

Influence of Post-cure Time on the Microhardness of Self-Adhesive Resin Cements Inside the Root Canal

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Clinical Relevance

The microhardness of self-adhesive, dual-cure resin cements, when used to lute fiber posts, depends on the material brand, with higher values of microhardness verified at the coronal third. Because changes in microhardness were detected between 24 hours and 7 days after luting, clinicians should take this into account to prevent damage to the biomechanical bonding of the post cement-dentin immediately following cementation.

SUMMARY

Purpose: To compare the microhardness of several dual-cure, self-adhesive resin cements used to lute fiber posts at 24 hours and seven days after cementation.

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Methods: Bovine incisors were selected to lute 15 fiber posts that were 12 mm long (FRC Postec Plus size 3, Ivoclar-Vivadent). Five resin cements were tested: Multilink Automix (Ivoclar-Vivadent), without light-curing, and the self-adhesive resin cements Maxcem Elite (Kerr), RelyX Unicem (3M ESPE), G-Cem (GC), and Smartcem 2 (Dentsply), which were light-cured for 40 seconds (LED Bluephase, Ivoclar-Vivadent). Each root was embedded in chemically cured acrylic resin and stored at 37°C for 24 hours. The roots were transversally sectioned into nine specimens that were each 1 mm thick, with three specimens corresponding to each root third. Indentations (100g, 30 seconds) were performed on each section in the resin cement, at 24 hours and seven days after cementation, using a Vickers digital microdurometer (Buehler). Data were analyzed by two-way analysis of variance, Stu-

dent-Newman-Keuls test, and paired *t*-test ($p < 0.05$).

Results: A significant influence was found ($p < 0.05$) for the resin cement evaluated, the root third, and their interactions on microhardness values at 24 hours and seven days after post cementation. RelyX Unicem and G-Cem exhibited the highest microhardness values, whereas Multilink Automix presented the lowest. All resin cements suffered a decrease in microhardness according to root canal depth, with the exception of G-Cem and Multilink Automix at 24 hours and Smartcem 2 after seven days. After seven days, the evaluated resin cements showed a significant increase in microhardness values, with the exception of Maxcem Elite and Smartcem 2 at the coronal third.

Conclusions: Microhardness of the self-adhesive resin cements when used to lute fiber posts was material-dependent and higher values were obtained in the coronal third, revealing their sensitivity to light irradiation. More information regarding the polymerization reaction of these cements is warranted. According to the current results, microhardness values were significantly higher one week after post luting.

INTRODUCTION

The restoration of endodontically treated teeth with large coronal loss generally requires the use of intraradicular posts to hold a core and eventual coronal restoration.

Translucent fiber-reinforced composite posts are often the preferred option because they exhibit mechanical properties that are more similar to dentin than do metal or ceramic posts, reducing the stress inside the root canal and preventing the risk of radicular fracture.¹ The similar moduli of elasticity among dentin and the materials that constitute the core and the post would contribute to establish a monoblock restoration, with the ability of the restorative materials to bond strongly and mutually to root dentin.² However, these monoblock restorations, which create mechanically homogeneous units with root dentin, are difficult to attempt and would be compromised if the resin cement used does not reach an adequate degree of conversion. Translucent posts are suggested as a better option over opaque posts to improve the curing of resin cements and, consequently, the bond strength³⁻⁵ and

microhardness.⁴⁻⁶ However, some widely used translucent posts have been shown to ineffectively transmit light to the apical region.⁷

To compensate for light attenuation by the post and root depth, a selected luting agent should be a dual-cure or self-cure resin material. Dual-cure resin cements possess a chemical-curing system that can achieve a more extensive polymerization in dark locations⁸ while also providing a light-curing mechanism that allows for an extended working time and a rapid initial hardening of the resin cement to stabilize the restoration.⁹ Both the light- and chemical-curing mechanisms are complementary and independent.⁶ Although chemical curing is responsible for curing at sites not reached by light exposure, the chemical component in some dual-cure resin composites has been described as slower, less effective,⁹⁻¹¹ or virtually ineffective.^{8,12,13}

