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Influence of Luting Material Filler Content on Post Cementation

ABSTRACT

Luting of fiber posts to intra-radicular dentin represents the worst-case scenario in terms of control of polymerization shrinkage. This study tested the hypothesis that filler content of resin cements does not influence luting of fiber posts to intra-radicular dentin, by assaying polymerization stress, push-out bond strength, and nanoleakage expression. The polymerization stress of experimental cements containing 10%, 30%, 50%, or 70% in filler content was investigated. Post spaces were prepared in endodontically treated teeth, and fiber posts were cemented with the experimental cements. A push-out test was performed, and interfacial nanoleakage expression was analyzed. Results showed that luting cements with higher filler content were related to increased polymerization stress ($p < 0.05$), decreased push-out bond strength ($p < 0.05$), and increased interfacial nanoleakage expression ($p < 0.05$). Conversely, lower-stress luting materials increased bonding of fiber posts to intra-radicular dentin. Further *in vivo* studies are needed to investigate the long-term clinical performance of these materials.

KEY WORDS: fiber post, resin-based cement, push-out bond strength, nanoleakage, polymerization stress.

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INTRODUCTION

While bonding to coronal dentin is more reliable, several factors have been described to affect intra-radicular bonding of resin-based materials (Schwartz and Robbins, 2004; Schwartz and Fransman, 2005; Schwartz, 2006; Breschi *et al.*, 2008). The peculiar histological characteristics of root dentin (Ferrari *et al.*, 2000), the presence of primary and secondary endodontic smear layers (*i.e.*, created either by endodontic instruments and modified by irrigants or by post-space calibrated burs) (Schwartz and Fransman, 2005), negative clinical factors (*i.e.,* minimal residual dentin structure), possible incompatibilities between simplified adhesives and dual-cure resin-based cements (Tay *et al.,* 2003b), and adverse geometric factors (Tay *et al.,* 2006) are consistent problems that affect bond strength within the endodontic space.

While the negative influence of some parameters may be reduced, the extremely high C-factor (defined as the ratio of bonded to unbonded surfaces of the restoration; Feilzer and Dauvillier, 2003) that characterizes the endodontic space cannot be solved. The endodontic C-factor has been estimated to be higher than 200, while coronal restoration values range between 1 and 5 (Bouillaguet *et al.,* 2003). Since polymerization shrinkage is an unavoidable adverse effect occurring in resin-based materials, a high C-factor may generate sufficient stress to cause debonding of the luting material from the intra-radicular dentin, thereby decreasing retention and increasing microleakage (Tay *et al.*, 2006). Additionally, since the unbonded surface area within the root canal is reduced, the direct consequence is insufficient stress relief (Tay *et al.*, 2006). Previous reports revealed that cavity geometric factors influence contraction stress, and cavity depth has a stronger influence than diameter (Braga *et al.*, 2006). Thus, considering the configuration of the endodontic cavity, the development of high contraction stress during polymerization cannot be avoided. Contraction stress and microleakage formation also depend on factors related to the rheological and viscoelastic properties of luting materials: Resin materials with high elastic modulus produce high shrinkage stress and consequently increase microleakage formation at the adhesive interface (Davidson and Feilzer, 1997; Moreira da Silva *et al.*, 2007). Contraction stress and elastic modulus are directly proportional to filler content; however, the role played by filler content is controversial. While a significant correlation between filler content and contraction stress has been reported (Condon and Ferracane, 2000), hypothesizing a relevant influence of material stiffness on stress development, it has also been demonstrated that highly filled composites present lower volumetric shrinkage and lower contraction stress (Baroudi *et al.,* 2007).

