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Microhardness of a Resin Cement Polymerized by Light-Emitting Diode and Halogen Lights through Ceramic

Tabassom Hooshmand, DDS, PhD,[1] Nasrin Mahmoodi, DDS,[2] & Alireza Keshvad, DDS, PhD[3]

[1] Department of Dental Materials, School of Dentistry/Research Center for Science and Technology in Medicine, Tehran University of Medical Sciences, Tehran, Iran
[2] School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran
[3] Department of Prosthodontics, School of Dentistry, Shahed University, Tehran, Iran

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Correspondence
Tabassom Hooshmand, Department of Dental Materials, School of Dentistry, Tehran University of Medical Sciences, Ghods St., Tehran 14174, Iran. E-mail: hoshmand@sina.tums.ac.ir

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Abstract

Purpose: This study evaluated the curing efficiency of light-emitting diode (LED) and halogen [quartz tungsten halogens (QTH)] lights through ceramic by determining the surface microhardness of a highly filled resin cement.
Materials and Methods: Resin cement specimens (Variolink Ultra; with and without catalyst) (5-mm diameter, 1-mm thick) were condensed in a Teflon mold. They were irradiated through a ceramic disc (IPS Empress 2, diameter 5 mm, thickness 2 mm) by high-power light-curing units as follows: (1) QTH for 40 seconds (continuous), (2) LED for 20 seconds, and (3) LED for 40 seconds (5-second ramp mode). The specimens in control groups were cured under a Mylar strip. Vickers microhardness was measured on the top and bottom surfaces by a microhardness tester. Data were analyzed using analysis of variance (ANOVA) and a post hoc Bonferroni test at a significance level of $p < 0.05$.
Results: The mean microhardness values of the top and bottom surfaces for the dual-cured cement polymerized beneath the ceramic by QTH or LED (40 seconds) were significantly higher than that of light-cured cement ($p < 0.05$). The top and bottom surface microhardness of dual-cured cement polymerized beneath the ceramic did not show a statistically significant difference between the LED and QTH for 40 seconds ($p > 0.05$).
Conclusions: The efficiency of high-power LED light in polymerization of the resin cement used in this study was comparable to the high-power QTH light only with a longer exposure time. A reduced curing time of 20 seconds with high-power LED light for photopolymerizing the dual-cured resin cement under ceramic restorations with a minimum 2-mm thickness is not recommended.

In recent years, adhesive ceramic restorations have become popular due to the increased interest in esthetic dentistry. Resin-based luting agents are typically used for cementation of ceramic veneers, inlays, onlays, crowns, fixed partial dentures, and endodontic dowels. These resin cements are generally hybrid composites based on Bis-GMA chemistry. Polymerization is normally initiated chemically and/or by visible light using a wavelength of 400 to 500 nm.[1]

The success of adhesive bonding of a ceramic restoration is dependent on a number of factors, including the ceramic system, luting agent, curing light characteristics, and curing regimen. Among other factors, adequate polymerization of the resin-based luting cement is a critical factor for the stability and clinical performance of the ceramic restoration. Dual-cured resin cements have been developed to combine the advantages of chemically photoactivated materials in deep areas where the curing light cannot penetrate; however, many studies have shown that the self-curing mechanism of some dual-cured cements is inadequate.[2-4]

Halogen lamps, known as quartz tungsten halogens (QTH), are the most frequently used light sources for polymerization of resin-based dental materials. They emit a continuous spectrum only a small part of which is useful for curing. Other wavelengths are filtered out to prevent undesirable side effects;[5] however, the spectral impurities of halogen lights deliver several wavelengths that are highly absorbed by dental materials, inducing heating of the tooth and resin during the curing process.[6] Other drawbacks are a decline of irradiance over time,[7] limited depth of cure, and relatively long exposure time.[8] The recently introduced light-emitting diode (LED) lights offer a much more narrow emission spectrum (around 470 nm, with a bandwidth of about 20 nm) that falls closely within the absorption range of camphoroquinone, the most frequently employed photoinitiator in resin composites.[9] In general, the LED light has the advantages of extended lifetimes of over 10,000 hours, little degradation of light output over time, preventing overheating, and resistance to shock and vibration.[10]

