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Bond strength to radicular dentin of two experimental luting cements

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ABSTRACT

Aim The aim of this study was to test two different of experimental cements based on two types of polymerization techniques comparing them with one already well known in the market.
Materials and methods Thirty intact central incisors, extracted for periodontal reasons, were selected and endodontically treated, then were randomly divided into 3 groups of 10 samples: Group 1, light cured composite experimental material with self-etch adhesive and dual polymerization activator; Group 2, dual experimental core build-up with self-etch adhesive and dual-polymer activator; Group 3 (control group), dual cement with self-etch adhesive and dual polymerization activator. One fiber post was luted into the root canal strictly following manufacturer's instructions. Each sample was cut in slices in order to perform the push-out bond strength test with a testing machine. To express the bond strength in MegaPascals (MPa), the breaking load recorded in Newton (N) was divided by the area of the bonded interface (A) in mm^2. The area of the bond interface was calculated as the area of the surface of a truncated cone using the formula: $A = \pi (R + r)$ [h2 + (Rr) 2] 1/2, where R represents the major radius of the coronal post, r represents the minor radius of the apical post and h is the thickness of the slice in millimeters. The diameters and thickness of the slice were measured individually using a digital caliper with 0.01 mm precision. After the test, each slide was observed to detect the type of failure and classified as adhesive between cement and dentin (AD); adhesive between the cement and the post (AP); fracture of the sample, cohesive inside the post and dentin (FR); cohesive in cement (CC); mixed (M).
Results Group 2 recorded the highest values of adhesion strength, group 1 the lowest. There were no statistically significant differences among groups 1 and 2 and controls. The most common failure mode was the mixed one and the less frequent was the adhesive type between the post and the cement.
Conclusions Within the limitations of this study, it can be concluded that the bond strength of experimental resin cements is comparable to that of a cement marketed by the same manufacturer, used here as a control and well known in the market.

INTRODUCTION

Resin-based cements are the material of choice for the cementation of root posts (1-5): good clinical performances and high success rates of teeth restored with fiber posts combined with various resin cements and adhesive systems were recorded (6-8). Nonetheless, evidence from clinical trials indicates that debonding of the post is among the most common failure modes (9). Apparently, obtaining an effective and lasting bond between the fiber and the tooth is the result of the meticulous execution of the operative protocol.
Advances in the study of biomaterials have led to the continuous development of new materials that have inspired researchers to focus on their performance in various clinical applications. However, before starting the clinical trials, the materials must first be checked with *in vitro* tests, both to perform a preliminary screening between similar materials and because of ethical and practical aspects (10). There are many materials that can be used, each one with their advantages and disadvantages; the objective of this study is to test two types of experimental cements with different polymerization techniques comparing them with a well known one, available in the market. The null hypothesis was that there were no statistically significant differences in bond strength, assessed with the push-out, between three different systems for post cementation tested.

MATERIAL AND METHODS

Specimen preparation

Thirty intact central incisors, extracted due to periodontal reasons were selected for the study. Teeth were stored in 0.5% chloramine-T solution at 4 °C and used within 3 months. The patency was checked

