Evaluation of the push-out bond strength of self-adhesive resin cements to fiber posts

Claudia Mazzitelli[1], Francesca Monticelli[2]

Abstract

Objective: To assess the push-out bond strength of self-adhesive resin cements bonded to epoxy-resin based fiber posts. **Methods and Materials:** Thirty single-rooted premolars were endodontically treated and post-spaces were prepared to receive RelyX Fiber posts #1. Three self-adhesive cements were used for luting the fiber posts: 1. RelyX Unicem; 2. Multilink Sprint; 3. Max-Cem. The bonded specimens were stored at 37° C and 100% relative humidity for up to 1 month. The force required to dislodge the post (MPa) in an apical-coronal direction was measured with the push-out test. Data were statistically analyzed with one-way ANOVA ($p<0.05$) and Tukey tests ($p<0.001$). Representative fractured slices were observed under a scanning electron microscope (SEM). **Results:** Differences in bond strength were recorded according to the cement used for luting fiber post ($p<0.05$). Push-out bond strength values of RelyX Unicem were higher than those of Multilink Sprint and Max-Cem. **Conclusions**: Differences exist in the bonding performance of self-adhesive cements when luting fiber posts. The delivery system used for placing the cement into the root canal plays an important role in short-term bond strength evaluations.

Clinical significance: RelyX Unicem appeared adequate to be used for fiber post luting. The use of an elongation tip would prevent the occurrence of voids and bubbles within the cement layer.

Key words: self-adhesive cement, push-out test, fiber post, dispensing method.

Short title: Bonding of self-adhesive cement to fiber posts.

Introduction

Fiber posts are used increasingly for the restoration of endodontically treated teeth with massive coronal destruction. Their mechanical properties and clinical behavior have been widely investigated.[1-4] The dislocation resistance of fiber posts into the root canal is significantly influenced by the selected luting agent and the cementation procedures.[5] Resin cements allow superior post retention and increase the fracture resistance of the post-restored tooth when compared to conventional cements.[6-8]

Several factors contribute to render post luting procedures difficult: the lack of direct vision and the limited access to the bonding substrate make cementation procedures very technique-related.[5] Moisture control within root canals represents an additional limitation during the management of multi-step resin cements.[9]

Self-adhesive resin cements have simplified luting procedures of indirect restorations and have been designed to be less technique-sensitive than their multi-step counterparts. Laboratory investigations found comparable bond strength values between RelyX Unicem and resin cements that utilized two or three-steps adhesives.[6,10] RelyX Unicem is one of the most investigated self-adhesive materials, notwithstanding the variety of products that have been launched by different

1 PhD student, Department of Fixed Prosthodontics and Dental Materials, University of Siena, Policlinico "Le Scotte", Viale Bracci 1, 53100, Italy;
2 Professor, Department of Dental Materials, Faculty of Sports and Health Science, University of Zaragoza, Huesca, Spain.

***Corresponding author:** Dr Claudia Mazzitelli, Department of Fixed Prosthodontics and Dental Materials, University of Siena, Policlinico "Le Scotte", viale Bracci, 1; 53100, Siena, Italy. Fax: +390577233131; Tel: +393383152058; email: claudiamazzitelli@yahoo.it*

Table 1
Manufacturer's, chemical composition and application modes of the cements investigated.

Material	Composition	Delivery system	Instructions for use
Rely X Unicem (3M ESPE,) *Batch n°:270644* *pH= 2.1*	Powder: glass fillers, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxy compound. Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators	Capsule and Aplicap Elongation Tip	Mix cement. Apply, self-cure (5 min) and light-cure (40s)
Multilink Sprint (Ivoclar-Vivadent) *Batch n°:j22739* *pH: 4.2*	Dymethacrylates, adhesive monomers, fillers, initiators/stabilizers	Paste/paste dual syringe with a mixing tip	Automix cement. Apply, self-cure (5 min) and light-cure (40s)
Max-Cem (Kerr Dental) *Batch n°:* *pH: 2.2*	Base: Uretanedymethacrylate, Camphoroquinone, Fluoroaluminosilicate, others. Catalyst: Bis-GMA, Triethyleneglycoldimethacrylates, Glycerophosphatedimethacrylates, Bariumaluminopolosilicate glass, Others	Paste/paste dual syringe with a mixing tip	Automix cement. Apply, self-cure (5 min) and light-cure (40s)

manufacturers. Self-adhesive cements possess different chemical compositions and dispensing modalities that could influence their mechanical properties and bonding performances.