A number of studies have shown the advantages and limitations of LED curing systems compared with the halogen light sources for the polymerization of light-activated dental restorative materials.[11-16] Many of the newer LED lights with high-power irradiance and recommended shorter exposure times have been introduced to the dental market; however, efficiency of newer generations of LED lights in polymerization of different resin-based cements with different chemical and filler contents under ceramic restorations has not been fully investigated. The efficiency of light-curing systems may be assessed by the degree of conversion and the depth of polymerization of resin-based materials using various methods such as Fourier transform infrared spectroscopy (FT-IR), nuclear magnetic resonance (NMR), and microhardness test. It has been shown that the hardness test was more sensitive than FT-IR to detect small changes in degree of conversion after the network was cross-linked.[17]

The purpose of this study was to evaluate the curing efficiency of the high-power LED and halogen light-curing units through ceramic by determining the surface microhardness of a highly filled resin cement with two modes of activation.

Materials and methods

A hot-pressed lithium disilicate-based glass-ceramic (IPS Empress 2 ingots, Lot DO7432; Ivoclar-Vivadent, Schaan, Liechtenstein) was selected as a simulated ceramic restoration in this study. A glass-ceramic disc specimen of 2-mm thickness and 5 mm in diameter was fabricated according to the manufacturer's instructions. The 2-mm thickness consisted of a 1-mm thick framework material (IPS Empress 2, Shade 400) and 1-mm thick layering material (IPS Empress 2, Shade 410/D3).

For specimen preparation, a clear glass slab on top of a black background was used as a supporting surface and to decrease the reflectivity of the underlying surface toward each specimen. A polytetraflouroethylene (PTFE) mold of 5 mm in diameter and 1 mm in height was placed on the glass slab. A dual-cured resin cement (Variolink Ultra Base, shade A3, Lot no: H25295, Ivoclar-Vivadent) either with or without a self-curing catalyst (Variolink Ultra Catalyst: high viscosity, 210/A3, Lot no: H25293, Ivoclar-Vivadent) was filled into the PTFE mold. Then, the resin cements in control groups were covered with a clear Mylar strip and a glass microscopic slide to obtain a flat polymerized surface. For the resin specimens cured beneath ceramic, the IPS Empress 2 ceramic disc previously described was placed on top of the Mylar strip to prevent adhesion of the resin cement to the ceramic disc.

Light curing of the resin cements was carried out with a QTH curing light (Coltolux 75, Coltene, Whaledent, Mahwah, NJ) at 800 mW/cm^2 for 40 seconds (continuous) and an LED curing light (radii, SDI, Victoria, Australia) at 1100 mW/cm^2. Photopolymerization modes for the LED light were 20 seconds (5 seconds ramp, 15 seconds full cure) and 40 seconds (5 seconds ramp, 35 seconds full cure). Power density of light-curing units was monitored using a radiometer (Demetron L.E.D. radiometer, SDS/Kerr, Orange, CA). The light tips were in close contact with either the microscopic slide or ceramic disc. Using two light-curing units, two activation modes of resin cement, and two curing times for the LED light provided six experimental and six control subgroups (Table 1). Each experimental and control subgroup contained five specimens.

Table 1 Distribution of the test specimens (n = 5)

	Resin cement material	Irradiation mode and time
Control (Mylar strip)	With catalyst	QTH (40 sec)[a]
		LED (20 sec)[b]
		LED (40 sec)[c]
	Without catalyst	QTH (40 sec)[a]
		LED (20 sec)[b]
		LED (40 sec)[c]
Experimental (under ceramic)	With catalyst	QTH (40 sec)[a]
		LED (20 sec)[b]
		LED (40 sec)[c]
	Without catalyst	QTH (40 sec)[a]
		LED (20 sec)[b]
		LED (40 sec)[c]

[a]Continuous mode: 40-seconds at 800 mW/cm^2.
[b]Ramp mode: exponential increase to 1100 mW/cm^2 within 5 seconds, 15 seconds at 1100 mW/cm^2.
[c]Ramp mode: exponential increase to 1100 mW/cm^2 within 5 seconds, 35 seconds at 1100 mW/cm^2.