A current trend in dental marketing is the use of dual-cure post luting cements that may also act as core build-up materials, due to their high filler content, which increases their physical properties. However, no studies have previously investigated the effect of filler content within resin-based cements

Table 1. Study Design, Adhesive System, Composition of the Experimental Luting Materials (expressed in wt%), and Application Steps Used to Lute the Posts into the Endodontic Space

		Experimental Luting Cement				
				Filler Composition		
				Fluoroaluminosilicate Glass		
Group	Bonding System	Resin Matrix Composition	Filler wt%	Particle Size 5.3 μm	Particle Size 0.7 mm	Silicon Dioxide
1	XP Bond: Lot #0604001288	Base: UDMA 80% wt, Methacrylic Acid Ester 20% wt, amines < 1% wt, photo catalyst < 1%	10%	7.2%	2.4%	0.4%
2			30%	21.6%	7.2%	1.2%
3	Self-cure Activator: Lot #070119	Catalyst: UDMA 80% wt, Methacrylic Acid Ester 20% wt, BPO < 1% wt	50%	36.0%	12.0%	2.0%
4			70%	50.0%	17.0%	3.0%

used to lute fiber posts to intra-radicular dentin in relation to polymerization stress development, bond strength, and quality of the adhesive interface. The hypothesis tested in this study was that stress occurring during polymerization, push-out bond strength of fiber posts, and interfacial nanoleakage expression along the bonded interface to intra-radicular dentin are regardless of the filler content of dual-cure resin-based cements.

MATERIALS & METHODS

Contraction Stress of Dual-cure Cements

The polymerization stresses of 4 experimental dual-cure cements with different filler content (10%, 30%, 50%, and 70%; composition reported in Table 1; Experimental Cement, GC, Tokyo, Japan) were analyzed by the use of stainless steel cylinders (6 mm in diameter and 40 mm in height) sandblasted with 150-200 µm alumina as bonding substrates. Two cylinders were fixed to the upper and lower clamps of a universal testing machine (Sun 500, Galdabini, Cardano al Campo, Italy). A silicon mold (6 mm in diameter) was adapted around the lower rod and filled with the mixed cement. After the insertion of the composite, the upper cylinder was lowered and inserted into the upper hole of the mold; the distance between the two cylinders was set to 1 mm. The C-factor of this study design was 3 (according to Feilzer *et al*., 1987). An extensometer (model 2630-101, Instron, Canton, MA, USA) attached to the rods kept specimen height constant during the test, with 0.1 µm accuracy. Five measurements were made for each cement immediately after materials were mixed. The force (N) recorded by the load cell necessary to keep specimen height constant was recorded for 2 hrs during self-polymerization; polymerization stress (MPa) and stress rate (expressed as MPa/sec) were calculated with data-analysis software (Logger Pro 3.5, Vernier Software & Technology, Beaverton, OR, USA).

Data were analyzed with a one-way analysis of variance (ANOVA) test, and differences between groups were calculated by Tukey's *post hoc* test. Statistical significance was pre-set at $p = 0.05$.

Push-out Bond Strength

Twenty single-rooted premolars, extracted for orthodontic reasons, were selected for the study after informed consent was obtained from the donors under a protocol approved by the University of Siena. Treatment and filling of the endodontic space were done in accordance with previously described procedures (Salameh *et al*., 2006). A standardized 7-mm post space was prepared by means of a low-speed calibrated bur (as recommended for RelyX Fiber Post size #2, 3M ESPE, St. Paul, MN, USA) in each root canal, with at least 4 mm of apical seal maintained. Specimens were randomly and equally assigned to 4 groups (N = 5). The experimental cements were used in combination with an etch-and-rinse bonding system (XP Bond, DeTrey-Dentsply, Kostanz, Germany) applied following the manufacturer's instructions (Appendix). Light-curing was performed with the use of a conventional quartz–tungsten–halogen light (600 mW/cm^2 output), with the light tip placed perpendicular to the post for 40 sec. The bonded roots were then placed in individually labeled containers in 100% humidity for 24 hrs at 37°C.