Apart from their curing mode, resin cements have also been classified according to their mechanism of interaction with the smear layer. Therefore, resin cements require either the application of an etch-and-rinse adhesive system or of a self-etching primer.^{5,14} Recently, a new subgroup was introduced into the self-etching category: self-adhesive resin cements. These new materials are applied to enamel and dentin without a previous application of an adhesive system.^{9,14} The first self-adhesive cement was RelyX Unicem, which was launched into the market in 2002. Since then, new products have been constantly introduced. The keys to their clinical success are based on their ability to adequately bond to different substrates¹¹ and their reduced technique and operator sensitivity.¹⁴ Self-adhesive resin cements are characterized as being dual-curing cements. However, their behavior as a group is not fully understood due to their complex and sometimes unknown compositions.¹⁵

Microhardness testing has been described as a valid indirect method to determine the degree of cure because it presents a good correlation with the spectroscopy approach.¹⁶ Therefore, the aim of this current study was to evaluate the microhardness along the depth of the root canal post space of a dual-cure resin cement, which requires the application of an adhesive system, and four self-adhesive resin cements used to lute a fiber post at 24 hours and seven days after cementation. The research hypotheses were that the self-adhesive resin cements attain higher microhardness; their microhardness decreases through the root length; and their microhardness increases after seven days.

METHODS AND MATERIALS

Fifteen bovine teeth that had been stored at 4°C in thymol for a maximum of six months were selected to lute 15 glass fiber-reinforced posts.

Post Space Preparation

Roots were sectioned perpendicularly to the long axis of the tooth to a length of 16 mm from the apex, using a diamond bur under copious water cooling.

Root canals were manually instrumented using the step-back technique, and the master apical K-file used was an ISO 080 (Dentsply-Maillefer, Ballaigues, Switzerland), due to the anatomy of the bovine teeth. Each root canal was flushed with 2.5% sodium hypochlorite and dried with ISO-standardized paper points (Dentsply-Maillefer).

Post preparation was carried out with low-speed FRC Postec size 3 drills (Ivoclar-Vivadent, Schaan, Liechtenstein) under water cooling, creating a 12-mm-deep post space and leaving the remaining 4 mm to the apex untouched. Before cementing the posts, the external root surfaces were painted with black nail varnish to prevent external light from interfering with resin-cement curing.

FRC Postec Plus Cementation

Each post was cut to a length of 16 mm. Therefore, 12 mm of the post was inside the root canal and 4 mm out of the root canal, which was determined to be the amount needed to place the coronal core. Prior to luting, the posts were checked to fit in the post space and conditioned according to the manufacturer's instructions. The post surfaces were cleaned with 35% phosphoric acid for 60 seconds (Coltène Whaledent, OH, USA), washed, and dried. The posts were silanized with Monobond-S (Ivoclar-Vivadent) for 60 seconds and dried with compressed air.

Five resin cements were evaluated (Table 1). The self-cured resin cement Automix (Ivoclar-Vivadent) and the self-adhesive resin cements Maxcem Elite (Kerr Corp, Orange, CA, USA), RelyX Unicem (3M ESPE, St Paul, MN, USA), G-Cem (GC Corporation, Tokyo, Japan), and SmartCem 2 (Dentsply-Detrey, GmbH, Konstanz, Germany) were used to lute glass fiber-reinforced posts (FRC Postec Plus, size 3, Ivoclar-Vivadent) inside the root spaces. All resin cements were applied following the manufacturers' instructions and similar shades were selected, according to the available products (opaque or yellow shades). The self-adhesive resin cements were light-cured with the LED polymerization unit Bluephase (Ivoclar-Vivadent), set for the high curing program

(1200 mW/cm²). To standardize the curing procedure, cements were light-cured for 40 seconds by contacting the lamp with the external portion of the post. The output intensity of the LED light-curing unit was checked (>600 mW/cm²) before every five luting procedures.

