Light-curing effect on microhardness of dual-cure resin cements used for cementation of intraradicular posts

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Introduction: *Insufficient curing of dual-cure cements used for cementation of intraradicular posts may compromise the restorative retention system, which could lead to restoration failure.* ***Objective:*** *This study aimed at assessing the microhardness of dual-cure resin cements used for glass fiber posts (DT Light Post^{TM}) cementation, whether subjected or not to a light-curing source, according to the different root thirds.* ***Methods:*** *Forty bovine incisors roots were selected and divided into eight groups, according to the cement being used: G1/G5 = Panavia^{TM}; G2/G6 = Variolink II^{TM}; G3/G7 = RelyX Unicem^{TM}; G4/G8 = Duo-link^{TM}. G1 to G4 were light cured; whereas G5 to G8 were not. The roots were longitudinally sectioned and submitted to microhardness tests. Data were tested for significant differences by three-way ANOVA and Tukey's tests ($p<0.05$).* ***Results:*** *Microhardness and mean ± standard deviation values obtained for cervical, middle and apical thirds were, respectively: G1 = 56.00±8.35, 51.72±11.39, 48.28±7.45; G2 = 42.94±4.92, 37.46±6.23, 35.28±5.96; G3 = 46.56±3.89, 48.12±4.88, 51.48±5.74; G4 = 42.24±2.85, 40.60±5.76, 39.24±3.84; G5 = 39.47±5.99, 37.33±3.15, 40.45±6.41; G6 = 6.72±1.70, 36.78±3.05, 35.25±0.59; G7 = 42.96±6.78, 43.12±7.56, 42.40±4.21; G8 = 7.78±7.11, 34.88±6.30, 34.84±6.03. Light-cured specimens showed higher microhardness values; Panavia^{TM} and RelyX Unicem^{TM} cements presented significantly higher values compared to others (ANOVA/Tukey,* $p<0.05$*). There were no differences among groups (ANOVA,* $p > 0.05$*).* ***Conclusions:*** *Light-curing positively influenced the results, regardless of the root section evaluated.* ***Keywords:*** *Resin cements. Dental posts. Hardness tests.*

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Introduction

Resin cements have been widely recommended for cementation of ceramic restorations. More recently, fiber-reinforced resin posts have been recommended due to their low solubility and superior mechanical and adhesive properties.[1,2,3] However, intraradicular adhesive cementation still poses a significant challenge to clinicians due to the technical variables involved and little knowledge about the clinical predictability of this material in the long term.[4,5]

Dual-cure cements have been developed with the qualities of light and chemical polymerization, effective control of working time and adequate conversion of monomers where light cannot reach.[6,7] These cements have been indicated for situations in which the opacity of the restoration or the depth of the cavity do not allow light to reach the root towards the apex.[5,6,8] Although light and chemical polymerizations are present in these cements, they are additional and independent mechanisms. Chemical polymerization will not activate the photosensitive portion of the cement, if light exposure is insufficient.[1,2,4,6]

Some studies have shown a significant decrease in the potential polymerization of composites in the intraradicular environment due to reduction in light irradiation,[5,9-12] resulting in inadequate conversion of monomers and low values of microhardness. In this context, the use of translucent fiber-reinforced composite resin posts has been reported as an auxiliary method of polymerization of dual-cure cements in the medium and apical thirds. However, the real effectiveness of these posts remains unknown.[2,10,11,13-16]

This study evaluated the changes in the microhardness of resin cements used to lute translucent fiber-reinforced composite posts with two modified curing protocols. The null hypotheses tested were that: 1) the curing protocols do not affect microhardness; 2) the type of resin cement used for cementation post does not affect microhardness; 3) microhardness does not vary along the root thirds.

> “When microhardness tests are used as an indirect measurement of the degree of conversion of resin cements, it is important to consider the different chemical composition of the evaluated brands.”

