Evaluation of bonding strength between selected polymer cements, root dentin and glass fiber reinforced composite posts

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Abstract: The aim of the study was to assess the value of the breaking force of the bonding between selected polymer cements, root dentin and fiber reinforced composite (FRC) posts. Five resin based luting cements were selected for use in the study. Tests were performed on a total of 30 teeth, which were divided into two groups. Fifteen teeth were installed vertically and 15 with an angle of 45°. All samples were exposed to crushing forces in an Instron 4411 device. After the strength tests, each sample was analyzed by micro-CT (micro computed tomography) in order to verify that the forces did not cause defects in the areas of the adhesive connection. The differences observed in the present study only involved the posts that failed at different values of the acting forces – in the case of the vertical sample, x = 532.5 N and in the case with an angle of 45°, x = 117.9 N. For the micro-CT images, there were no defects in the analyzed areas of the adhesive connections. Under the conditions of the current study, all selected polymer cements showed the required level of bonding.

Keywords: polymer cements, fiber reinforced composite posts, bonding, strength tests, micro computed tomography.

Ocena wytrzymałości połączenia wybranych cementów polimerowych z zębiną kanałową i wkładem wzmocnionym włóknem szklanym

Streszczenie: Oceniono wytrzymałość połączenia cementów polimerowych z zębiną kanałową i wkładem wzmocnionym włóknem szklanym. Zbadano pięć rodzajów polimerowych cementów stosowanych do stałego osadzania wkładów. Przeprowadzono test wytrzymałościowy 30 próbek zębów, podzielonych na dwie grupy: 15 zębów osadzonych pionowo i 15 zębów osadzonych pod kątem 45°. Wszystkie próbki poddano działaniu siły zgniatającej za pomocą urządzenia Instron 4411, a następnie każdą próbkę analizowano metodą mikroobrazowej tomografii komputerowej (mikro-CT) w celu sprawdzenia stanu połączenia między zębiną i cementem oraz wkładem i cementem.

W żadnym wypadku nie stwierdzono zerwania połączenia. Zaobserwowano jedynie różnice wartości siły uszkadzającej wkład. W wypadku próbki ustawionej pionowo wyniosła ona średnio 532,5 N, a próbki ustawionej pod kątem – 117,9 N. W obszarach analizowanych metodą mikro-CT nie stwierdzono zerwania połączenia. W warunkach przeprowadzonego badania wszystkie wybrane cementy polimerowe wykazały oczekiwany poziom połączenia.

Słowa kluczowe: cementy polimerowe, wkłady wzmocnione włóknem szklanym, połączenie, wytrzymałość na zgniatanie, mikroobrazowa tomografia komputerowa.

The reasons for using fiber reinforced composite posts (FRC posts) in the restoration of endodontically treated teeth are the simplicity of the application technique, as well as favorable biomechanical and esthetic effects [1–4]. However, the clinical success of this method depends upon the optimum retention of the post in the root canal [5, 6].

A material intended for FRC post cementation should have the following essential features: an ability to be bonded with both the dentin and the post, a high flexural and compressive strength, an elastic modulus similar to that of dentin, minimal to no solubility in water, and an agreeable appearance [7–9]. Only polymer materials for cementation fulfill the above requirements [10, 11].

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Dual-cured (contains two kinds of initiators – photo and chemical) resin based luting cements and core build up materials are recommended for the cementation of prefabricated and individually made FRC posts [12, 13]. An optimal level of adhesion of these materials to dentin is ensured by the use of appropriate bonding materials that require the dentin to be treated by acid – 2-aminoethylisothiouronium bromide (Acid Etch Technique, AET) – or by self-etching techniques. In addition, self-adhesive and self-etching cementation polymer materials are also available [13–15].

The aim of the study was to assess the value of the breaking force of the bonding between selected polymer cements, root dentin and FRC posts.

