Application of a Novel Modification of the Microbond Test for Evaluation of Adhesive Bond Strength Between Fiber Posts and Dual-Cure Dental Resin Cement

Authors' Contribution:
Study Design A
Data Collection B
Statistical Analysis C
Data Interpretation D
Manuscript Preparation E
Literature Search F
Funds Collection G

ABCDEF 1 Aleksandra Maletin
A 1 Dubravka Markovic
B 1 Isidora Neskovic
C 1 Bojana Ramic
E 1 Tanja Veljovic
ABCDE 2 Ivan Ristic

1 Department of Dentistry, Faculty of Medicine, University of Novi Sad, Novi Sad, Serbia
2 Department of Materials Engineering, Faculty of Technology, University of Novi Sad, Novi Sad, Serbia

Corresponding Author: Aleksandra Maletin, e-mail: aleksandra.maletin@mf.uns.ac.rs
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Background: While several tests can be used in the laboratory evaluation of composite resin-based cement materials, the push-out test remains the most prevalent. Due to difficulties in sample preparation, as well as a highly complex procedure, we attempted to develop an alternative method for testing the bond strength of dental resin cement materials.

Material/Methods: Ninety-six experimental samples of 2 dual-cure resin cements and 1 fiber post system were prepared for the 2 testing procedures: the push-out test and the modified Microbond test. The degree of monomer conversion was measured by Fourier-transform infrared spectroscopy.

Results: The push-out test results indicated that the bond strength of dual-cure resin cement differs depending on the tooth root region to which it is applied ($p<0.05$), In addition, our findings show that Variolink II exhibits a much lower bond strength relative to RelyX ARC. These findings were confirmed by the modified Microbond test results. The monomer conversion rate results indicate average conversion rates of 85.81% and 61.35% for RelyX ARC and Variolnik II, respectively.

Conclusions: Our study confirms the practical utility of the modified Microbond test in the assessment of bond strength of dental cement resin-based materials. The proposed test method is particularly useful given that, relative to the push-out test, it requires a much smaller number of preparation and execution steps, thus reducing the potential for introducing errors, while increasing the reliability of the obtained findings.

MeSH Keywords: Dental Bonding • Dental Debonding • Resin Cements

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Background

In dentistry it is difficult to predict the adhesion between dentin and cement or restorative materials due to the complex substrate composition, as well as presence of saliva, biofilm, and organic debris on the tooth surface, and the use of water during the procedure. Satisfactory adhesion of cement material to mineralized tooth tissue reduces the risk of secondary caries and marginal microleakage, which can induce bacteria colonization and ultimately lead to clinical failure [1]. For this reason, special attention is devoted to the study of the adhesion mechanism and to the determination of the mechanical parameters characterizing the tooth-cement material bond. One of the greatest challenges in dental practice is achieving adequate adhesion when cementing different fiber post systems in the endodontic dentition restoration. Several factors can influence the bond strength, including post shape, resin cement type, choice of cementation procedure, root canal preparation, and the type of light source adopted in the polymerization process [2–14].

In order to assess the clinical performance of different adhesive materials, the bond strength is usually verified [15]. In most extant studies on this subject, the difference in bond strength between the posts and cement materials, as well as between the cement and root dentin, was obtained.

When examining the adhesive bond strength in dental materials, tensile and shear tests are typically employed [16–19]. In tensile tests, the force applied to the material is perpendicular to the specimen surface, thus subjecting the tooth tissue or adhesive material to traction forces. Conversely, in shear tests, the applied force is parallel to the specimen surface, which results in lateral stress and strain in the tested material [20]. Depending on the specimen size, both tensile and shear tests can be classified as micro or macro tests, with the latter requiring a specimen surface of at least 3 mm^2 [21].

Shear bond strength [21], macro-tensile [2,12,22], pull-off [23], push-out [9,14,24,25] and micro-tensile tests are the usual bond strength testing methods applied to dental composite resin-based materials. While different approaches can be adopted in the assessment of bond strength between dental resin-based cement materials and fiber posts, the push-out test remains the most dominant [21]. However, as it involves a series of very intricate and demanding steps, and requires extensive sample preparation, there is evident need for developing a simplified testing method that can be easily applied in everyday dental practice and that yields comparable results in terms of precision and reliability.

This was the motivation behind the present study, as a part of which, we developed and applied a modified Microbond test, with the aim of making it available for standardized evaluation of polymer material characteristics.

