Clin Oral Invest (2012) 16:909–915
DOI 10.1007/s00784-011-0576-1

ORIGINAL ARTICLE

Effect of thermal cycling on the bond strength of self-adhesive cements to fiber posts

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Received: 4 September 2009 / Accepted: 26 May 2011 / Published online: 14 June 2011

Abstract The aim of this study was to assess the push-out bond strengths of self-adhesive resin cements to epoxy resin-based fiber posts after challenging by thermocycling. Thirty-six single-rooted premolars were endodontically treated, and the post-spaces were drilled to receive RelyX Fiber posts #1. Three self-adhesive resin cements (RelyX Unicem, G-Cem, and Breeze) were used for luting fiber posts. The bonded specimens were either stored for 1 month in a moist field (37°C) or submitted to thermocycling (5,000 times) prior to push-out test. The maximum force required to dislodge the post via an apical–coronal direction was recorded (megapascal). The data were statistically analyzed with two-way ANOVA and Tukey tests ($p<0.05$). The factors "luting cement" and "thermocycling" significantly influenced bond strengths. The initial push-out values of RelyX Unicem and Breeze were higher than those of G-Cem. After thermocycling, the bond strength of G-Cem increased and no differences were found between groups. RelyX Unicem and Breeze bond strengths were not affected by the thermal challenge. Thermal cycling and cement type differently influence the bond strengths of self-adhesive resin cements. Self-adhesive cements can represent an option for luting fiber posts into root canal

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Keywords Self-adhesive cements · Fiber post · Thermocycling · Bond strength

Introduction

Fiber posts are routinely used in dental practice for increasing the retentive strength of postendodontic restorations in the cases of massive coronal destruction [1–3]. Due to their passive retention into root canals, the dislocation resistance of fiber posts is mainly ascribed to the luting agents and cementation techniques [4, 5]. Resin-based luting materials are preferred for fiber post cementation, as an increase in post retention and higher fracture resistance are expected when compared to conventional cements [6, 7].

Post debondings have been indicated as the most unfavorable situations for post-restored teeth [1, 8]; in this context, the use of different resin cements can influence the results [9, 10]. Self-adhesive cements represent a subgroup of resin cements and are characterized by one-step, simple, and standardized adhesive procedures. These one-step cements do not require pre-conditioning of the post-space walls. Due to the variability of the substrate, bonding to intra-radicular dentin has been considered a challenge. Simplifying luting procedures would be helpful in overcoming some technical problems observed with multi-step cements systems [9], such as the difficult control of moisture and/or the chemical incompatibility between simplified adhesives and dual-cured methacrylate-based resin cements [11–14]. Self-adhesive composite cements exhibited similar push-out bond strength values [4, 15] and higher displacement resistance [16, 17] when compared to resin cements that rely on multi-step adhesive systems.

The bond strength of dental biomaterials can be quantitatively assessed with the push-out test. The push-out test has been considered appropriate for measuring the bond strength inside the root canal, as the shear movements are created parallel to the cement/dentin and cement/post interfaces and seems to procure similar stresses to those occurring in clinical conditions [18, 19]. A push-out design combined to dynamic stresses (i.e., thermocycling) allows for a better understanding of dental materials properties in vitro. Thermal cycling is conventionally used to simulate the thermal changes occurring in the oral cavity during eating, drinking, or breathing which may concur in stressing the adhesive interfaces [20–22]. A previous investigation tested the effect of 5,000 thermal cycles on the bond strength of RelyX Unicem in the endodontic space [23]. However, little information is present regarding the bonding ability of other auto-adhesive luting agents employed for the cementation of fiber posts inside the root canal [24]. Self-adhesive cements possess different chemical compositions that can differentiate their bonding mechanism and a comparison between differently branded luting materials is warranted.

