

In-Depth Polymerization of a Self-Adhesive Dual-Cured Resin Cement

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Clinical Relevance

Polymerization of a dual-cured resin cement is significantly affected by ceramic thickness but not affected by activation modes. Increased irradiation times could potentially lead to higher hardness values in applications where light is not completely blocked by the overlying restoration.

SUMMARY

The aim of this study was to assess Knoop hardness at different depths of a dual-cured self-adhesive resin cement through different

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thicknesses of Empress Esthetic® ceramic. Flattened bovine dentin was embedded in resin. The cement was inserted into a rubber mold (0.8×5 mm) that was placed between two polyvinyl chloride plastic films and placed over the flat dentin and light cured by Elipar Trilight-QTH (800 mW/cm²) or Ultra-Lume light-emitting diode (LED 5; 1585 mW/cm²) over ceramic disks 1.4 or 2 mm thick. The specimens (n=6) were stored for 24 hours before Knoop hardness (KHN) was measured. The data were submitted to analysis of variance in a factorial split-plot design and Tukey's test ($\alpha=0.05$). There was significant interaction among the study factors. In the groups cured by the QTH unit, an increase in ceramic thickness resulted in reduced cement hardness values at all depths, with the highest values always being found in the center (1.4 mm, 58.1; 2 mm, 50.1) and the lowest values at the bottom (1.4 mm, 23.8; 2 mm, 20.2). When using the LED unit, the hardness values diminished with increased ceramic thickness only on the top (1.4 mm, 51.5; 2 mm, 42.3). In the group with the 1.4-mm-thick disk, the LED curing unit resulted in

similar values on the top (51.5) and center (51.9) and lower values on the bottom (24.2). However, when the cement was light cured through the 2-mm disk, the highest hardness value was obtained in the center (51.8), followed by the top (42.3) and bottom (19.9), results similar to those obtained with the QTH curing unit (center > top > bottom). The hardness values of the studied cement at different depths were dependent on the ceramic thickness but not on the light curing units used.

INTRODUCTION

Resin cements have been widely used for cementation of indirect restorations,¹ as they have improved mechanical, physical, and adhesive properties when compared with conventional luting agents.² Furthermore, when used for cementing ceramic systems, resin cements increase the fracture strength and stability and show excellent esthetic results.^{3,4} However, when this material is inadequately polymerized, clinical problems may arise, such as inadequate biocompatibility,⁵ microleakage and recurrent caries,⁶ susceptibility to degradation, marginal ditching,⁷ discoloration, and reduction of mechanical properties.⁸ Therefore, the clinical success, longevity, and biocompatibility of indirect restorations are largely dependent on an appropriate degree of conversion of the resin cement.

Nevertheless, in several clinical situations, the resin composite used for cementation is only partially polymerized, or adequate polymerization is impossible to achieve with visible light. Metal or ceramic restorations or even intraradicular abutments attenuate the transmission of visible light to begin the polymerization reaction of resin cement.⁹ They can also interfere in the light spectrum transmitted.¹⁰ In these cases, an appropriate degree of conversion with greater polymerization depth and hardness will be obtained by increasing the time of light exposure¹¹ or by using dual-cured resin cements with a chemical reaction that would theoretically ensure maximum conversion of monomers. However, any dual-cured resin material used for cementation is dependent on the action of visible light with adequate irradiation to obtain an optimum degree of conversion.¹²⁻¹⁴

A resin cement with dual-cured self-adhesive components, RelyXTM Unicem (3M ESPE, Seefeld, Germany), was introduced in 2002. According to the manufacturer, the cement contains new dimethacrylate monomers and innovative technology for initi-

ating polymerization in an acid medium by exposure to visible light or the mechanism of oxyreduction.¹⁵ However, when this self-adhesive cement was polymerized by the chemical system of oxyreduction alone, a reduced degree of conversion of between 30% and 54% was shown when compared with polymerization performed by visible light and measured in the first minutes after polymerization begins.^{14,16} Substantial evidence of chemically induced polymerization after light curing has been confirmed after 24 hours and more intensely after seven days.¹⁷ Other studies conducted with different dual-cured resin cements have shown the need to wait 24 hours before fixed indirect restorations are submitted to masticatory forces when using these types of materials when they have been activated mainly by the chemical system.¹⁸⁻²⁰

