

Comparative Study of Bond Strength of Four Resin Cements Used for Endodontic Glass-fibre Posts

Xin DENG¹, Jian Guo TAN¹, Jian Feng ZHOU¹, Li CHEN¹

Objective: To assess the interfacial bond strength of total-etch, two self-etch and self-adhesive resin cement luted endodontic glass-fibre posts.

Methods: Sixteen recently extracted human teeth were collected for this study. The teeth were randomly and equally allocated to four groups: group 1, Single Bond 2 (3M ESPE, Seefeld, Germany) in combination with RelyX ARC (3M ESPE, Seefeld, Germany); group 2, ED Primer with Panavia F (Kuraray Co., Okayama, Japan); group 3, ParaBond with ParaCem (Coltène/Whaledent, Altstätten, Switzerland); and group 4, RelyX Unicem (3M ESPE, Seefeld, Germany). Fibre posts were cemented in endodontically treated teeth within the groups. One week later, the cemented posts in each group were processed for thin-slice push-out tests after thermal cycling (1000 cycles, 5/55 °C).

Results: The interfacial bond strength was not different among the four groups. The bond strength was significantly higher at the coronal third of the root canal than at the middle–apical third for group 4. The rest of the groups did not show regional differences in bond strength.

Conclusion: Single Bond/RelyX ARC, Panavia F, ParaCem, and RelyX Unicem demonstrated an equal potential for adhesion of fibre posts to intraradicular dentine.

Key words: adhesion, fibre post, intraradicular dentine, bond strength, push-out test

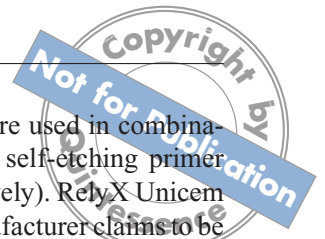
Since the 1950s, teeth with extensive loss of tissue have been saved using endodontic treatment performed widely and successfully in clinical practice. For reconstruction of an endodontically treated tooth, a post and core system is often necessary to achieve sufficient retention for a crown. Fibre posts bonded to root canal dentine via resin cements are now widely employed

because of several favourable properties. But the dislodging of the posts from the root canals is a frequent mode of failure with fibre posts.

Compared with enamel, bonding to dentine is a more difficult process due to the anatomical and histological characteristics of dentine as well as the smear layer on a cut dentine surface¹. Bonding to root canal dentine has proven to be even more difficult. The peculiar conditions of hydration in dentine on endodontically treated canal walls², the degradation of dentine collagen³⁻⁷, the effect of irrigation and eugenol-containing root-filling material, the regional differences in the density of the dentinal tubules⁸, and the fluidity of the bonding materials⁹ are all variables that can possibly influence the quality of adhesion at the post–cement–adhesive–dentine interfaces.

¹ Department of Prosthodontics, Peking University School and Hospital of Stomatology, Beijing, P.R. China

Corresponding author: Dr Jian Guo TAN, Department of Prosthodontics, Peking University School and Hospital of Stomatology, #22 Zhongguancun Nandajie, Haidian District, Beijing, 100081, P.R. China. Tel: 86-10-62179977 Ext 2347; Fax: 86-10-62173402. E-mail: kqtanjg@bjmu.edu.cn



Resin cements increase retention^{10,11} and tend to leak less than other cements¹²⁻¹⁴. Unfortunately, resin cements have some disadvantages. Resin cements are more 'technique sensitive' than most other luting cements. And they require several steps in the handling procedure. In order to solve these problems, some steps have been combined. A new resin luting material, RelyX Unicem, is a self-adhesive resin cement that does not need any primer or adhesive for pretreatment of the tooth substrate and requires only one step in cementation. Clinicians are concerned about the effectiveness of the adhesive systems with this simplified processing. There are many resin bonding systems commercially available, but which one is better in terms of mechanical characteristics? Consequently, four representative resin luting materials were selected for evaluation of their mechanical characteristics.