The top surfaces of all specimens were wet-ground slightly by a 1200 grit size silicon carbide paper to remove uncured resin. All specimens were stored in light-proof containers in distilled water at 37°C for 24 hours. The microhardness was measured using a microhardness tester (Micromet 2100, Buehler, Lake Bluff, IL) with a marker for Vickers unit. Microhardness indentations were made on top and bottom surfaces of each specimen. Three readings with a 25-g load for 10 seconds were taken on each surface, and the average was converted into a Vickers hardness number (VHN). Hardness ratios were computed by dividing the mean bottom by mean top hardness within each group. Data were analyzed statistically using analysis of variance (ANOVA) and a post hoc Bonferroni test (SPSS for Windows 11.5; SPSS, Chicago, IL) at a significance level of $p < 0.05$. Independent sample t-tests ($\alpha = 0.05$) were used to define differences between groups by the specific interacting variables.

Results

The average top and bottom surface microhardness data are presented in Tables 2 and 3, respectively. The bottom-to-top surface hardness ratios are shown in Table 4.

Table 2 The average top surface Vickers hardness data (VHN ± SD)

Groups	Cement material	QTH (40 sec)	LED (20 sec)	LED (40 sec)
Control	Light cured	52.93 ± 3.1	55.37 ± 2.64	55.49 ± 3.83
(Mylar strip)	Dual cured	68.19 ± 1.53	54.13 ± 1.9	59.87 ± 2.2
Experimental	Light cured	39.33 ± 3.53	38.69 ± 4.47	38.69 ± 2.28
(under ceramic)	Dual cured	48.51 ± 1.21	41.72 ± 6.56	49.57 ± 1.45

Control and experimental groups

The mean top and bottom surface microhardness values for all control groups cured under the Mylar strip with any of the light-curing modes (QTH: continuous 40 seconds, and LED lights: total exposure of 20 or 40 seconds; ramp mode within first 5 seconds) were significantly higher than that of experimental groups cured through the ceramic disc ($p < 0.05$).

Light-curing unit

Light-cured cement

There was no statistically significant difference in the top and bottom surface microhardness values among the three light-curing modes for the specimens cured through ceramic or under Mylar strip ($p > 0.05$).

Dual-cured cement

The mean microhardness values of top and bottom surfaces for the control group polymerized with the QTH were significantly higher than that of the LED for 20 or 40 seconds ($p < 0.05$). No significant difference in the bottom surface microhardness values of specimens in control groups existed between the LED lights for 20 seconds and 40 seconds ($p > 0.05$); however, the mean microhardness value of top surfaces for specimens in the control group polymerized with the LED for 40 seconds was significantly higher than that of the LED for 20 seconds ($p < 0.05$).

The mean top and bottom microhardness values for specimens cured under ceramic by the LED for 40 seconds were not significantly different from that of the QTH light ($p > 0.05$). The lowest microhardness values of top and bottom surfaces were obtained with the LED light for 20 seconds through the ceramic disc, significantly different from that of the QTH and LED lights for 40 seconds ($p < 0.05$).

Cement material

Control

The top and bottom surface microhardness values for the dual-cured resin cement polymerized with the QTH were significantly higher than that of the light-cured cement ($p < 0.05$); however, no statistically significant difference in the top and bottom surface microhardness values between the light- or dual-cured cement polymerized by the LED for 40 seconds or 20 seconds was found ($p > 0.05$).

Experimental (under ceramic)

The top and bottom surface microhardness values for the dual-cured resin cement polymerized with the QTH and LED for 40 seconds were significantly higher than that of the light-cured cement ($p < 0.05$). No significant difference in the mean top and bottom surface microhardness values was obtained with the LED for 20 seconds between the light and dual-cured resin cements ($p > 0.05$).

