

Light curing resin cements containing iodonium salts promote suitable apical bonding of posts to radicular dentin

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Abstract: The aim of this study was to analyze the efficiency of experimental light-curing resin cements (ERCs) with a ternary photo-initiator system containing diphenyliodonium hexafluorophosphate (DPI) and different amines on retention of glass-fiber posts to dentin (GFP). ERCs formulations: a 1:1 mass ratio of 2,2-bis[4-(2-hydroxy-3-methacryloxypropoxy) phenylpropane and triethyleneglycol dimethacrylate. Camphorquinone was used as initiator. Six experimental groups were established according to the amine used: [ethyl-4-(dimethylamino)benzoate-EDMAB or 2-(dimethylamino)ethyl methacrylate-DMAEMA] and the concentration of DPI (0, 0.5 mol%, 1 mol%). The resin cements Variolink II (dual- and light-cured versions) were used as commercial reference. Eighty recently extracted bovine incisors (n = 10) were selected for this study. The roots were prepared and the fiber posts were cemented with the resin cement specified for each experimental group. Specimens from coronal, middle, and apical thirds of the root were subjected to push-out bond strength test 24 hours after bonding. Data were subjected to split-plot ANOVA and the Tukey test (p = 0.05). ERCs containing DPI showed statistically significant higher bond strengths compared with ERCs without DPI. ERCs containing DPI were statistically similar to VARIOLINK II – dual-cured and superior to VARIOLINK II – light-cured (except for EDMAB – 1DPI in the medium third and DMAEMA – 1DPI in the coronal third). Different amines did not influence post retention. The apical root region showed the lowest bond strength for the groups EDAB-0DPI, DMAEMA-0DPI and VARIOLINK II light-cured. Light-cured ERCs containing DPI were efficient for GFP retention to radicular dentin, with similar behaviour to that of dual-curing commercial resin cement.

Keywords: Resin cements; Onium salt; Light-curing

Introduction

Pre-fabricated fiber posts have been widely used in restorative procedures for better repair of restorations after extensive coronal damage. Advantages including low cost, easy installation, and esthetic properties compared with those of metallic materials make fiber posts the first choice for the restoration of fractured teeth in many clinical situations.¹ Nevertheless, their main advantage is a mechanical similarity to dentin and composites,

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which drastically reduces the risk of tooth fracture under occlusal load, and helps re-establish strength similar to that of non-prepared teeth due to better tension distribution.^{2,3,4,5}

Because of reduced light transmission in the root canal,⁶ dual-cured resin cements should be used for the luting of pre-fabricated posts. The resin cements with dual-polymerization are capable of promoting suitable bonding to tooth structure; however, they also have the disadvantage of reduced working time, which can make it difficult to place the post correctly for a subsequent restoration. Glass fiber post cementation is a critical procedure with greater technical sensitivity to the operator, the development of simplified and less sensitive materials would be interesting to preserve longevity of restorations.

Optimal bond strength after cementation can be related to adequate polymerization and a high degree of conversion of the resin cement.^{7,8,9} Nevertheless, light attenuation occurs across the length of the root canal, impairing the degree of conversion in the deepest regions.^{10,11} To overcome this situation, some alternatives have been demonstrated to improve the polymerization of dental resin materials, such as increasing the light exposure time^{12,13} or the reactivity of the photo-initiator systems,^{14,15} improving the light-cure without extending the activation time.

Previous studies tested the addition of diphenyliodonium hexafluorophosphate (DPI) to a photo-initiator system to improve the polymerization kinetics and the degree of conversion of resin materials.^{16,17,18,19,20} Regarding experimental light-curing resin cements, the presence of DPI resulted in improved mechanical properties without increased polymerization stress.¹⁵ Although this sensitizer is excited only by UV light (300 nm), in the presence of another initiator, such as camphorquinone (CQ), decomposition of this salt may occur, increasing the ways of free-radical release for the polymerization of methacrylates,¹⁶ acting as a catalyst for the reaction.