Therefore, the present laboratory study was conducted to evaluate the influence of thermal cycling and cement type on the bond strength of translucent glass fiber posts luted with different self-adhesive resin cements. The null hypothesis tested was that thermal cycling and the type of cement do not affect the bond strength of the tested self-adhesive resin cements when luting fiber posts into root canals.

Materials and methods

Specimen preparation

Thirty-six extracted, single-rooted human premolars stored in 0.5% chloramine T solution at 4°C were collected after the informed consent of the donors was obtained. Exclusion criteria were presence of caries, cracks, or resorptions on the root. The crown of each tooth was removed 1 mm above the CEJ by means of a slow-speed diamond saw (Isomet; Buehler, Lake Bluff, IL, USA) under copious water cooling. Working length was established at 1 mm from the root apex. Cleaning and shaping of the root canal were performed with Protaper Ni-Ti rotatory instruments (size S1, S2, S3. Dentsply Maillefer, Ballaigues, Switzerland) following the crown-down technique. Irrigations with 2.5% sodium hypochlorite were performed between the instrumentations. The roots were dried with paper points, filled with gutta-percha cones (Coltène/Whaledent, Langenau, Germany) and cemented with a resin sealer (AH Plus Jet; Dentsply DeTrey, Konstanz, Germany) according to the lateral condensation technique. The canal access was sealed with a temporary restorative material (Fuji VII; GC Corporation, Tokyo, Japan; batch no. 0410221) and stored in a laboratory incubator (100% relative humidity; 37°C). After 24 h, the coronal seal was abraded by means of #240 SiC paper under water cooling, and the gutta-percha was removed with a pre-shaping drill (Dentsply DeTrey), leaving a 5-mm-long apical seal. A 10-mm-deep post-space was prepared with a universal drill (3 M ESPE, Seefeld, Germany) to match the size of the corresponding epoxy resin-based RelyX Fiber Post (#1; 3 M ESPE; Lot: 02363200603). The post was tried-in the prepared post-space and then cut with a bur mounted on a water-coolant handpiece to the selected coronal length. Three self-adhesive materials were employed for fiber post cementation (n=12): (1) RelyX Unicem (3 M ESPE), (2) G-Cem (GC Corporation, Tokyo, Japan), and (3) Breeze (Pentron Clinical Technologies, Wallingford, CT, USA).

Each material was handled according to manufacturer's instructions. Application modes, chemical composition, and batch numbers of the materials investigated are presented in Table 1.

Self-adhesive cements polymerized in a dual-cure mode. Immediately, 2 s of light curing allowed the removal of the extruding cement with a spatula. After the first 5 min of auto-cure, during which the post was seated to full depth in the prepared spaces using finger pressure, additional 40 s of light polymerization through the top of the translucent fiber post was performed (Astralis 7; Ivoclar-Vivadent, Schaan, Liechtenstein; output 500 mW/cm^2). A core buildup was created with a resin composite core material (CoreX; Dentsply DeTrey; Konstanz, Germany; batch no. 0703000080). All bonded specimens were stored for 1 month in a flower sponge slightly moist with deionized water in a laboratory stove at 37°C and relative humidity.

Prior to push-out test, half of the specimens (n=18) were additionally thermocycled for 5,000 cycles in deionized water from 5°C to 50°C. The dwelling time at each temperature was 30 s in each bath; the transport time between the water baths was 2 s.

Push-out bond strength test

The coronal portion of each root containing the fiber post was sectioned perpendicular to the long axis with a low-speed diamond saw (Isomet; thickness 0.1 mm) under water cooling to create 1.00±0.05-mm-thick slices. The apical side of each slice was marketed with an indelible marker, and the slice was then placed on the universal test machine. The cylindrical plunger of the testing machine (Triax 50; Controls, Milan, Italy) dislodged, via an apical–coronal

Table 1 Chemical composition, application modality, and manufacturers' instruction of the tested materials