The purpose of the present study was to assess the applicability of the proposed modified Microbond test method for the evaluation of the bond strength of a dual-cure resin cement.

Material and Methods

Push-out test

We used 20 extracted human maxillary central incisors for conducting the push-out test. The study was conducted in accordance with the Declaration of Helsinki, and the protocol was approved by the Ethics Committee of the Faculty of Medicine, University of Novi Sad, Republic of Serbia. The experimental sample for the push-out test consisted of human maxillary central incisors that had been extracted due to periodontal problems from patients ages 30–50 years. All chosen teeth were free from crown or root caries, cracks, and fractures. Care was also taken to select teeth with roots of regular and uniform shape, as well as of similar length. The external debris was removed and the teeth were stored in a 0.5% chloramine solution (supplied by NS Laboratory d.o.o. Novi Sad, Republic of Serbia) for up to 7 days following the extraction. They were subsequently placed into deionized water (supplied by GRAM, Belgrade, Republic of Serbia) at 4°C for further storage of up to 3 months. The aforementioned process was in full accordance with the ISO standard 11405.

The crowns of the selected teeth were cut 1 mm below the enamel-cement interface with a diamond bur (No. 850 012C FG 61896, NTI-Kahla GmbH, Kahla, Germany) under copious water cooling. The endodontic treatment was performed using NiTi instruments (ProTaper, Dentsply, York, PA, USA), after which definitive obturation was performed using gutta-percha points and AH plus sealer (both purchased from Dentsplay De Tray GmbH, Konstanz, Germany).