Material and Methods

Specimens preparation

Forty bovine incisors were transversally sectioned with a low-speed saw (Buehler, Lake Bluff, IL, USA) under constant irrigation with deionized water to obtain roots 17-mm in length. Endodontic access was obtained, with working length established at 16 mm. The step-back technique was used to prepare all root canals with a #45 K-file (Maillefer-Dentsplay, Ballaigues, Switzerland). Root canals were irrigated with deionized water. After instrumentation, irrigation was performed with EDTA (Inodon, Porto Alegre, RS, Brazil) for 5 minutes, followed by rinsing with deionized water and drying with absorbent paper cones. The root canals were filled with gutta-percha (Endo Points, Paraíba do Sul, RJ, Brazil) associated with a calcium-hydroxide-based sealer (Sealer 26/Dentsply, Rio de Janeiro, RJ, Brazil), using the lateral condensation technique (Figs 1A-C). The post space was created with 1-mm diameter and 13-mm long #3 drill (D.T. Light Post^{TM}, Bisco Inc., Schaumburg, Illinois, USA), followed by a 1.5-mm diameter #4 drill (D.T. Light Post^{TM}, Bisco Inc., Schaumburg, Illinois, USA), with a resulting cement line thickness of approximately 0.25 mm around the post. The cementation procedures were performed with four different dual-cure resin cements, Panavia^{TM}; Variolink II^{TM}; Rely X-Unicem^{TM} and Duo-link^{TM} (Figs 1D-F). The post was luted according to the protocol described by the manufacturer of each cement (Table 1).

For cementation the roots were randomly divided into four groups of 10 specimens per cement (n = 10), and then further divided into two groups of five specimens according to the curing protocol: G1/G5-Panavia^{TM}; G2/G6-Variolink II^{TM}; G3/G7-Rely X-Unicem^{TM}; G4/G8-Duo-link^{TM} (Table 1). G1, G2, G3 and G4 were light-activated for 20 seconds (Optilux 501 Polymerization Unit, 750 mW/cm^2,New York, USA) with the tip of the lamp placed at the post. G5, G6, G7 and G8 groups were not light-activated.

After cementation, the specimens were stored in dark recipients containing deionized water at 37 °C for seven days (Fig 1G).

Treatment of specimens for microhardness tests

The roots were longitudinally sectioned with a diamond disk (Extec Corp., Enfield, CT, USA). The cut was made tangential to the post so that the cement line could be exposed (Fig 1H). The sectioned surfaces were ground flat with water-cooled 600 and 1200 grit SiC abrasive papers (Carbimet Paper Discs; Buehler, Lake Bluff, IL, USA), and polished with felt paper wet by diamond spray (1 µm; Buehler, Lake Bluff, IL, USA). Between each polishing step, the specimens were rinsed with deionized water for 30 seconds and subjected to ultrasound in deionized water for 2 minutes (Fig 1I-J)

Microhardness assessment

Microhardness tests were performed with a Shimadzu Microdurometer Micro Hardness Tester HMV-2.000 (Shimadzu Corporation, Japan), coupled to CAMS-WIN software (NewAge Industries, USA), with a Knoop indenter under a static load of 50 g for 10 seconds (Fig 1K). Indentations were made in the middle of the cement line, 1 mm apart from the cervical to the apical third. In each specimen, nine indentations were made, three for each third (cervical, middle and apical). The representative microhardness for each third was obtained as the average of the three indentation values.

Table 1: Resin cements used in the study.