The push-out test is an appropriate method to measure the bond strength inside root canals. The shear stresses are created parallel to the cement/dentin and cement/post interfaces, resulting in a better simulation of the stresses occurring in clinical conditions.[11,12]

The purpose of the present study was to assess the push-out bond strength of three self-adhesive resin cements used for the cementation of epoxy resin-based fiber posts. The null hypothesis tested was that no differences in bond strength are present among the tested self-adhesive resin cements independently from their chemical composition and application mode.

Materials and Methods

Specimen preparation

Thirty extracted, single-rooted, caries-free human premolars, stored in 0.5% Chloramine T solution at 4° C for preventing bacterial growth, were selected for the study after obtaining informed consent of the donors. The crown of each tooth was removed 1 mm above the CEJ by means of a slow speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under copious water cooling. Working length was established at 1 mm from the root apex. Cleaning and shaping of the root canal were performed with Protaper Ni-Ti rotatory instruments (size S1, S2, S3; Dentsply Maillefer, Ballagues, Switzerland) following the crown-down technique. Irrigation with 5% sodium hypochlorite was performed between instrumentations. Gutta-percha cones (Coltène/Whaledent, Langenau, Germany) were used for filling the root canal and cemented with an epoxy-resin based sealer (AH Plus Jet, Dentsply DeTrey, Konstanz, Germany) according to the lateral condensation technique. Roots were coronally sealed with a temporary restorative material (Fuji VII, GC Corporation, Tokyo, Japan; batch n° 0410221) and stored in a laboratory oven at 37° C and 100% relative humidity. After 24 hours, the temporary seal was abraded by means of #240 SiC paper under water cooling, and the coronal gutta-percha was removed with a pre-shaping drill (Dentsply DeTrey, Konstanz, Germany), leaving a 5 mm-long apical seal. A 7 mm-deep post space was prepared with a universal drill (3M ESPE, Seefeld, Germany) to match the size of the co-respective RelyX Fiber Post (#1; 3M ESPE, Seefeld, Germany; LOT: 02363200603). The

Table 2

Push-out strengths and the percentage of slices with their respective failure modes. Numbers are means (MPa), values in brackets are standard deviations. Different letters show statistically significant differences ($p<0.001$). AD: adhesive failures between dentin and luting agent; AP: adhesive failures between post and cement; C: cohesive failures within the post; M: mixed failures.

Experimental groups	Sample size	Failure mode				Mean (SD)
		AD	AP	C	M	
Rely X Unicem	**32**	**69%**	**19%**	**6%**	**6%**	**10.27(2.18) A**
Multilink Sprint	**31**	**42%**	**29%**	**0%**	**29%**	**7.55(3.78) B**
Max-Cem	**30**	**33%**	**57%**	**0%**	**10%**	**3.86(2.94) C**

drilled canal was rinsed with water and gently air-blowed in order to eliminate any residual gutta-percha. Three self-adhesive composite cements were used for fiber post cementation (n=10): 1. Rely X Unicem (3M ESPE, Seefeld, Germany); 2. Multilink Sprint (Ivoclar-Vivadent, Schaan, Liechtenstein); 3. Max-Cem (Kerr Corp, Orange, CA, USA).

Materials were handled according to manufacturer's instructions. Application modes, chemical compositions and batch numbers of the investigated materials are listed in Table 1.