Materials and Methods

Sixteen single-rooted teeth, which had been extracted for periodontal or orthodontic reasons, were collected for the study. The crown portion of each tooth was removed perpendicular to the long axis of the tooth using a water-cooled diamond bur (Mani, Tokyo, Japan) below the cemento-enamel junction. The roots were endodontically instrumented at a working length of 1 mm from the apex with a #35 master apical file. A step-back technique was used with stainless-steel K-files, and the root canal was irrigated with 2% chloramine. The roots were obturated with gutta-percha (Gapdent, China) and eugenol-free root-filling material (VitaPex, Morita, Tokyo, Japan). Part of this filling material was then removed with Gates Glidden drills #2, and the canal wall of each specimen was enlarged with low-speed post drills provided by the post manufacturer to create a 9 mm deep post space, measured from the cemento-enamel junction on the buccal aspect of the tooth. One 1.25 mm diameter translucent glass-fibre post (ParaPost Fiber Lux, Coltène/Whaledent, Altstätten, Switzerland) was tried in and then cleaned with alcohol.

The teeth were randomly allocated to four groups with four teeth each, for different resin cements to lute the post: group 1, Single Bond 2 (3M ESPE, Seefeld, Germany) in combination with RelyXTM ARC (3M ESPE); group 2, ED Primer with PanaviaTM F (Kuraray Co., Okayama, Japan); group 3, ParaBond[®] with ParaCem[®] (Coltène/Whaledent, Altstätten, Switzerland); group 4, RelyXTM Unicem (3M ESPE, Seefeld, Germany). RelyX ARC is a dual-cured cement that requires phosphoric acid for substrate conditioning and the application of light-activated Single Bond 2. Panavia F and ParaCem

are dual-cured resin cements that are used in combination with the proprietary one-step self-etching primer (ED Primer and ParaBond respectively). RelyX Unicem is a dual-cured cement that the manufacturer claims to be self-adhesive in nature and does not require pretreatment of the tooth substrates. All of the materials were used strictly according to the manufacturers' instructions.

All of the cemented posts were placed in distilled water at room temperature for 1 week. The bond strength of cemented fibre posts was assessed with a thin slice push-out test. Before the test, the specimens underwent thermal cycling for a total of 1000 cycles (5/55 °C; dwell time: 30 s).

Thin slice push-out test

The top surface of each root was cut vertically to the post. The specimens were embedded in epoxy resin and then the portion of each root in which the post extended was sectioned perpendicular to the post axis from the coronal to the apical direction with a low-speed diamond saw (Isomet 1000, Buehler Ltd, New York, USA) under water cooling (Fig 1). From each specimen, five or six serial slices of 1 mm thickness were obtained. The thickness of each slice was individually measured by means of digital callipers and then firmly fixed with cyanoacrylate glue to a loading fixture. A compressive load was applied on the apical aspect of the slice via a universal testing machine (DCS5000, Shimadzu, Kyoto, Japan) equipped with a 1 mm diameter cylindrical plunger (Fig 2). The plunger tip was sized and positioned to touch only the bonded post, without stressing the surrounding root canal walls. The loading force was applied in an apical-coronal direction, in order to move the post towards the larger part of the root slice. Loading was performed at a speed of 0.5 mm/min until failure. Bond failure was manifested by the extrusion of the post from the root section.

In order to express the bond strength in megapascals, the load at failure recorded in newtons was divided by the area of the bonded interface, which was calculated through the following formula:

$$A = 2\pi rh$$

where π is the constant 3.14, r is the post radius and h is the thickness of the slice in millimetres.