Experimental groups	Resin cements	Composition	Adhesive system	Batch #	Manufacturer
G1 and G5	Panavia F 2.0	Paste A 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), hydrophobicaromaticdimetrhacrylate, hydrophobicaliphaticdimethacrylate, silanated silica filler, silanated colloidal silica, dl-camphorquinone, catalysts, initiators. Paste B Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic methacrylate, hydrophilic aliphatic dimethacrylate. silanated barium glass filler, catalysts, accelerators, pigments.	ED Primer A&B ED Primer A: 2-Hydroxyethyl methacrylate (HEMA), 10-Methacryloyloxydecyl dihidrogen phosphate, (MDP), water, N-Methacryloyl-5-aminosalicylic acid (5-NMSA), accelerators. ED Primer B: N-Methacryloyl-5-aminosalicylic acid (5-NMSA), water, catalysts, accelerators	51198	Kuraray Medical Inc, Japan
G2 and G6	Variolink II	Monomermatrix(Bis-GMA), urethanedimethacrylate, andtriethyleneglycoldimethacrylate and inorganic fillers are bariumglass, ytterbiumtrifluoride, Ba-Al-fluorosilicateglass, andspheroid-mixed oxide. Additional contents: catalysts, stabilizers, and pigments.	Excite DSC (smallendo) Phosphonic acid acrylate, hydroxyethyl dimethacrylate, methacrylate, highly dispersible silicon dioxide, ethanol (solvent), catalysts,stabilizers, applicator impregnated with initiators.	J27832	Ivoclar-Vivadent, Liechtenstein
G3 and G7	RelyX Unicem	Powder: glassfiber, initiator components, silica, calcium hydroxide, pigments. Liquid: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, initiator components, stabilizers.	Unnecessary	56818	3M ESPE, Germany
G4 and G8	Duolink	Base: Bis-GMA, triethylene glycol dimethacrylate urethane dimethacrylate, glass filler. Catalyst: Bis-GMA, triethylene glycol dimethacrylate, glass filler.	All Bond 2 Primer A: Acetone, ethanol, NTG-GMA salt Primer B: Acetone, ethanol, BPDM Dentin/EnamelBondingResin: Bis-GMA, hydroxyethylmethacrylate Pre-Bond Resin: Bis-GMA, triethyleneglycoldimethacrylate, benzoylperoxide. Uni-etch: phosphoricacid 37%	0600002208	BiscoInc, USA

