



## Evaluation of push-out bond strength of different adhesive system applications

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**Objective:** To compare interfacial strength and failure modes of glass-fibre endodontic posts luted with three different adhesive luting agents.

**Methods:** A total of 30 extracted human maxillary incisors were randomly divided in three groups and restored using glass-fibre posts and the following luting agents: an experimental self-etching primer/resin-cement MSM-107 (EXP), ExciTE® F DSC adhesive/Variolink II (VII), Gradia Core self-etching bond/Gradia Core cement (GC). Five sections of  $1.00 \pm 0.05$  mm thickness were prepared from each specimen, and the post in each section was subjected to a push-out test. Failure modes of root slices after push-out testing were examined with stereomicroscope. The level of significance was set at  $\alpha=0.05$ .

**Results:** EXP achieved the highest bond strength. The mean value recorded for GC was significantly lower than EXP ( $p < 0.05$ ) and did not differ from VII ( $p > 0.05$ ). There was also no significant difference between EXP and VII ( $p > 0.05$ ). Bond failure was mainly mixed failure for VII and GC. Adhesive failures between the cement and the post, cohesive failures within the post and the dentin were mostly observed in EXP.

**Conclusion:** The new experimental resin cement, MSM-107 showed promising results in term of bonding ability and might be used efficiently to restore endodontically treated teeth.

**Keywords:** Bonding agent; fiber post; luting cement; push-out bond strength.

Fibre-reinforced polymer posts have been employed to restore endodontically treated teeth that have suffered considerable loss of coronal structure since 1990s.<sup>[1,2]</sup> They offer a number of advantages over custom-fabricated cast alloy posts and core or prefabricated alloy posts. Fibre posts more closely match the modulus of elasticity of sound root dentin, thus distributing occlusal stresses more evenly in the root dentin and providing higher fracture resistance to weakened roots.<sup>[3,4]</sup> Other attractive features of fibre posts include resistance to corrosion, aesthetic appeal, and nonhypersensitivity.<sup>[5]</sup>

Selecting an appropriate adhesive and luting procedure for bonding posts to root dentine is challenging. Various luting agents and corresponding adhesive systems have been proposed for bonding fiber posts to root canal dentin. These materials can be divided into self-etching adhesives and etch & rinse systems.<sup>[6]</sup> Results on the effectiveness of self-etching adhesive systems when compared with etch & rinses are contradictory.<sup>[7,8]</sup> Some studies show similarities between these systems<sup>[8,9]</sup> whilst others suggest a superiority of the etch & rinse materials.<sup>[7,10]</sup> In a confocal laser scanning microscopy study, the application

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Submitted: March 28, 2016 Accepted: May 13, 2016

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of etch & rinse and adhesives resulted in a higher number of resin tags and an increased hybrid layer thickness compared with self-etching adhesive systems.<sup>[11]</sup>

A new experimental etch & rinse resin system (MSM-107; GC, Tokyo, Japan) has been recently developed. However, no scientific information is so far available regarding its adhesive potential when used for fiber post luting.

The purpose of this study was to compare interfacial strength in the root canal amongst glass-fibre endodontic posts luted with different luting agents, using the push-out test method. The null hypothesis to be tested was that bond strengths to root canal dentin do not vary with type of luting system.

## Materials and methods

A total of 30 intact human maxillary incisors with one straight root and, extracted because of periodontal reasons, were selected. Each tooth was placed in 5.25% sodium hypochlorite (NaOCl) for 2 h for surface disinfection, and then stored in distilled water.

The crown of each tooth was sectioned at the cemento-enamel junction (CEJ) using a water-cooled diamond disk to obtain 14-mm long root. The working length was determined visually by subtracting 1 mm from the length of a size 10 file (K-Files Limas K; MANI, Tochigi, Japan) at the apical foramen. The middle and coronal thirds were prepared using 1, 2, 3, 4, 5 and 6 drills (Gates Glidden; Produits Dentaires, Vevey, Switzerland).