Specimen Preparation

Each root was embedded in a transparent chemically cured epoxy resin (Buehler, Lake Bluff, IL, USA) and stored for 24 hours at 37°C. Then, nine specimens of 1 mm width were obtained from each root through transverse sectioning (three from the coronal third, three from the middle third, and three from the apical third) using an Isomet 5000 with a diamond blade (Buehler).

Each specimen was sequentially polished with Beta Polisher (Buehler) using 320-, 800-, 1200-, and 4000-grit polishing disks at 300 rpm for 30 seconds. Afterward, specimens were stored at 37°C and 100% humidity in a light-free container.

Microhardness

Measurements were carried out at 24 hours and seven days after post cementation, applying a 100g load for 30 seconds with a Vickers digital microhardness tester (Buehler 2101). Indentations were performed on the resin cement avoiding artifacts due to luting procedures, such as voids. A minimum distance corresponding to the length of two indentations was maintained between indentations and between the indentations and the post or dentin.

Statistical Analysis

Mean and standard deviation values were determined for each experimental group. The influence of the independent variables, resin cement and root third, on the dependent variable, microhardness at 24 hours and seven days after luting, was evaluated by two-way analysis of variance. A *post hoc* test was performed using the Student-Newman-Keuls test.

Microhardness values of resin cements obtained at 24 hours were compared with those measured at seven days using a paired *t*-test. All statistical testing was performed at a preset α of 0.05 by means of IBM SPSS statistics software, version 19.0 (IBM, New York, USA).

RESULTS

Microhardness values (VHNs) obtained for the self-cured resin cement and the self-adhesive resin cements have been divided into three sections:

Table 1: Composition of the Resin Cements

Resin Cement	Manufacturer	Composition	
		Resin Matrix	Filler
MultilinkAutomix Shade: Yellow	Ivoclar-Vivadent, Schaan, Liechtenstein	DM, HEMA	Ba-glass, SiO ₂ , YF ₃ 40 vol.%, 68.5 wt.%
Maxcem Elite Shade: Yellow	Kerr Corp. Orange, CA, USA	GPDM, comonomers, mono-, di-, tri-functional methacrylate monomers	F-Al-Si-glass, Ba-glass, SiO ₂ 46 vol.%, 67 wt.%
RelyX Unicem Shade: AO3	3M ESPE St. Paul, MN, USA	PAE, TEGMA, BisGMA	SiO ₂ , glass 54 vol.%, 72 wt.%
G- Cem Capsule Shade: AO3	GC Corporation Tokyo, Japan	UDMA, PAE, 4-META, DM	F-Al-Si-glass, SiO ₂ 56.6 vol.%, 71 wt.%
SmartCem 2 Shade: Opaque	Dentply-Detrey GmbH Konstanz, Germany	UDMA, Urethane Modified BisGMA, DM, DPP	Ba-B-F-Al-Si-glass, SiO ₂ 46 vol.%, 69 wt.%

DM: dimethacrylate, HEMA: hydroxyethyl methacrylate, GPDM: glyceroldimethacrylate dihydrogen, PAE: phosphoric acid ester monomer, TEGDMA: triethyleneglycol dimethacrylate, Bis-GMA: bisphenol A dimethacrylate, UDMA: urethane dimethacrylate, 4-META: 4-methacryloyloxyethyl trimellitate anhydride, DPP: dipentaerythritol pentaacrylate phosphate.

microhardness evaluated at 24 hours, microhardness evaluated at seven days, and microhardness variation according to time elapsed after post luting.

Microhardness Evaluated at 24 hours

Figure 1 shows mean (SD) values obtained for each resin cement evaluated at the coronal, middle, and apical root thirds.

According to the statistical analysis, a significant influence on microhardness of the resin cement ($p < 0.001$), root third ($p < 0.001$), and their interaction ($p < 0.01$) was detected.