Discussion

To obtain better mechanical properties of the light-activated resin-based materials, appropriate energy density of the light-curing units is required. The energy density is obtained by the emitted light intensity and the exposure time. High-power light-curing units provide higher energy density in a shorter period of time; however, they are unlikely to provide a higher depth of cure in the same short period.[18] This could be of major concern when curing the resin composite cements under the indirect esthetic restorations.

The results from this study showed a significantly decreased microhardness value in the top and bottom surfaces for all specimens (light- or dual-cured cements) cured under ceramic by any of the high-power light-curing mode compared with that of control groups ($p < 0.05$). This confirms the observations of other studies[19-21] in which light curing through ceramic as compared with direct irradiation reduced the values for most mechanical parameters and materials. Attenuation of the curing light by passing through the ceramic disc could decrease the light intensity and thereby reduce the degree of polymerization of resin cements.

Light transmission through an indirect restoration is critical, and an adequate polymerization at the bottom of the cavity could be theoretically obtained by the ongoing chemical

Table 3 The average bottom surface Vickers hardness data (VHN ± SD)

Groups	Cement material	QTH (40 sec)	LED (20 sec)	LED (40 sec)
Control	Light cured	52.01 ± 2.53	50.53 ± 1.12	52.17 ± 2.3
(Mylar strip)	Dual cured	58.82 ± 3.07	52.29 ± 2.15	53.39 ± 2.66
Experimental	Light cured	34.37 ± 3.99	30.28 ± 4.58	35.89 ± 2.37
(under ceramic)	Dual cured	45.80 ± 2.6	34.38 ± 5.08	41.9 ± 1.53

Table 4 Bottom-to-top surface microhardness ratio (%)

Groups	Cement material	QTH (40 sec)	LED (20 sec)	LED (40 sec)
Control	Light cured	98.31	91.25	94.22
(Mylar strip)	Dual cured	86.35	96.6	89.21
Experimental	Light cured	87.65	78.73	92.85
(under ceramic)	Dual cured	94.39	82.67	84.56

reaction within dual-cured resin cements where access for the curing light is limited. Similar to reports by other investigations,[1,19,22-24] the dual-cured resin cement polymerized better beneath the ceramic disc than the light-cured mode in this study. This was statistically significant for the specimens polymerized by the high-power halogen and LED lights with a 40-second curing time ($p < 0.05$). Thus, the use of catalyst was a decisive factor for improving the polymerization and surface hardness when the resin cement was irradiated through ceramic.

Highly filled resin composites may be expected to have improved mechanical properties. Several factors affect the degree of cure in light-activated resin composites, including type, size, and load of filler, shade and thickness of resin restoration, the monomer composition and type, the concentration of initiator and accelerator in the resin materials, amount of light transmission, light intensity, and curing time.[18,25] In addition, a correlation between volumetric filler content and hardness has been demonstrated.[26] The chemical composition of Variolink Ultra resin cement used in this study is similar to that of Variolink II cement but with a higher filler content of 79% in weight or 56% in volume. It has been reported that Variolink II has a relatively weak chemical curing component and relies mainly on its light-curing capabilities.[27] A significant chemically induced continuation of the polymerization process after light initiation is difficult to achieve. The initial light exposure causes a rapid increase in conversion of the resin, resulting in a very viscous gel. This rapid increase in viscosity hinders the migration of active radical components that would be responsible for further chemically induced polymerization. Therefore, the duration of inhibition and the level of initial conversion caused by the light exposure are highly influential factors upon the final cure of a dual-cured resin.[1,28] The thickness of ceramic overlay in this study was 2 mm, and the resin cement used was highly filled. This may explain why there was no significant difference in the surface microhardness values between the light-cured and dual-cured cement specimens polymerized through ceramic by the LED light for 20 seconds ($p > 0.05$). The chemical-activating part of the dual-catalyst system was not effective in improving the surface hardness of the resin cement significantly. Thus, the reduced exposure time of the high-power LED light (20 seconds) could not compensate for the attenuation of light through the 2-mm thickness of ceramic and the highly filled resin cement and resulted in much lower hardness values than that of other light-curing modes. It is difficult to compare the results of our study with other reports in the literature, as the materials, the light-curing lights, and exposure times selected are different. Nevertheless, many studies have shown that sufficient curing time as well as adequate light source energy would result in higher degree of conversion, polymerization depth, and hardness values.[29-31]