with the insertion of K-file #10 (FlexOFiles; Dentsply Maillefer, Ballaigues, Switzerland) as patency file, until achievement the apical foramen. Endodontic treatment was performed using a standard chemomechanical disinfection protocol with ideal irrigants (11). All samples were prepared by the same operator using Reciproc™ (Dentsply-VDW, Munich, Germany) R25 for root canal instrumentation, up to the working length. The Reciproc® blue 25 has a diameter of 0.25 mm at the tip and a taper of 8% (0.08 mm/mm) in the first 3 mm from the tip. The use of this mechanical instrument, mounted on an endodontic micromotor (VDW Silver) by selecting the "Reciproc All" setting, provides a reciprocating movement (counterclockwise rotation of 140° and release with 20° clockwise rotation).
The mechanical instrument was alternated with irrigation cycles with the use of 2 ml of NaOCl at 5.25%. Before starting the preparation, the length of the root canal was estimated with the help of an X-ray or through a manual file #06. The silicone stop was set on the Reciproc® instrument at two-thirds of working lenght. The Reciproc® instrument was introduced into the canals with a slow pitching movement without completely extracting it from the canals. The amplitude of the incoming and outgoing movements (peaks) did not exceed 3-4 mm. After 3 peaks, or if resistance was encountered before the 3 movements were completed, the instrument was taken out of the canals and cleaned.
A manual file #10 was used to verify the patency of the estimated length. The canals have been abundantly irrigated. The Reciproc® instrument was then reused in the same way up to two thirds of the estimated length. A file # 10 was used to determine the working length. The Reciproc® instrument was reused as described until the working length was reached. At the end of the procedure, 10 ml of EDTA (Produits Dentaires SA, Vevey, Switzerland) left for a total time of two minutes were inserted into the canals. Final irrigation was then carried out with 10 ml of 5% NaOCl followed by 3 ml of distilled water. The canals were then dried with paper cones and obtured with gutta-percha cones (Roeko, Coltène Whaledent, Langenau, Germany) and Canal Seal AH Plus™ (Dentsply DeTrey, Konstanz, Germany) according to the manufacturer's instructions.
The root canal obturation was performed with the technique of continuous condensation waves; the gutta-percha cone was then cut and compacted 4 mm from the apex and the remaining portion of the canal was filled with the Obtura syringe with injection of fluid gutta-percha (BeeFill® 2 in 1, VDW).
The obturated roots were sealed with a fluid composite (GC America's G-aenial Universal Flo) and stored in water for 48 hours to allow the sealant to fix.
After removing the coronal filings, the post space was prepared in each root through Gates Glidden burs No. 3, No. 4, No. 5 at low speed to leave 5 mm of apical gutta-percha. Radiopaque aesthetic post (GC Fiber Posts, diameter 1.6 mm, GC) were used for the study. The surface of the post was treated with a silanizing agent (Ceramic Primer II, GC) before cementation following the manufacturer's instructions and air-dried.
The samples were randomly divided into three groups of ten samples each and they were classified according to the cement used, as follows.

- Group 1: Light cured composite experimental material with self-etch adhesive (G-Premio Bond, GC) and dual polymerization activator G-Premio Bond DCA, GC).
- Group 2: dual experimental core build-up with self-etch adhesive (G-Premio Bond, GC) and dual-polymer activator (G-Premio Bond DCA, GC).
- Group 3, control group: dual cement (Gradia Core, GC) with self-etch adhesive (G-Premio Bond, GC) and dual polymerization activator (G-Premio Bond DCA, GC).

After the cavity was prepared for the post, the canal was washed and dried with a jet of air and paper cones. G-Premio Bond and G-Premio Bond DCA were mixed in 1:1 portions, applied to the canal for 20 sec, then dried for 5 sec and cured for 10 sec.
Photopolymerization was performed using a LED lamp (B.a. International) with a ligt intensity of 1080-1320 mW/cm^2, positioning the tip on the coronal part of the post. In every group the cement used was dispensed with a special tip for the root canals: throughout the delivery it can remain completely inside the cement avoiding the formation of air bubbles. The posts were inserted within 1 minute from the application of the cement, maintained with a moderate pressure and polymerized with the same lamp used previously on the two opposite sides for 20 seconds each side and 20 seconds in the coronal part. One GC Fiber Post was luted into the root canal strictly following manufacturer's instructions. The samples were placed in water at room temperature for at least 24 hours before their use.

Push-out test

The crowns were removed at the enamel-cement junction (CEJ) using a low speed water-cooled diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). Based on the length of the root, the roots were cross-sectioned from three to seven 1 mm thick slices with the Isomet saw in water cooling. The first section was made 1 mm from the CEJ. In total, 47 slices were obtained for the first group, 49 for the second group and 56 for the third group. All the slices obtained were used for the statistical evaluation of the push-out bond strenght. The test was performed using a machine (Triax Digital 50; Controls, Milan, Italy) operating at a speed of 0.5 mm/min. Each slice was positioned on the loading machine with the apical part facing the plunger, in order to ensure the application of the loading force in the apical-coronal direction, so as to move the post towards the larger part of the slice. The plunger was positioned so that it was in contact only with the post during the test. To express the bonding strength in MegaPascals (MPa), the breaking load recorded in Newton (N) was divided by the area of the bound interface (A) in mm². The area of the bound interface was calculated as the area of the surface of a truncated cone using the formula: A = ϖ (R + r) [h2 + (Rr) 2] 1/2, where R represents the major radius of the coronal post, r represents the minor radius of the apical post and h is the thickness of the slice in millimeters. The diameters and thickness of the slice were measured individually using a digital caliper (Orteam, Milan, Italy) with 0.01 mm precision. All specimens were analyzed using a stereomicroscope (Nikon SMZ645, Nikon, Tokyo, Japan) at 40 magnifications and the failure modes were classified as follows:

- adhesive between cement and dentin (AD);
- adhesive between the cement and the post (AP);
- fracture of the sample, cohesive inside the post and dentin (FR);
- cohesive in cement (CC);
- mixed (M).