Twenty-four hours after finishing the endodontic treatment, root canal preparation for the application of fiber posts was performed using System FRC Postec Plus (fiber post size 3, purchased from Ivoclar Vivadent, Schaan, Liechtenstein). For fiber post surface preparation, 37% phosphoric acid (supplied by Ivoclar Vivadent, Schaan, Liechtenstein) was applied for 60 s, followed by rinsing with water and drying. A single-component primer Monobond S (purchased from Ivoclar Vivadent, Schaan, Liechtenstein) was applied for 60 s, after which the surface was thoroughly dried. Two dual-cure resin cements – Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein) and RelyX ARC (3M ESPE, Starnberg, Germany) – were used in the study. Polymerization was enabled using an LED curing lamp (Smartlite IQ 2, Dentsply Caulk, Milford, DE, USA).
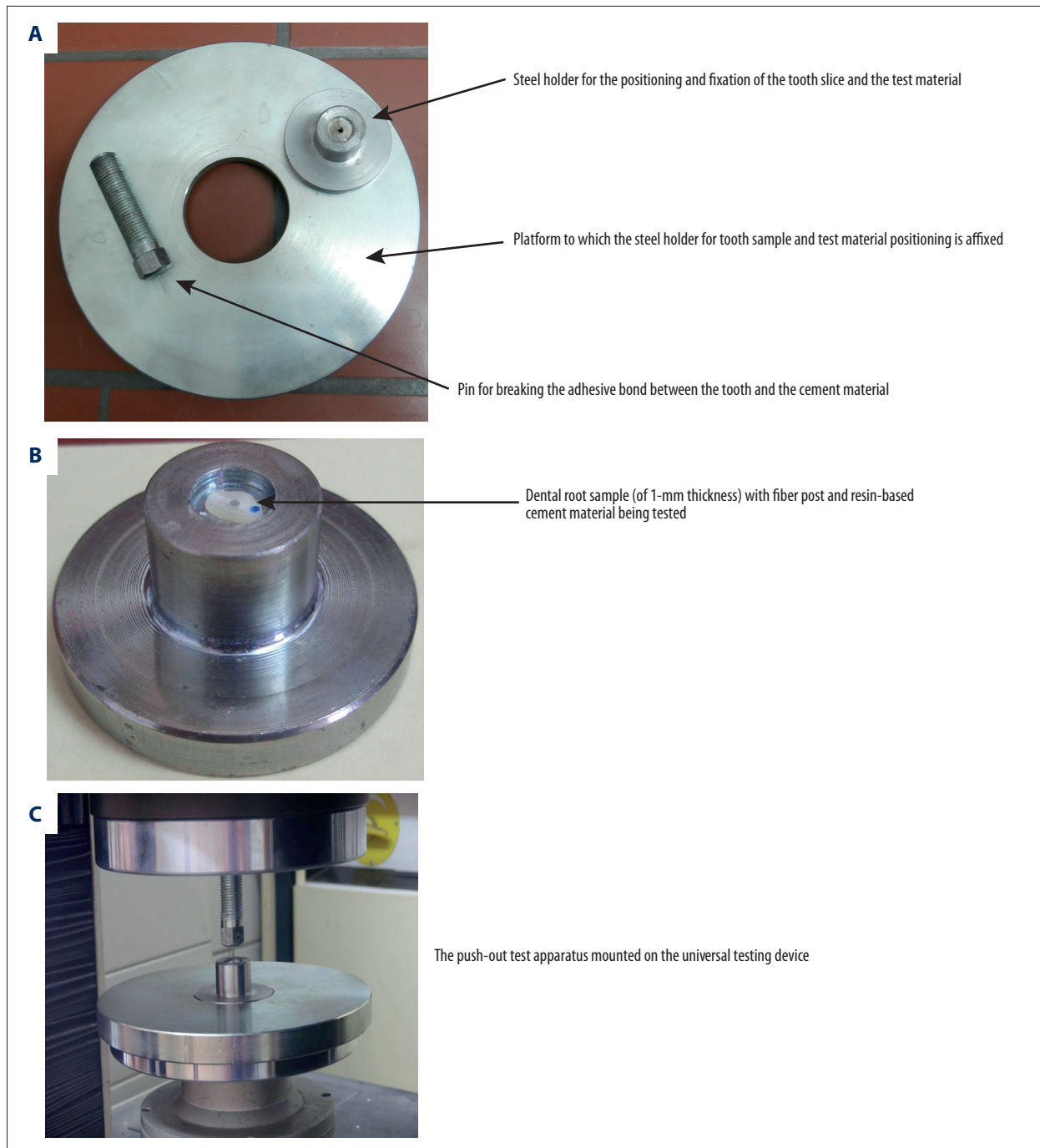
Figure 1. (**A**) Steel holder for the positioning and fixation of the tooth slice and the test material. Platform to which the steel holder for tooth sample and test material positioning is affixed. Pin for breaking the adhesive bond between the tooth and the cement material. (**B**) Dental root sample (of 1-mm thickness) and resin-based cement material being tested. (**C**) The push-out test apparatus mounted on the universal testing device.