EXPERIMENTAL PART

Materials

Dual-cured self-adhesive polymer cements:

– Breeze (Pentron Clinical) – BISGMA (bisphenol A-glycidyl methacrylate), UDMA (urethane dimethacrylate), TEGDMA (diluent monomer triethylene-glycol dimethacrylate), HEMA (monomer 2-hydroxyethyl methacrylate), 4-META (4-methacryloxyethyl trimellitate anhydride), barium borosilicate glasses, silica with initiators, stabilizers and UV absorber, organic and/or inorganic pigments and opacifiers;

– G-CEM (automix) (GC) – 7,7,9(or 7,9,9)-trimethyl-4,13-dioxo-3,14-dioxa-5,12-diazahexadecane-1,16-diyl bismethacrylate, 2-hydroxy-1,3-dimethacryloxypropane, diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide, 6-*tert*-butyl-2,4-xylenol, methacryloyloxydecyl dihydrogen phosphate, *tert*-butyl hydroperoxide, diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide, silicon dioxide, pigment;

– G-CEM (capsules) (GC) – UDMA, 4-META, 2-hydroxy-1,3-dimethacryloxypropane, 7,7,9(or 7,9,9)-trimethyl-4,13-dioxo-3,14-dioxa-5,12-diazahexadecane-1,16-diyl bismethacrylate, methacryloyloxydecyl dihydrogen phosphate, 6-*tert*-butyl-2,4-xylenol, hydroquinone, potassium persulfate, fluoroaluminosilicate glass, water, inhibitor, and pigment;

– SoloCem (Coltene) – UDMA, TEGDMA, 4-META, HEMA, dibenzoyl peroxide, and zinc oxide.

Polymer cement requiring the use of AET and bonding material [Ena Cem HF (Micerium)] – UDMA, 1,4-butanediol dimethacrylate, tetramethylene dimethacrylate, dibenzoyl peroxide, *N*,*N*-bis(2-hydroxyethyl)-*p*-toluidine, cristobalite powder, highly dispersed silicone dioxide, and pigments.

Glass fiber reinforced composite post [GC Fiber Post (GC) by Ø 1.2 mm] – 1,6-hexanediyl bismethacrylate, dibenzoyl peroxide, (1-methylethylidene)-bis[4,1-phenyleneoxy(2-hydroxy-3,1-propanediyl)] bismethacrylate, and glass fiber.

Dual-cured single-component adhesive [ExciTE F DSC (Ivoclar-Vivadent)] – HEMA, dimethacrylate, phosphonic acid acrylate, highly dispersed silicone dioxide, and initiators.

36 % H_3PO_4 DeTrey Conditioner 36 (Dentsply DeTrey).

Silane coupling agent [GC Ceramic Primer (GC)] – tetrakis(trimethylsilyl)silane.

5.25 % sodium hypochlorite solution [CHLORAXID (Cerkamed)].

Villacryl SP (Zhermapol) (self-curing polymer) – liquid (methacrylic resin, dimethacrylate ethylene glycol, *N*,*N*-dimethyl-*p*-toluidine) and powder [poly(methyl methacrylate), dibenzoyl peroxide, pigments].

Sample preparation

The canals of 30, previously extracted, single-rooted premolars were prepared using Gates-Glidden drills (N° 3 and 4) and a GC Fiber Post Drill (Ø 1.2 mm). Next, the crowns of the teeth were leveled to the height of the cervix. Upon preparation, the teeth were placed in special containers filled with unpolymerized, acrylic Villacryl SP. Fifteen teeth were installed vertically and 15 with an angle of 45 degrees (respectively, 3 samples for all examined luting materials). Twenty-four hours after polymerization of the acrylic, all the canals were prepared for adhesive bonding following the indications of the producers of the selected, resin-based, luting cements. In the case of the Ena Cem HF material, the canals were etched with 36 % H_3PO_4, rinsed with water, dried for 2 seconds with an air stream and paper points, and the canal dentin was coated with bonding material, ExciTE F DSC. The bonding material was dispersed with a weak stream of air, and any excess was removed with paper points. In the case of Breeze, G-CEM (capsule and automix) and SoloCem materials, the post spaces were chemically cleaned with 5.25 % NaOCl, rinsed with water and dried for 2 seconds with an air stream and paper points. After activation of the applicator device, GC capsules (G-CEM) were placed in a mixer [Silamat S5 (Ivoclar/Vivadent)] for 10 seconds. Subsequently, the cementing materials under examination were placed inside the canals by means of a dedicated mixing and/or application tips. Before cementing, the posts were covered with silane coupling agent that was dispersed with a weak stream of air and then coated with a thin layer of cementing material. Each post was placed in a canal with its crown part protruding ≈ 10 mm above the surface of the tooth. Finally, the bonding and/or cementing materials' photoinitiators were activated with a halogen polymerization lamp [Astralis 7 (Ivoclar/Vivadent)] by curing all surfaces for 20 seconds.