After 24 hrs, the specimens were transversely sectioned into 5 or 6 1-mm-thick serial slices by means of a low-speed saw (Isomet, Buehler, Lake Bluff, IL, USA) under water-cooling. The push-out test was performed on these slabs in a universal testing machine (Controls S.P.A., Milano, Italy) connected to a load cell operating at a crosshead speed of 0.5 mm/min in accordance with Radovic *et al*. (2008). To evaluate the area of the debonded interface and the mode of failure, we then carefully investigated each slab by stereomicroscopy (Nikon type 102, Tokyo, Japan). Images were taken from both the upper and lower sides of each specimen, and by means of image analysis software (Image ProPlus 5.0, Media Cybernetics, Silver Spring, MD, USA), the failure limit was traced with a closed line, then measured after software calibration. Preliminary calculations revealed that the shape of the failed area (either elliptical or circular) did not influence the extent of the lateral surface and thus had no influence on push-out bond strength values (MPa calculation). Failure modes were classified as (A) adhesive between dentin and cementing agent, (M) mixed, (PA) adhesive between post and cementing agent, or (C) cohesive in cementing agent failures.

Statistical analysis of the data was performed, with significance level set at $p < 0.05$. Since the tooth of origin was not a significant factor for bond strength (regression analysis), push-out slices were considered as statistical units. Since the data distribution was not normal according to the Kolmogorov-Smirnov test, we applied the Kruskal-Wallis Non-parametric Analysis of Variance to assess the statistical significance of the differences among the groups, followed by Dunn's Multiple-range test for *post hoc* comparisons.

Interfacial Nanoleakage Analysis

Additional post-luted roots (N = 4 *per* group) were selected for nanoleakage interfacial evaluation in accordance with previously described procedures (Saboia *et al.*, 2008). Undemineralized and unembedded specimens were immersed in a 50 wt% ammoniacal $AgNO_3$ solution, then in photo-developing solution (Tay *et al.*, 2003a). Sections were ground down to approximately 40 μm in thickness with wet carbide papers mounted on a specially designed grinding machine (Micromet, Remet, Bologna, Italy). Slides were stained with acid fuchsin and observed with a transmitted light microscope (Nikon Eclipse, Nikon). Images of all interfaces were obtained at a x100 magnification, and we evaluated the amount of tracer along the interface by scoring nanoleakage interfacial expression by two observers in accordance with previously described procedures (Saboia *et al.,* 2008; Table 2).

We used the Kruskall-Wallis Non-parametric Analysis of Variance to assess the significance of the differences among the groups, followed by Dunn's Multiple-range test for *post hoc* comparisons. The level of significance was set at $p < 0.05$.

Table 2. Means and Standard Deviations of Push-out Bond Strength (N = 5 *per* group; expressed in MPa), Contraction Stress (N = 5 *per* group; expressed in MPa), and Stress Rate (expressed in MPa/min) for Each Tested Material

				Nanoleakage*		
Filler Content	Stress (MPa)	Stress Rate (MPa/min)	Push-out Mean ± SD (median) MPa	Score	N of Images (total)	Median (range)
10 wt%	$1.19 \pm 0.25^{a\dagger}$	0.10 ± 0.02^{A}	9.2 ± 5.5^{1} (7)	0	13(38)	1 (0-2)$^{\#}$
				1	22(38)	
				2	3(38)	
				3	0(38)	
				4	0(38)	
30 wt%	1.40 ± 0.22^{b}	0.12 ± 0.02^{A}	9.8 ± 5.7^{1} (8.5)	0	9(40)	1 (0-3)$^{\#}$
				1	27(40)	
				2	2(40)	
				3	2(40)	
				4	0(40)	
50 wt%	1.84 ± 0.02^{c}	0.09 ± 0.01^{A}	6.9 ± 3.9^{2} (5.5)	0	2(37)	3 (0-3)$^{\$}$
				1	6(37)	
				2	7(37)	
				3	22(37)	
				4	0(37)	
70 wt%	2.36 ± 0.04^{c}	0.22 ± 0.02^{B}	4.5 ± 2.5^{2} (4.5)	0	0(40)	3 (0-4)$^{\$}$
				1	2(40)	
				2	4(40)	
				3	29(40)	
				4	5(40)	