Subsequently, samples were stored in closed mini-test tubes in distilled water at 37°C for 24 h.

Roots with a cemented post were cut perpendicularly to the longitudinal axis of the tooth with a linear precision saw (IsoMet 4000 Precision Cutter, Buehler, Lake Bluff, IL, USA). Three 1-mm thick (with ±0.1 mm fault tolerance) segments were obtained from each tooth, in accordance with the root region (i.e., the cervical, the middle, or the apical). The slice thickness was measured with digital calipers (Digital Caliper,
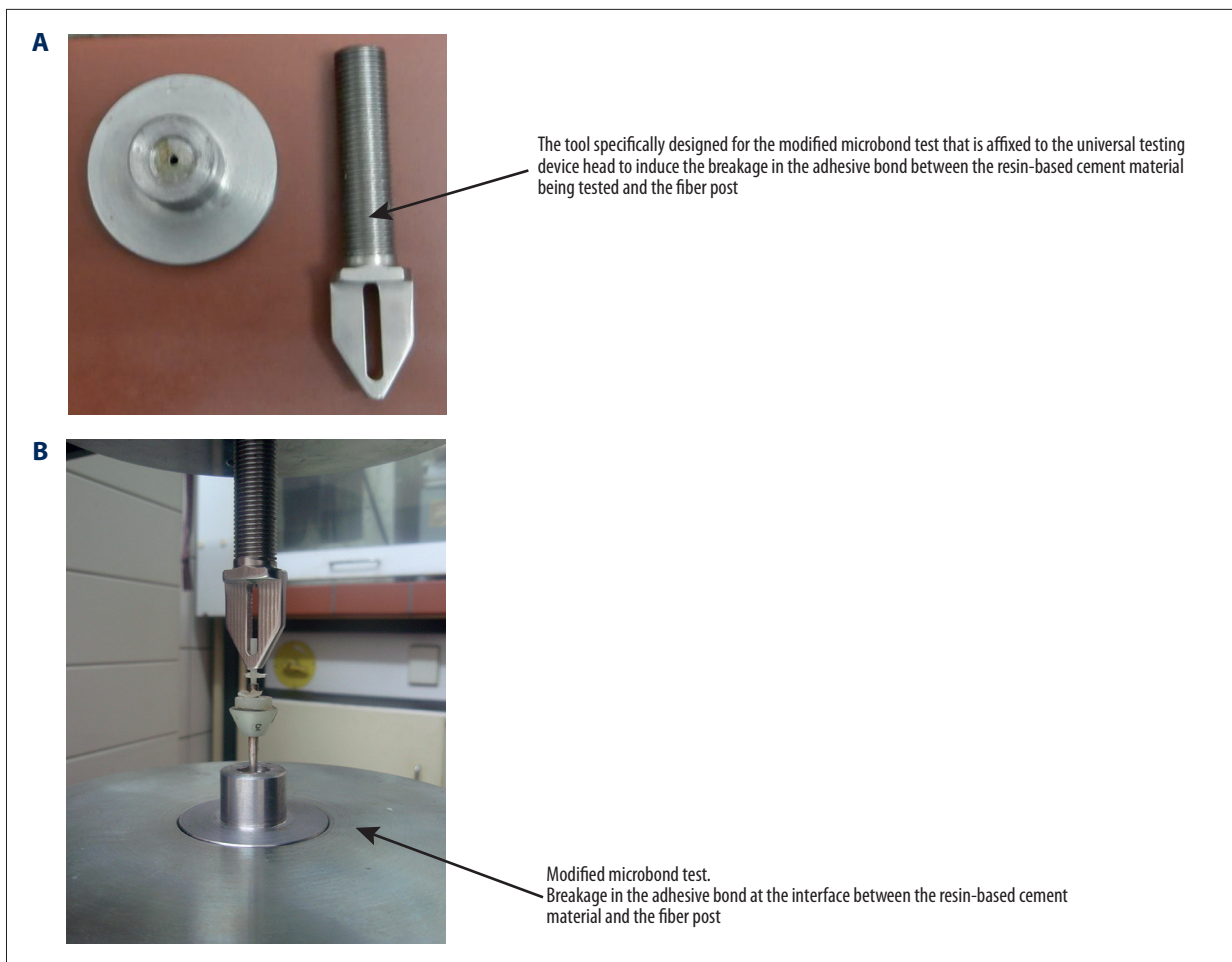
Figure 2. (A) The tool specifically designed for the modified "microbond" test that is affixed to the universal testing device head in order to induce the breakage in the adhesive bond between the resin-based cement material being tested and the fiber post. **(B)** Modified "microbond" test. Breakage in the adhesive bond at the interface between the resin-based cement material and the fiber post.

Mitutoyo, Japan). The push-out test was performed in the apical-coronal direction in the universal testing device (Figure 1) equipped with a previously prepared steel holder and a bespoke steel pin of 7 mm length and 1 mm diameter, designed specifically for the present study.

The bond strength was calculated by applying Equation (1),

$$\text{Bond strength} = F/2r\pi h \ \ldots\ldots \tag{1}$$

where r denotes the post radius in mm (i.e., the cervical third of $2r=2$ mm, the middle third of $2r=1.5$ mm, and the apical third of $2r=1$ mm) and h is the tooth segment thickness in mm.

Modified microbond test

For the modified microbond test performed as a part of the present study, fiber posts were prepared following the

manufacturer's instructions. Three cement material samples were applied to each of the previously prepared 12 posts at 3 positions (i.e., the cervical third, the middle third, and the apical third), thus obtaining 36 samples. The cement material was affixed at 5 mm, 10 mm, and 15 mm distance from the flattened part of the post. Thus, for each dental resin cement material examined in this study, 3 samples of each tooth were prepared, pertaining to 3 different root regions (the cervical, the middle, and the apical) to identify any variations in the bond strength in relation to the position on the root. Silicon molds with a central hole of different diameters (2 mm, 1.5 mm, and 1 mm) and 1 mm thickness were prepared. Polymerization was activated with an LED curing light (Smartlite IQ 2, Dentsply Caulk, Milford, DE, USA) that was applied to each specimen for 40 s. The specimens were then stored in distilled water at 37°C in closed mini-test tubes for 24 h. To perform the modified Microbond test, the authors used a steel holder prepared for the push-out test and the cylindrical cone-shaped steel

part with a central opening that was specifically designed for this investigation, both of which were placed on the universal testing device (Instron Model 1122 Reconditioned, Norwood, MA, USA), as shown in Figure 2.

