Influence of light-curing units and restorative materials on the micro hardness of resin cements

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ABSTRACT

Aim: The aim of this study was to evaluate the effect of indirect restorative materials (IRMs) and light-curing units (LCUs) on the micro hardness of dual-cured resin cement.
Materials and Methods: A total of 36 cylindrical samples (2 mm thick) were prepared with dual-cured resin cement (Relyx ARC) photo-activated with either a QTH (Optilight Plus) for 40s or a LED (Radii) light-curing unit for 65s. Photo-activation was performed through the 2-mm-thick IRMs and the samples were divided into six groups (n = 6) according to the combination of veneering materials (without, ceramic and indirect resin) and LCUs (QTH and LED). In the control group, the samples were light-cured with a QTH unit without the interposition of any restorative material. Vickers micro hardness test was performed on the top and bottom surfaces of each sample (load of 50 g for 15 secs). The data were statistically analyzed using a three-way ANOVA followed by Tukey × s post-hoc test ($P < 0.05$).
Results: There were no statistically significant differences on the top surface between the light curing-units ($P > 0.05$); however, the LED provided greater hardness on the bottom surface when a ceramic material was used ($P < 0.05$). The mean hardness in photo-activated samples, in which there was no interposition of indirect materials, was significantly greater ($P < 0.01$).
Conclusions: It may be concluded that the interposition of the restorative material decreased the micro hardness in the deeper cement layer. Such decrease, however, was lower when the ceramic was interposed and the cement light-cured with LED.

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The cementation stage is extremely important in esthetic rehabilitations using indirect restorative materials (IRMs). Resin cements bond indirect materials to tooth structure and according to their mode of activation may be classified as: Auto-polymerized, photo-polymerized, and dual-polymerized (chemical and physical activation).[1]

Factors, such as light-curing method, exposure time, use of an IRM and the luting agents have been shown to influence the final quality of restorations.[2,3] Some mechanical properties of resin-based materials can be assessed by an indirect measurement of the quality of polymerization. Microhardness tests show good correlation with Fourier infrared spectroscopy analysis[4,5] and have been commonly used to indicate the degree of conversion of resin-based cements.[6] The degree of conversion in a polymerization reaction is normally dependent on the energy delivered during light curing as a function of light intensity and exposure time.[7]

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Dual cements are indicated for the cementation of inlays, onlays, and total crowns fabricated with composites or ceramic material, since they compensate for the light attenuating effects produced by light curing through IRMs.[2] This compensation is possible by the chemical polymerization, which guarantees the cure of the material even in the deeper regions where light access is limited.[8] The polymerization of dual resin cements occurs in both the presence and absence of light. However, the chemical activation mechanism alone is less effective than dual-cure,[1,9,10] with the best results being achieved when the photo-activation process is also carried out.[3]

Halogen lamps are the most frequently used light source for polymerization of resin-based dental materials. The conventional Quartz Tungsten Halogen (QTH) lamp possesses the advantage of low cost technology; however, with drawbacks that include production of high temperatures and irradiance decline over time due to bulb and filter ageing.[11] QTH units have a useful lifetime of 40 to 100 hours.[12] An alternative curing device that has been investigated in an attempt to overcome the problems of halogen lamps is the light-emitting diodes (LED). These light sources operate at a wavelength of around λ 470 nm, and therefore, have the spectral purity for high efficiency curing. LEDs have a useful lifetime superior to 10,000 hours and undergo little degradation over time.[13]

Both the optimal curing time for LEDs and whether it has the ability to adequately cure all resin-based materials are unknown. There is a need to transfer a number of clinical settings to the laboratory since it has still not been determined whether the LED has been fine-tuned sufficiently to replace halogen-based visible curing units. Uncertainties remain regarding the effectiveness of photo-activation of resin cements and the capacity of LEDs to harden the luting material through an IRM. The purpose of this study, therefore, was to evaluate the micro hardness of dual-cure resin cement subjected to photo-activation by either QTH or LED and through either ceramic or indirect resin. Thus, the null hypothesis of this study is that similar resin cement hardness is obtained in presence of different IRMs, using LCUs and at different cement depths.