Until recently, most studies conducted with this self-adhesive cement have analyzed its adhesive properties on different substrates (enamel, dentin, ceramic, and intraradicular abutments, among others).²¹⁻²⁴ Some of the chemical and physical properties were also analyzed,^{25,26} and the degree of conversion was assessed by Fourier transform spectrophotometry.^{14,16,17} However, those studies did not examine the influence of different light curing units and the interference of ceramic materials in the polymerization efficiency of RelyXTM Unicem.

Therefore, the aim of this study was to investigate the influence of ceramic thickness and light curing units on the dual-cured resin cement RelyXTM U100 polymerization at different depths by means of the Knoop hardness test. It should be remembered that, according to the manufacturer, the only difference between the self-adhesive cements RelyXTM Unicem and RelyXTM U100 is the distribution system. While Unicem requires an activator, triturator, and applicator, U100 can be manipulated manually because of its clicker system.

MATERIALS AND METHODS

Fabrication of Ceramic Disks

Empress Esthetic® covering ceramic (Ivoclar Vivadent, Schaan, Liechtenstein) was used to make two disks measuring 8 mm in diameter and 1.4 mm thick or 2 mm thick in shade ETC2. Metal matrixes were used to make the disks. The ceramic powder was mixed with the modeling liquid on a glass plate using a flexible ceramic spatula (KOTA) until the mixture was homogeneous and a pasty, shiny consistency was obtained. The ceramic was inserted into the

matrix with the use of a no. 3 hair paint brush (Ivoclar Vivadent) wet with deionized water. The ceramic condensation process was carried out under vibration, the excess material was removed, and sintering was performed in an EP600 furnace (Ivoclar Vivadent) in accordance with the temperatures recommended by the manufacturer. The ceramic disks were stored in a dry place at room temperature.

Substrate Preparation

To simulate the condition of cementing an indirect restoration using dentin reflectance, a bovine tooth was used. The root was removed using a diamond disk mounted on a bench lathe (Nevone, São Paulo, Brazil), making it possible to remove the coronal pulp tissue with a no. 5 probe (Duflex, São Paulo, Brazil). The incisal and proximal surfaces of the crown remnants were ground using a universal polishing machine APL-4 (Arotec, Cotia, Brazil) with 120- and 200-SiC abrasive paper (Carborundum Saint-Gobain, Recife, Pernambuco, Brazil), in sequence, under water cooling, in order to fit the specimens in the three-quarter-inch polyvinyl chloride (PVC) matrixes. The bovine incisors were embedded in polypropylene resin with the buccal surface facing out, which was ground flat using 200-, 400-, and 600-SiC abrasive papers under water cooling.

Fabrication of Samples

The samples were fabricated from self-adhesive dual-cured resin cement RelyX™ U100 (3M ESPE), which was light cured with either a halogen light appliance (QTH, Elipar Trilight, 3M ESPE) that emitted a light intensity at 800 mW/cm² and using a light curing time of 40 seconds or a blue light-emitting diode (LED) fitted with additional ultraviolet lamps (5 Ultralume, Ultradent Products, South Jordan, UT, USA) emitting light intensity at 1585 mW/cm² and using a light curing time of 20 seconds. It should be noted that both light curing units produced nearly equivalent light energy (LED = 31.7 J/cm² and QTH = 32 J/cm²).

