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Influence of Different Exposure Times Required to Stabilize Hardness Values of Composite Resin Restorations

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Abstract

Aim: The purpose of this study was to evaluate if Knoop hardness values (KHN) for top and bottom surfaces of resin composite materials can reach a plateau within a clinically acceptable photoactivation time.

Methods and Materials: Four light-curing units (LCUs) were evaluated in this study (n=5): QTH (Optilux501: 550 mW/cm^2) and LEDs (FreeLight2: 1100 mW/cm^2; UltraLume5: 900 mW/cm^2; and Radii: 750 mW/cm^2). Composite resin discs (4 mm x 2 mm) of Heliomolar (Ivoclar/Vivadent) and Herculite XRV (Kerr) were tested using five photoactivation times (20, 40, 60, 80, and 100 seconds). KHN were obtained for each test specimen and comparisons between LCUs, depths, and photoactivation times were analyzed using two-way analysis of variance (ANOVA) and polynomial regression analysis.

Results: Data for Heliomolar discs using linear regression found a relationship between the independent variables KHN and time with the Optilux501 at the top and bottom surfaces (r^2=0.68/ r^2=0.66). Radii presented a linear regression at the top surface (r^2=0.75) and a quadratic regression at the bottom (r^2=0.94). A quadratic regression was also detected for UltraLume5 and FreeLight2 at both top (r^2=0.84/ r^2=0.94) and bottom surfaces (r^2=0.97/ r^2=0.90), respectively, reaching a plateau at 80 seconds in all cases. For Herculite XRV, a quadratic regression was observed for all LCUs at the top and bottom surfaces and 80 seconds irradiation time was needed to reach a plateau in KHN.

Conclusion: There is a specific, but not clinically acceptable, photoactivation time that KHN at both top and bottom surfaces can reach a plateau and is dependent on LCUs and the resin-composite tested.

Clinical Significance: The LCUs and the resin-composite formulation affected the exposure time required to stabilize hardness values. The overall performance of LED LCUs was better than the QTH LCU regardless of the material evaluated.

Keywords: Light curing units, Knoop hardness, resin-composites, curing time, LED, irradiance

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Introduction

Advances in light-curing technology have contributed to the extended use of resin composites in restorative dentistry. The most commonly used light curing unit (LCU) is the Quartz-Tungsten-Halogen (QTH) bulb, though light-emitting-diode curing units (LED) are gaining popularity. Most of the "first generation" LED units were unable to cure resin composites as accurately as QTH units.[1-3] However, LED technology has advanced allowing high power LED units to polymerize some resin composites as well as or better than some QTH units.[4,5] LED emitted irradiance is approximately 470 nm; this corresponds to the absorption peak of the camphoroquinone (CQ) spectrum.[3,6] However, concerns exist this emerging technology may not be able to fully cure different photo initiators to the proper depth. The quality of cure is an important parameter in determining the ultimate physical and mechanical properties of the resin-composite. Inadequate polymerization can result in inferior physical-mechanical properties such as poor resistance to wear, colour instability, increased rates of water absorption, increased solubility; all of which can result in poor clinical performance.[2,7,8]

There are concerns regarding the photoactivation times recommended by the manufacturers. These photoactivation times may not be adequate to fully cure all types of resin composites using some of the available LCUs.[3,4,9,10]

In order to produce resin-composite restorations with better physical-properties an increase in the photoactivation time has been proposed.[3,9,11] However, it is not clear if the hardness values can reach a plateau within clinically acceptable photoactivation times. On the other hand, some studies have shown an adequate cure with QTH curing units requires an irradiance of at least 300 to 400mW/cm^2, an increment no more than 2 mm, and a photoactivation time of 40-60 seconds.[9] Some curing units deliver irradiances higher than 400 mW/cm^2, and it has been shown these LCUs may offer a greater depth of curing in reduced photoactivation times.[4] High intensity LEDs may achieve acceptable performance reducing the photoactivation time.[4,12]

The purpose of this study was to determine if the photoactivation time for top and bottom surfaces of resin composites where hardness values can reach a plateau is clinically acceptable and if the amount of time is dependent on resin-composite and/or LCU. Three LEDs (all of high irradiance)

Table 1. Resin composites tested.