MATERIALS AND METHODS

Testing the Vickers micro hardness of Relyx ARC dual resin cement was performed when light cured using either QTH or LED light-curing units (LCUs), and through two different IRMs (Noritake feldspathic ceramic and Solidex indirect composite resin) [Table 1].

A total of 36 disk-shaped samples with dimensions of 5 mm in diameter and 2 mm thick were prepared in a teflon cylindrical mould rested onto a glass plate and a Mylar polyester strip (Polidental Ind., São Paulo, Brazil). The cement was mixed and inserted in the mold according to ISO 4049. The interior of the mold cavity was filled with a bulk increment of resin cement using a TD4X spatula (Dental Thompson, Manufacturing Company Incorporation). The cement was dispensed using a "clicker" system (which ensures a precise volume ratio of the two pastes) and manipulated for 10 seconds using a metal spatula (Heraeus Kulzer, Wehrhein, Germany), according to the manufacturer's instructions. After filling the whole mold, the mold was then covered with another polyester strip to separate the samples of resin cement for hardness testing. Over this set, a disc of indirect restorative material was digitally compressed to act as interposition between the cement and the light source and to flatten samples' top surface.

Two types of disks (7 mm in diameter and 2 mm thick) were fabricated, both with A2 shade (Vita shade guide, Vita - Germany) of ceramic and indirect composite resin. The thickness of the disk chosen closely simulates that of inlay/onlay indirect restorations, whereas the diameter selected was used to ensure that the light passed only within the boundaries of the disk (as the diameter of the light tip is larger than 5 mm). Disk measurements were assessed with a digital caliper (Starret, MA, USA).

Two LCUs were used, one was QTH based (Optilight Plus, Gnatus, SP, Brazil, with 400-500 mw/cm^2 intensity) and the other was LED based (Radii LED curing light, SDI Limited, Victoria, Australia, with 1,200-1,400 mw/cm^2 intensity. Both QTH and LED units were used for 40 and 65 secs, respectively, according to their manufacturer's instructions. During light activation, the light tip was in contact with the veneering material to allow adequate light exposure. After light curing, each specimen was protected with aluminum foil and stored in the dark at 37°C during 24 hours. Six groups (n = 6) were formed by the combination of veneering materials (without, ceramic and indirect resin) and LCUs (QTH and LED). The group in which light-curing was performed with the QTH unit and without the interposition of any restorative material served as a control group.

Before and during light curing, the light intensity of each curing unit was monitored by means of a light meter (Hilux Ledmax Curing Light Meter, SDI Limited, Victoria, Australia). The wavelength of the emitted light was measured using a spectrophotometer (Jobin Yvon, model U-1000, France) for both QTH and LED curing units.

A hardness tester with a Vickers diamond indenter (Shimadzu microdurometer (Shimadzu Corporation, Kyoto, Japan; 50 g load for 15 secs) was used for testing the hardness of each specimen. Three indentation readings were obtained from each of the two surfaces (top and bottom). Mean Vickers hardness numbers (VHNs) were then calculated. Data were subjected to a three-way ANOVA followed by Tukey-Kramer tests at a significance level of 5%.

Table 1: Description of resin-based and ceramic materials used in this study

Classification	Product	Composition	Shade	Manufacturer
Dual resin cement	Relyx ARC	Inorganic filler load of 67.5% by weight, with an average size of 1.5 μm, inserted into a Bis-GMA and TEGDMA mold	A1	3M ESPE, St. Paul, USA
Indirect composite resin	SHOFU SOLIDEX® light-cured laboratory composite	53% inorganic and ceramic microfilaments, 25% co-polymers of multi-functional resins and 22% conventional resins/light-initiators	A2	Shofu Inc., Kyoto, Japan
Feldspathic ceramic material	NORITAKE EX-3	Aluminum silicate, pure silica, caolim and quartz	A2	NORITAKE Co., Inc., Nagoya, Japan

Bis-GMA = Bisphenol A-Glycidyl Methacrylate; TEGDMA = Try Etileno Glicol Dimetil Methacrylate

RESULTS

There were significant differences in mean VHNs between interposition materials (ANOVA, $P < 0.01$) and cement depths (ANOVA, $P < 0.01$). The analysis also revealed two interactions: Interposition material *vs.* cement depth and interposition material versus light-curing unit [Table 2].

