

Effect of Light-Activation with Different Light-Curing Units and Time Intervals on Resin Cement Bond Strength to Intraradicular Dentin

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The aim of this study was to assess the bond strength of a resin cement to intraradicular dentin varying the light-curing unit and the moment at which the light was applied. Post spaces of endodontically treated canines were prepared. The roots were distributed into 6 groups (n=10) according to the light-curing unit and the moment of light exposure: I) Quartz tungsten halogen-600 mW/cm² (QTH) + immediate light activation (t0); II) QTH + light activation after 10 min (t10); III) Light-emitting diodes (LED)-800 mW/cm² (LED-800)+ t0; IV) LED-800 + t10; V) LED-1,500 mW/cm² (LED-1500)+ t0; VI) LED-1500 + t10. After post cementation, slices from coronal, middle and apical post/root regions were submitted to the push-out test and failure evaluation. It was verified that LED-800 (4.40 ± 3.00 MPa) and LED-1500 (4.67 ± 3.04 MPa) provided bond strength statistically superior to QTH (3.13 ± 1.76 MPa) (p<0.05), and did not differ from each other (p>0.05). There was no significant difference between t0 and t10 (p>0.05). Coronal post/root region (4.75 ± 3.10 MPa) presented significantly higher bond strength than the apical (3.32 ± 2.30 MPa) (p<0.05) and middle regions (4.14 ± 2.99 MPa) showed intermediate values. Adhesive failures were predominant when using QTH. Adhesive and mixed failures occurred more frequently in the apical region. Higher adhesion of the resin cement to intraradicular dentin was observed in the coronal region with LED light-activation, regardless of the moment of light exposure.

Key Words: dental bonding, dental cements, curing lights, dental, dentin.

INTRODUCTION

The use of intraradicular posts enables rehabilitation of endodontically treated teeth with partially or totally destroyed clinical crown that requires prosthetic or restorative treatment (1).

The retention of glass-fiber-reinforced posts is performed by materials with ability to adhere to dentin. The properties of these materials, such as adhesive systems and resin-based luting agents, and aspects related to the technique and protocol can influence the success of the adhesive cementation of fiber posts (2-4).

Dual-polymerized resin cements are dependent on light-activation to achieve favorable mechanical properties, satisfactory clinical performance (5,6) and

to allow for initial stability of the material to resist masticatory stresses (7). It has been speculated that a delay in the light-activation of dual-cured materials would enhance their properties (7,8), as the immediate exposure to light after cement handling could compromise the self-curing reaction by entrapping polymerization promoters and unreacted monomers (6-9).

Assuming that the resin-based luting agents whose self-curing mechanism is deficient or compromised by the immediate light-activation can present changes in their adhesive characteristics (10), the delay in light exposure could favor the chemical reaction, modifying the polymer structure and increasing the degree of conversion (8,11). Consequently, the material's properties could be reinforced by the further conversion

of monomers by light-activation. Additionally, the degree of conversion and the properties of adhesive cements are influenced by the power density delivered by the curing units (12,13).

Quartz-tungsten halogen (QTH) and light emitting-diodes (LED) are the most commonly employed light-curing units in the clinical practice (14). QTH emits light from a halogen bulb, which is filtered by a dielectric pass-band filter to remove the undesirable wavelengths (14). LED is formed by junctions of doped semiconductors to generate light and requires no filters to produce blue light with a narrow specter (440-490 nm) and falls within the camphoroquinone absorption spectrum (470 nm) (15).

The impact of light curing-units and delaying the light-activation on bond strength of resin-based luting agents to intraradicular dentin is not well established. The null hypotheses tested in this study were that neither the light-curing unit nor the moment at which the light is applied interferes with the bond strength of resin cement to the root dentin at different post/canal regions. The objective in this study was to assess the push-out bond strength of dual-cured resin cement after light-activation with QTH 600 mW/cm², LED 800 mW/cm² and LED 1,500 mW/cm² immediately or 10 min after placing the post inside the root canal.

MATERIAL AND METHODS

This study was independently reviewed and approved by the institutional Ethics Committee.