Composite	Classification	Filler Size	Composition
Heliomolar (Ivoclar/Vivadent)	Microfilled	0.04 – 0.2µm	**Filler:** 46 vol% silicon dioxide, ytterbium trifluoride and copolymer **Resin:** Bis-GMA, Bis-EMA, UDMA, TEGDMA
Herculite XRV (Kerr)	Microhybrid	0.6µm	**Filler:** 79 vol% Ba-Al-F-B, silicate glass **Resin:** Bis-GMA, TEGDMA

and one QTH were used to cure two resin composites materials. The null hypotheses tested were:

1. Hardness values cannot reach a plateau at top and bottom surfaces within clinically acceptable time regardless of LCU or resin-composite material.
2. Top and bottom composite surfaces cannot reach a plateau using the same photoactivation times.

Methods and Materials

Two commercial resin-composite materials (Table 1) and four light-curing units (LCUs) using five photoactivation times were investigated, resulting in eight experimental groups, each with five subgroups as presented in Figure 1 (n=5).

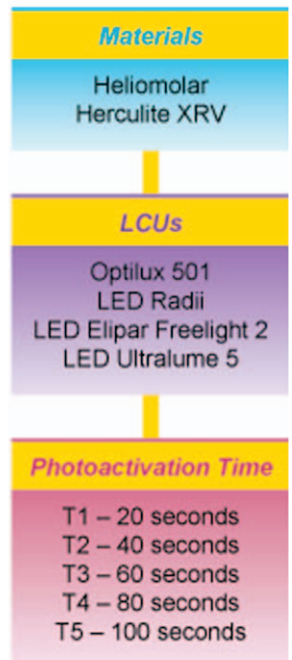

Figure 1. Experimental Groups Tested (n=5).

The optical power, in mW, of the four LCUs was measured using a power meter (Ophir Optronics Inc., Danvers, MA, USA) and the irradiance, in mW/cm^2, was calculated by dividing optical power by the area of the light guide tip. The spectral distributions of the LCUs were obtained using a spectrometer (USB 2000, Ocean Optics, Dunedin, FL, USA). The total irradiance data and the spectral distributions of the sources were tabulated in the software Origin 6.1 (OriginLab Corp. Northampton, MA, USA) to obtain, by integrated calculus, the specific light irradiance at the 450-490 nm wavelength range (Table 2).

The resin-composite materials were placed in metallic moulds with cylindrical cavities 4 mm in diameter and 2 mm deep. After insertion, the material was covered with a Mylar strip in order to provide a flat surface. Then, the samples were cured for 50% (20 seconds), 100% (40 seconds), 150% (60 seconds), 200% (80 seconds), and 250% (100 seconds) of the recommended photoactivation time. The tip of the curing light was positioned on the resin-composite surface. After light curing, the Mylar strip was removed and the specimens were immersed in water and kept in lightproof containers for 24 hours at 37°C before testing.

The Knoop hardness test was performed using a 25 g load for 20 seconds (Microhardness Tester, Future Tech FM-1E, Future Tech Corp., Tokyo 140, Japan). The indentations were placed at five points on both the top and the bottom surfaces. The larger diagonal length of the indentation was measured with a monitor (9M 100A Teli, Tokyo 140, Japan) and the values transformed in Knoop hardness numbers (KHN). The mean KHN for

Table 2. Light irradiance of the LCU's.

LCUs	Irradiance (mW/cm^2)
Optilux 501- Demetrom	550
Radii – SDI	750
Elipar Freelight 2 – 3M ESPE	1100
Ultra Lume 5 - Ultradent	900

each surface and for each experimental group was calculated.

The surfaces were imaged by high vacuum SEM (Quanta 200, FEI, Hillsboro, OR, USA), operating at 1 Torr pressure and 30 KeV and x 5000 magnification.

A two-way analysis of variance (ANOVA) with Tukey's post-hoc test (p=0.05) and polynomial regression analysis were used to compare Knoop hardness between LCUs, depths, and photoactivation times.

Results

The two-way ANOVA and Tukey's post-hoc test revealed differences among the LCUs for top and bottom surfaces (p=0.05). The influence of photoactivation time in the hardness values for each LCU and (top or bottom) surface was evaluated using the regression analysis.