At the coronal third, RelyX Unicem showed the highest microhardness values, followed by G-Cem. Multilink Automix, a self-cured resin cement, exhibited the lowest mean values. Intermediate and statistically similar mean values were obtained by Maxcem Elite and SmartCem 2.

At the middle and apical thirds, RelyX Unicem and G-Cem showed the highest microhardness values, with no statistical differences. SmartCem 2 exhibited intermediate mean values that were significantly higher than those obtained for Maxcem Elite and Multilink Automix.

Regarding the variation of microhardness values of the resin cements evaluated according to the root third, G-Cem and Multilink Automix were the only

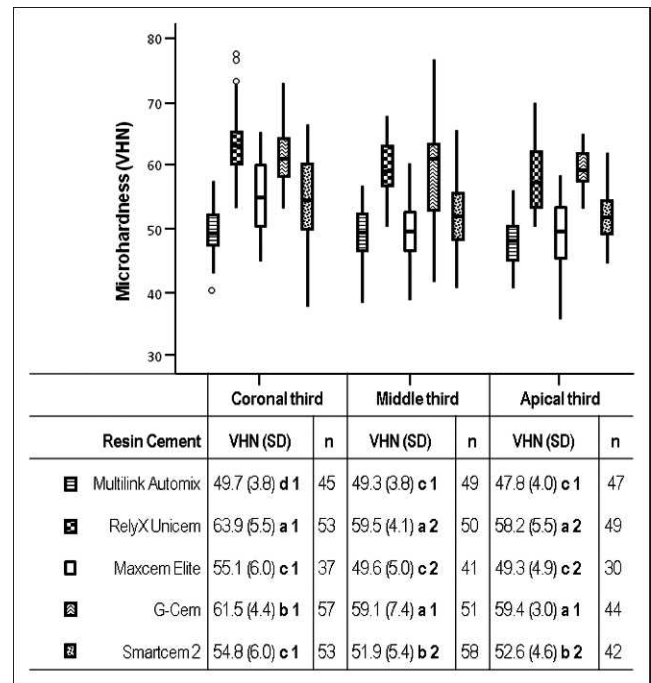


Figure 1. VHNs means and standard deviations (sd) of resin cements evaluated at 24 hours. Same letters in the same column mean no statistically significant differences among cements at each root third ($p < 0.05$). Same numbers in the same row mean no statistically significant differences among root thirds for each cement ($p < 0.05$).

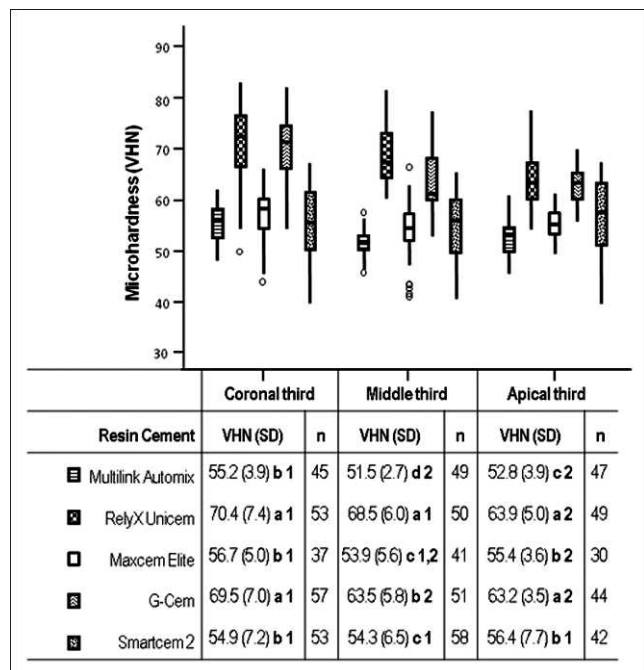


Figure 2. VHNs means and standard deviations (sd) of resin cements evaluated at 7 days. Same letters in the same column mean no statistically significant differences among cements at each root third ($p < 0.05$). Same numbers in the same row mean no statistically significant differences among root thirds for each cement ($p < 0.05$).

ones that showed a homogeneous behavior among the root thirds, whereas the other cements suffered a significant decrease in microhardness from the coronal to the middle thirds.