Statistical analysis

First, a one-way analysis of variance was used to assess the significance of the differences in thickness of the slices among the four groups. Then, in order to assess the significance of the differences in interfacial strength

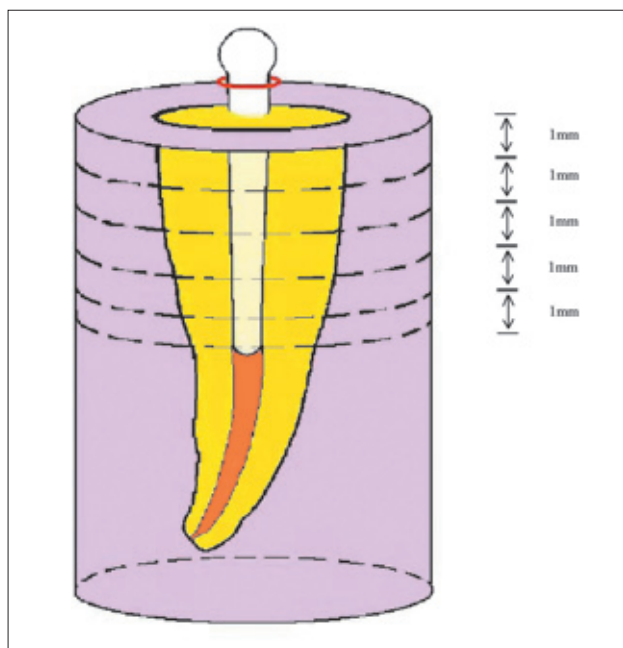


Fig 1 The portion of each root in which the post extended was sectioned perpendicular to the post axis from the coronal to the apical direction into five or six 1-mm-thick serial slices.

among the four resin cements, a one-way analysis of variance was applied. A further objective of the statistical analysis was to test the hypothesis that the coronal, middle and apical portions of the root canals provide different conditions of adhesion. For this purpose, the interfacial strength data from the same root level of each group were pooled together, regardless of the root of origin, and the levels of adhesion achieved in the different distinct portions of the roots were compared for each material. The level of significance was set at $P < 0.05$.

Results

Thickness of sample slices

There were no statistical differences in thickness of the slices among the four groups. ($P > 0.05$).

Push-out strengths

The results of the push-out strength measurements are shown in Table 1. The interfacial strengths achieved by the four adhesive systems were not statistically different from each other ($P > 0.05$).

When the data were pooled together for root level, it appeared that, for the self-adhesive cement, the bond strength was significantly higher at the coronal third than that at the middle–apical third. There were no significant

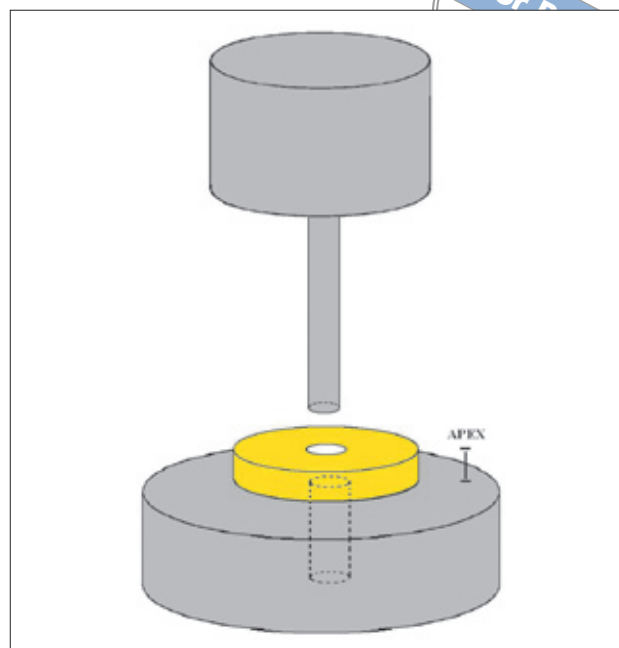


Fig 2 A compressive load was applied on the apical aspect of the slice via a universal testing machine equipped with a 1 mm diameter cylindrical plunger.

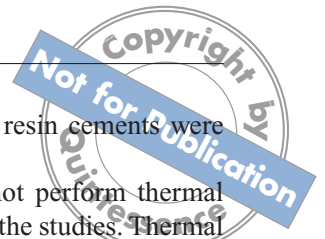
regional differences in bond strength in the rest of the groups.