Heliomolar Resin Composite

The Heliomolar discs in all cases presented lower hardness values than Herculite XRV discs independently of LCU or photoactivation time (Figures 2a-d).

Figure 2 shows the KHN for Heliomolar discs at the top (Figure 2a) and bottom (Figure 2b) in each combination of LCU and photoactivation time. UltraLume 5 LCU presented the highest values at the top surface. No differences were detected among Raddi, Elipar FreeLight 2, and Optlux 501 LCUs. By using 50% of the manufacturer's recommended time (20 seconds) no differences among the LCUs were observed at the bottom surface. For 80 and 100 seconds, UltraLume 5 LCU produced the highest values at top surface while no statistically significant differences were observed among the other LCUs. At the bottom surface, in general, the Optilux 501 LCU produced lower hardness mean values.

Figure 3 shows the results of the regression analysis. A linear regression of the independent variables KHN and photoactivation time was observed for Optilux 501 at the top and bottom surfaces (r^2=0.68/ r^2=0.66). Radii produced linear regression at the top (r^2=0.55) and quadratic regression at the bottom (r^2=0.94) with 80 seconds irradiation time needed to stabilize KHN values. A quadratic regression was also detected for UltraLume 5 and Elipar FreeLight2 at top (r^2=0.84/ r^2=0.94) and bottom (r^2=0.97/ r^2=0.90) with 80 seconds required to reach a plateau in KHN values in both cases.

Overall, a plateau was reached for LEDs only after 80 seconds for both top and bottom surfaces.

Herculite XRV Resin-Composite

Figure 2 shows the KHN for the Herculite XRV discs at both top (Figure 2c) and bottom (Figure 2d) in each combination of LCUs and photoactivation time.

According to the results using 20 and 40 seconds of photoactivation time, no statistically significant differences were observed at the top or bottom surface among the LCUs tested.

For 60, 80, and 100 seconds at the top surface, LEDs always presented higher values than QTH for either top or bottom surface.