Specimen Preparation

Sixty maxillary canines stored in 0.1% thymol solution at 4 °C were selected. All teeth had straight roots, completely formed apices, single canals, no calcification or radiographically confirmed resorption. The teeth were washed in running water for 24 h to completely remove thymol residues and examined under ×20 stereomicroscope magnification (Leica Microsystems, Wetzlar, Germany) to exclude those with fracture lines or fissures.

The clinical crowns were sectioned transversally close to the cementoenamel junction leaving a root length of 15 mm. The working length was determined by subtracting 1 mm from the root length using a #15 K-file. Instrumentation was performed with K3 instruments (SybronEndo Corporation, Glendora, CA,

USA) up to a #40/.02 instrument. During this process, root canals were irrigated with 2 mL of 1% sodium hypochlorite between each file, followed by final irrigation with 17% EDTA for 5 min. Final irrigation was done with 10 mL of distilled water, and the canals were dried with absorbent paper points (Dentsply Ind. e Com. Ltda., Petrópolis, RJ, Brazil). The root canals were filled using thermomechanical condensation with McSpadden instruments (Moyco Union Broach, York, PA, USA), gutta-percha points (Dentsply Ind. e Com., Ltda.) and AH Plus sealer (Dentsply DeTrey, Konstanz, Germany). Radiographs were taken to verify the quality of filling. Specimens were sealed with temporary coronal filling material (Coltosol; Vigodent, Rio de Janeiro, RJ, Brazil) and stored at 37°C in relative humidity for 72 h.

Post Space Preparation

Gutta-percha was initially removed by using heated endodontic pluggers, maintaining at least 4 mm of filling material in the apical third. Post spaces were prepared to a depth of 10 mm by using a Largo drill at a low-speed handpiece (Dabi Atlante; Ribeirão Preto, SP, Brazil). Final irrigation was done with distilled water and the post spaces were dried with paper points.

Post Cementation

Parallel glass fiber-reinforced composite posts (Reforpost #3; Angelus, Londrina, PR, Brazil) were used.

For the adhesive luting procedure, a bonding agent (ED Primer II; Kuraray, Tokyo, Japan) was applied inside the root canal as well as onto the posts for 30 s. The adhesive was gently air dried and the excess material was removed with paper points. The pre-treated posts were coated with the resin luting agent (Panavia F 2.0; Kuraray) and immediately seated into the root canals.

The specimens were randomly distributed into 6 groups (n=10) according to the light-curing unit and the moment at which light was applied: I) quartz-tungsten halogen (QTH) with 600 mW/cm² power density (Ultralux; Dabi Atlante) + immediate light-activation (t0); II) QTH + light-activation after 10 min (t10); III) LED with 800 mW/cm² power density (Spaceled; Ecel, Ribeirão Preto, SP, Brazil) (LED-800) + t0; IV) LED-800 + t10; V) LED with 1,500 mW/cm² power density (Radii Plus; SDI Ltd, Bayswater, Victoria, Australia) (LED-1500) + t0; VI) LED-1500 + t10.

Excess material was removed and the resin luting

agent was light-cured with the allocated light-curing unit at the determined moment by placing the light tip perpendicularly to the post for 40 s. The light-tip distance was standardized as being the coronal remaining portion of the post. Before each luting procedure, the light output of each curing unit was measured with a light curing meter (Ecel) to ensure accurate light intensity.

Push-out Test

The specimens were serially sectioned horizontally using a precision cutting machine (Isomet 1000, Buehler, Hong Kong, China) with water cooling. Post/dentin sections with 1 mm thickness were obtained from each coronal, middle and apical regions and subjected to the push-out test. Each section was marked on its apical side and fixed in a stainless steel base, with a central hole attached to the lower portion of a universal testing machine (Instron Model 4444; Instron, Canton, MA, USA). The specimen was positioned in the machine, ensuring that the coronal surface faced the metallic base and the resin/cement/post was centered over the hole in the base. The push-out test was performed by applying a compressive load to the apical aspect of each slice by using a 0.6-mm-diameter cylindrical plunger attached to the upper portion of the Instron machine. A crosshead speed of 0.5 mm/min was applied until bond failure occurred. To express the bond strength in MPa, the load at failure recorded in N was divided by the area (mm²) of the post/dentin interface.