Material	Composition	Delivery system	Instructions for use
RelyX Unicem (3 M ESPE), batch no. 270644	Powder: glass fillers, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxy compound. Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators	Capsule and Aplicap Elongation Tip	Mix cement. Apply, self-cure (5 min) and light-cure (40 s)
Breeze (Pentron Clinical Technologies), batch no. 161936	Mixture of BisGMA, UDMA, TEGDMA, HEMA, 4-META resins, silane-treated barium borosilicate glasses, silica with initiators, stabilizers and UV absorber, organic and/or inorganic pigments, opacifiers	Paste/paste dual syringe with a mixing tip	Automix cement. Apply, self-cure (5 min) and light-cure (40 s)
G-Cem (GC Corp.), batch no. 0707051	UDMA; phosphoric acid ester monomer; 4-META; water; dimethacrylates; silica powder; initiators/stabilizers; fluoroaminosilicate glass	Capsules	Mix cement. Apply, self-cure (5 min) and light-cure (40 s)

direction, each inverted, truncated fiber post from the root dentin. A load (cross-head speed 0.5 mm/min until failure) was then applied to the post surface that resulted in shear stresses along the cement/dentin–cement/post interfaces. The retentive strength of the post fragment (megapascal) was calculated by dividing the load at failure (Newton) by the interfacial area of the post segment (S_L). The formula used for measuring the troncoconical area was as follows:

$$S_L = \pi(R + r)[(h^2 + (R - r)^2]^{0.5}$$

In which π was equal to 3.14, R and r were the coronal and the apical post radius, respectively, and h the root slice thickness. The diameters of the post and the thickness of the slice were individually measured using a digital caliper with 0.01 mm accuracy (Orteam; Milan, Italy). Failure modes were evaluated by a single operator under a stereomicroscope (Olympus SZ-CTV; Olympus, Tokyo, Japan) at ×40 magnification and classified as cohesive (within the cement, *C*), adhesive (between the post and the cement, AP, or at the cement/root dentin level, AD), or mixed (adhesive and cohesive fractures occurred simultaneously, *M*).

Statistical analysis

The normal and equal distributions of the push-out bond strength data were first checked and verified by the Kolmogorov–Smirnov and Levene's test, respectively. A two-way ANOVA was executed to determine the effect of the type of cement, thermocycling, and interactions ($p<0.05$). Mean bond strengths of the three cements were analyzed with the Tukey test for post hoc multiple comparisons ($p<0.05$). Calculations were handled by the SPSS 15.0 software (SPSS; Chicago, IL, USA).

Results

Bond strength was significantly influenced by the luting material ($F=14.640$; $p<0.0005$) and thermocycling ($F=18.205$; $p<0.0005$); interactions were also significant ($F=3.836$; $p=0.001$). Mean push-out bond strengths and standard deviations (SD) of the tested cements (megapascal) are shown in Table 2.

Initially, the push-out bond strengths of RelyX Unicem and Breeze were statistically comparable and higher than those exhibited by G-Cem. Thermocycling did not affect the bond strengths of RelyX Unicem and Breeze. After the thermal challenge, increased push-out values were regis-

Table 2 Mean bond strength (SD) values (megapascal) and post hoc comparisons results

Material	Initial	TC
RelyX Unicem	10.3 (2.2) A	13.7 (8.2) A
Breeze	10.3 (4.6) A	9.9 (6.3) A
G-Cem	6.8 (2.6) B	10.0 (4.3) Aa

Different uppercase letters in each column and lowercase letters in each row indicate statistically significant differences among the tested groups

Table 3 Percentage of failure registered in each experimental group

Experimental groups	Initial				TC			
	AD (%)	AP (%)	C (%)	M (%)	AD (%)	AP (%)	C (%)	M (%)
RelyX Unicem	69	19	6	6	62	21	0	17
Breeze	54	46	0	0	83	17	0	0
G-Cem	61	15	0	24	97	3	0	0

tered for G-Cem when compared to the initial group, and no differences were then found among the tested groups.