The cements were used in the dual-cured modality. After the first 5 min of auto-curing mode in which the post was loaded under finger pressure, additional 40 s of light polymerization through the translucent fiber post were performed (Astralis 7, Ivoclar-Vivadent, Schaan, Liechtenstein; output: 600 mW/cm2). The cement in excess was carefully removed with a spatula. A core build-up was performed with Fuji VII (GC corp; LOT: 0703071). Specimens were maintained for 1 month in a flower sponge slightly moist with demonized water and stored in a laboratory incubator (37±1 °C) in order to better simulate a clinical hydration condition.

Push-out bond strength test

The portion of the root containing the fiber post was subsequently sectioned into four to six 1 mm-thick slices with a diamond saw (Isomet) under water cooling. The force of the cylindrical plunger of the testing machine (Triax 50, Controls S.P.A, Milan, Italy) was used to dislodge each inverted, truncated fiber post from the root dentin in an apical-coronal direction. A load (0.5 mm/min until failure) was then applied to the post surface that resulted in shear stresses along the cement/dentin – cement/post interfaces. The retentive strength of the post fragment (MPa) was calculated by dividing the load at failure (Newton) by the interfacial area of the post segment (S_L). The formula used for measuring the tronco-conical area was:

$$S_L = \pi (R+r) [(h^2+ (R-r)^2]^{0.5}$$

where π was equal to 3.14, R and r were the coronal and the apical post radius respectively, and h the root slice thickness. The diameters of the post and the thickness of the slice were individually measured using a digital caliper with 0.01 mm accuracy.

Failure modes were evaluated by a single operator under a stereomicroscope (Olympus SZ-CTV, Olympus, Tokyo, Japan) at 40x magnification and classified as cohesive (within the post), adhesive (between the post and the cement or at the cement/intra-radicular dentin level) or mixed (adhesive and cohesive fractures occurred simultaneously).

One stressed-to-failure slice per group was used for scanning electron microscopy (SEM) evaluation. Specimens were immersed in ascending ethanol solutions, gently air-dried, mounted on metallic stubs, gold-sputter coated (Polaron Range SC 7620, Quorum Technology, Newhaven, UK) and observed under a scanning electron microscope (JSM-6060LV, Jeol, Tokyo, Japan).

Statistical analysis

The normal and equal distribution of the push-out bond strength data were first checked and verified by the Kolmogorov-Smirnov and Levene's test respectively. A one-way ANOVA was performed to verify the differences in push-out bond strengths between the tested luting cements ($p<0.05$). A Tukey test was then executed for post-hoc comparisons ($p<0.001$). Calculations were handled by the SPSS 15.0 software (SPSS Inc.; Chicago, IL, USA).

Results

The mean push-out bond strengths (SD) and failure modes of the tested cements are shown in Table 2. Differences in bond strengths exist among the self-adhesive cements used for luting fiber posts ($p<0.05$).

RelyX Unicem exhibited significantly higher bond strengths than the other tested cements. The push-out values of Multilink Sprint were lower than those of RelyX Unicem, but higher than Max-Cem that recorded the lowest push-out bond strength values.

The failure modes recorded were mostly adhesive in nature, both between dentin and cement and at the post/cement interface. Cohesive failures were only observed for RelyX Unicem. Mixed failures also occurred in the three self-adhesive cements investigated. No cohesive failure within the fiber post were observed in the present study.

Discussion

The results of this study require the rejection of the null hypothesis since differences in push-out bond strength exist between the tested self-adhesive cements.

The bonding mechanism of self-adhesive cements rely on chemical interactions and micromechanical retentions with the bonding substrate, but concerns still exist on the effective adhesive potential of these simplified cements.