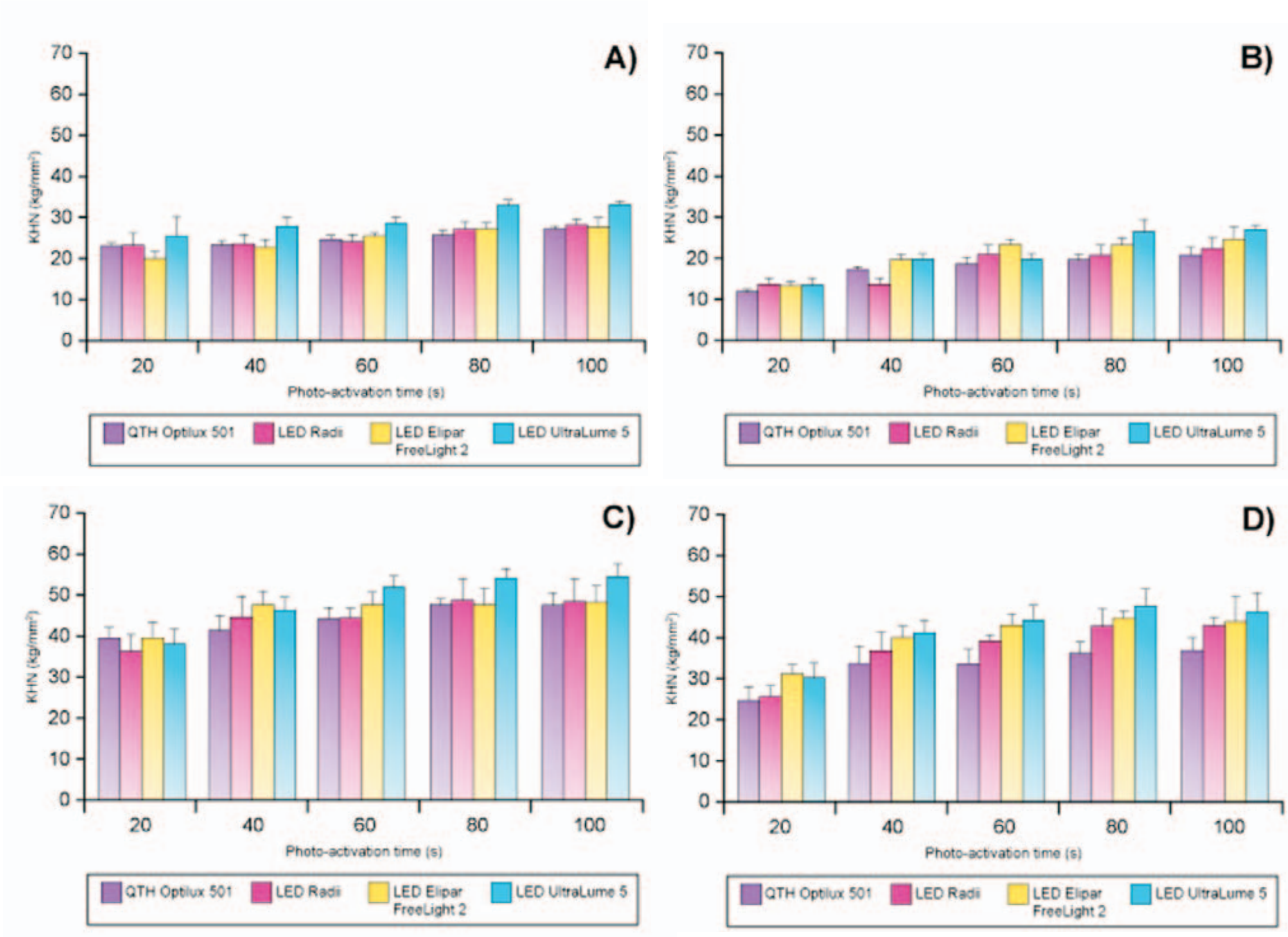

Figure 2. KHN values at (a, c) top and (b, d) bottom: LCUs versus photoactivation times.
(a) Heliomolar: top surface; **(b)** Heliomolar: bottom surface; **(c)** Herculite XRV: top surface; **(d)** Herculite XRV: bottom surface

Figure 3 shows the results of the regression analysis. A quadratic regression of the independent variables KHN and photoactivation time was observed for all LCUs tested at the top and bottom surfaces, 80 seconds irradiation time was needed to reach a plateau in KHN values (Optilux 501 r^2= 0.70/ r^2=0.76; Radii r^2= 0.98/ r^2=0.89; Elipar FreeLight 2 r^2= 0.81/ r^2=0.90; and UltraLume 5 r^2= 0.99/ r^2=0.99).

Overall, a plateau was reached for all LCU's after 80 seconds for both top and bottom surfaces. The spectra for all LCUs are shown in Figure 4. Figure 5 illustrates the SEM images for both resin-composite materials.

Discussion

There is limited information in the literature evaluating if there is a specific and clinically acceptable photoactivation time at which hardness values reach a plateau. According to the results of this study, the hardness values can, in some cases, reach a plateau within a specific photoactivation time, but these values are LCUs and resin-composite formulation dependent. Hence, the first hypothesis was rejected.

A plateau in hardness values was reached for all resin-composite/LCUs' combinations with the exception of Heliomolar/Optilux 501 (top and bottom) and Heliomolar/Radii (top). In all cases where the plateau was reached, it was achieved at the same photoactivation time (80 seconds) for top and bottom surfaces, regardless of the LCUs or resin-composite formulation tested. Hence, the second hypothesis was rejected. The maximum difference between top and bottom hardness values was at the 20 seconds and 40 seconds photoactivation times (Figure 2) for all LCUs and both resin-composite materials.