The hardness means (VHN) on the top surface for QTH and LED LCUs with each IRM material are shown in Table 3. Vickers hardness on the top surface was not affected by the interposition material or light-curing unit ($P > 0.05$). However, the bottom surface hardness was dependent on either the interposition material or light-curing unit used ($P < 0.05$). There were significant differences between QTH and LED units when there was no interposition of material. However, in the Noritake ceramic veneering material, superior Vickers hardness was obtained when the cement was light cured with LED as opposed to QTH.

The groups without interposition of indirect materials presented significantly higher VHN means than groups in which the cement was light cured through the Solidex or Noritake ceramic (TK, $P < 0.01$). There were no significant differences between the Solidex and Noritake ceramic IRMs ($P > 0.05$).

General VHN means for the top surface, regardless of the interposition material or light source used, were 31.13 (±1.59). This is 21% greater than the VHN on the bottom surface, 25.69 ± 3.81). Moreover, there was an interaction between interposition materials versus cement depths. This interaction shows that the top surface demonstrated greater hardness values than the bottom surface (TK, $P < 0.01$), except for light activation without interposition of a veneering material. In this group, the hardness on the top and bottom surfaces was similar. On the top surface, the results showed no significant differences between the interposition conditions evaluated.

Table 2: Denomination and characteristics of the groups

Group	n	Veneering material	Light-curing unit	Exposure time (s)
1 - WIH*	6	Without interposition	QTH	40
2 - WIL	6	Without interposition	LED	65
3 - SH	6	Solidex	QTH	40
4 - SL	6	Solidex	LED	65
5 - NCH	6	Noritake ceramic	QTH	40
6 - NCL	6	Noritake ceramic	LED	65

*Control group: The dual cement was light cured without interposition of veneering indirect material

However, on the bottom surface, photo-activation through the Noritake ceramic or Solidex veneering showed lower VHN means than on the top surface.

DISCUSSION

The mechanical properties of resin-based materials can be evaluated by methods that evaluate diametral tensile strength and micro hardness. The diametral tensile test is usually carried out to determine the effect of filler content, monomer composition, different curing times, and polymerization procedures.[14] The effectiveness of material cure may be directly or indirectly assessed. Direct methods, such as infrared spectroscopy, are complex, expensive, and time-consuming.[15] Indirect methods have included visual, scraping, and hardness testing. A good correlation between hardness and infrared spectroscopy[4] as well as hardness and monomer conversion degree[5] has been previously reported. In this study, micro hardness measurements were used as a means to estimate the quality of resin curing under indirect restorative materials, since the mechanical properties of resin-based materials can be directly related to the extent of the conversion of the polymer network.[16]

The present study evaluated the micro hardness of dual resin cement using different LCUs and distinct veneering interposition materials. Dual-cured resin cements are widely used because they allow better control during the cementation procedure, are efficient in the deeper areas where the curing light cannot penetrate and the self-curing mechanism hardens the cement. As the self-curing mechanism of some dual-cure cements have been reported to be inadequate,[17] the light curing would function as an additional means of curing for these materials.

The results of present study demonstrate that the interposition of IRMs and the use of different LCUs, in specific conditions, influenced directly the curing depth of the dual-cured resin cement used. The interposition of 2 mm of indirect veneering material decreased the VHN of cement, which in present study was light cured through Solidex or Noritake ceramic. This confirms the light attenuating properties of IRMs on resin cement polymerization previously reported by Hasegawa *et al.*[18] and Tango *et al.*[2] The indirect materials limited the penetration of light and only about 0.13% of the light emitted by the light-curing unit passed through a 1-mm-thick ceramic veneer.[19]

Table 3: Comparison of hardness means (Vickers hardness numbers) at the evaluated depths

