Microtensile Bond Strength of Luting Materials to Coronal and Root Dentin

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ABSTRACT

Purpose: The purpose of this study was to evaluate the microtensile bond strength (μTBS) of two dual-cured resin cements and a glass ionomer cement to coronal dentin versus root dentin.

Materials and Methods: RelyX Unicem (3M ESPE, St. Paul, MN, USA) and Panavia F (Kuraray Medical Inc., Tokyo, Japan) were the resin cements used and FujiCEM (GC Corp., Tokyo, Japan) was the glass ionomer cement used. Once separated, the labial coronal and root surfaces of six bovine incisors were ground with 600-grit SiC papers to expose middle dentin. Then, the dentin surfaces were treated following the manufacturers' instructions and a 1 mm thick layer of each material was applied to the flattened coronal and root surfaces. Each material was cured following the manufacturers' recommendations and a composite buildup was made over the cured luting materials for testing purposes. After 24 hours in water at 37°C, the teeth were sectioned into 1 mm × 1 mm × 6 mm beams and tested for μTBS. The data were analyzed by one- and two-way analysis of variance and Fisher's Protected Least Squares Differences test ($p < .05$).

Results: The μTBSs to coronal and root dentin were similar within each cement. Comparing the materials, RelyX Unicem presented the highest μTBS, followed by Panavia F and FujiCEM, respectively ($p < .0001$).

Conclusions: Although there were differences in μTBS among the materials tested, no significant differences were found between bond strengths to coronal and root substrates.

CLINICAL SIGNIFICANCE

Since bond strengths of luting materials to coronal and root dentin showed comparable results, there is no need to treat those surfaces differently prior to luting of indirect restorations. Nevertheless, because significant differences existed among the different luting materials, the choice of a luting material should be based on the type of preparation and restoration as well as the need for fluoride release.

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Since the introduction of composites and cements, dentin adhesion has been a major issue in restorative dentistry. The variety of commercially available luting materials has significantly increased and, consequently, so has the necessity to better understand the interaction between each material and the tooth substrate. Procedures such as luting of posts, inlays and onlays, and crowns have specific method-

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ologies. In addition, surface treatment, type of restorative material, and cavity preparation are aspects that have to be considered prior to selection of a luting material.

In 1972, glass ionomers were introduced as luting cements for indirect restorations, as liners to protect the pulp complex, and as restorative materials.[1] In the 1990s, resin-modified glass ionomers were introduced with a reformulated composition. Part of the original glass ionomer formulation was replaced by alternative filler particles and/or matrix setting reactions to make it more similar to composites.[2] This newly developed resin-modified glass ionomer cement would combine the good properties of both the conventional glass ionomer and resin composites, that is, sustained fluoride release, increased physical and mechanical strength, less moisture sensitivity and improved adhesion to the tooth structure.

Since introduced in restorative dentistry as polycarboxylate cements in 1968,[3] resin cements have had their composition modified. Currently, their formulation is similar to resin composite restorative materials; however, they contain a lower concentration of filler particles.[4] Although most resin cements require the use of a bonding system, which might make them unattractive to the clinician, they are the primary choice for use with restorations such as all-ceramic restorations. This is due to the fact that glass ionomer cements may expand, fracturing and/or compromising the integrity of the restoration or tooth structure.[5]

Currently, the choice of a luting material is based on the type of restoration and preparation. However, it is important to better understand the interaction between different dentin locations and type of luting material. Most of the preparations for full-coverage crowns and inlays/onlays are within coronal dentin. However, certain restorations include segments or all margins in root dentin. Crown-root fractures, for instance, represent up to 5% of dental traumas.[6] In addition, a recent review by Goodacre and colleagues showed a mean incidence equal to 18% of abutments that become carious in fixed partial dentures.[7] The restorative treatment in that situation would be to incorporate some of the root structure in the preparation of the tooth.

When shear bond strengths of conventional glass ionomers to coronal and root dentin were compared, it was found that the bond strength of the former was greater than that of the latter.[8] However, the literature has limited information on the adhesion of glass ionomers or other types of cements (resin-modified glass ionomers and resin cements) to different areas of the tooth. Although the majority of the tooth preparations are within coronal dentin, it is important to determine whether these cements truly behave differently on coronal and root dentin. Therefore, the purpose of this study was to evaluate the microtensile bond strength (μTBS) of two commercially available dual-cured resin cements and a glass ionomer cement to coronal and root dentin.

The null hypothesis of this study was that bond strength of the glass ionomer and the resin cements to coronal and root dentin would not be statistically significantly different.