The type of tertiary amine may also influence the mechanical properties of resin cements.²⁰ Usually ethyl-4-(dimethylamino) benzoate (EDAB) or 2-(dimethylamino)ethyl methacrylate (DMAEMA) is used, combined with CQ as a reducing agent. Both have different chemical properties: EDAB has an

aromatic ring and a higher ability to donate electrons compared with DMAEMA, which presents an aliphatic structure and hydrophilic behavior.

As demonstrated in a previous study,¹⁵ DPI may lead light-curing resin cements to better polymerization. Nevertheless, it is unknown if these better results can improve the efficacy of this agent in clinical procedures when reduced irradiance reaches the resin materials (e.g. middle and apical regions of the root canal). Previous study evaluated the effect of DPI on bond strength of fiber posts.²¹ However, the study evaluated different concentration of DPI using only one amine in the initiation system, without any commercial reference to compare the efficiency of the experimental cements with the current ones. Therefore, the aim of this study was to investigate the influence of different DPI concentrations combined with two tertiary amines on the bond strength of glass-fiber posts fixed with an experimental light-curing resin cement (ERC), comparing the bonding efficacy with that of commercial light-curing and dual resin cements under different regions of the root canal. The hypotheses tested were: a three-component photo-initiator system containing an onium salt and different tertiary amines influence the regional bond strength of experimental photo-activated resin cements.

Methodology

Formulation of the experimental resin cements

A model blend based on a 1:1 ratio (wt) of 2,2-bis[4-(2-hydroxy-3-methacryloxypropoxy) phenyl]propane (Bis-GMA) and triethyleneglycol dimethacrylate (TEGDMA) (Esstech Inc., Essington, USA) was prepared with 1 mol% of camphorquinone (CQ-Esstech Inc.) as initiator. The experimental groups were established according to the amine used (2 mol% of DMAEMA or 2 mol% of EDAB, both from Sigma-Aldrich, St. Louis, USA) or the concentration of DPI (Sigma-Aldrich, St. Louis, USA) used (0, 0.5, or 1 mol%). Butylated hydroxytoluene (BHT, Sigma-Aldrich, St. Louis, USA) 0.1 mol% was added as inhibitor. Each formulation was loaded with 60% by weight of silanated barium borosilicate glass fillers (Esstech, 0.7µm average

size). The chemicals were used without further purification. The manipulation of the ERC was carried out in an environment with controlled humidity and temperature, under yellow fluorescent light.

Specimen preparation

Eighty recently extracted bovine incisors with similar root sizes and lengths were selected for this study. The crowns were removed 1 mm below the cemento-enamel junction (CEJ) by means of a water-cooled low-speed diamond saw (Isomet; Buehler, Lake Bluff, USA) to obtain 15-mm root segments. Roots with opened apices and/or a curvature greater than 10° were not included in the study. The post space was prepared with a Largo drill #4 (Dentsply Maillefer, Ballaigues, Switzerland) for the cementation of glass-fiber post #3 (1.5 mm diameter, Reforpost - Ângelus Produtos Odontológicos, Londrina, Brazil).

The roots were filled with gutta-percha (Dentsply Indústria e Comércio Ltda, Petrópolis, Brazil) using the lateral condensation technique. Each root was then stored in distilled water (1.5 mL) for 24 hours at 37°C. Thereafter, the gutta-percha was removed with Largo drill #5 (Dentsply Maillefer, Ballaigues, Switzerland), and the post space was prepared to depths of 12 mm from the cervical margin, leaving an apical seal of 3 mm. The roots were randomly divided into eight groups (n = 10) according to the experimental formulation determined by the photoinitiator system used (Table 1).

Distinct uppercase letters indicate statistically significant differences between resin cements in the same column; and distinct lowercase letters indicate statistically significant differences between the dentin root regions in the same row. Split-plot ANOVA and Tukey's test ($p < 0.05$).