During the test, the aforementioned cylindrical cone-shaped steel part was moved towards the cement material at 0.5 mm/min speed in an attempt to sever the cement-post bond. The bond strength (in MPa) was calculated according to the formula:

$$\text{Bond strength} = F/2r\pi h \ \dots \dots \qquad (1)$$

where F is the force applied to the material in N, r is the post radius in mm (with the cervical third of $2r=2$ mm, the middle third of $2r=1.5$ mm, and the apical third of $2r=1$ mm), π is 3.14, and h is the thickness of the resin cement segment in mm.

Statistical analysis

The results were evaluated statistically using analysis of variance (ANOVA) at 0.01 significance levels and post hoc test at 0.05 significance levels.

The monomer conversion degree measurement via Fourier-transform infrared spectroscopy (FT-IR)

The conversion degree of resin-based cement monomer was measured by Fourier-transform infrared spectroscopy (FT-IR). For this purpose, 10 specimens of each examined cement material (Variolink II and RelyX ARC) were prepared, in line with the manufacturer's instructions. Briefly, circular silicone molds of 6 mm diameter and 2.5 mm height were employed and light polymerization was achieved by exposing the samples to an LED lamp positioned at fixed height from the mold surface. Prepared samples were stored in mini-test tubes in a 37°C water bath for 24 h. The FT-IR spectra of the samples were recorded in a potassium bromide tablet (0.5 mg of sample with 150 mg potassium bromide) within the 400–4000 cm^{-1} wavelength range, using the FT-IR spectrophotometer Bomem Hartmann & Braun MB-series. FT-IR spectral analyses were conducted 24 h after cement material polymerization.

Fourier transform is a mathematical operation that, when applied to an interferogram (using a computer software), outputs the intensity of radiation transmitted through the sample as a function of frequency I(ν), which corresponds to the IC spectrum recorded by a monochromatic instrument (Figure 3).

The dependence of transmittance (T[%]=I/I$_0$×100) on frequency I(ν) provided directly by classical bichromatic instruments, I(ν) is divided by the reference function I$_0$(ν), which is recorded under the same conditions as I(ν) without a sample in the beam path.
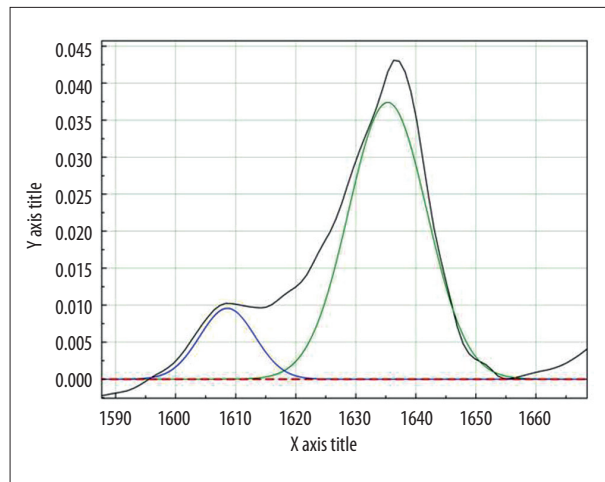

Figure 3. Fourier transform IC spectrum.

The change in the monochromatic interference radiation intensity as a function of X (displacement) is referred to as an interferogram.

Results

The results yielded by the ANOVA statistical analysis, when applied to the push-out test data, indicated that the adhesive bond strength yielded by the Variolink II resin-based cement material was statistically significantly weaker than that produced by RelyX ARC (Table 1, $p<0.01$). The mean adhesive bond strength values for the 2 examined cement materials ranged from 0.6420 to 4.5920 MPa (Variolink II) and from 5.2650 to 16.4230 MPa (RelyX ARC). Moreover, the push-out test indicated that the bond strength was dependent on the root segment, whereby the bond strength values of resin cements in the apical third and middle third were significantly higher than those in the cervical third (Table 1, post hoc test $p<0.05$). Modified microbond test results confirmed those yielded by the push-out test. Specifically, adhesive strength of the Variolink II dental cement was significantly weaker than that obtained for the RelyXARC cement (Table 1, $p<0.01$). For these 2 materials, the mean bond strength values based on the Microbond test were in the 3.7000–4.6750 MPa (Variolink II) and 3.3367–10.5483 MPa (RelyX ARC) range. Similarly, the modified microbond test results indicated that the bond strength values of the resin cement materials measured in different fiber post segments were significantly different (Table 1). Specifically, the bond strength of both Variolink II and RelyX ARC was higher in the cervical third relative to the apical third.