† Means ± SD of stress measurements followed by the same superscript small letter indicate no statistical difference at the 95% confidence level ($p > 0.05$). Means ± SD for stress rate followed by the same superscript capital letter indicate no statistical difference at the 95% confidence level ($p > 0.05$). Means ± SD of push-out followed by the same superscript number indicate statistical difference at the 95% confidence level ($p > 0.05$). Nanoleakage medians followed by the same superscript symbol indicate no statistical difference at the 95% confidence level ($p > 0.05$).

* Nanoleakage distribution was analyzed by two independent observers using light microscopy at x100 and scored as 0 = no nanoleakage, 1 = < 25% of interface showing nanoleakage, 2 = 25 ≤ 50% of interface showing nanoleakage, 3 = 50 ≤ 75% of interface showing nanoleakage, and 4 = > 75% of interface showing nanoleakage.

RESULTS

Contraction Stress of Dual-cure Cements

As the filler concentrations of the cements increased from 10 to 70 wt%, the stress values increased significantly ($p < 0.05$; Table 2). The stress rate was significantly higher for the 70 wt% cement in comparison with the other 3 materials tested, among which no difference was found. The analysis of the contraction force/time curves showed that contraction force sharply increased in the first 2000 sec for 70 wt% cement (Fig. 1), while the curves' slope for the other materials was less steep.

Push-out Bond Strength

When luting cements containing 10 and 30% were used, significantly higher bond strengths were found than those observed in specimens luted with higher filler content (50% and 70% of filler; $p < 0.05$; Table 2). Failure modes of tested adhesive interfaces showed that the majority of the bonds failed in a mixed mode (around 65-75% in each group; around 5% were adhesive between post and cement, and between 20 and 30% were adhesive between dentin and cement [data not shown]).

Nanoleakage Interfacial Analysis

The statistical analysis demonstrated that specimens luted with cements filled by 10% and 30% wt exhibited significantly lower nanoleakage than specimens luted with cements having 50% and 70% wt of filler (Fig. 2, Table 2).

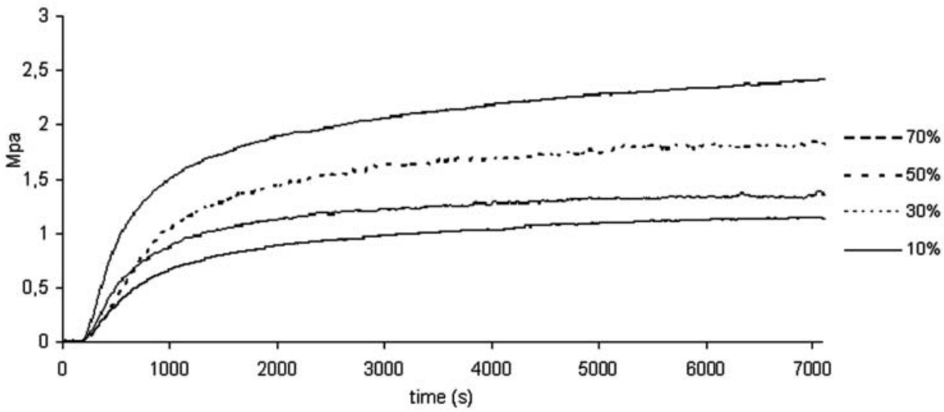

Figure 1. Representative curves of stress expressed in MPa of the different cements tested in the study during light exposures (sec).

Regardless of filler content, specimens showed that nanoleakage was greatly reduced in areas in which the cement thickness was lower than 50 µm, or the surface of the post was in contact with the adhesive layer (Fig. 2). Conversely, areas revealing greater cement thickness showed higher silver nitrate penetration (Fig. 2).