A description of the composition of commercial materials used in this study is shown in Table 2.

Table 1. Bond strength means (standard deviations) of experimental groups according to the different root canal regions.

Resin cements	Dentin root region		
	Coronal	Middle	Apical
EDAB-0mol% DPI	9.4 (2.9) ^{Da}	6.2 (2.3) ^{CDab}	4.7 (2.7) ^{BCb}
EDAB-0.5mol% DPI	23.3 (7.3) ^{Aa}	16.0 (4.8) ^{Ab}	6.6 (3.0) ^{ABCc}
EDAB-1mol% DPI	20.6 (3.3) ^{ABa}	10.0 (1.8) ^{BCb}	9.54 (2.7) ^{ABb}
DMAEMA-0mol% DPI	13.1 (2.6) ^{CDa}	3.7 (1.2) ^{Db}	3.9 (2.1) ^{Cb}
DMAEMA-0.5mol% DPI	20.4 (6.5) ^{ABa}	14.4 (5.8) ^{ABb}	9.9 (4.1) ^{ABc}
DMAEMA-1mol% DPI	17.8 (5.5) ^{BCa}	12.6 (3.5) ^{ABb}	9.9 (3.9) ^{Ab}
VARIOLINK II Ligth-cured	17.1 (6.0) ^{BCa}	9.8 (4.1) ^{BCb}	3.6 (2.1) ^{Cc}
VARIOLINK II DUAL	20.4 (8.0) ^{ABa}	17.5 (3.8) ^{Aa}	7.7 (3.7) ^{ABCb}

Post cementation

The adhesive protocol used for all groups was as follows: After acid-etching with 35% phosphoric acid for 15 s (Scotchbond Etchant Gel, 3M ESPE, St. Paul, MN, USA), the root canal was washed with distilled water for 15 s, and the excess of water was removed with absorbent paperpoints (Dentsply Maillefer, Ballaigues, Suisse). The adhesive system (Adper Scotchbond Multipurpose, 3M ESPE, St. Paul, USA) was then applied following the manufacturer's instructions: primer agent, followed by a 5-second gentle air-spray, and then the hydrophobic layer of

Table 2. Commercial materials used in this study.

Material	Manufacture	Chemical composition
Scotchbond Etchant gel	3M ESPE - St. Paul, MN, USA	Water, phosphoric acid, poly(vinyl alcohol)
Adper scotchbond multipurpose	3M ESPE - St. Paul, MN, USA	Primer – water, 2-hydroxyethyl metacrylate (HEMA), copolymer of acrylic and itaconic acids
		Adhesive – bisphenol A diglycidyl ether dimethacrylate (BISGMA)
Variolink II	Ivoclar Vivadent AG – Schaan, Liechtenstein	The monomer matrix is composed of Bis-GMA, urethane dimethacrylate (UDMA), and triethylene glycol dimethacrylate (TEGDMA). The inorganic fillers are barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, and spheroid mixed oxide. Additional contents: catalysts, stabilizers, and pigments. The particle size is 0.04–3.0 µm. The mean particle size is 0.7 µm
Ceramic primer	3M ESPE - St. Paul, MN, USA	Ehtyl acohol, water, methacryloxymetpropyltrimetoxisilane

adhesive agent. The adhesive was light-cured for 20 s with a light-emitting diode (LED-Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein) with irradiance 1200 mW/cm².

The experimental resin cement and the commercial reference were inserted into the root canal by means of a syringe (Centrix, Inc., 770 River Road, Shelton, CT, USA) to fill the entire root canal. The commercial resin cement (Variolink II, Ivoclar Vivadent, Schaan, Liechtenstein) was used as a commercial reference in light-cured (base paste) and dual-cured (base paste and catalyst paste) modes, prepared following the manufacturer's instructions.