Methods of testing

All samples were exposed to crushing forces in an Instron 4411 device (Instron, 825 University Ave, Norwood, MA, U.S.) (Fig. 1). Samples were loaded in accordance with ISO 4049 standards (pressure of 50 +/- 16 N with a head speed of 0.5 mm/min) until the protruding part of the post failed by being crushed.

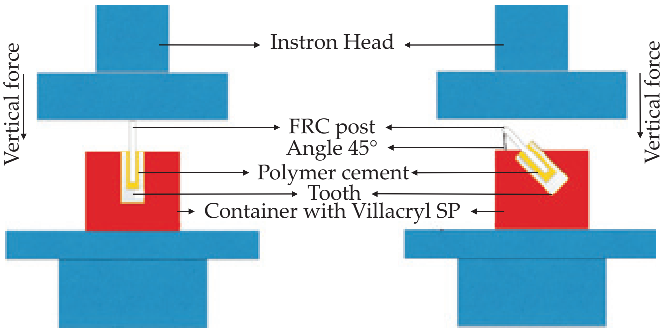

Fig. 1. Scheme of the sample in the Instron device

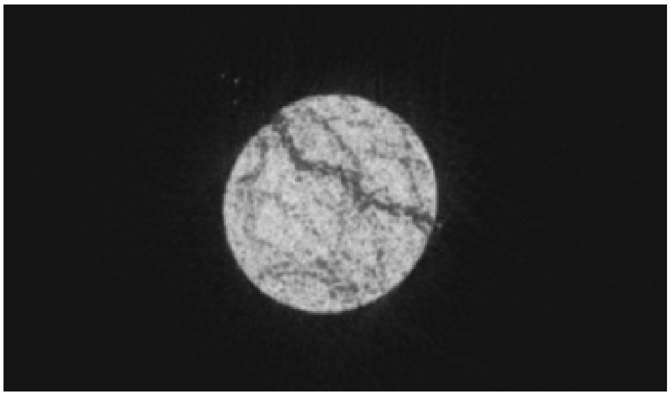

Fig. 2. An exemplary image obtained using micro-CT showing a damaged post

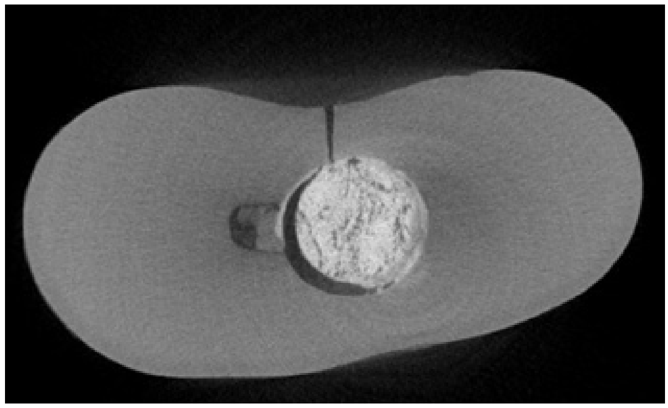

Fig. 3. An exemplary image obtained using micro-CT showing a tooth fracture; air bubbles of different sizes are visible in the intact cement

After the strength tests, each sample was analyzed in the micro-CT (SkyScan 1172, Bruker microCT, Kartuizersweg 3B, 2550, Kontich, Belgium) in order to verify that the forces do not cause defects in the post-cement-dentin interface.

All statistical analyses were performed using the STATISTICA (version 10.0) software package (StatSoft, Inc., Tulsa, OK, U.S.). The data was analyzed using an unpaired *t*-test, and a *p*-value < 0.05 indicated a statistically significant difference.

