BASIC SCIENCE RESEARCH

Effect of Light-Curing Method and Cement Activation Mode on Resin Cement Knoop Hardness

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Purpose: **To evaluate the Knoop hardness (KHN) of the resin cement Enforce activated by chemical/physical mode or physical mode solely; light-cured directly or through a 1.5 mm thick ceramic disc (HeraCeram) on shade DD2.**

Materials and Methods: **Light-curing was carried out using a conventional quartz tungsten halogen light (QTH) (XL2500) for 40 seconds at 700 mW/cm^2; light-emitting diodes (LED) (Ultrablue Is) for 40 seconds at 440 mW/cm^2; and Xenon plasma arc (PAC) (Apollo 95E) for 3 seconds at 1600 mW/cm^2. Bovine incisors had their buccal faces flattened and hybridized. A mold was seated on these surfaces and filled with cement. A disc of the acid-etched and silanized veneering material was seated over this set for light-curing. After dry storage (24 hours at 37°C), specimens ($n = 10$) were sectioned for KHN measurements performed in a microhardness tester (50 gf load for 15 seconds). Data were submitted to ANOVA and Tukey's test ($\alpha = 0.05$).**

Results: **The highest KHN values were obtained with LED, for both dual-cured and light-cured cement. The lowest KHN value was obtained with light-cured PAC. Light-curing with QTH resulted in hardness values similar to PAC in dual-cured groups.**

Conclusions: **Light-curing through HeraCeram can influence resin cement hardness.**

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INDEX WORDS: resin-based cement, resin composite, ceramic, light transmission

THE use of resin cements has grown in the last few years due to greater use of indirect restorative materials, such as ceramics and resin composites. The advantages of these cements are adhesion to substrates, silane agent and adhesive system compatibility, low solubility, easy manipulation, and favorable aesthetics when used with all-ceramic systems. The application of these cements can still result in higher fatigue compressive strength of all-ceramic crowns compared with glass ionomer cements and zinc phosphate cements.[1]

Despite a variety of available cements, there is no ideal cement for all clinical situations. Therefore, the choice of luting agent must rely on its physical and biological properties, as well as the characteristics of the prosthesis and the remainder of the prepared tooth.[2]

Factors such as light-curing method, exposure time, indirect restorative material, and the luting agent can influence the final quality of restorations.[3-9] Inlays, onlays, laminated veneers, and all-ceramic crowns are commonly cemented with dual-cured resin cements, because light transmission through an indirect restoration is critical, and the chemical reaction theoretically would guarantee a satisfactory polymerization. Linden et al[10] verified that the light transmission spectrum through ceramics is influenced by the ceramic's thickness and opacity. Longer light-curing exposure time results in higher polymerization depth, conversion degree, and hardness values,[3,9,10] and

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therefore in improved mechanical and esthetic properties.[11] Consequently, the exposure time recommended by the manufacturer should be treated with caution.[10,12]

The hardness test is commonly used as a simple and reliable method to indicate the degree of conversion of resin cements.[13] The degree of conversion in a polymerization reaction depends on the energy supplied during light-curing, characterized as a product of the light intensity and exposure time.[14,15] In the same brand, dual-cured resin cements when light-activated present higher hardness values than those light-cured solely.[3,16] Witzel et al[17] verified that the dual-cured resin cements, when not light-cured and associated with one-bottle adhesive systems, resulted in about 51% and 64% lower values of bond strength compared with those obtained with light-cured, dual-activated cements.

Light-curing is usually carried out with quartz tungsten halogen (QTH) light-curing units (LCUs). Other technologies, such as Xenon plasma arc (PAC)[18,19] and light-emitting diodes (LEDs),[20-23] are also available. These systems are still under development, but are increasingly being used. There are still doubts about the effectiveness of resin cement light-activation with different methods using these LCUs. This study aims to evaluate the influence of HeraCeram ceramic and different light-curing units on resin cement's Knoop hardness (KHN). Thus, the null hypothesis of this study is that similar resin cement hardness values would be obtained with different veneering materials, light-curing units, and cement activation modes.

Materials and Methods

For the present study, 60 disc-shaped specimens (1.5 mm in height, 7 mm in diameter) were prepared with a feldspathic ceramic (HeraCeram, Heraeus Kulzer, Wehrhein, Germany) in shade DD2.