Discussion

The resistance to dislocation of fibre posts bonded to intact root canals with resin-based cements may be considered a net sum of micromechanical interlocking, chemical bonding and sliding friction¹⁵. So, a push-out test has the benefit of more closely simulating the clinical condition¹⁶. However, it was suggested that a highly nonuniform stress may be developed at the adhesive interface when the push-out test is performed on the whole post¹⁶ or on thick root sections¹⁷⁻¹⁹. Using a thin-slice specimen, the thin slice push-out test permits a more uniform stress distribution along the bonded interface²⁰⁻²². This technique allows the assessment of regional differences in bond strength inside the root canals²⁰.

Table 1 Thin slice push-out test results

Group	No. specimens tested	Push-out strength (MPa) (mean \pm SD)
1	20	13.20 \pm 4.49
2	20	13.01 \pm 4.12
3	20	12.04 \pm 3.40
4	20	13.37 \pm 3.09



With small-sized specimens, the microtensile method has already been applied to evaluate bond strength to root canals. Goracci et al²⁰ compared the microtensile technique with the thin slice push-out test in the ability to measure accurately the bond strength of fibre posts luted inside root canals. The great number of premature failures and the finding of high standard deviation values make questionable the reliability of the microtensile technique. In contrast, with the push-out test, no premature failure occurred and the variability of the data distribution was acceptable. This study showed that the push-out test appeared to be more reliable than the microtensile technique when measuring the bond strength of luted fibre posts.

Long-term water storage and thermal cycling are the conditions most often used to test the durability of resin bonds. Both tests are considered to be clinically relevant aging parameters. Thermal cycling was used in this study to simulate the clinical condition more closely, keeping in mind that the relatively short observation time (1000 min) in this study was not long enough to provide information on the long-term stability of the post bond.

In this study, there were no significant differences in the interfacial strength achieved by the four adhesive systems. However, in two studies by Goracci et al^{15,20}, it appeared that with Excite-Variolink II the interfacial strength was significantly higher than that with RelyX Unicem. On the contrary, Bitter et al²³ reported significantly higher bond strengths for RelyX Unicem compared with the total-etching materials (Excite DSC/Variolink II, PermaFlo DC, Clearfil Core) and the self-etching materials (Panavia F, Multilink). Conflicting results were obtained from previous studies, and differences in luting procedures for the same material may be the key. The methods used in the present study are different from those in the studies mentioned above in the following aspects.

First, the handling methods of the resin cements are different. According to the manufacture's instruction, Lentulo Spirals could not be used to insert RelyX Unicem into the root canal, as this can excessively accelerate setting. So, Goracci et al^{15,20} only applied the cement onto the post and inserted directly into the dry root canal. However, other cements used in the studies were inserted into the root canal with Lentulo Spirals. Fakiha et al²⁴ reported that when Lentulo Spirals and syringes were used, the bond strength of post-to-root canal dentine was statistically higher than that of the group in which the cement had only been applied onto the post. In the study by Bitter et al²³, all resin cements were only applied onto the post. In the present study, RelyX Unicem was injected into the root canal with

'Elongation Tips', while the other resin cements were inserted with Lentulo Spirals.

Second, Goracci et al^{15,20} did not perform thermal cycling prior to the push-out test in the studies. Thermal cycling is the condition most often used to test the durability of resin bonds. The test is considered to be a clinically relevant aging parameter. Two recent studies found that the bond strength of some resin cements could be affected by thermocycling^{23,25}. So, in the present work the samples were subjected to thermal cycling (1000x) in water (5/55 °C) prior to push-out testing.

Finally, in one of studies by Goracci et al²⁰, RelyX Unicem was only chemically initiated. Foxton et al²⁶ have reported a significant reduction in bond strength when polymerisation of the dual-cure resin composite was chemically initiated. This might be the reason for the difference in bonding strength between Excite-Variolink II and RelyX Unicem found in the Goracci et al study.