The fiber post was pre-treated with 35% phosphoric acid (Scotchbond Etchant Gel, 3M ESPE, St. Paul, USA), washed, and dried, and then a silane layer was applied (Ceramic Primer, 3M ESPE, St. Paul, USA). After this, the post was inserted into the root canal filled with the resin cement established for each group, and excess luting material was removed. The cements (experimental and commercial reference) were light-cured continuously for 60 s from the top of the glass fiber post, directed perpendicular to the root, by means of the previously mentioned LED source. After cementation, each root was kept in the dark, maintained in relative humidity at 37°C.

Twenty-four hours after the cementation, the roots were sectioned perpendicular to their long axis in three slices (1 mm thick), using a low-speed diamond saw (Isomet 1000, Buehler) with constant water coolant. The first slice was discarded to obtain standardized and smooth samples. The samples were divided in accordance with the region (1 mm - coronal, 5 mm - middle, and 9 mm - apical).

Push-out bond strength test

For the push-out test, the slices were fixed in a base with a central hole (3 mm) in a load-testing machine (EMIC DL 500, São José dos Pinhais, Brazil) and stressed to failure at a crosshead speed of 0.5 mm/min. The data obtained in kgf were converted to MPa, as described in a previous study.²²

Failure analysis

The failure mode was evaluated by stereomicroscopy (×50, Meiji 2000, Meiji Techno, Saitama, Japan) at ×50

magnification. Fracture mode categories were classified into three groups: AP – adhesive between fiber post and luting agent; AD – adhesive between dentin and resin cement; and M – mixed failure.

Statistical analysis

Data obtained from push-out bond strength tests (MPa) were subjected to split-plot ANOVA two-way (resin cement and root region) after analysis of the normal distribution of data and homoscedasticity of variances. Multiple comparisons were performed by the Tukey post-hoc test. The level of significance was set at 5% (SAS Institute Inc., Cary, USA).

Results

Push-out bond-strength test

The interaction between the factors “resin cement” and “root region” was significant ($p < 0.001$). The push-out bond strength decreased with the increase of the root canal depth (Table 1), but in some instances the apical region was similar to medium third, as observed in the groups EDAB – 1mol% DPI, DMAEMA 0 and 1 mol%DPI. In DPI-containing cements, the presence of salt (0.5 mol%) was capable of significantly increasing the push-out bond strength of fiber-posts compared with ERCs without DPI, excepting the apical region for EDAB-containing cements. The cements with DPI also presented results statistically similar to those of the commercial reference Variolink II – dual-cured, except for groups containing DMAEMA – 1mol% DPI in the coronal region and EDAB – 1 mol% DPI in the middle region, which were comparable with Variolink II – light-cured. In the apical region, the commercial reference Variolink II – light-cured mode presented the lowest results, similar to groups EDAB and DMAEMA without DPI.

Fracture mode analysis

It was noted that the most prevalent failure occurred in the adhesive layer between dentin and resin cement (AD) (Figure). Fractures occurred on the adhesive layer between post and cement, and mixed failures were also observed. However, the frequency of this kind of failure was significantly lower than the AD mode.