All canals were further prepared with R40 RECIPROC (40/0.06) file (VDW, Munich, Germany) as follows: The instrument was installed on a gear reduction handpiece powered by a torque-controlled motor (Gold; VDW GmbH, Munich, Germany) and was introduced into the canals with back-and forth movements. After pecking for 3 times and when the blocking sensation was felt, the instrument was removed, cleaned and the canal was irrigated with 5% NaOCl solution (NICLOR 5, Ogna, Maggio, Italy). This cycle was repeated until reaching the working length. Finally, the canals were rinsed with distilled water, dried with paper points and filled with sealer (Kerr Pulp Canal Sealer; Sybron Dental Specialties, CA, USA) and .06 taper gutta-percha points (RECIPROC-Gutta-Percha points; VDW GmbH, Munich, Germany) using the warm vertical condensation technique (BeeFill 2in1 system; VDW GmbH, Munich, Germany). The filled roots were sealed with glass-ionomer cement (Fuji II; GC, Tokyo, Japan), and stored in water for 48 h to allow the sealer to set.

The post space was prepared with a low-speed drill

provided by the post manufacturer up to a fixed depth of 10 mm from the CEJ. Following preparation, the canals were rinsed with 5% NaOCl solution, followed with distilled water, and then canals were dried with paper points. The specimens were randomly divided into three groups of 10 teeth each. The translucent and radiopaque methacrylate-based glass-fiber posts (GC Fiber Posts, diameter 1.4 mm; GC America Inc., Alsip, IL, USA) were luted with three different adhesive luting systems: An experimental self-etching primer/resin-cement MSM-107 (GC, Tokyo, Japan) (group 1), Excite® F DSC adhesive/Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein) (group 2), Gradia Core self-etching bond/Gradia Core cement (GC, Tokyo, Japan) (group 3). All products were handled according to the manufacturer's instructions. The batch numbers, adhesive strategy, curing method, application mode of the materials used in the study are reported in Table 1. Light curing was performed using a LED light (1200 mW/cm<sup>2</sup> output; B.A. Optima 10; B.A. International Ltd., Northampton, UK). The exposed dentin along the coronal part of the root of all prepared specimens was sealed with flowable composite (G-aenial Bond/G-aenial Flo; GC America Inc., Alsip, IL, USA). All the post-cemented roots were placed in water at room temperature for 2 h until further use.

## Preparation of roots for push-out bond strength testing

Five sections of 1.00±0.05 mm were obtained per specimen (150 sections in total) using a water-cooled diamond blade (Isomet; Buehler, Lake Bluff, IL, USA). The actual thickness of each slice was measured with a digital calliper (Orteam, Milan Italy) with an accuracy of 0.01 mm.

Each slice was marked on its coronal side with an indelible marker and attached to a horizontal stainless steel platform with an adhesive mounting tape (Scotch; 3M, St. Paul, MN, USA). Loading was performed on a universal testing machine (Triax Digital 50; Controls, Milan, Italy) at a speed of 0.5 mm/minute until bond failure occurred. Each slice was loaded with a 1 mm diameter cylindrical plunger tip for the cervical and middle thirds and with a 0.7 mm diameter cylindrical plunger for the apical thirds that provided the maximum coverage over the post without touching the canal wall. The load was applied to the apical aspect of the root slice and in an apical-coronal direction, so as to push the post toward the larger part of the root slice, thus avoiding any limitation to the material movement. Bond failure load was noted when a sharp decline was observed on the graph and/or complete dislodgement of the post.

To express the bond strength in megapascals (MPa),

**Table 1.** Batch numbers, adhesive strategy, curing method, application mode of the materials used in the study

Luting agent	Bonding agent	Manufacturer	Adhesive strategy	Curing method	Application mode
MSM-107 Clear, 130208	Self etching primer A, A130208 Self etching primer B, B130208	GC, Tokyo, Japan	Etch-and-rinse	Dual-curing	Etching of the root canal with phosphoric acid for 15 s. Mixing self etching primer A and B for 5 s. Applying to the post space and waiting for 30 s. Drying with medium air pressure for 10 s. Dispensing resin cement into the post space. Seating the post into the root canal. Light curing for 40 s.
Variolink II, shade transparent Base, S04129 Catalyst, S01620	Excite® F DSC, R64753	Ivoclar Vivadent, Schaan, Liechtenstein	Etch-and-rinse	Dual-curing	Etching of the root canal with phosphoric acid for 15 s. Activation of Excite® F DSC. Applying Excite® F DSC for 10 s. Mixing the base and the catalyst of Variolink II and applying the mixed material to the post. Seating the post into the root canal. Light curing for 40 s.
Gradia Core Dual-cured radiopaque composite, 1305071	Gradia Core self etching bond A, 1405031 Gradia Core self etching bond B, 1405121	GC, Tokyo, Japan	Self-etch	Dual-curing	Mixing self etching bond A and B for 5 s. Applying to the post space and waiting for 30 s. Drying with medium air pressure for 10 s. Light curing for 10 s. Dispensing dual-cured luting cement into the post space. Seating the post into the root canal. Light curing for 20 s.