After the push-out test, the fragments were examined under $\times 20$ stereomicroscope magnification (Leica Microsystems LTD) to assess the failure mode, which was considered adhesive (when occurred in the dentin/adhesive cement interface), cohesive (when the rupture occurred in the cement), and mixed (when both modes were combined).

Statistical Analysis

After checking the assumption of homogeneity of variance and normal distribution, bond strength data were analyzed statistically by ANOVA with a significance level of 5%. Tukey's test was applied where significant differences were detected. The SPSS 17 software (SPSS Inc., Chicago, IL, USA) was used to perform the statistical analyses.

RESULTS

There was a statistically significant effect for the factors light-curing units ($p=0.003$) and post/canal region ($p=0.011$). Neither the immediate or delayed light-activation ($p=0.057$) nor the interaction among the factors ($p=0.44$) had significant effect.

QTH provided the lowest bond strength values (3.13 ± 1.76) and differed significantly from LED-800 (4.40 ± 3.00) and LED-1500 (4.67 ± 3.04) ($p<0.05$), which did not differ from each other ($p>0.05$).

For the post/canal region, the coronal region presented higher bond strength values (4.75 ± 3.10) of the adhesive luting system to the dentin, and it was statistically different from the apical region (3.32 ± 2.30) ($p<0.05$). The middle region (4.14 ± 2.99) had intermediate values that were not statistically different from both the coronal and apical regions.

In the failure analysis, prevalence of adhesive failures was observed in the group light-activated with QTH, regardless of the post/canal region and the moment at which the light was applied. For the group LED-800, there was predominance of mixed failures in the coronal and middle regions and adhesive in the apical region, regardless of the moment of light exposure. It was also verified that the immediate light-activation with LED-

Table 1. Failure modes observed in each group after the push-out test (%).

Failure mode	QTH						LED - 800						LED - 1500					
	Immediate			After 10 min			Immediate			After 10 min			Immediate			After 10 min		
	C	M	A	C	M	A	C	M	A	C	M	A	C	M	A	C	M	A
Adhesive	50	60	70	60	60	80	10	20	60	20	30	60	0	10	40	10	10	50
Cohesive	30	0	0	20	10	0	30	30	0	0	0	0	50	50	10	50	30	10
Mixed	20	40	30	20	30	20	60	50	40	80	70	40	50	40	50	40	60	40

C: coronal post/canal region; M: middle post/canal region; A: apical post/canal region.

1500 presented a higher amount of cohesive and mixed failures in the coronal region, cohesive in the middle, and mixed in the apical region. When the adhesive cement was light-activated after 10 min, cohesive failures were predominant in the coronal region, mixed failures in the middle third and adhesive failures in the apical post/canal region. The percentage of failures is given in Table 1.

DISCUSSION

The null hypotheses were partially not accepted since only light curing-units affected the bond strength of resin-based luting agents to the intraradicular dentin.

The results of the present study revealed that the light-activation with QTH light-curing unit provided lower bond strength of the resin-based luting agents to the intraradicular dentin than LEDs. Although the bond strength values cannot be used as predictors of the degree of conversion of monomers into polymers, the findings of this study suggest an adequate monomer conversion after light-activation with LED, as verified by Carvalho et al. (16). This is probably due to the higher power density (12,13) of the LED unit used in this investigation, which may have favored a more effective cure of the resin-based material (13). The prevalence of adhesive failures in the QTH light-activated group is in agreement with the lower bond strength. An adequate degree of conversion implies absence or reduced residual monomers(16), which decreases the occurrence of porosities (17) and may contribute to limit the loss of adhesion observed in this study. In this sense, light-curing units of high intensity could be employed to achieve efficient polymerization of resin materials, especially in deep cavities (15).

Conversely, the push-out bond strength of the adhesive cement was not influenced by the immediate and delayed light-activation. Faria-e-Silva et al. (18) found that the Panavia F did not present any significant polymerization reaction for 5 min, suggesting low concentration of initiators of chemical reaction. This fact could also justify the lack of difference in the adhesion of the resin cement light-activated immediately or after 10 min. Delaying light-activation did not benefit the chemically activated reaction and did not influence the bond strength of cement to the root dentin. Considering that immediate exposure to the light commonly occurs in the clinical practice, it seems that there is no justification for adopting the delayed curing protocol, as no difference was found to the immediate activation.