On the dentin surface, a PVC packaging film (PVC Film, Goodyear do Brasil Produtos de Borracha Ltda, São Paulo, Brazil) was seated, and over this a rubber mold (0.8-mm height and 5-mm diameter) was bulk filled with the cement prepared following the manufacturer's recommendations. Another plastic film was seated over this set, and a ceramic disk of one of the predetermined thicknesses (1.4 or 2 mm) was digitally compressed to promote extrusion

and removal of excess material. The PVC films were used to prevent the cement samples from adhering to the substrate and to the ceramic. Next, the cement was light activated with either a QTH or an LED appliance, resulting in cement specimens 800 µm thick. The experimental groups were formed by combinations between the factors of ceramic disk thickness and light curing appliance, totaling four groups with n=6. Figure 1 shows the experimental setup of the study.

After light curing, the samples were stored in a dry oven at 37°C for 24 hours. To measure the Knoop hardness of the cement, a water-cooled diamond disk was used to section the samples longitudinally (Exttec model 12205, Exttec Corp, Enfield, CT, USA). The cut surface resulting from sectioning was polished under water cooling in a universal polishing machine model APL-4, using 400-, 600-, and 1200-grit silicon carbide abrasive paper for 15, 30, and 60 seconds, respectively.

Knoop Hardness Measurement Data and Statistical Analysis

After polishing, indentations were made on the samples with a microhardness tester model HMV-2 (Shimadzu, Tokyo, Japan). A 50-gf load was applied for 15 seconds. Three equidistant indentations were made at each predetermined depth from the surface, which was in contact with the PVC film closest to the ceramic disk (top, 50 µm; center, 400 µm; bottom, 750 µm) in the center and periphery of the longitudinal section. For each sample, the direction in which the indentations began was inverted so that no region would be favored.

A mean hardness was obtained for each depth in each sample (three measurements). The data were submitted to analysis of variance applied in a split-plot factorial design. The plots represented the light

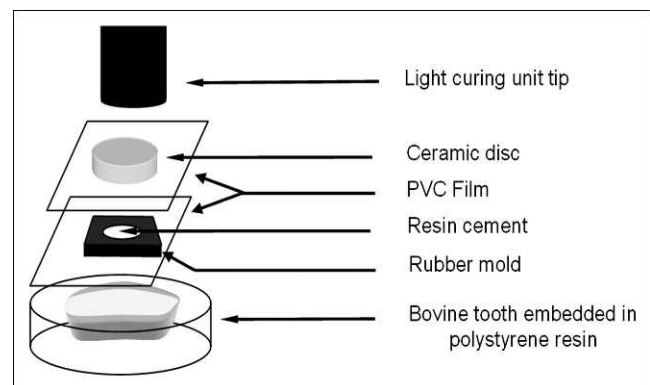


Figure 1. Schematic representation of specimen preparation.

Table 1: Mean Hardness Values (Knoop Hardness Test) of RelyX™ U100 with Regard to the Light Curing Unit, Thickness of the Interposed Ceramic, and Cement Depth Assessed (Standard Deviation)^a

Light Curing Unit	Depth	Thickness	
		1.4 mm	2 mm
QTH	Top	42.0 (3.5) Ab	40.6 (5.4) Bb
	Center	58.1 (9.1) Aa	50.1 (5.7) Ba
	Bottom	23.8 (4.4) Ac	20.2 (2.7) Bc
LED	Top	51.5 (7.6) Aa	42.3 (5.5) Bb
	Center	51.9 (4.8) Aa	51.8 (6.6) Aa
	Bottom	24.2 (2.3) Ab	19.9 (3.3) Ac

^a Means followed by different letters differ within each light curing unit by analysis of variance and Tukey tests ($p \leq 0.05$); capital letters in the horizontal and lowercase letters in the vertical. There was no significant difference between the curing modes ($p=0.4484$).

curing unit \times ceramic thickness, and subplots represented the measured depths. The level of significance considered was 5%.