the load at failure recorded in newtons (N) was divided by the area of the bonded interface (A) as calculated by the following formula  $A = \pi(r_1+r_2)[(h^2+r_1-r_2)^2]^{0.5}$ , where  $\pi$  is the constant 3.14,  $r_1$  is the coronal post radius,  $r_2$  is the apical post radius and  $h$  is the thickness of the slice in millimeters. All debonded specimens were analyzed using a stereomicroscope (Nikon SMZ645; Nikon, Tokyo, Japan) at 40x magnification, and failures were classified as follows: adhesive between the cement and the dentin (AD); adhesive between the cement and the post (AP); fracture of the specimen (cohesive within the post and the dentin [FR]); and mixed failure (M).

### Statistical analysis

A preliminary linear regression analysis was conducted in each group to check if the root of origin was a significant factor for differences in push-out strengths of root slices.

The results revealed that in none of the groups did the root of origin significantly influence the measured bond strength. Therefore, the slices were considered as independent statistical units within each experimental group. The results of tests were expressed as the number of observations (n), mean±standard deviation, median and min-max values. The results of the homogeneity (Levene's Test) and normality tests (Shapiro Wilk) were used to decide which statistical methods to apply in the comparison of the study groups. Normally distributed and with homogeneous variances groups were compared three or more groups by Analysis of Variance. Multiple comparison tests, the Tukey HSD test was used. According to those tests results parametric test assumptions were not available for some variables, so the comparisons three independent groups were performed by Kruskal Wallis test. Multiple comparison tests, the adjusted Bonferroni test was used. All statistical analyses were performed with the SPSS

**Table 2.** Descriptive statistics of the push-out bond strength values. Different letters indicate statistically significant differences among groups ( $p < 0.05$ )

Group	Sample size	Mean	Standard deviation	Significance $p < 0.05$
MSM-107	10	9.61	2.98	A
ExciTE® F DSC/Variolink II	10	6.57	3.54	AB
Gradia Core	10	4.79	2.47	B

**Table 3.** Failure mode distribution

Group	Failure mode (n)			
	AD	AP	FR	M
MSM-107	9	15	10	16
ExciTE® F DSC/Variolink II	3	17	0	30
Gradia Core	12	12	0	26

AD: Adhesive between the cement and the dentin; AP: Adhesive between the cement and the post; FR: Fracture of the specimen (cohesive within the post and the dentin); M, and mixed failure.

software (SPSS Ver. 17.0; SPSS Inc., Chicago IL, USA). P value of  $< 0.05$  was considered statistically significant.

## Results

The push-out bond strengths of resin cements are shown in Table 2. There were significant differences in bond strength amongst groups. Experimental resin cement MSM-107 in combination with etch and rinse application (group 1) achieved the highest bond strength ( $9.61 \pm 2.98$ ). The mean value recorded in group 3 (Gradia Core) ( $4.79 \pm 2.47$ ) were significantly lower than group 1 ( $p < 0.05$ ) and did not differ from group 2 (ExciTE® F DSC/Variolink II) ( $6.57 \pm 3.54$ ) ( $p > 0.05$ ). There was also no significant difference between groups 1 and 2 ( $p > 0.05$ ).

## Failure modes

Stereomicroscopic examination of 150 samples revealed that the bond failure to be mainly mixed failure in ExciTE® F DSC/Variolink II and Gradia-Core groups. Adhesive failures between the cement and the post, cohesive failures within the post and the dentin were mostly observed in MSM-107 group (Table 3).