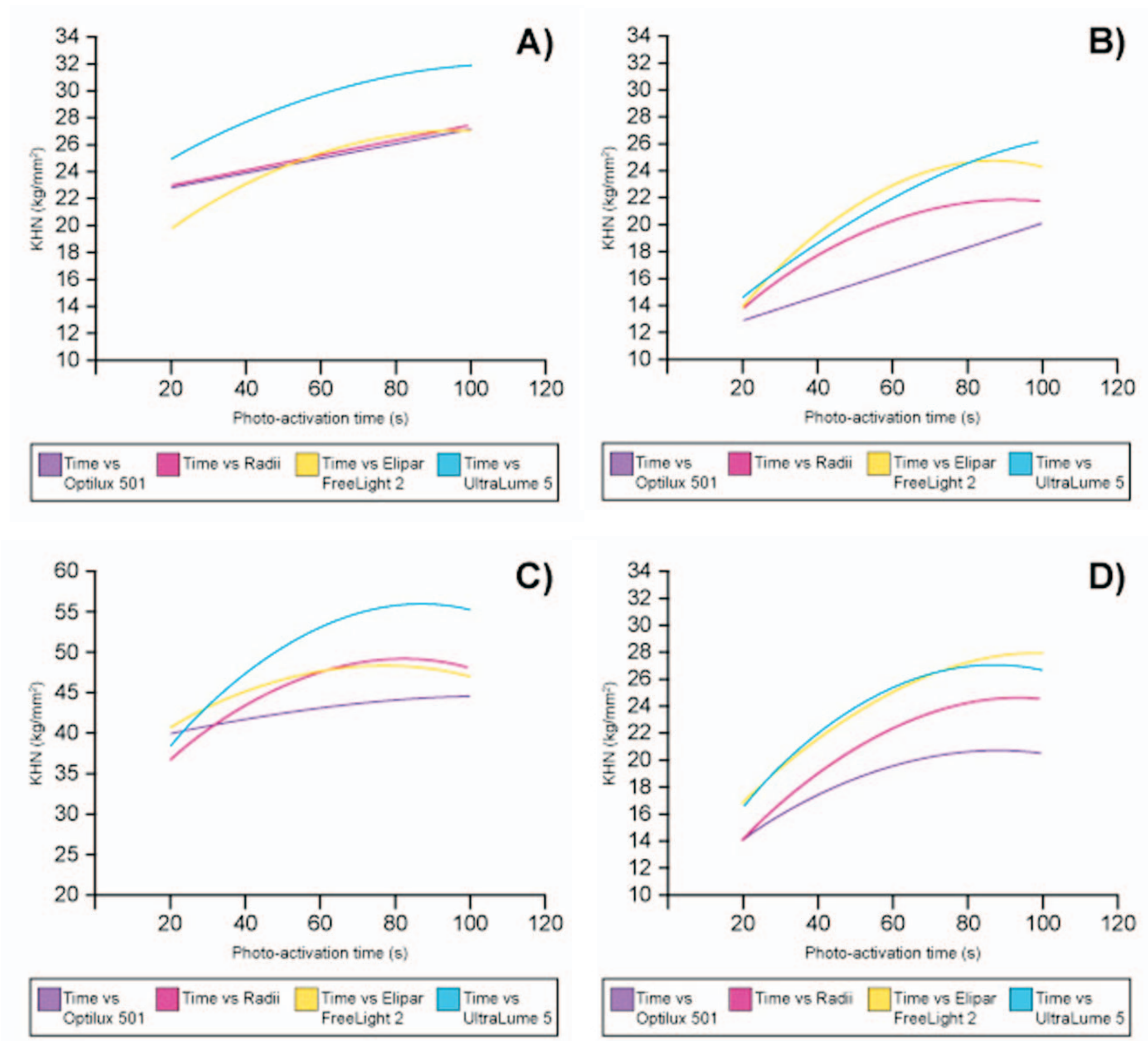

Figure 3. KHN of the resin composites at top and bottom surfaces versus photoactivation times and polynomial regression analysis by using different LCUs. **A.** Heliomolar: top surface B. Heliomolar: bottom surface. **C.** Herculite XRV: top surface. **D.** Herculite XRV: bottom surface.