The most frequently recorded modes of failure were adhesives at the cement/dentin interfaces (Table 3). Debonded specimens between cement and fiber posts were also recorded both in the initial and in the thermocycled groups. Cohesive failures within the cement were only registered for RelyX Unicem prior to thermocycling. Mixed failures were observed for RelyX Unicem and G-Cem before being thermally challenged.

Discussion

The present investigation dealt with the push-out bond strengths of three differently branded self-adhesive resin cements used to lute fiber posts into root canal. The tested materials were selected as they possess different chemical composition that would differentiate their bonding mechanism [25]. Additionally, RelyX Unicem was the most investigated self-adhesive luting agent, while little information is present in literature regarding the bonding performances of Breeze and G-Cem before and after being thermally challenged. The null hypothesis was rejected as bond strengths were influenced by the type of cement and thermocycling.

The push-out test is a practical method to test the multiple variables that can affect the retention of posts, resulting in shear stresses at the cement/dentin and cement/fiber post interfaces. The push-out design is considered more reliable than the microtensile technique for testing the bond strength inside the root canal. Lower incidence of premature failures has been reported when using the push-out test instead of the microtensile method [18, 26]. Due to the substantial premature failures registered when self-adhesive cements were used on dentin and tested with a microtensile test [27, 28], the push-out technique was used in the present investigation.

Quasistatic evaluations seemed to not be enough for a complete understanding of dental material properties, and in this context, dynamic stresses become of great importance. Although in vivo studies are the ultimate testing stages, laboratory tests and aging simulations are important sources for reproducing intra-oral conditions [18, 29, 30]. Thermocycling test is conventionally used to simulate the thermal changes and water exposure that may occur in the oral cavity during eating, drinking, or even breathing [21]. The ISO TR 11450 [31] reports that 500 thermocycles in water (5°C and 55°C) are an appropriate method to test thermal stability of a dental material. To date, concerns still arise on the ability of the test to simulate intra-oral aging. In the present study, the measurements of push-out bond strengths were performed prior and after thermal cycling. The challenging parameters applied in the investigation are in accordance with previous studies [23, 32, 33]. Bitter et al. reported higher push-out bond strengths with RelyX Unicem, after being submitted to 5,000 thermal cycles [23]. An important difference is that in the present study, the roots were not isolated in order to directly expose the bonded interfaces to the different temperatures.

No differences in push-out values were encountered for RelyX Unicem and Breeze before or after being thermally challenged. An increase in bond strength was previously reported for RelyX Unicem when used for luting fiber posts or ceramic restorations [34, 35]. Although the authors admit that this finding is difficult to explain, it was speculated that the thermal stress occurring during the laboratory test would enhance the chemical polymerization of the materials, promoting their complete setting reactions [23, 34, 35]. In the present study, the roots were not embedded in acrylic resins and no isolation was created around the bonded interfaces. The direct exposure of the root to the different temperatures may have promoted weak adhesive–dentin interfaces counting for the differences registered between the investigations. Some questions arise on the degree of monomer conversion of self-adhesive cements when used in an auto-cure mode, as an incomplete polymerization is expected and it will jeopardize bond strength [36, 37]. If so, dual-curing self-adhesive cements have become imperative for achieving reliable bonding. Conversely, Mazzoni et al. reported inferior bond strengths of RelyX Unicem when compared to resin cements that rely on multi-step adhesive systems and after being thermally challenged for 40,000 cycles revealing an increase of silver nitrate deposition at the cement/dentin interface [38]. Limited demineralization/infiltration into dentin was previously observed for several self-adhesive cements, and no hybrid layers were observed at the adhesive interfaces [39, 40].

The lack of an effective hybrid layer and the high viscosity of the materials resulted in the lowest bond strength of selected self-adhesive cements [26, 40], renewing concerns regarding the effective sealing ability of self-adhesive cements. Future studies are warranted to fully investigate on this topic.

