 2. All the parameters except compaction techniques had significant interactions ($P < 0.\ 05$) (Table 2). Resilon had significantly less push-out bond strength than gutta-percha ($P < 0.\ 05$) and the push-out bond strengths of the sealers were ordered decreasingly as Ketac-Endo, AH Plus and Epiphany.

Duncan's Multiple Range tests demonstrated statistically significant differences amongst the interfacial bond strengths of the groups ($F = 18.542$; $P = 0.000$) (Table 1). Whilst the micropush-out bond strength of groups 2 and 1 were significantly higher than the other groups ($P < 0.\ 05$), there was no statistically significant difference between them ($P > 0.\ 05$). The lowest micropush-out bond strength values were seen in groups 8, 9, 10 and 11 ($P < 0.05$) and there was no statistically significant difference amongst them ($P > 0.05$).

Stereomicroscopic examination of 432 samples revealed that cohesive failures (59.95%) within the filling material occurred more often than adhesive failures (40.05%) at the filling–dentine interface ($P < 0.001$). Adhesive failure at the filling–dentine interface was mostly observed in group 6 (gutta-percha/Epiphany/combined warm vertical compaction) (75.0%). Cohesive failures within the filling material were mostly observed in group 1 (gutta-percha/AH Plus/cold lateral compaction) (91. 7%) (Table 1).

Discussion

In the light of the push-out test results, the null hypothesis that Resilon/Epiphany provides higher interfacial strengths than gutta-percha/conventional sealer combinations was rejected. Resilon/Epiphany used with combined warm vertical compaction had low, but not significantly lower bond strength values than gutta-percha/AH Plus used with the same filling technique. Gutta-percha/Ketac-Endo combinations had higher bonding strength which can be explained by the bonding ability of glass–ionomer-based sealers to both dentine (0.74 MPa) and gutta-percha (0.14 MPa) (Chung *et al.* 2001, Lee *et al.* 2002). In the present study, stereomicroscopic findings of Ketac-Endo/gutta-percha groups revealed mostly cohesive failures in the filling (69.44%) corresponded to the good bonding of Ketac-Endo to dentine rather than gutta-percha. It is well known that the chemical reaction increases between dentine and glass–ionomers with time and provides a stronger bonding (Prosser *et al.* 1984, Davis *et al.* 1993).

In contrast to previous studies reporting significantly higher bond strengths with epoxy resin-based sealers (Lee *et al.* 2002, Saleh *et al.* 2002, Gogos *et al.* 2004) in the present study no significant difference was found between Ketac-Endo and AH Plus when used with gutta-percha. These different results can be related to the variety of samples and methods to test the bond strength in these various studies. Taking into consideration that Ketac-Endo binds chemically to dentine, the lower bond strength of Resilon/Ketac-Endo combination can be dependent on the lack of adhesion between the filling materials as seen in the microscopic evaluation. No study investigating the Resilon/Ketac-Endo combination appears in the literature with the result that the lower bond strengths found in the present study could not be compared.

It can be concluded that the compaction technique can influence the type of failure (Table 1). Warm techniques resulted in more adhesive failures between root dentine and filling that can be related to accelerated polymerization of the sealers. In resin-based sealers, rapid setting causes increased stiffness and does not allow time for the relief of shrinkage stress via resin flow (Tay *et al.* 2005). However, warm techniques did not affect the push-out bond strengths in the present study ($P > 0.05$).

Low bond strength values of Resilon/Epiphany combinations can be the result of two factors. First, the chemical union between Resilon and Epiphany was not as high as expected. This finding might be due to an insufficient amount of dimethacrylate (polycaprolactone/dimethacrylate is 10 : 1) in Resilon (United States Patent & Trademark Office 2005, Tay *et al.* 2006). This weak bonding cannot resist the polymerization stress of the dual-cured resin sealer. Furthermore, light polymerization for immediate coronal sealing hinders the relief of resin flow and increases the stress in the material (Ferracane 2005). Secondly, the smear layer impeding the tubular penetration of adhesive materials might also be a causal factor (Gesi *et al.* 2005).

The most important handicap for adhesive systems used in root canals is the high cavity configuration factor (Feilzer *et al.* 1989, Alster *et al.* 1997). As the unbonded surface area becomes small in long narrow root canals, there is insufficient stress relief by flow and a high risk of pull off or debond at the interfaces (Tay *et al.* 2005).

The results of push-out tests in previous studies (Gesi *et al.* 2005, Ungor *et al.* 2006) indicate that Resilon/Epiphany combinations had significantly lower bond strengths compared with Gutta-percha/epoxy resin-based sealer combinations agreeing with the results of the cold lateral compactions groups of the present study. However, in the present study, there was no statistically significant difference amongst the warm vertical compaction groups. Interestingly, the gutta-percha/Epiphany/cold lateral compaction group had significantly higher bond strength than the Resilon/Epiphany/cold lateral compaction group. Ungor *et al.* (2006) speculated that the higher bond strength was due to higher compactability of gutta-percha than that of Resilon. However, to explain this result, further studies investigating the bonding relation between gutta-percha and Epiphany are necessary.

The push-out test is based on the shear stress at the interface between dentine and cement as well as between post and cement (Van Meerbeek *et al.* 2003), which is comparable with stresses under clinical conditions (Frankenberger *et al.* 1999a). Taking into account the relative weakness of post-dentine bonding, Goracci *et al.* (2004) reported that the push-out test was the most accurate and reliable technique to measure the bonds of fibre posts to root dentine compared with conventional and modified microtensile tests. Nonuniform stress distribution is a drawback of the push-out test when it is performed on the whole post (Gallo *et al.* 2002) or on thick root sections (Sudsangiam & Van Noort 1999). To overcome this problem, the original push-out technique was modified by slicing the root into 1-mm-thick specimens (Frankenberger *et al.* 1999b, Goracci *et al.* 2004). Therefore, this technique was preferred for the present study. The contributing effect of friction on the bond strengths of fibre posts during push-out tests speculated by Goracci *et al.* (2005) can also be relevant for the filling materials; this claim requires further investigations. Furthermore, punch diameter which is much less than the canal diameter can cause nonuniform stress distribution over the interfaces. It might be more reliable to use punches with closer diameters to those of root canals.

Conclusions

Within the limitations of the present study, it can be concluded that:

1. Gutta-percha/Ketac-Endo/cold lateral compaction had the highest push-out bond strength ($P < 0.05$).

2. The push-out bond strengths of Resilon/Epiphany combinations were not higher than those of gutta-percha/conventional root canal sealer combinations. This contradicts the suggestions of monoblock formation.

3. Core materials and sealers may affect the push-out bond strengths of filling materials.

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