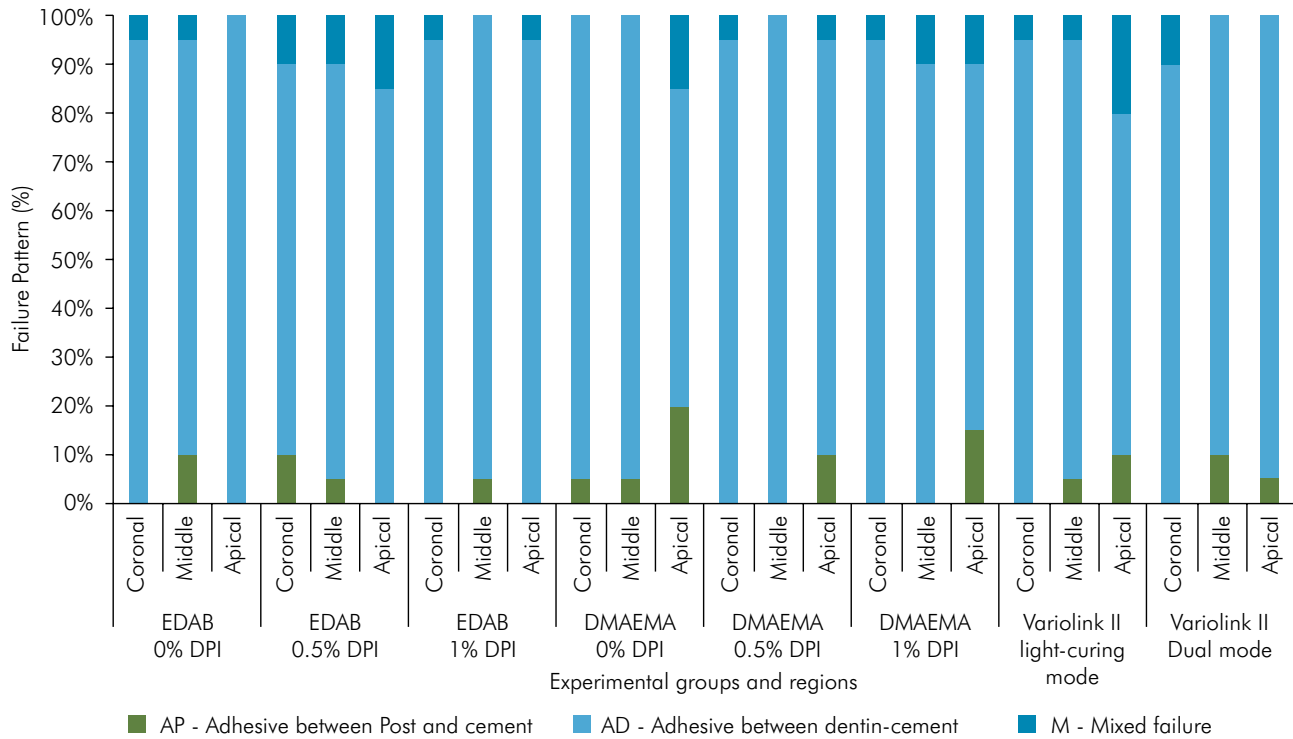


Figure. Distribution of the failure mode according to the experimental group and region evaluated.

Discussion

The first hypothesis of the present study, that a three-component initiator system containing DPI and different tertiary amines influence the regional bond strength of experimental photo-activated resin cements, was not accepted. Although the bond strength of ERCs containing 0.5mol% DPI was significantly increased compared with that of ERCs without iodonium salt, regardless of the root canal region, there was no statistical difference between EDAB - 0mol% DPI and EDAB - 1mol% DPI at the middle and apical thirds, as well between DMAEMA - 0mol% DPI and DMAEMA - 1mol% DPI at the coronal third. The potential of DPI salt to promote better polymerization and mechanical properties has been demonstrated in previous studies.^{15,16,23} Although DPI cannot be activated by blue light, it reacts with excited CQ, producing a triplet state, and an active phenyl radical is generated, which is effective in initiating the polymerization reaction. Another mechanism involving the salt is that the free radicals produced by the interaction between CQ and

the amine can also react with DPI, breaking the C-I bond and releasing another reactive phenyl radical, which can further react with residual amines, abstracting a proton and generating a new amine free radical. As a consequence of this improved polymerization, the ERCs containing DPI were able to achieve bond strength similar to that of the dual-cured commercial reference tested, and superior to that of the light-cured commercial reference. This better performance of DPI-containing cements can be explained based on the better polymerization, which promotes polymers with mechanical properties superior to those of cements without DPI, as demonstrated previously.¹⁵