Microhardness Evaluated at Seven Days

Figure 2 shows mean (SD) values obtained for each resin cement evaluated at coronal, middle, and apical root thirds.

According to the results, a significant influence on microhardness of the resin cement ($p < 0.001$), root third ($p < 0.001$), and their interaction ($p < 0.001$) was detected.

At the coronal third, RelyX Unicem and G-Cem exhibited similar microhardness values, which were statistically higher than those determined for Multi-link Automix, Maxcem Elite, and SmartCem 2, which were statistically similar.

At the middle third, RelyX Unicem exhibited the highest microhardness values, followed by G-Cem. Maxcem Elite and SmartCem 2 attained intermediate values with no statistical differences. The lowest microhardness values were determined for Multilink Automix.

At the apical third, RelyX Unicem and G-Cem obtained the highest microhardness values with no statistical differences. Maxcem Elite and SmartCem 2 obtained similar and intermediate microhardness values. The lowest values were attained with Multilink Automix.

The evaluation of microhardness along root thirds was also evaluated, showing that SmartCem 2 was the only resin cement that had microhardness values that were homogeneous through the root space. The other cements showed a decrease of microhardness when comparing the mean values at the coronal and middle thirds.

Microhardness Variation According to Time Elapsed From Post Cementation

Table 2 shows mean (SD) values obtained for each resin cement evaluated at the coronal, middle, and apical root thirds.

Table 2: VHN Means and Standard Deviations (SD) of Resin Cements Evaluated at 24 Hours and Seven Days, Together With the Comparison Between Both Times

Resin Cement	Coronal Third			Middle Third			Apical Third		
	24 h	7 d	24 h vs 7 d	24 h	7 d	24 h vs 7 d	24 h	7 d	24 h vs 7 d
Multilink Automix	49.7 (3.8)	55.2 (3.9)	$p < 0.001$	49.3 (3.8)	51.5 (2.7)	$p < 0.01$	47.8 (4.0)	52.8 (3.9)	$p < 0.001$
RelyX Unicem	63.9 (5.5)	70.4 (7.4)	$p < 0.001$	59.5 (4.1)	68.5 (6.0)	$p < 0.001$	58.2 (5.5)	63.9 (5.1)	$p < 0.001$
Maxcem Elite	55.1 (6.0)	56.7 (5.0)	$p = 0.279$	49.6 (5.0)	53.9 (5.6)	$p < 0.001$	49.3 (4.9)	55.4 (3.6)	$p < 0.001$
G-Cem	61.5 (4.4)	69.5 (7.0)	$p < 0.001$	59.1 (7.4)	63.5 (5.8)	$p < 0.01$	59.4 (3.0)	63.2 (3.5)	$p < 0.001$
Smartcem 2	54.8 (6.0)	54.9 (7.2)	$p = 0.922$	51.9 (5.4)	54.3 (6.5)	$p < 0.05$	52.6 (4.6)	56.4 (7.7)	$p < 0.01$

All resin cements tested showed a significant increase in microhardness values after seven days, with the exception of Maxcem Elite and SmartCem 2 at the coronal third.

DISCUSSION

The results of the present study indicate that microhardness values depend on resin luting material brand, root third, and time elapsed from post cementation. Thus, the research hypotheses formulated must be accepted.