FT-IR analysis showed that the monomer conversion rates were 85.81% and 61.35% for RelyX ARC and Variolink II, respectively (Table 2).

Table 1. Mean bond strength values, standard deviations and p-values of comparisons of bond strength of two tested cement materials and thirds (in MPa).

Variolink II# (p<0.001)					
PT			MMT		
N (samples)	MBS ±SD (MPa)	p-Value##	N (samples)	MBS ±SD (MPa)	p-Value##
CTh N=20	0.6420±0.55483	CTh *vs.* MTh 0.013* CTh *vs.* ATh <0.001***	CTh N=12	4.6750±2.49192	CTh *vs.* MTh 0.140 CTh *vs.* ATh 0.008**
MTh N=20	2.7830±1.83161	MTh *vs.* CTh 0.013* MTh *vs.* ATh <0.001***	MTh N=12	3.5433±1.10569	MTh *vs.* CTh 0.140 MTh *vs.* ATh <0.001***
ATh N=20	4.5920±2.60371	ATh *vs.* CTh <0.001*** ATh *vs.* MTh <0.001***	ATh N=12	3.7000±0.87560	ATh *vs.* CTh 0.008* ATh *vs.* MTh <0.001***
RelyX ARC# (p<0.001)					
PT			MMT		
N (samples)	MBS ± SD (MPa)	p-Value##	N (samples)	MBS ±SD (MPa)	p-Value##
CTh N=20	5.2650±1.56820	CTh *vs.* MTh 0.013* CTh *vs.* ATh <0.001***	CTh N=12	6.9483±1.48610	CTh *vs.* MTh 0.140 CTh *vs.* ATh 0.008**
MTh N=20	6.8540±2.21277	MTh *vs.* CTh 0.013* MTh *vs.* ATh <0.001***	MTh N=12	10.5483±3.40107	MTh *vs.* CTh 0.140 MTh *vs.* ATh <0.001***
ATh N=20	16.4230±3.74859	ATh *vs.* CTh <0.001*** ATh *vs.* MTh <0.001***	ATh N=12	3.3367±1.35194	ATh *vs.* CTh 0.008** ATh *vs.* MTh <0.001***

PT – push-out test; MMT – modified microbond test; MBS – mean bond strength; SD – standard deviation; CTh – cervical thirds; MTh – middle thirds; ATh – appical thirds; # ANOVA; ## Post Hoc test; * significant at 0.05; ** significant at 0.01; *** significant at 0.001.

Table 2. Mean degree of conversion values for the two tested cement materials.

Experimental groups Mean degree of conversion ± SD		
N (samples)	Variolink II	RelyX ARC
10	0.6135202±0.24510902	0.8581615±0.9002248

SD – standard deviation.

Discussion

According to the available data, in more than 26% of the extant studies on adhesion between dentin and resin-based dental materials (adhesive agents in particular), the shear bond strength method was used due to its simplicity and efficiency [21]. However, shear bond test results are highly dependent on a significant number of parameters, such as the type of substrate being tested, the rigidity of the test material, sample preparation, and storage [21].

When aiming to assess the adhesion strength between dentin and a particular resin-based material, both pull-off and push-out macro-tensile tests can be applied, whereby the specimen is extracted or expelled from the apparatus, respectively. These tests are rarely used in practice, as they are protracted and require extensive and complex specimen preparation.