RESULTS

The hardness results are shown in Table 1. Statistical analysis showed that the factors “ceramic thickness” ($p=0.0057$) and “measurement depths” ($p<0.0001$) were significant, while the factor “light curing unit” ($p=0.4484$) was not. There was a significant interaction among the three factors ($p=0.0109$). Table 1 shows the result obtained by breaking down the interactions.

In the groups light cured with the halogen lamp, an increase in ceramic thickness reduced the hardness values (KHN) of the cement at all depths, with the highest values always being found in the center (1.4 mm, 58.1; 2 mm, 50.1) and the lowest values at the bottom (1.4 mm, 23.8; 2 mm, 20.2). When the LED appliance was used, an increase in ceramic thickness resulted in diminished hardness values only on the top (1.4 mm, 51.5; 2 mm, 42.3). In the group in which the 1.4-mm disk was used, LED induced similar values at the top (51.5) and center (51.9) and lower values at the bottom (24.2). However, when the 2-mm disk was used, the highest hardness value was obtained in the center (51.8),

followed by the top (42.3) and bottom (19.9), results that were similar to those obtained with the halogen light curing unit (center > top > bottom).

DISCUSSION

A hardness test is commonly used as a simple and reliable method for indirectly indicating the degree of conversion of resin cements⁶, and the Knoop microhardness test is the most indicated for polymeric materials since the dimensions used as reference for this calculation do not undergo elastic recovery after removing the load.²⁷ The degree of conversion in a polymerization reaction depends on the energy supplied during light polymerization, characterized by the product of light intensity and exposure time.¹¹ Additionally, the location of the indentation made in laboratory tests is extremely important since hardness values are usually higher in the center of the material than at its extremities or edges. This can be explained by the fact that, in the center of the material, the free radicals of the monomer are tridimensionally surrounded by possible reactive partners, whereas a free radical located at the extremity of the test specimen will find reactive partners located on only one side of the hypothetical sphere in which the free radical is the center.²⁸ This study assessed the polymerization depth of an 800- μ m film in order to indicate its conversion potential, although the film thickness obtained in accordance with the ISO 9917 specification for RelyX™ Unicem cement was 23.2 μ m.²⁹

When cured by QTH unit, the 2-mm-thick ceramic disk decreased the hardness values of RelyX™ U100 at all depths studied. The lowest hardness values and, consequently, the lowest degree of conversion are usually attributed to the attenuation of light caused by the distance from curing light tip^{30,31} and by the increase in opacity of the material resulting from the increase in thickness of the prosthesis.^{8,10,14,19,32} The translucence of ceramics is related to their thickness, microstructure (crystalline content), number of firing cycles carried out in their processing, and the presence of porosities.³³ Empress Esthetic, used in this current study, is a leucite-reinforced vitreous ceramic, with higher translucence, smaller grain size, and more homogeneously distributed leucite crystals than its predecessor, Empress.³⁴ Pazin and others³² found that the Empress Esthetic ceramic, at the thicknesses of 1.4 and 2 mm, showed no influence on the emission spectrum of halogen and LED curing units (including the same brands used in this present study), maintaining the peak wavelength of the curing units

in the same position in the curve. However, this ceramic was capable of reducing the level of irradiance of a QTH unit to approximately 50% at the same thicknesses as those used in the current study.³⁵ Further studies are necessary to assess the activation of RelyXTM U100 through other ceramics of different colors and opacities and with the use of other levels of irradiance of light curing appliances.