MATERIALS AND METHODS

Six bovine incisors stored frozen were used in this study. The teeth were randomly allocated to three groups of two teeth each: group 1, in which RelyX Unicem (RU) was used; group 2, in which Panavia F (PF) was employed; and group 3, in which FujiCEM (FC), a glass ionomer cement, was used. Product descriptions and manufacturers are presented in Table 1. In addition, each group was subdivided into two subgroups: coronal and root dentin.

The crown was separated from the root at the cementoenamel junction using a diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under running water. The labial surfaces were ground with a 600-grit SiC paper to produce a flat dentin surface. Dentin was treated according to the manufacturers' instructions. Equal amounts of ED Primer

TABLE 1. MATERIALS USED IN THE STUDY.

Product	Type	Manufacturer	Shade	Composition	Batch No.
RelyX Unicem	Dual-curing, self-adhesive resin cement	3M ESPE, St. Paul, MN, USA	A2	Powder: glass powder, initiator, silica, substituted pyrimidine, calcium hydroxide, peroxy compound, pigment Liquid: methacrylate phosphoric ester, dimethacrylate, acetate, stabilizer, initiator	132336
Panavia F	Dual-curing, adhesive resin cement	Kuraray Medical Inc., Tokyo, Japan	Opaque	Paste A: MDP, BIS-GMA, filler, benzoyl peroxide, photoinitiator Paste B: BIS-GMA, filler, sodium fluoride, amine	61152
ED Primer	Conditioning system	Kuraray Medical Inc., Tokyo, Japan	—	HEMA, MDP, 5-NMSA, sodium benzene sulfinate, *N,N*-diethanol *p*-toluidine, water	Liquid A: 00149A Liquid B: 00034A
FujiCEM	Resin-modified glass ionomer luting cement	GC Corp., Tokyo, Japan	Yellow	Paste A: fluoro-amino silicate glass, HEMA, dimethacrylate, pigment, initiator Paste B: polyacrylic acid, distilled water, silica powder, initiator	0203072
GC Cavity Conditioner	Polyacrylic acid	GC Corp., Tokyo, Japan	—	Polyacrylic acid, aluminum chloride hexahydrate	090461

5-NMSA = 3% *N*-methacryloyl 5-aminosalicylic acid; BIS-GMA = bisphenol glycidyl methacrylate; HEMA = 2-hydroxyethyl methacrylate; MDP = 10-methacryloyloxydecyl dihydrogen phosphate.

A and B were mixed and applied to the dentin surface of the PF group. The primer was left undisturbed for 60 seconds before the excess was gently air dried. FC samples were treated with GC Cavity Conditioner for 10 seconds and then rinsed and dried with a gentle airflow to leave the dentin moist. No conditioning step was required for RU.

Subsequently, a thin layer of each luting material was mixed and applied to the coronal and root dentin surfaces. To standardize the amount of luting material placed on each specimen, identical spacers were fabricated and wrapped around each tooth. A ruler was used to measure the height of the template from the top of the tooth to the top of the spacer, which was 1 ± 0.2 mm for all specimens. The resin cements were light cured with a Demetron Optilux 501 unit (Kerr Corp., Orange, CA, USA) at 600 mW/cm^2 for 40 seconds. FC was allowed to set for 4 minutes and 30 seconds, following the manufacturer's instructions. For testing purposes, Single Bond Dental Adhesive (3M ESPE) was applied to the top of the luting material layer and light cured. Then, a composite buildup was made over the luting materials with Filtek Z250 (3M ESPE) shade A2, which was placed in three 2 mm increments and light cured for 20 seconds each. After 24 hours in water at 37°C, the teeth were sectioned into 1 mm × 1 mm beams, according to the "non-trimming" technique proposed by Shono and colleagues,[9] and tested for µTBS with a tabletop tester (EZTest, Shimadzu Corp., Tokyo, Japan) using a Ciucchi jig (Figure 1) at a crosshead speed of 1 mm/min.[10,11]

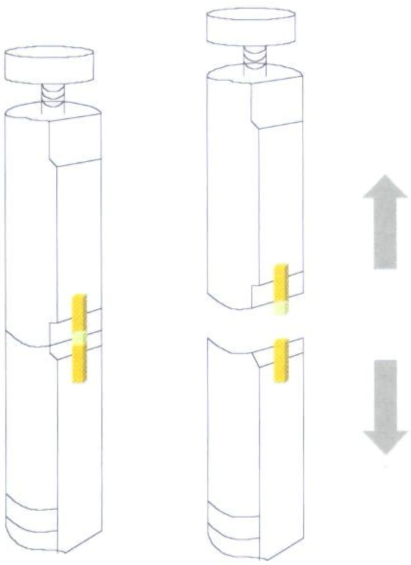

Figure 1. The Ciucchi jig: the device used in this study to hold the specimens during the test of microtensile bond strength.