	Interposition material (VHN ± SD)					
	Without		Solidex		Noritake ceramic	
Light-curing unit	QTH	LED	QTH	LED	QTH	LED
Top surface	32.60 (± 1.01) Aa	31.67 (± 0.71) Aa	31.30 (± 1.23) Aa	30.83 (± 1.75) Aa	29.77 (± 2.14) Aa	30.62 (± 0.7) Aa
Bottom surface	29.90 (± 0.98) Aa	30.37 (± 0.54) Aa	23.13 (± 1.38) Ba	23.80 (±1.64) Ba	21.50 (± 1.92) Ba	25.40 (± 0.61) Bb

VHN - Vickers hardness number; SD - standard deviation; Identical letters indicate no significant differences ($P > 0.05$). Capital letters compare values per row. Lower cases compare values per column, between QTH and LED

There was no difference between the Solidex and Noritake ceramic materials as interposition materials. These materials have distinct compositions. Whereas, Solidex is an indirect composite resin, Noritake is feldspathic porcelain. However, Tango *et al.*[2] has shown that when resin cement was light cured through HeraCeram, too using a QTH unit, it presented lower hardness in comparison to Artglass indirect resin. The authors attributed this phenomenon to the different refraction indices and opacity of the IRMs due of their distinct nature. Koch *et al.*[20,8] showed that ceramic translucency has an important effect on the VHN of dual-curing composite resins. The authors found that higher translucency of the ceramic restoration resulted in a high depth of conversion and VHN values.

A number of studies have confirmed that IRMs decrease the depth of polymerization of resin cements.[18,21-23] This is clinically relevant because an increase in ceramic thickness has a negative effect on the curing depth and hardness of light-cured cements, regardless of the light-curing unit used, with hardness decreasing dramatically when material thickness is greater than 2 mm.[22]

Although there were no significant differences between the two IRMs used in the present study, there was statistical interaction with the light sources. There was no significant difference between the QTH and LED units when the cement was photo-activated through Solidex or without an interposition material. However, in the Noritake ceramic, higher Vickers hardness was obtained when cement was light cured with LED. Tango *et al.*[3] also found greater VHN values for light cured dual cements photo-activated with LED than for those activated with QTH. This may be attributed to the high-intensity LED (1,200-1,400 mw/cm^2) used in this study, when compared to QTH (400-500 mw/cm^2). As difference between LCUs was not observed with the Solidex resin, it is plausible to speculate that the LED can promote a more efficient polymerization through the ceramic material. The specificity and intensity of light could interact with the refraction indices and opacity of the veneering material.

Vickers hardness on the top surface was not affected by the interposition materials or the different LCUs. The light intensity in this area was sufficient to harden the dual-cure cement, regardless of interposition material used. However, the top surface always presented higher VHNs than the bottom surface, except for light activation without interposition of the IRM. In this latter condition, hardness on the top and bottom surfaces was similar. The greater hardness on the top surface is in agreement with a number of studies.[24,25] This may not necessarily represent a problem in clinical terms since cement thickness was sufficiently thin. However, indirect restorations do not always possess uniform thickness due to the indirect material used, occlusion aspects and cavity shape. Moreover, indirect restorations may have areas of inadequate adaptation that inevitably results in an increase in resin cement thickness. This may justify the assessment of hardness in deeper regions as a means to simulate thicker cement layers.

The use of a self-curing catalyst is recommended over the light-curable portion only, because it produces greater or equivalent hardness and depth of cure with all light polymerization modes.[22] This is mainly true for restorations greater than 1 mm in thickness.[21] High-intensity LCUs when used for an adequate time are clearly a better option, as this curing mode will ensure sufficient hardening of the lower surface of thick increments and hence thorough polymerization.[24,26] IRMs may promote light intensity attenuating conditions and required the photo-activation are optimal. In this regard, the use of dual-cure cements and high-intensity LCUs are necessary. Thus, resin cements under specific conditions may present adequate mechanical properties and clinical performance.

CONCLUSIONS

Based on the results above, the following conclusions may be drawn:

- On the top surface, cement hardness was not influenced by either indirect materials (resin or ceramic) or LCUs (QTH or LED).
- The interposition of IRMs decreased the micro hardness of the dual resin cement on the bottom surface due to their light intensity attenuating properties.
- Cement hardness was not dependent on LCUs on the top surfaces; however the LED increased the hardness in deeper layers of the cement when the ceramic material was used.

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