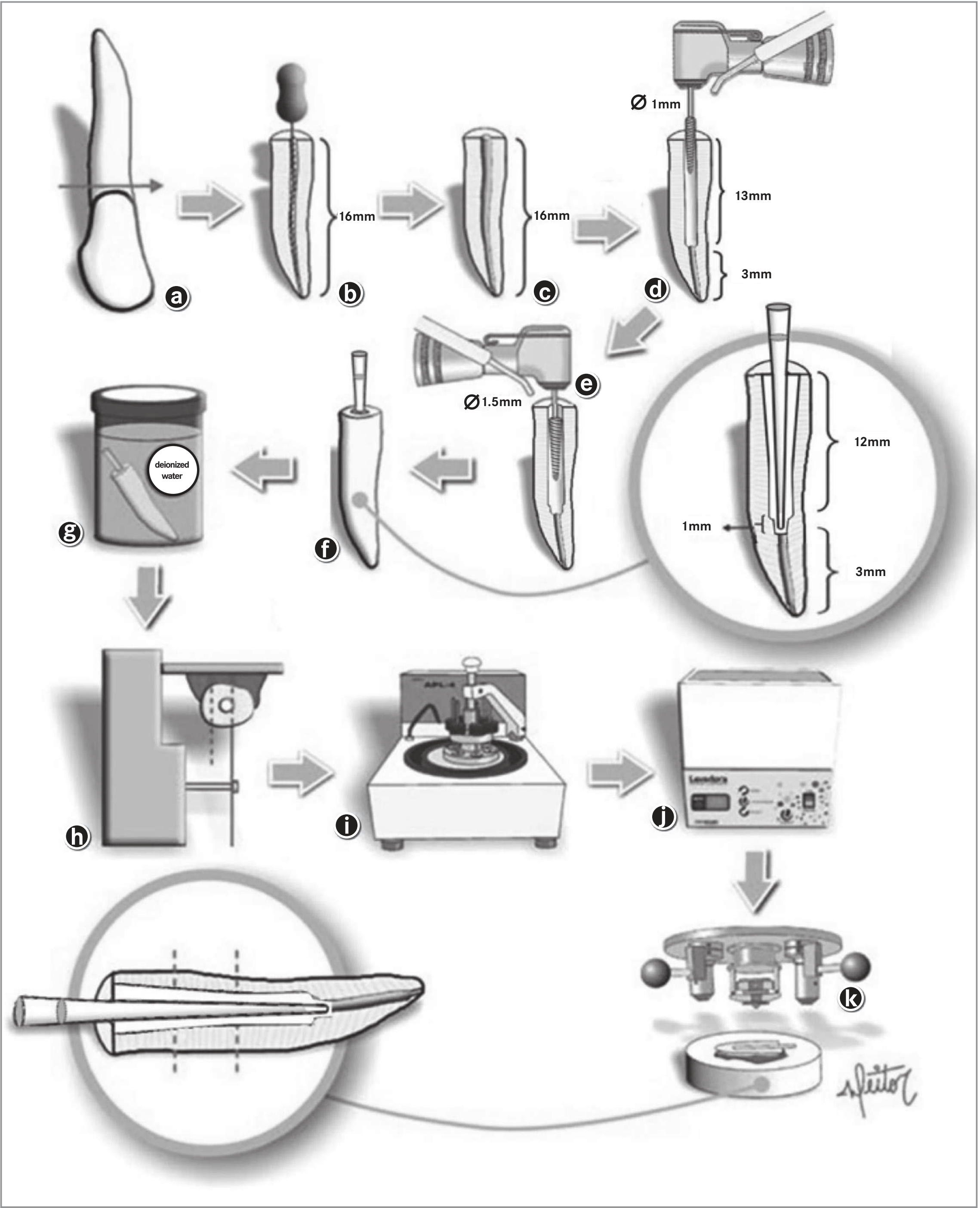

Figure 1: Schematic drawing of the experimental design: **A**) sectioning the bovine roots to the length of 17 mm; **B**, **C**) endodontic treatment; **D**) post space preparation with 1-mm diameter drill to the length of 13 mm; **E**) enlargement of post space with 1.5 mm-diameter drill to the length of 12 mm; **F**) post cemented; **G**) specimens stored in water for seven days; **H**) root sectioning to expose the cement line; **I**) surface polishing; **J**) ultrasound cleaning of specimens; **K**) microhardness indentations placed along the cement line.

Statistical analysis

Data analysis was carried out by Statistica 5.1 statistical package (StatSoft Inc., Tulsa, USA). The assumptions of equality of variances and normal distribution of errors were checked for all variables tested. As normal distribution was satisfied, three-way ANOVA (dual-cure cement, activation mode, radicular thirds) and Tukey's post hoc tests were carried out for statistical comparisons, applying a significance level of 5%.

Results

The mean Knoop hardness number (KHN) and its standard deviation along with intergroup comparison are given in Table 2. The results of ANOVA revealed that the light-activation groups showed higher KHNs values than no light-activation groups ($p < 0.05$). In comparison between resin cements, Panavia$^{\text{TM}}$ and Rely X-Unicem$^{\text{TM}}$ showed higher KHNs values than Variolink II$^{\text{TM}}$ and Duo-link$^{\text{TM}}$ ($p < 0.05$). No differences were detected for root third comparisons ($p > 0.05$).

Table 2: KHN and mean (standard deviation) values for resin cements, according to the type of dual-cure cement, activation mode and root third.

Group - Resin cement	Activation	Cervical third	Middle third	Apical third
G1 - Panavia	Light	56.00 (8.35)^{ac}	51.72 (11.39)^{ac}	48.28 (7.45)^{ac}
G2 - Variolink II	Light	42.94 (4.92)^{ad}	37.46 (6.23)^{ad}	35.28 (5.96)^{ad}
G3 - RelyX Unicem	Light	46.56 (3.89)^{ac}	48.12 (4.88)^{ac}	51.48 (5.74)^{ac}
G4 - Duolink	Light	42.24 (2.85)^{ad}	40.60 (5.76)^{ad}	39.24 (3.84)^{ad}
G5 - Panavia	Chemical	39.40 (5.99)^{be}	37.33 (3.15)^{be}	40.45 (6.41)^{be}
G6 - Variolink II	Chemical	36.72 (1.70)^{bf}	36.78 (3.05)^{bf}	35.25 (0.59)^{bf}
G7 - RelyX Unicem	Chemical	42.96 (6.78)^{be}	43.12 (7.56)^{be}	42.40 (4.21)^{be}
G8 - Duolink	Chemical	37.78 (7.11)^{bf}	34.88 (6.30)^{bf}	34.84 (6.03)^{bf}

* Values in the same column followed by distinct letters indicate statistical significance ($p < 0.05$).