In the present study, RelyX Unicem attained higher bond strength values when compared to the other materials. In line with previous studies, these values may be comparable to those of multi-step luting agents.[6,10] When compared to another self-adhesive material, RelyX Unicem registered a higher bond strength.[13] The use of the cement/post combination as recommended by the manufacturers may have accounted for the results obtained.[10,14] In the present study, only one type of fiber post was used (RelyX Fiber Post, 3M ESPE), as the main purpose was to estimate the bond strengths of different self-adhesive cements. Future studies should be addressed to test the influence of different branded post systems on the bond strength of self-adhesive cements. Differences in the application mode may have similarly influenced the results. RelyX Unicem was inserted into the root canal utilizing an elongation tip, resulting in less chance of bubble formation and air-entrapment, which would lead to an improvement in the marginal adaptation of the material, both to the dental substrate and to the fiber post.[15,16] Simonetti and colleagues addressed the higher sealing ability of RelyX Unicem dispensed with the elongation tip in comparison with Max-Cem and other multi-step cements which were inserted into root canal with a lentulo spiral or directly applied on post surface.[16] The two self-adhesive cements employed in the present study have a paste-to-paste composition. Base and catalyst are mixed together through an auto-mixing tip on a glass pad and the material then inserted into the root canals with a lentulo spiral. This method appeared less feasible as it could increase the risk of air entrapment causing the formation of voids and interfacial defects that would expedite premature failure in presence of cyclic stresses.

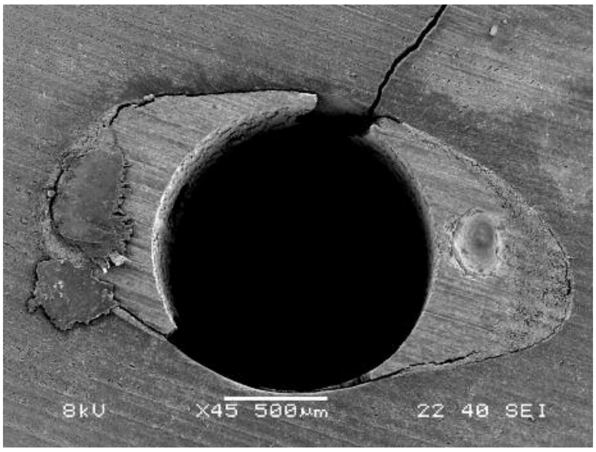

Figure 1: Representative SEM image of Max-Cem after the push-out test (original magnification 45X). Voids are detected within the cement bulk, possibly due to air-entrapment during the mixing and insertion procedures.

Max-Cem attained the lowest push-out values (Table 2). The cement layer appeared inhomogeneous with clear voids and bubbles incorporated into the bulk (Fig. 1). Max-Cem is considered extremely technique-sensitive and any errors occurring during the mixing process can determine its physical and mechanical properties.[17] A prevalence of adhesive decementation at the cement/post side were recorded. Max-Cem possesses an acidic pH that is maintained at a high level, even 48 hours after its application.[17] This may exert a detrimental effect on its physical properties, diminishing the possibility to establish effective micro-mechanical retention. Moreover, the presence of an acidic layer on the post surface may jeopardize the formation of hydrogen bonds between the cement and fiber post, limiting the bonding potential of the cement itself.[18]

Multilink Sprint bond strengths were lower than those of RelyX Unicem, but higher than those of Max-Cem. The material was able to partially demineralise the dental substrate, although discrepancies between the degree of

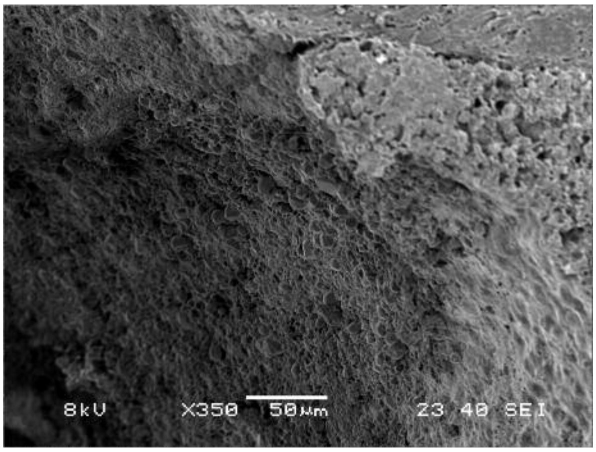

Figure 2: SEM microphotographs of Multilink Sprint (20 bar, original magnification: 350X). A detachment of the fillers from the resinous matrix was noticed. The cement showed a porous appearance afterward the push-out bond strength test.