RESULTS

The descriptive statistics of the bond strength (mean ± standard deviation) are shown in Table 1. In particular, group 2 recorded the highest values of adhesion strength, while group 1 recorded the lowest values.
There were no statistically significant differences between the control group and groups 1 and 2.
It follows that group 2, Core build-up dual with self-etch adhesive (G-Premio Bond, GC) and dual polymerization activator (G-Premio Bond DCA, GC) showed higher values than group 1, Light cured composite material with self-etch adhesive (G-Premio Bond, GC) and dual polymerization activator (G-Premio Bond DCA, GC) and control group.
Regarding the failure mode, the most common was the mixed one and the less frequent ones were the adhesive one between the post and the cement and the fracture of the sample. The results of each type of fracture per group are shown in Figure 1.
Regarding the distribution of the fracture type, no significant differences were observed between the groups (Table 2).
The most frequent mode of fracture is the mixed one in all groups, with percentages of 61.70%, 67.34% and 64.28% respectively. As expected, when the cement used was dual-cure, the type of failure are even more similar (group 2 and 3). In the first group the most common failure was always mixed, but with a slightly higher prevalence of the adhesive failure between dentin and cement (AD = 25.5%) followed by detachment of the post (AP) (Fig. 2).

	Mean±SD (MPa)
Group 1	10.46±5.99
Group 2	12.92±6.02
Group 3 Control	11.99±4.22

TABLE 1 Bond stregth values of the three groups.

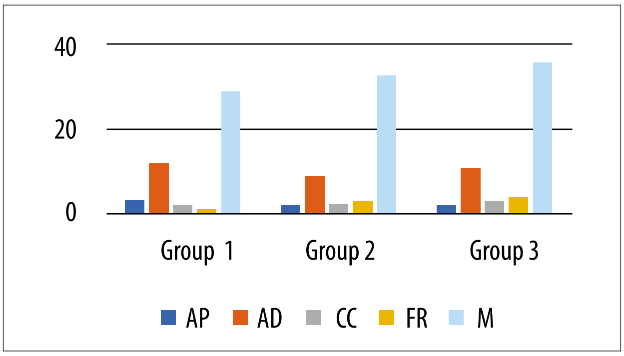

FIG. 1 Type of failure in each group.

	AP	AD	CC	FR	M
Group 1	3	12	2	1	29
Group 2	2	9	2	3	33
Group 3	2	11	3	4	36
Total	8	31	7	8	98

TABLE 2 Failure modes between groups.

DISCUSSION

Several laboratory tests have been developed to evaluate the bond strength (microtensile test, pull-out and push-out) of adhesive post retention (12). However, the bond strength measured between a fiber adhesive post and root dentin depends considerably on the test method used for its assessment. Most studies recommend the push-out test as a method for determining the bonding force of fiber posts to dentin (12-14). In fact, it is considered more similar to the clinical conditions than the pull-out test (15). Moreover, the push-out test demonstrated a more homogeneous stress distribution in the analysis of the elements (14) and a lower variability of the data compared to the microtensile test (12-14). The microtensile technique