However, the groups containing 1 mol% of DPI had intermediary bond strength, with both DMAEMA and EDAB. The increased concentration of DPI in these cements can probably promote a greater amount of free radicals, promoting premature termination, reducing the physical properties of the polymer, as shown in a previous study.¹⁵ We also speculate that the higher polymerization stress of the ERCs containing 1 mol%¹⁵ can influence the bond strength to dentin, which, allied

to the high C-factor of the root canal,²⁴ can be responsible for the intermediary values obtained. Therefore, based on the present results, the best concentration to obtain the highest bond strength of fiber posts to the root canal is 0.5 mol%. As demonstrated in a previous study, high concentrations of DPI (1 and 2 mol%) can reduce the mechanical properties and increase the polymerization stress of the cement. These characteristics, allied with the intermediate results of the effects of cements containing 1 mol% of DMAEMA and EDAB on bond strength, observed in the present study, highlight the ideal concentration as 0.5 mol% of DPI.

The impaired performance of glass-fiber posts subjected to push-out bond strength tests in the apical region has been well-documented,^{25,26} corroborating the findings of the present study, due to the reduced irradiation reaching the resin cement in this region. This negative effect was more evident in groups using cements without DPI and in the light-cured commercial reference. Moreover, many adverse factors in this region should be considered, such as reduced density of dentin tubules,²⁷ highest C-factor, and, consequently, increased stress of polymerization.²⁴ However, it should be highlighted that the presence of DPI improved the bond strength in the apical region, equalling, in some cases, the results in the middle region and better than the results in groups using cements without DPI and in the light-cured commercial reference.

Previous study evaluated the effect of push-out bond strength of resin cements containing DPI to root canal dentin, showing contrasting results.²⁸ Despite the higher polymerization stress presented by the cement without DPI, the previous study demonstrated better results for this cement in the cervical region.²⁸ In the present study, the experimental cements without DPI had lower bond strength compared to the cements containing the salt, probably due to the better polymerization reaction. In addition, the present study evaluated two different amines in the initiator system, comparing with light- and dual polymerization commercial references, demonstrating the efficacy and promisor results of DPI cements to bond fiber posts to dentin. Despite the molecular structural differences both amines behaved similarly in the different root regions.

Satisfactory adhesion to radicular dentin is still a challenge; therefore, the selection of the adhesive system

is an important step in glass-fiber post cementation. In this study the protocol was evaluated with a three-step light-curing adhesive system so that there was no negative interaction between acid monomers present in superficial layer of two-step etch and rinse adhesive systems with tertiary amines of the dual-cured commercial reference Variolink II. The use of dual adhesive systems was discarded since the components would possibly influence resin cement polymerization, modulating the results of the different formulations and jeopardizing the analysis of resin cement composition. In analysis of the failure mode, it can be noted that the higher frequency of the AD mode for all groups may indicate that the hybrid layer was the weakest link of the adhesion. These results probably occurred due to the difficult curing of the dental adhesive. Nevertheless, when we observed the results and distinct behavior of the cements, even with these characteristics of the bonding interface, the comparison between the different formulations was not compromised.

Dual-cured resin cement used as commercial reference attained the highest result for bond strength to root canal dentin. However, the light-curing resin cements containing DPI had statistically similar results to the dual-cure resin, clearly indicating the efficiency of ternary photo-initiator systems containing DPI for optimization of the polymerization reaction of light-cured resin cements, even in deep regions. The use of light-curing resin cements containing DPI can allow for reliable bonding with better working time compared with the dual-cure resin cements, indicating their promise as materials for the repair of posts and ceramic restorations, even in situations with reduced irradiation.

Conclusions

Retention of glass-fiber posts luted with experimental light-cured resin cement containing DPI was similar to that of those fixed with the dual-cured commercial reference.

The concentration of 0.5 mol% of DPI allowed for the best bond strength in most conditions tested.

Different tertiary amines may not cause different bond strength values in the light protocol tested.

The apical region presented the lowest bond strength values.

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