The mean length of the beams was 6 mm, varying according to the tooth location. Prior to testing, the specimens were macroscopically analyzed and those with interfacial gaps, voids within the material, or any other relevant defects were excluded from the study. No extra specimens were made to replace excluded specimens.

Analyses of the fracture modes were performed using a stereomicroscope at ×40 magnification. Fracture modes were classified as "interface dentin/cement" when the fracture occurred exclusively at the interface between the dentin and the luting material, without any involvement of the substrates. Fractures classified as "cohesive in cement" were entirely within the luting material, and "cohesive in dentin and cement" included both dentin and cement fractures during the μTBS test.

The data were analyzed by one- and two-way analysis of variance (ANOVA) and Fisher's Protected Least Squares Differences (PLSD) test ($p < .05$) to determine the effect of the substrate on each material.

RESULTS

One-way ANOVA showed no statistical significant difference between coronal and root dentin within each material. When comparing μTBS values of coronal and root dentin, Fisher's PLSD test revealed p values of .2807 for RU, .7479 for PF, and .8452 for FC.

RU showed higher bond strengths to crown (20.2 ± 4.6 MPa [n = 24]) and root (19.1 ± 3.7 MPa [n = 24]) when compared with bond strengths of PF (9.7 ± 4.6 MPa [n = 19] and 10.1 ± 4.8 MPa [n = 23]) and FC (4.5 ± 0.7 MPa [n = 24] and 4.3 ± 1.1 MPa [n = 20]) (Figure 2). The effect of material on μTBS was observed with two-way ANOVA at a significance level of confidence > 99%.

Evaluation of the fracture modes by means of a stereomicroscope revealed more than 50% of the fractures at the dentin-luting material interface, suggesting that the bond strengths represented the real values of the adhesion between dentin and the materials tested (Figure 3).

DISCUSSION

Differences among coronal and root dentin could be due to a variety of factors such as the number, diameter, and direction of the dentinal tubules. In addition, differences in the mineral content between coronal and root dentin might be of importance. As a heterogeneous tissue, dentin structure varies at different anatomic locations. Comparisons of the number and diameter of dentinal tubules in human and bovine teeth have revealed that crowns and roots have a difference in tubule distribution.[12,13] In the crown, dentinal tubules can be found in a greater number closer to the pulp than near the dentin-enamel junction (DEJ) because of their convergence toward the pulp.[14] Moreover, an increase in tubule diameter from 0.5 to 3.2 μm occurs toward the pulp chamber, and the peritubular dentin disappears as approaching the predentin.[15,16] It has recently been reported that the mineral content is lower near the DEJ than at the pulpal side of the crown.[17] Other studies have suggested that mineral content is slightly lower in the crown compared with that in the root.[18] Besides the anatomic and compositional differences present in dentin, aging and pathologic events such as caries can modify the dentin structure and composition.[19] The differences described might have some influence

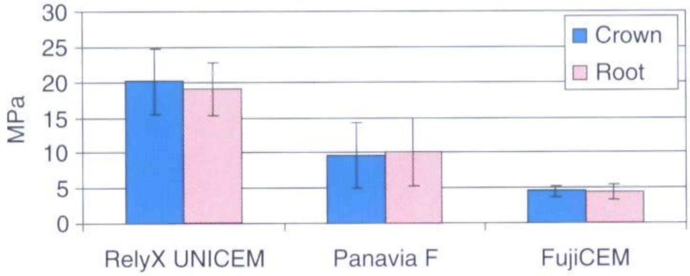

Figure 2. Microtensile bond strengths and standard deviations of each cement to coronal and root dentin. No statistical significant difference was found between coronal and root dentin within each cement. However, the cements were statistically different from each other.

when comparing bond strengths between coronal and root dentin.

Bond strengths of luting materials to root and coronal dentin were initially compared by Berry and Powers.[8] They reported greater shear bond strengths to root dentin than to coronal dentin when a conventional glass ionomer cement was used. Limited reports are available comparing bond strengths of luting materials to coronal and root dentin. Most studies are performed using only coronal dentin as the substrate; therefore, there is a lack of data regarding the bond strength to root dentin.

In the present study, there was no difference between the μTBS values to coronal and root dentin within each luting material tested. This is not in agreement with the study done by Berry and Powers[8]; the reason for the discrepancy could be differences among the materials, techniques, and substrates used in the studies. In the study by Berry and Powers, crowns and roots of human third molars were used, whereas our study used bovine incisors.