Nonetheless, as a dynamic test, the push-out test is deemed appropriate for evaluating adhesive bond strength between fiber post and resin-based dental cement materials [21]. For example, in the majority of extant fiber post retention studies, the authors employed the push-out test [26–32], as it most closely simulates clinical conditions relative to other testing methods [31]. Still, the significant number of specimen preparation phases that need to be executed prior to testing make the push-out approach a highly technically demanding method for evaluating dental material bond strength [33]. Numerous problems can emerge during specimen preparation and testing process, including difficulty in obtaining human material, and a high likelihood of damage to the human material during storage, which can consequently deteriorate and become unsuitable for testing. In addition, the tooth preparation for cutting is complex, and there is a potential for damaging the specimen during cutting, thus reducing the number of samples
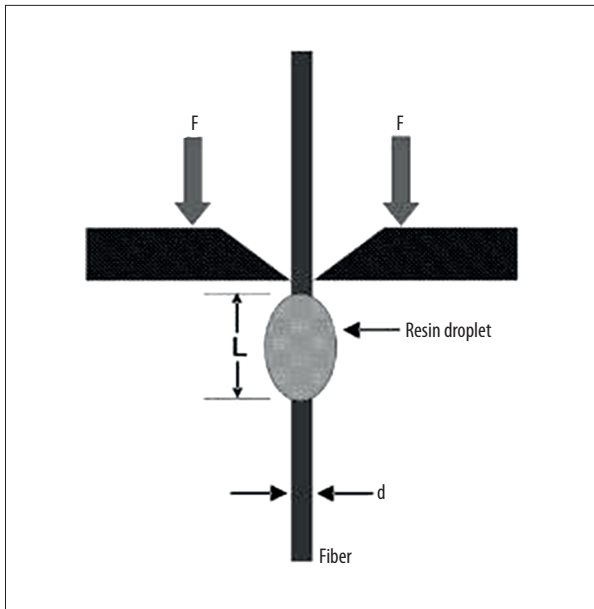
Figure 4. Schematic representation of the basic microbond test apparatus.

available for testing. Similarly, correct specimen positioning in the testing apparatus requires a high degree of dexterity, and both human and cement material can be damaged during positioning. Due to these issues, the testing procedure cannot be standardized; therefore, the data reported in the pertinent literature are difficult to compare, as the test results are influenced by a wide range of factors, including those noted above. Compared to the standard push-out test, the proposed microbond test is highly simplified, as it requires less specimen preparation and fewer test execution steps, thus reducing the potential for specimen damage, while limiting failure and error rates. Specifically, the proposed microbond test dispenses with the need for human material preparation and

storage, and eliminates the specimen cutting phase, thus not only simplifying the testing process, but also requiring fewer tools. Most importantly, the testing apparatus and procedure can be standardized, allowing the obtained results to be compared across materials or studies.

The microbond test, developed by Miller et al. in 1987, is a single fiber-matrix interfacial bond test widely used in polymer material studies. It consists of applying a polymer droplet on a single reinforcing fiber and pulling a fiber out in addition to the debonding at the fiber-matrix interface, as shown in Figure 4 [34]. This method is commonly used to determine polymer adhesive bond strength [35]. However, the microbond test method has not been previously applied as a method of choice in the bond strength testing of dental resin-based materials.

In the microbond test, the interfacial shear strength is calculated by determining the force required to debond the cement droplet from the contact area between the droplet and the fiber [36]. Liu et al. made a disc-shaped droplet using a pinhole, whereas Morlin and Czigany developed a new method for producing a cylindrical droplet [35]. In the present study, the size and shape of the resin materials deviate from those typically adopted in microbond tests, as the aim was to replicate the natural tooth slice shape and thickness that were used when conducting the push-out test (1.0±0.1 mm), as shown in Figure 5.

The results yielded by the push-out test indicated that the adhesive bond strength of the Variolink II resin cement material was weaker than that of RelyX ARC cement, which may be due to the difference in material composition, whereas Variolink II, unlike RelyX ARC cement, is a 2-component material prepared by mixing the base and the catalyst. Mixing of this cement is a highly sensitive procedure, as incorporation of oxygen can result in inadequate cement polymerization and insufficient
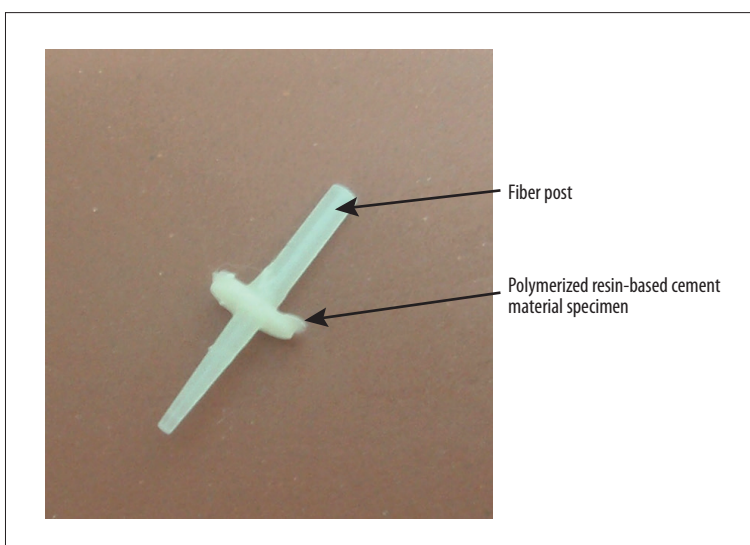

Figure 5. Fiber post with the resin-based cement material specimen.

bond strength [9,37]. The Variolink II bond strength values reported in the pertinent literature are similar to those obtained in the present study [9,10], whereas the values for RelyX ARC are higher [14].