DISCUSSION

The use of dual-cure cements with different filler contents to lute fiber posts, in association with a two-step etch-and-rinse adhesive, revealed differences among the tested groups, in both mechanical and morphological behavior of the adhesive interface. Thus, the tested hypothesis must be rejected, since the amount of filler within the luting material influenced the contraction stress occurring during polymerization, push-out bond strength, and nanoleakage interfacial expression. A significant positive correlation was found between polymerization stress and filler content within the luting agent. The analysis of the results showed that as filler concentrations of the cements increased from 10 to 50 or 70 wt%, the stress values significantly increased; thus, the highest stress values were related to stiffened materials. Interestingly, push-out strength values tended to be inversely proportional to the filler content percentage of the tested luting cements. In fact, as the filler content increased, bond strength was reduced even if statistical differences were evident only between cements containing 10 and 30% filler content *vs.* 50 and 70% filler content ($p < 0.05$). Additionally, the quality of the adhesive interface was affected by the filler content of the adhesive cement, since nanoleakage increased when filler was augmented. Conversely, more elastic materials (10-30% filler content) revealed improved sealing capability of the adhesive interface.

Thus, from the results of this study, it can be speculated that incorporating high percentages of filler within the luting agent can improve its rigidity and increase stress development during polymerization (Condon and Ferracane, 2000), which in turn affects the adhesive interface, reducing bond strength and increasing leakage. This is further emphasized by the high C-factor of the endodontic space, which is considered to be the main factor in the development of the contraction stress in composite resin restorations (Feilzer *et al.*, 1987; Peutzfeldt, 1987; Choi *et al.*, 2000, 2004). C-factor is affected by material thickness. Previous FEA studies showed that the smallest plastic deformations and contraction stresses are found in the thinnest layers (De Jager *et al.*, 2005).

Additionally, the results of this study emphasize the role of the polymerization contraction force occurring within the endodontic space, since reduced polymerization contraction stress can improve interfacial integrity of the endodontic restorations. For this reason, the use of more elastic/flowable materials (with a lower filler content) should be promoted, since better bonding can be achieved (10-30% filler content).

The magnitude of stress depends on the composition of the luting agent (filler content and matrix composition) and its ability to flow before gelatinization, which is related to the cavity configuration and curing characteristics of the material (Braga and Ferracane, 2004). Since the high C-factor that characterizes the endodontic space cannot be solved, filler content and resin matrix composition are claimed to dictate the amount of volumetric shrinkage and elastic modulus values of the material (Labella *et al.*, 1999; Feilzer and Dauvillier, 2003). Even though the resin matrix has a great influence on the behavior of the resin-based material, the stiffness and flowability of the luting cement remain two important factors affecting stress development, both related to the filler content. Indeed, the mechanical properties of materials greatly change by changing properties related to the filler, such as content, size, shape, composition, inter-particle spacing, and surface treatment (Tjandrawinata *et al.*, 2005). The progressive increase of bigger particles of filler might be related to the higher stress and interfacial nanoleakage and lower bond strength. In contrast, the shrinkage reduction observed in the low-filled cements may have been caused by the reduced polymerization rate of these materials, hypothesizing that the light did not scatter properly throughout the low-filled composite material. This hypothesis should be further investigated.

Previous studies reported the use of unfilled resin under composite to improve the marginal sealing of class V restorations by means of a significant reduction in stress values (Kemp-Scholte and Davidson, 1990; Choi *et al.*, 2000, 2004). However, the clinical use of unfilled resin in coronal restorations is problematic, due to its translucency, which may negatively interfere in radiographic diagnosis, in association with the fact that the clinical application in thick layers is difficult, due to high fluidity. Conversely, these negative aspects for coronal restorations may not affect the use of unfilled or less-filled (10-30%