One hundred and twenty fresh extracted bovine incisors were sectioned to separate their coronal portion. They were embedded with polystyrene resin in plastic molds, keeping their vestibular surfaces exposed. These surfaces were ground flat under water-cooling with SiC sandpapers with #200, #400, and #600 grit (Saint-Gobain, Pernambuco, Brazil), to obtain an exposed dentin area of at least 25 mm^2. Prior to cementation, the dentin surfaces were etched with 37% phosphoric acid (Condicionador Dental Gel, Dentsply, Petrópolis, Brazil) and submitted to hybridization with the adhesive system Prime & Bond 2.1 (Dentsply), according to manufacturer's instructions. Light-curing was carried out with a QTH LCU XL 2500 (3M ESPE Dental Products, St. Paul, MN), for 10 seconds for each layer at 700 mW/cm^2 for 20 specimens; another 20 specimens were light-cured with LED Ultrablue Is (DMC Equip. Ltda., São Carlos, Brazil) also for 10 seconds, but at 440 mW/cm^2. The remainder were light-cured with Xenon PAC (Apollo 95E, DMD Equip. Ltd., Westlake Village, CA) for 3 seconds at 1600 mW/cm^2 (PAC). The irradiances of light-curing units were measured with a digital handheld radiometer (Dental Hilux Curing Light, Dental Benlioglu, Inc., Binnaz SK 1-6 Kavaklidere, Ankara, Turkey).

The discs of veneering materials were etched with 10% hydrofluoric acid (Condicionador de porcelanas, Dentsply) and silanized (Silano, Dentsply), according to manufacturer's instructions.

The resin cement Enforce (Dentsply) in shade A2 was used for cementation with two activation modes: dual-cured and light-cured solely. By the combination of veneering materials, light-curing mode, and cement activation mode, 12 groups ($n = 10$) were tested (Table 1). The control groups were obtained with direct light-curing of resin cement.

For cementation, a rubber mold (5-mm diameter, 1-mm height) was seated over the hybridized dentin and bulk-filled with resin cement. Over this set, a disc of the veneering material was digitally compressed for cement excess flow and removal. Exposure time was 40 seconds for both QTH and LED and 3 seconds for PAC.

Table 1. Groups Tested

Groups	*Cement Activation Mode*	*Veneering Material*	*Light-Curing Mode*
1	Dual-cured	Direct (without material)	QTH
2	Dual-cured	Direct (without material)	LED
3	Dual-cured	Direct (without material)	PAC
4	Light-cured	Direct (without material)	QTH
5	Light-cured	Direct (without material)	LED
6	Light-cured	Direct (without material)	PAC
7	Dual-cured	HeraCeram	QTH
8	Dual-cured	HeraCeram	LED
9	Dual-cured	HeraCeram	PAC
10	Light-cured	HeraCeram	QTH
11	Light-cured	HeraCeram	LED
12	Light-cured	HeraCeram	PAC

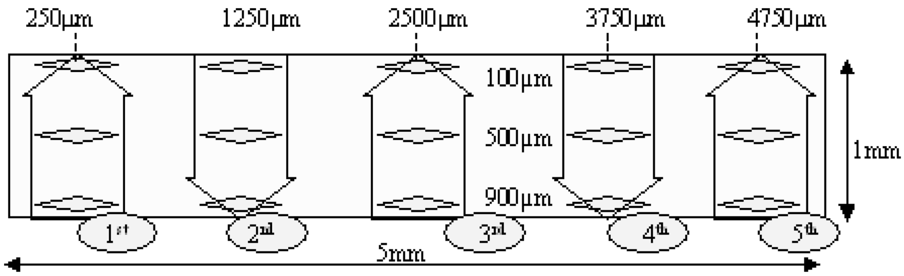

Figure 1. Sequence of indentations.

During light-curing, the LCU tip was in contact with the veneering material.

After light-curing, the samples were stored dry in the dark at 37°C, for 24 hours. To perform resin cement KHN measurements, samples were sectioned longitudinally under water-cooling with a diamond saw (Extec model 12205, Extec Corp., Enfield, CT). The surface obtained by sectioning was polished sequentially under water-cooling with SiC sandpapers with # 400, #600, and #1200 grit.

Indentations and microhardness measurements (KHN) were performed sequentially in columns, in a microhardness testing machine HMV-2000 (Shimadzu, Tokyo, Japan). Five indentations were performed in each depth at 100, 500, and 900 μm from the top surface (Fig 1), with a load of 50 gf for 15 seconds.

For each sample, a mean hardness value was obtained from 15 measurements, and data were submitted to 3-way ANOVA and Tukey's test, both with $\alpha = 0.05$.

Results

Analysis of variance showed that there was significant interaction of factors ($p = 0.00001$), which led to the comparison among groups by Tukey's test. Table 2 shows the results.

Discussion

Light-curing of resin composites has two goals: to fulfill clinical requirements and to provide reliable mechanical properties, such as high hardness and high conversion degree.[24] Regarding conversion degree, Ferracane[25] suggested that the use of indirect methods, such as hardness evaluation, is reliable for predicting the degree of conversion of composites. To perform hardness measurements, 50 gf load for 15 seconds was used in all tested conditions. Uhl et al[26] verified that with the variation of indentation load, the same material can present distinct hardness values. In the group where the physical-cured cement was light-activated with Apollo 95E through ceramic, it was not possible to measure hardness values due to low polymerization, which led to large indentations that exceeded the digress limit between the vertical bars of the microhardness tester viewfinder (Table 2). The decrease in load and in indentation time would produce a smaller indentation, allowing these values to be obtained; however, in surfaces with higher hardness, these small indentations could lead to the higher data variability.