Although there was no difference in mean bond strengths between coronal and root dentin within each material, there were significantly different values among cements. RU showed the highest mean bond strengths compared with those of the other materials. This might be due to the presence of phosphorylated methacrylates, which would allow for the simultaneous demineralization of the tooth surface

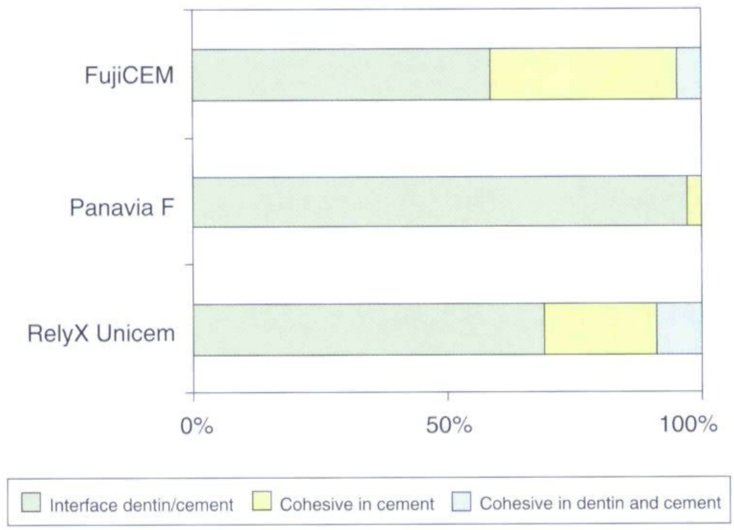

Figure 3. Fracture patterns under microscopic evaluation (×40) classified into three categories: interface dentin/cement, cohesive in cement, and cohesive in dentin and cement.

and penetration of the resinous component of the cement into dentin (information provided by the manufacturer).

In addition, it has been demonstrated that the ED Primer used with the PF cement permits water-induced interfacial changes that result in low cement-dentin bond strengths. The use of a more hydrophobic adhesive layer to cover the primed dentin could increase the bond strengths in 35%.[20] Jayasooriya and colleagues have also proposed the use of a coating technique when using PF.[21]

A recent review by Kramer and colleagues has shown that the newest luting materials exhibit excellent flow characteristics with mean film thicknesses ranging between 8 and 21 µm.[22] The thickness of the luting materials used in this study was not equal to the thickness usually obtained clinically beneath indirect restorations. In this study, in order to produce specimens viable for testing with a standardized thickness, a 1 mm thick layer was used. Therefore, these materials may produce different results applied in thinner layers. Following the manufacturers' indications and respecting the limitations of each luting material might compensate for the differences in bond strengths among materials.

In the present study, the majority of the fractures were at the interface for all materials and not cohesive within the cements as previously described.[23] This is probably due to the improved mechanical properties of the luting materials tested and the reduced bonded surface area used in the µTBS test.[10]

The null hypothesis has been accepted since the bond strengths of the different luting materials were not statistically different when comparing coronal and root dentin. It can be concluded that there is no need for treating coronal and root dentin differently when luting indirect restorations. Further studies are needed to evaluate those materials under different clinical conditions.

DISCLOSURE

The authors do not have any financial interest in the companies whose materials are discussed in this article.

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COMMENTARY

MICROTENSILE BOND STRENGTH OF LUTING MATERIALS TO CORONAL AND ROOT DENTIN

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The microtensile tensile testing method has become popular in the past decade. A survey of the key word *microtensile* in the MEDLINE database from 1996 to 2004 yielded more than 150 references. The test method has its advantages, one of which is that each tooth produces multiple specimens. However, it is much more labor intensive than the conventional shear and tensile techniques. An excellent review covered the methodology in great depth.[1]

This study used the microtensile technique to test bond strengths of resin luting materials to coronal and root bovine dentin. Although most crown preparations usually involve only coronal dentin, the research idea is nevertheless reasonable since, as the authors suggest, certain restorations have their margins in root dentin. The authors used bovine dentin as a substrate and hence were at a loss when comparing their results with published data using human dentin.[2] To their credit, a recent study by Reis and colleagues did find that bovine teeth proved to be a possible substitute for human teeth in either dentin or enamel bonding.[3] However, one must still be cautious in extrapolating the current result to human dentin and on to clinical relevance. Caution should also be used when comparing data obtained with different methodologies, for example, shear, tensile, and microtensile methods. As a general rule, microtensile bond strengths are usually higher than shear bond strengths.

The loss of specimens during microtensile specimen preparation is not uncommon. Other authors have assigned a minimal number to replace lost specimens.[1] The authors of this article left the groups uneven. Standard deviation with microtensile testing is usually smaller than with conventional techniques, yet it may be large enough to obscure the true difference. A larger sample size would alleviate that concern. The authors treated the bond strengths obtained from six bovine incisors as statistically independent data. This is a controversial area that might need further exploration.

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