Regardless, all the test specimens light cured by the halogen light curing unit through 1.4- and 2-mm-thick disks showed higher hardness values in the center region (400 μm) followed by the top (50 μm) and finally the bottom of the material (750 μm). The lower hardness values found in the bottom region may be attributed to two possible causes: 1) the attenuation of incident light, resulting from the absorption and scattering promoted by the ceramic spacer³³ and the organic and inorganic components of the resin cement,¹¹ and 2) the impossibility of the polymerization reaction continuing because of the increase in viscosity of the resin caused by the initial polymerization and entrapment of radicals and chemical promoters in the polymer network.³⁶ Thus, development of the polymer network could be affected both by the reduction in the conversion of monomers and by an interference in the type and degree of cross-linking.³⁷

In places where the light arrives with less intensity, a lower number of polymer growth centers is generated, and this is unfavorable to the formation of polymer networks that are more densely composed of cross-links.³⁷ Therefore, lower hardness values in the bottom region of the resin cement could also be related to its lower cross-link density. Thus, debonding of the prosthesis could occur clinically as a result of a cohesive failure of the luting agent.³⁸ In addition, one must consider that the present study analyzed the hardness values after 24 hours of dry storage, while the setting reactions via oxyreduction and acid base present in RelyXTM Unicem have been shown to be capable of modifying the values of the degree of conversion seven days after light activation.¹⁷ Further investigations are necessary to determine how important the chemical setting of this dual-cured cement in longer periods is with regard to the durability of indirect restorations cemented with RelyXTM U100.

Since the layer inhibited by oxygen usually does not exceed 20 μm ,^{39,40} it was not a factor with the topmost region evaluated in the current study (50 μm). The intermediate hardness values observed on the top region for QTH curing, compared to the center and bottom regions, could be attributed to the migration of

the organic polymer to the top of the luting agent, which occurs because of the compression exerted on the ceramic disk while seating it on the tooth preparation and light activation, promoting a superficial layer rich in resin both in modified ionomer materials and in resin materials.⁴¹ Clinically, one should be concerned about the additional use of light at the restoration margins in order to minimize the oxygen inhibition on the resin cement surface.

When the cement RelyXTM U100 was light cured by a LED light curing unit, one could observe two different situations with regard to the polymerization depth of the cement: 1) the hardness values on the top were similar to those found in the center when light cured through the 1.4 mm thick ceramic disk, and 2) hardness values on the top were dependent on the disk thickness, which does not occur with the center and bottom values. Therefore, the top region of RelyXTM U100 would be more dependent on the level of irradiance during light curing/activation/polymerization by LED. Ultra-Lume LED 5 is a high-powered, third-generation LED (1585 mW/cm^2), with a broader spectrum band than the second-generation LEDs.³² It is known that high-intensity light emission during the first seconds of light activation causes the rapid formation of the polymer network on the top layer of the composite, characterized by the formation of cross-links,⁴² which could increase the hardness values found under the previously mentioned conditions, including values at the top similar to those found in the center. Thus, it is expected that the reduction in mobility of the monomers in the body of the specimens would occur, making polymerization of the center and bottom regions dependent on the additional acid-base setting reactions, that is, the reaction between the metal ions of the vitreous particles of nonsilanized aluminum fluorine silicate and methacrylate phosphate radical.¹⁵

It is also known that the polymerization reactions via free radical and the acid base present in hybrid materials, such as in resin-modified glass ionomers and even RelyXTM U100, compete with and inhibit one another.⁴³ This is most likely the reason why the thickness of the ceramic disk did not influence the hardness values of the center and bottom regions when light cured by the LED curing unit in this current study. Therefore, an increase in light intensity could improve the polymerization efficiency of the luting agent and shorten the irradiation time, contributing to patient comfort and reducing clinical time for professionals. The additional polymerization reactions present in RelyXTM U100 will occur slowly,

but they will contribute to the formation of a polymer network with a high molecular weight.¹⁵ Therefore, the patient must be instructed to take care when exerting masticatory effort in the first 24 hours (or even seven days)¹⁷ after prostheses have been cemented with this cement.

CONCLUSIONS

Based on the results of the present study and considering its limitations, it was concluded that the thickness of the ceramic restoration negatively influenced the polymerization of the self-adhesive cement RelyXTM U100 when cured by QTH at all analyzed depths. However, only the top region of the specimens cured by the LED unit was affected by the ceramic thickness.

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