In dentine bonding materials, only total-etching systems can remove the smear layer completely, which is more likely to form a hybrid layer and resin tags according to the bonding mechanism. However, the clinician faces the problem of technique sensitivity of the luting procedures as well as the problem that different luting procedures have to be applied. Bonding to intraradicular dentine presents challenges due to the anatomy of roots, tooth position, the presence of coronal residual tissues, the handling characteristics of the adhesive system, the light curing technique and the wet bonding technique. These factors may account for the equal bond strength achieved by the total-etching system compared with the results of the self-etching systems and the self-adhesive cement in bonding fibre posts to root canal dentine.

Regarding tubule density in root dentine, Ferrari et al⁹ reported that the highest tubule density was found in the cervical region and it was significantly reduced in the middle and apical third regions. However, in this study there were no significant regional differences in interfacial strength in groups 1, 2 and 3. In the studies by Goracci et al^{15,20}, there were no significant regional differences in interfacial strength of bonding fibre posts to root canal dentine either. According to Foxton et al²⁷ and Gaston et al²⁸, bond strength may be related more to the area of solid dentine than to tubule density.

When the data for the self-adhesive cement were pooled together for root level it appeared that the interfacial strength was significantly higher at the coronal third than at the middle-apical third. RelyX Unicem was the only self-adhesive cement used in this study. The self-adhesive properties are claimed to be based upon phosphoric acid methacrylates that demineralise and infiltrate the tooth substrate, resulting in micromechanical

retention. Secondary reactions have been suggested to provide chemical adhesion to hydroxyapatite²⁹. The basic inorganic fillers are able to undergo a cement reaction with the phosphoric acid methacrylates. The cement is the only element that is responsible for the efficacy of the material. For the other three dentine bonding systems used in this study, the inner surfaces of the root canals were pretreated before the placement of the cements. The efficacy of the primer-adhesive can be controlled by the strict and uniform application protocols. But the uniform adaptation of the cement at the different levels of the root is impossible, because of the differences in shape between the post and the root canal. The space for the cement at the coronal third is bigger than at the middle–apical third. This factor may account for the lower bond strengths achieved by the self-adhesive cement in the middle–apical root sections.

Conclusion

Interfacial strengths demonstrated an equal potential for adhesion of fibre posts to intraradicular dentine with Single Bond/RelyxARC, Panavia F, ParaCem, and RelyX Unicem.