The results show that the dual-cured cement when activated with PAC presented higher hardness values compared with physical-cured cement (Table 2). With direct light-curing using LED, the same behavior was noted (Table 2). Kramer et al[27] suggested that the use of dual-cured cements could be favorable, because the chemical initiators would complement a possible deficiency of the resin cement light-curing; however, the light-curing of dual-cured cements has been neglected by professionals due to misunderstanding of the LCU characteristics they possess. Peutzfeldt,[28] Rueggeberg and Caughman,[29] and el-Mowafy et al[6] verified that when dual-cured cement had been light-cured, there was an increase of conversion degree compared with dual-cured cement polymerized only by chemical activation.

Table 2. KHN Comparison among Groups by Tukey's Test

	Direct Light-Curing Mean (SD)	*HeraCeram Mean (SD)*
Dual-cured		
QTH	45.0 (4.7) A, b	41.4 (5.9) B, b
LED	53.6 (4.6)* A, a	50.1 (5.2) B, a
PAC	38.1 (2.9)* A, c	39.1 (4.5)* A, b
Light-cured		
QTH	44.3 (1.2) A, b	34.8 (2.7) B, b
LED	50.8 (2.2)* A, a	51.2 (4.9) A, a
PAC	25.7 (5.1)* A, c	0.0 (0.0)* B, c

Different small letters in columns for each cement activation mode and different capital letters in rows represent statistically significant differences.
*Represents statistically significant differences between cements for the same light-curing unit ($p < 0.05$).

During this study, just one shade and one thickness of veneering material was used (shade DD2/1.5-mm thick), because as verified in the literature, the hardness of resin-based cements can be influenced by these factors.[4,30,31] The resin cement per se and the light transmission coefficient of ceramics can influence the degree of conversion of light-activated materials.[32] Warren[33] and el-Mowafy et al[6] showed that the thicker the veneering material, the softer the cement. The bond strength of cement–ceramics and tooth–cement interfaces can also decrease with an increase in thickness.[34]

In this study, it was possible to verify the influence of light-curing methods in the dual-cured and light-cured groups. On both conditions, higher hardness values were obtained with LED, followed by QTH and PAC (Table 2). The correlation between the light transmission spectrum of LED and the light absorption spectrum of camphorquinone could possibly explain these results,[22,23] although QTH and PAC emitted higher light intensity. It can also be hypothesized that the ceramics worked as a filter with QTH, absorbing light of its broad wavelength spectrum.

The lowest hardness values were obtained in the physical-cured group light-activated with PAC (Table 2). According to Danesh et al,[35] the polymerization efficiency using Apollo 95E depends on the type and brand of the material to be light-cured. For the resin cement Enforce, the LCU manufacturer's recommended exposure time is 3 seconds. This exposure time is very fast, and the energy density supplied by PAC (4.8 J) to resin cement is much smaller than the energy density supplied by QTH (28 J) and LED (17.6 J). The energy density is obtained by the exposure time and the light intensity emitted by the light-curing unit. Thus, the former could not provide enough energy for the polymerization reaction of the composite, which would present poor properties.[36] It was possible to verify that for the dual-cured cement light-activated through HeraCeram, PAC and QTH presented similar hardness values, even with the lower energy density supplied by PAC. It can be supposed that the chemical polymerization in this case complemented the setting reaction of the cement.

According to Moon et al,[37] appropriate exposure time and enough energy density should be applied to obtain better mechanical properties of the composites. In general, the degree of polymerization of a resin composite is proportional to the amount of light it is exposed to. Thus, in higher depths of resin composite restorations, where there is lower light penetration, there would be lesser conversion.[38,39] Rasetto et al[9] stated that the same could be applied for resin cements. Therefore, for indirect light-curing of resin cements using high intensity LCUs, manufacturer-recommended exposure time should be increased to obtain hardness values similar to those obtained with direct light-curing.[33,40] In this study, in general, higher hardness values were obtained with direct light-curing compared with light-curing through HeraCeram, except to dual-cured cement with PAC and light-cured cement with LED. It can be supposed that the light absorption and scattering by HeraCeram were very low with LED because of its narrow spectrum of output light. The similarity for PAC could be the result of the low power density supplied during light-curing. In this case, it can be hypothesized that almost full reaction occurred by chemical activation of the polymerization reaction.

Further studies are necessary to clarify the role of different types of indirect prosthetic materials in the attenuation and modification of light emitted by different LCUs for resin cement light-curing.

Conclusions

Considering the limitations of this study, it can be concluded that the null hypothesis was rejected, and:

1. Light-curing with PAC for 3 seconds is not reliable for resin cement hardness values.
2. Higher hardness values were obtained with LED, on both cement activation modes.
3. In general, light-curing through ceramics resulted in lower resin cement hardness values.

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