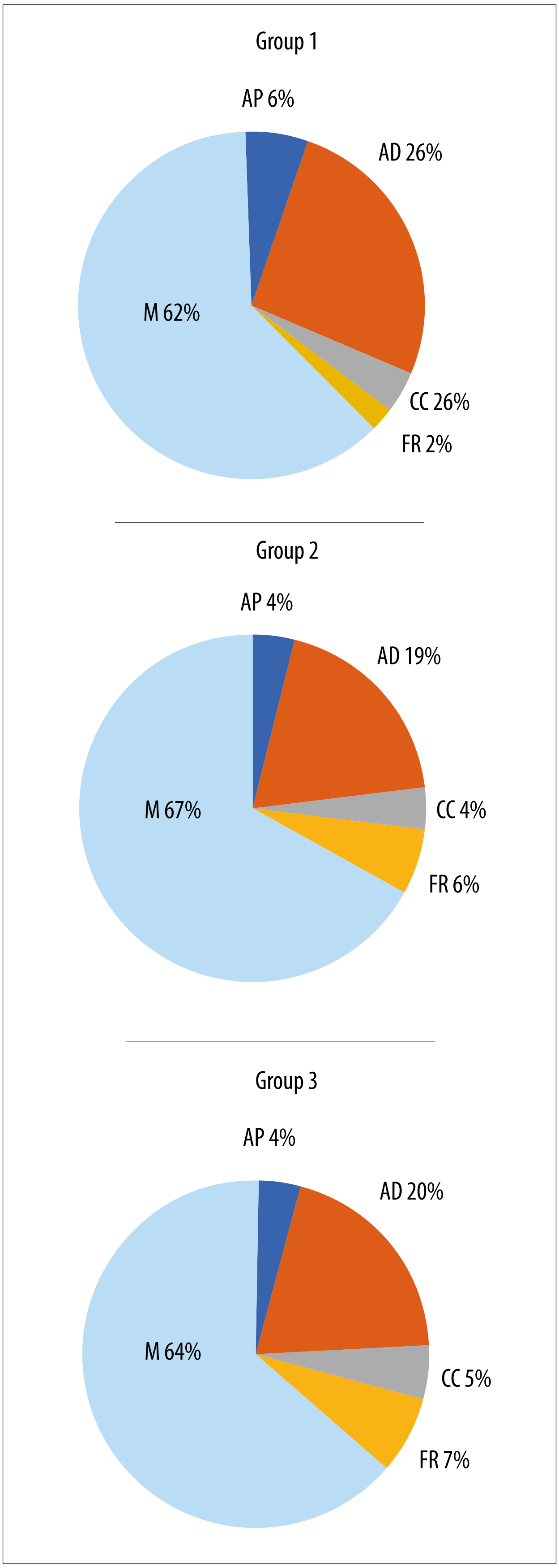

FIG. 2 Chart showing the percentages of failure.

has frequently premature failures compared to the push-out test (12,13). Therefore, this study employed the push-out test to evaluate adhesion of the post to the root's walls.
Based on the results of the study, the null hypothesis is accepted because no statistically significant differences in the bond strength between the tested cements have emerged. Maximum bond strength was achieved by the dual cure build-up experimental material in combination with self-etch adhesive (G-Premio Bond, GC) and dual polymerization activator (G-Premio Bond DCA, GC) (12.92-6.02 MPa). These material combinations showed also slightly higher values of group 3, dual cement (Gradia Core, GC) with self-etch adhesive (G-Premio Bond, GC) and dual polymerization activator (G-Premio Bond DCA, GC) (11.99±4.22). Lower values were measured when the cement used was group 1, light cure composite material with self-etch adhesive (G-Premio Bond, GC) and dual polymerization activator (G-Premio Bond DCA, GC) (10.46±5.99). However, the push-out values of this group can be considered clinically acceptable because no statistically significant differences between the groups were found. This can be explained based on the study by Goracci et al. (16), according to which the post (GC Fiber Posts, GC), was one of the best on the market for the passage of light intensity up to its peak and in any case of sufficient quantity to guarantee a good degree of conversion of the material.
The results concerning the type of failure, and in particular the predominance of mixed ones, are consistent with other previous push-out studies that evaluated dual cements with different percentages of filler, which reported that most of the bonds, about 65-75%, failed in a mixed way, about 5% was an adhesive failure occurred between the post and the cement, and 20-30% was adhesive failure between dentin and cement (17). Cohesive fractures have low percentages across all groups, suggesting that the bond between cement and dentin is less strong than the cement has with the dentin or the post.
Within the limits of this study, it can be concluded that the bond strength of experimental resin cements is comparable to that of a cement marketed by the same manufacturer (Gradia Core, GC, Tokyo, Japan), used here as a control and well known in the market. When the experimental cement is only light-curing, values of bond strengths were measured slightly lower, but still acceptable.
Finally, the results of this *in vitro* study are to be validated with randomized control longitudinal clinical studies (18-23), some of which are already under way.

CONCLUSION

The findings of this study showed that both experimental luting materials can have an equivalent bond strength to radicular dentin as products already in the market.

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