References

- Craig RG, Powers JM. Restorative Dental Materials, ed 11. St Louis: Mosby, 2002.
- Hayashi M, Takahashi Y, Hirai M, Iwami Y, Imazato S, Ebisu S. Effect of endodontic irrigation on bonding of resin cement to radicular dentin. *Eur J Oral Sci* 2005;113:70–76.
- Rivera EM, Yamanchi M. Site comparisons of dentin collagen crosslinks from extracted human teeth. *Arch Oral Biol* 1993;38:541–546.
- Kato G, Nakabayashi N. The durability of adhesion to phosphoric acid etched, wet dentin substrates. *Dent Mater* 1998;14:347–352.
- Hashimoto M, Ohno H, Kaga M, Endo K, Sano H, Oguchi H. *In vivo* degradation of resin–dentin bonds in humans over 1 to 3 years. *J Dent Res* 2000;79:1385–1391.
- Kitasako Y, Burrow MF, Nikaido T, Tagami J. The influence of storage solution on dentin bond durability of resin cement. *Dent Mater* 2000;16:1–6.
- Hashimoto M, Ohno H, Kaga M, Endo K, Sano H, Oguchi H. Resin–tooth adhesive interfaces after long-term function. *Am J Dent* 2001;14:211–215.
- Mjör IA, Smith MR, Mannocci F. The structure of dentine in the apical region of human teeth. *Int Endod J* 2001;34:346–353.
- Ferrari M, Vichi A, Grandini S. Efficacy of different adhesive techniques on bonding to root canal walls: an SEM investigation. *Dent Mater* 2001;17:422–429.
- Mezzomo E, Massa F, Libera SD. Fracture resistance of teeth restored cements: an *in vitro* study, Part I. *Quintessence Int* 2003;34:301–306.
- Nissan J, Dmitry Y, Assif D. The use of reinforced composite resin cement as compensation for reduced post length. *J Prosthet Dent* 2001;86:304–308.
- Mannocci F, Ferrari M, Watson TF. Microleakage of endodontically treated teeth restored with fiber posts and composite cores after cyclic loading: a confocal microscopic study. *J Prosthet Dent* 2001;85:284–291.
- Reid LC, Kazemi RB, Meiers JC. Effect of fatigue testing on core integrity and posts microleakage of teeth restored with different post systems. *J Endod* 2003;29:125–131.
- Bachicha WS, DiFiore PM, Miller DA, Lautenschlager EP, Pashley DH. Microleakage of endodontically treated teeth restored with posts. *J Endod* 1998;24:703–708.
- Goracci C, Sadek FT, Fabianelli A, Tay FR, Ferrari M. Evaluation of the adhesion of fiber posts to intraradicular dentin. *Oper Dent* 2005;30:627–635.
- Sudangiam S, van Noort R. Do dentin bond strength tests serve a useful purpose? *J Adhes Dent* 1999;1:57–67.
- Gallo JR, Miller T, Xu X, Burgess JO. *In vitro* evaluation of the retention of composite fiber and stainless steel posts. *J Prosthodont* 2002;11:25–29.
- Patierno JM, Rueggeberg FA, Anderson RW, Weller RN, Pashley DH. Push-out and SEM evaluation of resin composite bonded to internal cervical dentin. *Endod Dent Traumatol* 1996;12:227–236.
- Ngo EC, Pashley DH, Loushine RJ, Weller RN, Kimbrough WF. Effects of eugenol on resin bond strengths to root canal dentin. *J Endod* 2001;27:411–414.
- Goracci C, Tavares AU, Fabianelli A, Monticelli F, Raffaelli O, Cardoso PC et al. The adhesion between fiber posts and root canal walls: comparison between microtensile and push-out bond strength measurements. *Eur J Oral Sci* 2004;112:353–361.
- Drummond JL, Sakaguchi RL, Racean DC, Wozny J, Steinberg AD. Testing mode and surface treatment effects on dentin bonding. *J Biomed Mater Res* 1996;32:533–541.
- Goracci C, Fabianelli A, Sadek FT, Papacchini F, Tay FR, Ferrari M. The contribution of friction to the dislocation resistance of bonded fiber posts. *J Endod* 2005;31:608–612.
- Bitter K, Meyer-Lueckel H, Priehn K, Kanjuparambil JP, Neumann K, Kielbassa AM. Effect of luting agent and thermocycling on bond strengths to root canal dentin. *Int Endod J* 2006;39:809–818.
- Fakiha Z, Al-Aujan A, Al-Shamrani S. Retention of cast posts cemented with zinc phosphate cement using different cementing techniques. *J Prosthodont* 2001;10:37–41.
- Lüthy H, Loeffel O, Hammerle CH. Effect of thermocycling on bond strength of luting cements to zirconia ceramic. *Dent Mater* 2006;22:195–200.
- Foxton RM, Nakajima M, Tagami J, Miura H. Bonding of photo and dual-cure adhesives to root canal dentin. *Oper Dent* 2003;28:543–551.
- Foxton RM, Nakajima M, Tagami J, Miura H. Adhesion to root canal dentine using one and two-step adhesives with dual-cure composite core materials. *J Oral Rehabil* 2005;32:97–104.
- Gaston BA, West LA, Liewehr FR, Fernandes C, Pashley DH. Evaluation of regional bond strength of resin cement to endodontic surfaces. *J Endod* 2001;27:321–324.
- 3M ESPE. Technical data sheet: Espertise RelyX Unicem (2002). Seefeld Germany: 3M ESPE.