RESULTS AND DISCUSSION

The results concerning the scope of the breaking forces acting on the posts and their subsequent deflections are provided in Table 1. For each group of samples, minimum and maximum values were specified and the mean value, statistical variance, standard deviation, and standard error were calculated. In 21 cases, the posts failed (crushed or sheared) (Fig. 2), and in 9 cases, the tooth ruptured (Fig. 3).

In the statistical analysis of the obtained results, the value of the post force acting on the post and the maximum deflection of the system before failure were taken into consideration. Samples with cracked teeth were not taken into account.

The micro-CT images showed that there was no failure of the bonding between the selected, resin-based, luting cements and the post and/or dentin. In many images, air bubbles were observed that were enclosed in the structure of the polymerized cement. These bubbles appeared alone, in different places, and had different sizes, ranging from poorly to highly visible.

Statistically significant differences ($p < 0.01$) pertained solely to the strength of the posts themselves.

The use of prefabricated or individually made FRC posts in the restoration of endodontically treated teeth has been a successfully used therapeutic procedure for many years [4, 16]. Clinical studies have reported success rates of 95–99 % for teeth restored with this method [17, 18]. Their favorable biomechanical and esthetic effects were particularly emphasized as a result of their basic properties and their optimum level of retention. The retention of FRC posts depends on the bond strength between the resin luting agent, the area of the post, and the bond strength between the resin luting agent and the root dentin [19]. Debonding is a common cause of failure that is encountered with glass fiber-reinforced composite posts and usually occurs along the post space-dentin adhesive interface [20].

The retentive forces of FRC posts that have been inserted with polymer materials for cementation have

T a b l e 1. Ranges of forces creating post damage and ranges of post deflection when damaged

Variable	*n* important	Minimum	Maximum	Mean (x)	Variance	Standard deviation (SD)	Standard error
Vertical force, N	11	360.5	858.8	532.5	28024.0	167.4	48.3
Oblique force, N	10	101.6	144.7	117.9	138.5	11.8	3.5
Vertical deflection, mm	11	0.21	0.71	0.37	0.02	0.15	0.04
Oblique deflection, mm	10	1.71	4.71	2.93	1.04	1.02	0.31

been assessed in many studies. On the basis of the results obtained, it was found that the use of core build-up composites and polymer cements demonstrated the formation of a homogenous hybrid layer, the penetration into the dentinal tubules, and the optimal adhesion between polymer and post, which provides long-term retention of the glass-fiber reinforced composite posts [12, 14, 21, 22].

In the present study, regardless of the type of material or the type of sample or the size of the force applied, the value of bonding breaking force was not observed. This confirms the earlier findings of other authors who noted no statistically significant differences in the retention of posts bonded with different polymer cementing materials [12, 14, 21, 22]. However, the available reports contain statistically significant differences in the level of adhesion of different polymer cements to the root dentin [23, 24]. According to Dua *et al*., their report concerns not only the same cements, but also a specific area of dentin, where the adhesive strength decreases in the direction of the root apex [23]. Failure was also observed in the cement–dentine interface, followed by the post–cement interface, which shows difficulty in bonding in the post–cement and dentine–cement interfaces [24].

The differences observed in the present study only involved the posts that failed at different values of the acting force – in the case of the vertical sample x = 532.5 N (SD = 167.4) and in the case with an angle of 45° x = 117.9 N (SD = 11.8). The considerable spread (≈ 500 N) in the strength of posts with respect to the vertical forces could have been caused by the fact that the Instron head was not always parallel to the sample.

The observed deflection of the glass fiber-reinforced posts until their breaking point (at a vertical sample x = 0.37 mm, SD = 0.15 and at an angle of 45° x = 2.93 mm, SD = 1.02) indicates some flexibility of the posts, which is consistent with the declarations of the producer that their mechanical and physical properties are equal to those of dentin.

The results obtained in the present study cannot be compared with other findings due to the differences in the empirical model and the application of polymer cements and FRC posts manufactured by other producers. Furthermore, deflection assessment has not yet been the subject of research.

CONCLUSIONS

In the current study all selected polymer cements showed the required strength of bonding. The observed differences only involved to the behavior of the posts that failed at different values of acting forces.

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