Scarce information is present in literature regarding the bonding ability of Breeze as fiber post cementing agent. When compared to another self-adhesive cement and a glass-ionomer cement, Breeze showed the highest dislodgement force after being stored in water for 30 days [17]. Breeze is composed of a mixture of hydrophilic components (i.e., BisGMA and HEMA) and hydrophobic monomers (i.e., TEGDMA). The latter molecule furnishes the material hydrophobic characteristics necessary to withstand the moisture condition of dentin when submitted to thermal challenging and prevent excessive water sorption that would jeopardize the polymerization reaction of the material. This may have accounted for the lack of differences recorded for Breeze prior and after being aged. However, further studies are required to better define the bonding performance of Breeze.

G-Cem previously showed the worse push-out values when used for luting fiber posts in comparison to RelyX Unicem and multi-step resin cements [10]. Although the auto-adhesion mechanism of G-Cem follows a glass-ionomer technology (G-Cem technical information, GC Corp.), its high viscosity has been responsible of the scarce interaction with the adhesive substrates [10]. However, bond strength of G-Cem increased after thermal challenging. Thermal changes will cause expansion/contraction stresses within the material [22, 41], which may affect the adhesive stability, but the cement expansion will also create frictions along the root canals that are thought to improve its mechanical retention [42]. After setting, G-Cem showed a porous appearance at the adhesive interface [27]. The pores would function as stresses–absorbers that would prevent the premature degradation of the adhesive interfaces [43]. It remains to be proved if prolonged expansion/contraction phenomena would result in crack formations within the cement bulk hence diminishing the mechanical properties of the material and if the pores present in the material would resist to prolonged cycles and mechanical occlusal loading and their resistance to degradation when submitted to chemical solutions (i.e., sodium hypochlorite).

The importance of using the cement/post combination recommended by each manufacturer has been previously emphasized [9, 38]. Glass, quartz, zirconium, and/or titanium posts differ in terms of structure, composition, and mechanical properties that can influence their bonding mechanism as well as affect their responses to chemo/mechanical treatments. In the present investigation, only one type of post was used, which is made of glass fibers (60–70 vol.%) embedded in an epoxy resin (RelyX Fiber Post technical pamphlet, 3 M ESPE). Although no differences were found between cements after thermocycling, the bonding performance of self-adhesive cements to other differently branded fiber posts should also be evaluated.

The most frequently registered modes of failures were adhesives between cement and dentin, followed by adhesive at the cement/post interface both prior to and after thermocycling (Table 3). The self-adhesive cement–dentin joint represents the weakest point of the one-step cements. Although self-adhesive cements do not require any pre-treatment of the bonding substrate, their retention was increased once the post-space cavity was roughened, resulting in micromechanical grooves formations were the cement could flow and establish an improved bonding [15]. Doubts also exist on the efficacy of pre-treating fiber posts in the attempt to increase the retention of the auto-adhesive cements/fiber post bonds, such as with silane agents [44]. Due to the limited chemical interaction established between self-adhesive cements and fiber posts, chemomechanical post surface treatments could be proposed to increase the surface area available for bonding and enhance microretentions [45].

In general, bond strength of dual-cure self-adhesive luting agents is not compromised by physiologic thermal stresses. Although further studies are desirable to define the overall bonding process of the different self-adhesive luting agents, a combination of chemical adhesion and mechanical retention seemed to characterize the adhesion mechanism of these simplified cements.

Conclusions

Within the limits of this study, it can be concluded that self-adhesive cement type and thermal cycling influence the bond strengths to fiber posts into root canal. Push-out bond strength values of G-Cem increased after the thermal challenge.

Acknowledgments This study was partially supported by grants CICYT/FEDER #MAT 2008-02347\MAT, JA P07-CTS-2568, and JA P08-CTS-3944.

Conflict of interest statement The authors declare that they have no conflict of interest.

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