## Discussion

Bond-strength testing has become a popular method for determining the effectiveness of adhesion between endodontic materials and tooth structure. Roydhouse<sup>[12]</sup> introduce extrusion testing in operative dentistry with a system in which composite cylinders were pushed out from dentin disks.<sup>[13]</sup> The extrusion design generates polymerization stresses that similar to those that occur in

clinical situations.<sup>[14]</sup> Microtensile pull-out and push-out tests have been traditionally used to assess the retention of posts in the root canal.<sup>[15,16]</sup> The push-out test is based on shear stress at the interface between dentin and luting material.<sup>[17]</sup> In the present study, the push-out test was performed 24 hours after adhesive cementation procedures because bond strength can increase during this period.<sup>[18]</sup> On the other hand, no aging or rather simulation of clinical function, i.e. thermocycling, cyclic mechanical loading or long-term storage of the specimens was planned for the present study. This short interval fails to provide information about the long-term performance of the materials. It could be interesting to investigate the long-term behavior of the bond following storage in a stimulated oral environment, possibly in combination with thermocycling and/or mechanical loading, in order to mimic clinical function.

In the light of the push-out test results and the analysis of dentin interfaces, the null hypothesis, that there are no differences in bond strengths amongst the luting systems, was rejected. All three of the groups tested showed measurable adhesive properties. The new experimental etch & rinse resin system MSM-107 had the highest bond strength, whereas the Gradia Core self-etching bond/Gradia Core cement combination had the lowest values.

The reported bond strengths of etch and rinse adhesives to root canal compared to self-etch adhesives are inconsistent and seem to be dependent on the luting material used.<sup>[16,19,20]</sup> Nevertheless, it was assumed that combining all the fiber post system components (adhesive, resin cement, and fiber post) from the same manufacturer would prevent possible incompatibility between the materials and allow assessment of the full potential of each system under laboratory conditions.<sup>[20]</sup> The only exception was made in the case of ExciTE® F DSC adhesive/Variolink II group. This may partly serve an explanation for the insignificant difference in bond strength between ExciTE® F DSC adhesive/Variolink II group and GradiaCore group, as it was previously reported that push-out strength may be influenced by a type of fiber post to a greater extent than by a luting agent.<sup>[21]</sup>

A current trend in dental marketing is the use of dual-cure post luting cements that may also act as core build-up materials, due to their high filler content, which increases

their physical properties. Dual-cured cement, investigated in group 3, is a material formulated to be used for both post cementation and the core build-up procedure. A recent study reported that when resin cements with 10wt% or 30wt% filler load were used to lute fiber posts, higher push-out strengths and lower interfacial nanoleakage were recorded, in comparison with 50wt% or 70wt% filled cement.<sup>[22]</sup> Therefore it can be speculated that the high content of filler (70–75%)<sup>[23]</sup> may have negatively affected the bond strength of Gradia-Core. Furthermore, self-etch adhesives include acidic monomer solutions either making the smear layer permeable to allow a formation of hybrid layer interface or hybridized smear layer. The penetrating ability of the dentin smear layer is dependent to smear layer thickness and aggressiveness of self-etch monomers.<sup>[24]</sup> When the self-etch monomer does not etch profoundly enough, the adhesive will be unable to establish a firm bond with the intact dentin<sup>[19]</sup> Retention and 4-year survival rate of fiber posts cemented with Gradia-Core system was comparable with the results obtained with self-adhesive cements.<sup>[25–27]</sup> However, comparative studies between etch and rinse and Gradia-Core system is currently lacking in the literature.

Scarce information is also available on the resin cement MSM-107. The results of the present study are corroborated by other investigators, who evaluated the wear resistances of several resin cements using the three body wear test and obtained significantly higher wear resistance for the MSM-107, compared to self-etch adhesives and Variolink II.<sup>[28]</sup>

Although promising results for the use of fiber posts have been reported,<sup>[29]</sup> problems associated with cementation, bond integrity, and interface stability have not been entirely solved.<sup>[30]</sup> The bond strength between the fiber post and the radicular dentin was affected by the curing mode, the working time, the mixing method, the filler contents, the flow, the composition of the resin matrix, and the moisture control of the root canal. The degree of conversion of the resin cements, the shape of the root canal, the position of post in the root canal, and the space occupied by the resin cement between post and the radicular dentin are also influential factors that require further study.<sup>[31]</sup>

## Conclusion

Within the limitations of the present study, it may concluded that the push-out bond strength of fiber posts was significantly influenced by luting agents. In the test arrangement used, the self-etching approach may offer less favourable adhesion to root canal dentin in comparison with newly developed etch and rinse approach, MSM-107.

## Acknowledgement

The author would like to thank Prof. Marco Ferrari, Prof. Cecilia Goracci and Prof. Simone Grandini for the materials and research facilities they provided.

**Conflict of interest:** None declared.

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