demineralization and depth of resin penetration was assessed by light microscopy.[19] At the dentin site bond, the material appeared porous, probably due to an incomplete polymerization reaction (Fig. 2). The presence of residual acidic monomers at the bottom of the adhesive interface may represent weak areas as they can retain their etching potential, thus jeopardizing adhesion.[20,21] These areas and the presence of exposed collagen fibers at the adhesive joint would experience premature degradation, hence limiting the bonding potential of the material and reducing the service life of the restoration.[21,22] Further studies should be performed to assess the longevity of these self-adhesive cements.

Nowadays, many studies are performed using ageing tests to assess the longevity of bonded interfaces. Several authors found that thermocycling may increase the retentive strength, especially for RelyX Unicem. The thermal changes were intended to promote complete chemical polymerization, thus enhancing its bonding potential.[23,24] Self-adhesive cements work as dual-cure materials, where the chemical polymerization can be completed by light irradiation,[25] However, doubt exists on the degree of monomer conversion of the simplified cements. Some authors attained inferior bond strengths and decreased mechanical properties when RelyX Unicem was only auto-cured.[26-28] No differences in the degree of monomer conversion were found between RelyX Unicem and Multilink Sprint, whereas Max-Cem attained the lower values.[26] Several factors may account for the differences recorded, primarily in the chemical composition of the cement. However, little information is supplied by their respective manufacturers and more specific details are recommended in order to define their characteristics and bonding behaviour.

Conclusions

Within the limits of this study, it can be concluded that RelyX Unicem attained the higher push-out bond strength values among the tested cements. The use of an elongation tip is highly recommended for placing the material inside the root canal in order to limit the formation of defects at the adhesive interfaces.

References

1. Ferrari M, Vichi A, Mannocci F, Mason PN. Retrospective study of clinical behaviour of several types of fiber posts. Am J Dent 2003; 13: 14B-19B;

2. Grandini S, Goracci C, Monticelli F, Tay FR, Ferrari M. Fatigue resistance and structural integrity of fiber posts: three-bending test and SEM evaluation: Dent Mater 2005; 21: 75-82;

3. Cagidiaco MC, Radovic I, Simonetti M, Tay FR, Ferrari M. Clinical performance of fiber post restorations in endodontically treated teeth: 2-year resultas. Int J Prosthodont 2007; 20(3): 293-298;

4. Ferrari M, Cagidiaco CM, Goracci C, Vichi A, Mason PN, Radovic I, Tay FR. Long-term retrospective study of the clinical performance of fiber posts. Am J Dent 2007; 20(5): 287-291;

5. D'Arcangelo C, Zazzeroni S, D'Amario M, Vadini M, De Angelis F, Trubiani O, Caputi S. Bond strength of three types of fibre-reinforced post systems in various regions of root canals. Int Endod J, 2008; 42: 322-328.

6. Bitter K, Priehn K, Martus P, Kielbassa AM. In vitro evaluation of push-out bond strengths of various luting agents to tooth-colored posts. J Prosthet Dent 2006; 95: 302-310.

7. D'Arcangelo C, D'Amario M, Vadini M, Zazzeroni S, De Angelis F, Caputi S. An evaluation of luting agent application technique effect on fibre post retention. J Dent 2008; 36: 235-240.

8. Qualtrough AJE, Mannocci F. Tooth-colored post systems: a review. Oper Dent 2003; 28: 86-91

9. Chersoni S, Acquaviva GL, Prati C, Ferrari M, Grandini S, Pahley DH, Tay FR. In vivo fluid movement through dentin adhesives in endodontically treated teeth. J Dent Res 2005; 19: 223-227.