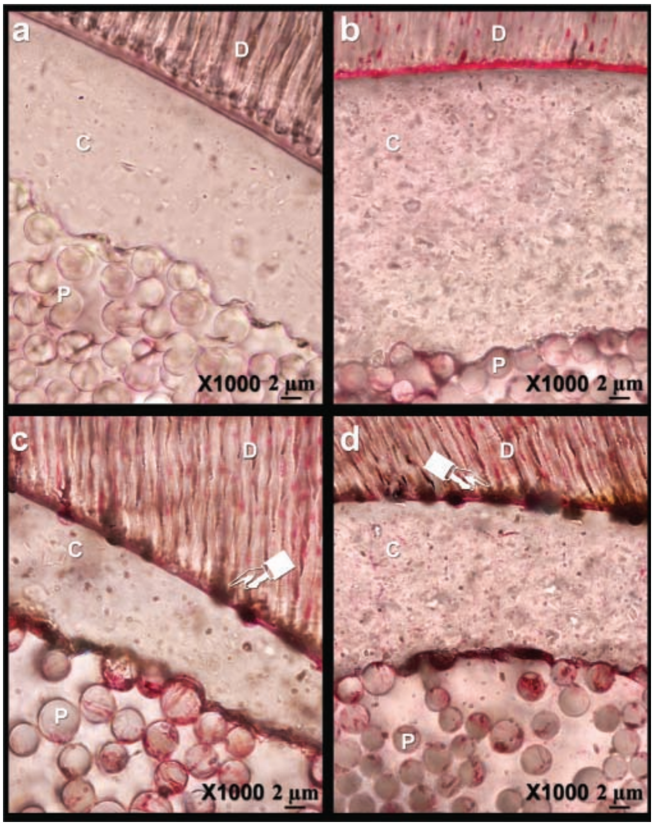

Figure 2. Light-microscopy expression of interfacial nanoleakage analysis of the fiber posts luted within the endodontic space with 4 dual-cure cements differing in their filler content (N = 4 *per* group). All images were taken at the original magnification of x1000 (scale bar = 10 microns). (**a**) Image of a fiber post (P) luted with the resin-based cement (C) containing 10 wt% filler. Excellent adaptation was achieved, and almost no nanoleakage was evident along the adhesive interface (representative image of score 0); intra-radicular dentin (D). (**b**) Adhesive interface created by a fiber post (P) luted with the 30% filler-containing cement (C). Nanoleakage was scattered along the interface, and silver aggregates were sometimes found (representative image of score 1); intra-radicular dentin (D). (**c**) Fiber post (P) cemented with 50 wt% filler-containing cement (C), revealing frequent silver deposits along the bonded interface, even in areas in which the cement layer was very thin (representative image of score 3). Silver deposits are clearly evident both at the intra-radicular dentin (D)/adhesive/cement interface (pointing hand) and along the cement/fiber post interface. (**d**) Image revealing a fiber post (P) luted with the cement (C) with the highest tested filler concentration (*i.e.*, 70 wt%). Nanoleakage was frequently visible along the adhesive interface, affecting both the intra-radicular dentin (D)/adhesive/cement interface (pointing hand) as well as the cement/fiber post interface, with extensive silver aggregates (representative image of score 4).

filler content) resin cements proposed in the present study to lute fiber posts in endodontically treated teeth.

Although the market has focused on the formulation of luting agents also suitable for core build-up, allowing for simultaneous post cementation and core fabrication, from a research perspective it is important to note that the increased percentage of filler in these materials needed for core build-up restoration and retention leads to an irreversible higher stress during polymerization, ultimately resulting in lower bond-strength values.

In conclusion, based on the satisfactory performance shown in the present *in vitro* study, a reduction in filler content for luting cements appears to offer a promising new approach to the luting of fiber posts in endodontically treated teeth. Further *in vivo* studies are currently ongoing to investigate the long-term clinical performance of these materials. Such studies are mandatory before a general recommendation can be made for their use.

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