Although the chemical reaction may not have been achieved immediately after cement manipulation and within 10 min, this might have occurred later. In this context, it is assumed that the appropriate moment to perform the light-activation varies with the activation potential of the resin-based cement (14). Materials with high potential for chemical reaction can reach appropriate degree of conversion delaying the light-activation for the chemical reaction to take place (14). For dual-cured resin cements with high photoinitiator concentration, the immediate light-activation may be preferable (14), which may be the case of the cement used in this study.

When the bond strength was compared among the post/canal regions, the findings showed that the coronal region was superior to the apical region and that the middle region did not differ from the others. Unlike Yoldas and Alaçan (19), who found that the polymerization of resin material has occurred at a 15 mm depth, the present results suggest that the polymerization of cement was compromised in the apical region, where the light has a reduced range. The differences among post/canal regions can be explained by the regional differences in the root dentin such as quantity, volume and orientation of the tubules toward the apical portion, apical sclerosis (20), the high cavity configuration factor, the difficulty in visualization and access to this region, restrictions in the flow and distribution of material in the apical region that makes more bubbles and voids within the cement (21) and presence of smear layer formed during the post space preparation, which could not be modified by the adhesive cement or removed by irrigation with sodium hypochlorite and EDTA (22). Another aspect to be considered is the limited light transmission capability of the fiber post used in the present study (22), with impaired polymerization of cement and bonding quality in the apical portion (5, 22).

Despite the methodological differences, previous studies (2,3,20,22-24) have also verified higher bond strength in the coronal root canal region. Analysis of the failure modes confirm the lower values of adhesion in the apical region, with predominance of adhesive and mixed-type failures in this region.

In the present study, LED units with 800 and 1,500 mW/cm² power densities provided higher bond strength of the resin cement to the root canal than QTH. Conversely, light activation performed immediately or after 10 min did not affect the bond strength between the resin cement and the root dentin. The coronal post/

canal region presented higher bond strength than the apical region.

RESUMO

O objetivo deste estudo foi avaliar a resistência de união do cimento resinoso à dentina intrarradicular em função da fonte fotoativadora e do momento de aplicação da luz. Os espaços para cimentação dos pinos foram preparados em caninos tratados endodonticamente. As raízes foram distribuídas em seis grupos ($n = 10$) de acordo com a fonte fotoativadora e momento de exposição à luz: I) Luz halógena (LH) com densidade de potência 600 mW/cm^2 + fotoativação imediata (t_0); II) LH + fotoativação após 10 min (t_{10}); III) LED com densidade de potência 800 mW/cm^2 (LED-800) + t_0 ; IV) LED-800 + t_{10} ; V) LED com densidade de potência 1.500 mW/cm^2 (LED-1500) + t_0 ; VI) LED-1500 + t_{10} . Após a cimentação dos pinos, slices das regiões cervical, média e apical foram submetidos ao teste de push-out e avaliação das falhas. Verificou-se que LED-800 ($4,40 \pm 3,00 \text{ MPa}$) e LED-1500 ($4,67 \pm 3,04 \text{ MPa}$) proporcionaram resistência de união estatisticamente superior à LH ($3,13 \pm 1,76 \text{ MPa}$) ($p < 0,05$), e não diferiram entre si ($p > 0,05$). Não houve diferença estatisticamente significativa da resistência de união entre t_0 e t_{10} ($p > 0,05$). A região cervical ($4,75 \pm 3,10 \text{ MPa}$) apresentou maiores valores de resistência de união que a apical ($3,32 \pm 2,30 \text{ MPa}$) ($p < 0,05$) e a região média ($4,14 \pm 2,60 \text{ MPa}$) mostrou valores intermediários. Falhas adesivas foram predominantes no grupo LH, independentemente da região pino/canal. A parte apical apresentou predomínio de falhas adesivas e mistas. Concluiu-se que a resistência de união do cimento resinoso à dentina intrarradicular foi maior no terço cervical, com fotoativação com LED, independente do momento de exposição à luz.

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