10. Radovic I, Mazzitelli C, Chieffi N, Ferrari M. Evaluation of the adhesion of fiber posts cemented using different adhesive approach. Eur J Oral Sci 2008; 116: 557-563.

11. Goracci C, Grandini S, Bossù M, Bertelli E, Ferrari M. Laboratory assessment of the retentive potential of adhesive

posts: A review. J Dent 2007; 35: 827-835.

12. Goracci C, Tavares AU, Fabianelli A, Monticelli F, Raffaelli O, Cardoso PC, Tay FR, Ferrari M. 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;

13. Zicari F, Coutinho E, De Munck J, Poitevin A, Scotti R, Naert I, Van Meerbeek B. Bonding effectiveness and sealing ability of fiber-post bonding. Dent Mater 2008; 24: 967-977.

14. Kececi AD, Kaya U, Adanir N. Micro push-out bond strengths of four fiber-reinforced composite post systems and 2 luting materials. Oral Surg Oral Med Oral Pathol Oral Radiol Endod 2008; 105: 121-128.

15. Watzke R, Blunck U, Frankenberger R, Naumann M. Interface homogeneity of adhesively luted glass fiber posts. Dent Mater 2008

16. Simonetti M, Coniglio I, Magni E, Cagidiaco MC, Ferrari M. Sealing ability and microscopic aspects of a self-adhesive resin cement used for fiber post luting into root canals. Int Dent S Afr 2008; 8: 24-30.

17. Behr M, Rosentritt M, Loher H, Kolbeck C, Trempler C, Stemplinger B, Kopzon V, Handel G. Changes of cement properties caused by mixing errors: the therapeutic range of different cement types. Dent Mater 2008

18. Wrbas KT, Altenburger MJ, Schirrmeister JF, Bitter K, Kielbassa AM. Effect of adhesive resin cements and post surface silanization on the bond strengths of adhesibvely inserted fiber posts. J Endod 2007; 33: 840-843.

19. Monticelli F, Osorio R, Mazzitelli C, Ferrari M, Toledano M. Limited decalcification/diffusion of self-adhesive cements into dentin. J Dent Res 2008; 87: 974-979.

20. Mazzitelli C, Monticelli F, Osorio R, Casucci A, Toledano M, Ferrari M. Effect of simulated pulpal pressure on self-adhesive cements bonding to dentin. Dent Mater 2008; 24: 1156-1163..

21. Spencer P, Swafford JR. Unprotected protein at the dentinal-adhesive interface. Quintessence Int 1999; 30: 501-507.

22. Wang Y, Spencer P. Evaluation of the interface between one-bottle adhesive systems and dentin by Goldner's trichrome. Am J Dent 2005; 18: 66-72.

23. Bitter K, Meyer-Lueckel H, Priehn K, Kanjuparambil JP, Neumann K, Kielbassa AM. Effects of luting agents and thermocycling on bond strengths to root canal dentine. Int Endod J 2006; 39: 809-818.

24. Reich SM, Wichmann M, Frankenberger R, Zajc D. Effect of surface treatment on the shear bond strength of three resin cements to a machinable feldspatic ceramic. J Biomed Mater Res Part B: Appl Biomater 2005; 74B: 740-746.

25. Radovic I, Monticelli F, Goracci C, Vulicevic ZR, Ferrari M. Self-adhesive resin cements: a literature review. J Adhes Dent 2008; 10: 251-258.

26. Vrochari AD, Eliades G, Hellwig E, Wrbas KT. Curing efficiency of four self-etching, self-adhesive resin cements. Dent Mater 2009; doi: 10.1016/j.dental.2009.02.015.

27. Piwowarczyk A, Lauer HC. Mechanical properties of luting cements after water storage. Oper Dent 2003; 28: 535-542.

28. Kumbuloglu O, Lassilla VJ, User A, Vallittu PK. A study of the physical and chemical properties of four resin composite luting cements. Int J Prosthodont 2004; 17: 357-363.