The modified microbond test results concurred with those obtained in the push-out test, showing that Variolink II produced a weaker adhesive bond than RelyX ARC cement. However, given that our extensive search of the pertinent literature failed to find any prior studies in which a modified microbond test was applied in the comparison of bond strengths of different dental resin-based cement materials, our findings could not be evaluated in relation to published data. For example, Miller et al. examined shear bond strength at the fiber-resin interface in polymer materials in general, rather than focusing specifically on dental materials [34].

Nonetheless, the weaker bond strength measured for the Variolink II dental cement is supported by empirical evidence indicating that the inorganic component of resin cement exerts a significant effect on the material's physical, mechanical, and chemical properties [38].

The increase of filler particles is believed to weaken the bond strength of resin cement materials [38]. The optimum percentage of filler is 10–30%. According to the Material Safety Data Sheet data, Variolink II has filler particles in an amount of up to 73.4%, which agrees with research results. Filler loading of the mixed RelyX ARC cement is approximately 67.5% by weight according to the 3M Technical Product Profile. The average particle size for the filler of RelyX ARC is approximately 1.5 µm.

As a part of the present study, adhesive bond strength was also evaluated in relation to the root segment (push-out test), or the fiber post segment (modified microbond test), whereby each root/post length was cut into thirds and the differences were statistically analyzed. Authors of several extant studies have shown that bond strength does not depend on the position on the root [15,39,40], while others reported a significantly higher bond strength in the cervical third [41,42]. A greater bond strength in the cervical third is attributed to a more successful light activation compared to chemical polymerization, as well as to orientation and density of dentinal tubules, better humidity control, and a more visible operating field [11,41]. The superior bond strength in the apical region in the present study may be ascribed to adequate preparation of the root canal for cementing fiber posts, whereby 5 mm of compact apical obturation was ensured, along with using light-transmitting fiber posts. Empirical evidence indicates that light-transmitting fiber posts can assist in efficient resin cement polymerization in regions that are distant from the light source [43]. The use of light-transmitting posts is promoted through the

"one-shot" technique that simplifies the entire post cementation process by allowing simultaneous polymerization of the resin cement and adhesive agent [44].

Moreover, during the push-out test, a complete loss of the cement-fiber post bond was observed, whereby the post would be ejected without any traces of cement material on its surface, which was not the case in the modified microbond test. Namely, during the microbond test, the adhesive bond between the cement material and the fiber post was severed, in line with the obtained results, thus confirming that a more realistic measure of the adhesive cement-fiber post bond strength is obtained in the microbond test.

These results further indicate that, owing to the complex tooth and cement material preparation procedure for executing the push-out test, as well as use of non-standardized equipment, the results of this test are highly influenced by the testing procedure parameters and are thus unreliable.

Conversely, the results yielded by the modified microbond test, in terms of higher adhesive bond strength values obtained for the RelyX ARC cement, are fully consistent with the monomer conversion degree findings obtained through the FT-IR method, indicating that a higher degree of monomer conversion provides better adhesion (stronger adhesive bond). Thus, these results can be considered valid.

The conversion degree of resin-based cement materials during polymerization is important for the longevity and quality of clinical outcomes obtained in any restorative procedure [11]. Insufficiently effective polymerization reaction of resin-based cement materials can result in adverse effects on mechanical and adhesive material performance [40]. Monomer conversion into polymer is rarely complete and a low conversion rate in composite materials and adhesives is generally observed [45]. It is worth noting that, for both materials, the conversion rate exceeds 50%, although no minimum acceptable polymerization success rate (i.e., the degree of resin-based cement monomer conversion) presently exists.

Conclusions

The modified microbond test method used in the present study was shown to be of high practical value, as it is simple to use and can yield reliable information on the adhesive bond strength of resin-based dental cements. Unlike in the push-out test, no special sample preparation is needed prior to conducting the test, and there is no potential for damaging the specimen.

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