For the specimens polymerized directly under the Mylar strip, significantly higher top and bottom surface microhardness values were obtained for the dual-cured cement than that of the light-cured cement when cured only by the halogen light ($p < 0.05$). These results may indicate that the halogen light with a continuous and broader emission spectrum was more capable of inducing and complementing the chemical reaction by the catalyst in the Variolink Ultra resin cement when irradiated directly, compared with the LED lights for 20 or 40 seconds (with a 5-second ramp mode). Although limitations of LED lights for complete polymerization of some light-activated resin composites due to having a narrower distribution of light have been shown,[13,31] this may also need to be explored further for different types of resin-based luting agents.

By using the same resin cement and simulated ceramic restoration (lithium-disilicate-based glass-ceramic) throughout this study, any differences found in the microhardness data are attributed to the light-curing units. From our results, no statistically significant difference ($p > 0.05$) in the Vickers hardness of top or bottom surfaces for the light-cured resin cements irradiated either directly or through ceramic was found among the QTH (continuous for 40 seconds) and the LED lights for 20 or 40 seconds. Thus, it can be concluded that the curing efficiency of the high-power LED light with a shorter exposure time of 20 seconds in polymerizing the light-cured resin cement tested (Variolink Ultra Base) with high filler content was comparable to that of the high-power LED for 40 seconds or QTH light in both control and experimental groups. This is inconsistent with other investigations that showed the ability of high-power LED or QTH lights to reduce exposure times in curing the resin-based materials.[30,32] High-power light sources produce more photons for absorption by photosensitizers and cause more camphorquinone molecules to excite and react with amine, resulting in production of more free radicals for polymerization.[33] The high-power LED light (1100 mW/cm^2) compensated for the short curing time, and the increase of exposure time from 20 to 40 seconds had no significant effect in improvement of surface hardness for the light-cured resin cement.

When the Variolink Ultra base was mixed with the catalyst (dual cured), the mean microhardness value of the top surfaces for the specimens in the control group polymerized with the LED for 40 seconds was significantly higher than that of the LED for 20 seconds ($p < 0.05$). Considering the same LED light-curing unit used in these two test groups (the same light intensity and curing mode), only a longer exposure time of 40 seconds could effectively produce a higher top surface hardness for the dual-cured cement when irradiated directly under the Mylar strip; however, there was a similar decrease in the bottom surface hardness values for the two curing times of the high-power LED light used in this study.

Studies have shown that soft-start polymerization methods could reduce polymerization strains and improve material properties.[33,34] On the contrary, we found no increase in the Vickers hardness of light- or dual-cured resin cements polymerized by the high-power LED light with a 5-second ramp mode (total exposure of 20 or 40 seconds) compared with that of high-power QTH light with a continuous 40-second curing time.

There is no internationally recognized standard for adequate depth of polymerization as measured by the relative hardness method. It has been suggested that for adequate depth of polymerization, a relative hardness value (hardness of lower surface/hardness of upper surface $\times$ 100) must be higher than 80%.[26] The bottom-to-top surface hardness ratios were higher than 80% in all groups tested in this study, except for the light-cured cement polymerized through ceramic with the LED for 20 seconds (78.7%).

Conclusions

Microhardness values were significantly reduced after irradiation through the glass-ceramic disc of 2-mm thickness by any of the light-curing modes tested. The results showed improved surface hardness of dual-cured resin cement polymerized under ceramic over the light-cured cement by the high-power QTH or LED light-curing unit. The efficiency of the high-power LED light in polymerization of the resin-based cement with high filler content through the simulated ceramic restoration was comparable to the high-power QTH light only with a longer exposure time. The clinical implication of the results is that a reduced curing time of 20 seconds with high-power LED light for photopolymerizing the dual-cured resin cement under ceramic restorations of minimum 2-mm thickness is not recommended; however, it should be noted that the results obtained in this study may not be applied to other types of resin-based cements with different filler load, monomer, and photoinitiator-catalyst compositions.

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