Self-adhesive resin cements possess a complex chemical composition. They contain conventional mono-, di-, and/or multi-methacrylate monomers, carboxylic or phosphoric acid-functionalized monomers, fillers, and photo-initiators. This unique composition allows them to combine a curing mechanism based on a free-radical redox polymerization and an acid-base reaction.¹⁷ The acid-base reaction occurs between the acidic functionality on the monomers and the acid-soluble glass or the mineralized tooth surface. As this reaction proceeds, ionic cross-links that form between acid groups and calcium or aluminium ions cause the pH to rise.¹⁷ This pH neutralization is a matter of relevance because redox initiators and photoinitiators have been described to be sensitive to acidic monomers.¹⁸

In the present study, two of the self-adhesive resin cements tested, RelyX Unicem and G-Cem, attained the highest microhardness values. Self-adhesive resin cements constitute an attractive alternative for post cementation because no dentin pretreatment is required. This lack of pretreatment simplifies the procedure and reduces technique and operator sensitivity.⁵ Although there are a dozen self-adhesive resin cements available, the majority of the studies regarding these materials concern RelyX Unicem. This self-adhesive cement has been reported to produce an effective adhesion with dentin, despite its very superficial interaction with this tissue, exhibiting similar¹⁹ or even higher bond-strength values to root dentin than conventional resin cements²⁰ and a better sealing ability.²¹ The setting of RelyX Unicem is characterized by a very rapid rise in pH, probably related to the presence of calcium hydroxide in its composition, achieving neutrality only 15 minutes after mixing when used in the dual-curing mode.²² This allows the cement to change from a hydrophilic paste into a hydrophobic mixture that exhibits better properties, such as limited post-cure swelling and material deterioration.¹⁷ Additionally, filler weight and volume percentage significantly influence the mechanical

properties of resin cements.²³ Accordingly, RelyX Unicem and G-Cem contain a higher filler content in comparison with the other resin cements (Table 1).

In contrast, Multilink Automix, a self-cured resin cement, consistently presents low microhardness values, which can be related to its reduced filler content. It is crucial to note that Multilink Automix is described by the manufacturer as a self-curing luting material with a light-curing option. However, several studies indicate that Multilink Automix behaves better when applied using the dual-curing mode instead of the self-curing mode. Vrochari and others,²⁴ in 2009, evaluated the degree of cure for Multilink Automix in the self-curing and dual-curing mode using micro-attenuated total reflectance (ATR) Fourier transform infrared spectroscopy (FTIR), obtaining a very low degree of conversion (14.47%) in the self-curing mode and almost a six times higher degree of conversion (61.36%) in the dual-curing mode. Accordingly, low Vickers microhardness values (5.79 VHN) were determined for Multilink Automix when this material was left to self-cure when compared with those obtained after 40 or 80 seconds of light irradiation through a 4-mm indirect composite restoration (16.75 VHN or 19.37 VHN, respectively).²⁵ Furthermore, Multilink Automix requires a self-etching adhesive containing acidic resin monomers. These acidic monomers may impair polymerization because they interact with the benzoyl peroxide of the dual-cure cement.²⁶ Therefore, given that the effectiveness of the Multilink Automix self-curable component does not appear to be very high, a dual-curing mode seems to be the preferred option, despite the manufacturer's description.

Regarding Maxcem Elite and SmartCem 2, both materials attained intermediate to low microhardness values, depending on the root third evaluated and time elapsed since cementation. Both self-adhesive resin cements were statistically softer than RelyX Unicem and G-Cem, according to a previous report.²⁵ The lower filler amount in Maxcem Elite and SmartCem 2, in comparison with RelyX Unicem and G-Cem, may explain their lower mechanical properties (Table 1). There is not much information regarding Maxcem Elite; however, the previous version, Maxcem, has been described in several studies to achieve deficient mechanical and physical properties when compared with other self-adhesive cements, like RelyX Unicem or G-Cem,^{22,27,28} including a lower degree of cure.²⁴ SmartCem 2 has also shown poor mechanical properties, such as unfavorable bond strength.²⁷ It has been shown that

Maxcem and SmartCem 2 do not have an important acid-base reaction while setting, as do other self-adhesive cements like RelyX Unicem,²⁸ maintaining a low pH for a long time^{22,28} that could adversely influence the adhesion to dentin and the formation of an optimal cross-linked polymer network.²⁹

Although microhardness is an indirect method widely used and accepted to determine the degree of cure,^{8,29} the data obtained cannot be linearly correlated if compared across different materials.³⁰ Other factors, such as the nature of the matrix, type of filler, filler load, the quantity of initiators, the amount of inhibitors, and the ratio between auto- and light-polymerizing components, strongly influence the final amount of reacted monomers.^{8,18,30} Thus, only microhardness data from the same resin cement should be compared according to the root third or time elapsed from luting.