Discussion

Based on the results of this study, KHNs values of dual-cure resin cements depend on the activation mode and manufacturer. Therefore, the first and third null hypotheses were rejected, whereas the second was accepted. Studies have shown a correlation between Knoop microhardness and infrared spectroscopy (gold standard) to evaluate the degree of conversion.[12,17,18] Nevertheless, infrared spectroscopy has a high cost and requires specialized knowledge for execution, limiting its application.[10,19] Predicting an absolute value of the degree of conversion by means of an absolute microhardness value is not achievable because of other factors, such as filler load,[20] monomer composition,[21,22] type and quantity of initiators, diluents concentration, and activation mode influencing the final quantity of reacted monomers.[6,23,24] Thus, microhardness values of the same resin cement should only be compared according to the depth of the root canal or the time elapsed since luting.[25] In this present study, KHN values were measured to investigate variations in the degree of conversion along the cement line surrounding the intraradicular post. The microhardness of resin cements could be assessed in each root third (cervical, middle and apical). Consequently, data indirectly indicate the real effectiveness of polymerization along the length of the root canal, not in agreement with other studies, that showed the effect.

When microhardness tests are used as an indirect measurement of the degree of conversion of resin cements, it is important to consider the different chemical composition of the evaluated brands. There is a large variation of the potential of dual-cure resin cements among products,[26] and this variation was also confirmed in this study. Panavia™ F 2.0 and Rely X-Unicem™ cements presented higher hardness than Variolink II™ and Duolink™ cements, regardless of the mode of activation (light or chemical). Four cements presented higher hardness when light-activation was provided. No difference in hardness was observed among root thirds (cervical, middle and apical) for all tested cements.

The similarity of the behavior of Panavia™ and Unicem™ cements is probably due to the effectiveness of chemical polymerization of these types of material in the apical portion of intraradicular luted posts. The Unicem™ cement, though entitled as a resin cement by the manufacturer, has the chemical combination of conventional glass ionomer cements and resin modified, to which were added silanized fillers, methacrylates and initiators.[27] Thus, this cement presents the radical polymerization initiated by light or redox system and the typical acid-base reaction of the glass ionomer cements.[28] This characteristic may have contributed to a homogeneous polymerization in the three thirds. However, the results of Variolink II™ and Duolink™ cements are not in agreement with other studies[6,9,11] showing the effect of attenuation of light intensity as a result of the distance from the light source and transmission through the cement, which progressively reduces the rate of polymerization.

The results of this study may suggest that the influence of light on the degree of conversion is material-dependent and can vary with the material composition. In fact, another explanation to be considered would be that translucent posts allow the effective action of the curing light to the apical third of root canals, minimizing problems caused by attenuation of light intensity along the root towards the apex. This experiment used the depth of 10 mm for evaluation of microhardness, according to the previous studies.[11,12] Roberts et al[10] evaluated the effect of a light-transmitting post on the depth of cure of a resin composite, and the results showed the presence of the post did increase Knoop hardness values in simulated apical regions, but only very near the post. The KHN values bottom/top cure ratio at depths below 3 mm did not achieve the 80% threshold value, suggesting inadequate polymerization. This study suggests that previous experiments,[9,11] may have overestimated the light-transmitting posts ability to increase the depth of cure.

Conclusion

Based on the results obtained, it could be concluded that:

1. Light-activation promoted a significant increase in hardness for the evaluated cements.

2. Panavia™ and Rely X-Unicem™ showed higher hardness values than Variolink II™ and Duolink™.

3. There was no difference in hardness among root thirds for all tested cements.

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