Regarding the variation of microhardness values with root canal depth, all of the cements evaluated achieved the highest values in the coronal third and decreased through the root canal space, with the exception of G-Cem and Multilink Automix at 24 hours and Smartcem 2 after seven days. Various studies agree that light attenuation along the root canal negatively affects the polymerization of resin cements^{10,31,32} and that the capacity of the translucent posts to transmit light is insufficient for clinical luminous activation of resin cement at the apical or middle thirds.³³ Therefore, the proximity of the irradiation source is a determinant in the extent of polymerization, despite the dual-cure nature of a material. G-Cem showed a homogeneous microhardness at different root thirds. It is possible that light attenuation is compensated for in this material by its chemical-curing component, indicating that G-Cem might present a higher amount of chemical-curing initiator in its composition when compared with the rest of the evaluated cements. High microhardness values have been reported for G-Cem even without light irradiation when applied under a 4-mm-thick indirect composite restoration.²⁵ Moreover, Multilink Automix was expected to behave homogeneously along the different root thirds because no light irradiation was applied.

In the present study, all resin cements evaluated had a post-curing effect seven days after luting, showing statistically higher microhardness values, with the exceptions of SmartCem 2 and Maxcem Elite at the coronal third. These results are in disagreement with the study by Yan and others,³⁴ who did not find changes in microhardness values after 24 hours postirradiation or postmix. However, the polymeriza-

tion reaction of dual-cured materials might be material specific,³⁵ and the resin cements tested in that study were not the same as those in the present study. In fact, no self-adhesive resin cement was evaluated in the former study and the luting of fiber posts inside the root canal was not simulated. Therefore, the curing mechanism of the resin cements was based only on a free-radical redox polymerization and no acid-base reaction was expected.

According to the current results, the degree of conversion was lower where the light did not reach, due to the dual-curing behavior. It has been reported that dual-cure resin cements are characterized by slow polymerization¹⁰ and, specifically for RelyX Unicem, increasing microhardness values have been reported even three months post luting.⁶ Only the microhardness values of SmartCem 2 and Maxcem Elite at the coronal third remained similar after one week. As explained above, the information regarding the curing mechanism of these self-adhesive resin cements is scarce, but it may be that their final curing mainly depends on the level of initial conversion obtained from light exposure at the coronal third.

This current study confirms that the microhardness of the self-adhesive resin cements, when used to lute fiber posts, is brand dependent. Moreover, this degree of conversion is affected by the proximity to the light irradiation source because the resin cements were significantly softer in the middle and apical thirds, regardless of their dual-cure nature. Nevertheless, the polymerization reactions seem to continue longer than 24 hours postmix, attaining a significantly higher microhardness one week later.

Although microhardness testing is a sensitive method to detect small changes during resin-cement setting, it is only one mechanical property and no value has been established to ensure clinical success. Further research is warranted to explain how the polymerization reaction of these self-adhesive cements occurs as a function of time, with other properties being considered, in order to estimate their clinical performance.

CONCLUSIONS

The studied resin cements showed statistically different microhardness values, with RelyX Unicem and G-Cem presenting the highest values. All resin cements suffered a decrease in microhardness from the coronal to middle thirds, except for G-Cem and Multilink Automix at 24 hours and Smartcem 2 at seven days. Additionally, microhardness values were

significantly higher seven days after post luting, with the exception of Maxcem Elite and Smartcem 2 at the coronal third, revealing a long postirradiation curing reaction.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

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