As the photoactivation time increased (60-100 seconds), this difference decreased. The increase in photoactivation time is directly related to an increase in radiant exposure (irradiance x photoactivation time). When irradiance and photoactivation time increase, the hardness and degree of cure values also increase. As a result, a material with better mechanical properties can be obtained as is evident from the results of this study and other studies in the literature.[13,14] Other studies have shown it is possible to reduce the photoactivation time when using LCUs with high intensities such as second generation LEDs.[4,12]

The reduction on photoactivation time is based on the total radiant exposure concept that a certain dose (irradiance x photoactivation time) of light is needed to adequately cure a specific material. However, this may not be applied for all kinds of composites as well as LCUs.[10] It has been shown a high irradiance combined with a short photoactivation time may reduce the degree of cure and the kinetic chain length.[15] This may also increase the frequency of cross-linking compared with curing with intermediary irradiances for long exposures that can be detrimental to the mechanical properties of the composite.[8,16]

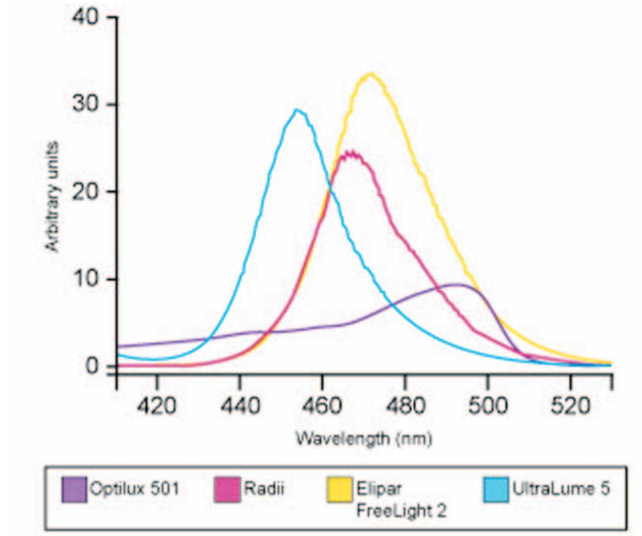

Figure 4. Spectrum distribution of light curing units.

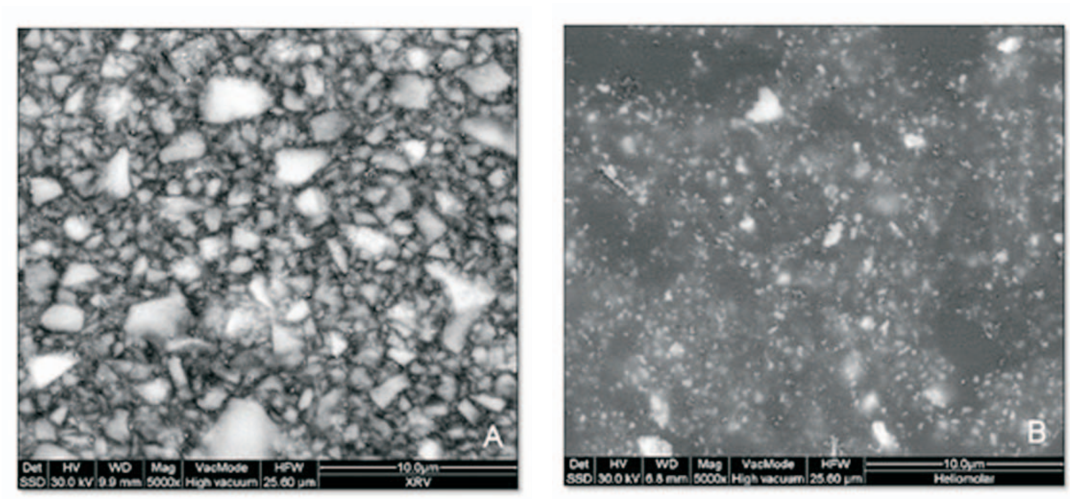

Figure 5. Filler content of composites tested (SEM). **A.** Herculite XRV. **B.** Heliomolar.

Absorption and scatter within the material are the major factors associated with light attenuation[17,18] and can result in a low depth of cure and reduced degree of conversion.[18] These factors are dependent on the material's composition and also on the LCUs' spectrum.[17,18] Light-scattering within the resin-composite material increases as the particle size of the fillers approaches the size of the wavelength of the activating light.[17,19,20] Ruyter has suggested scattering is maximized when the filler size is one-half of the incident's light wavelength.[19] Furthermore, 'Rayleigh scattering equation' states that higher scattering occurs at lower wavelengths.[19] Heliomolar is a microfilled resin-composite containing fillers of 0.04-0.2 μm size which corresponds to one-half of the QTH maximum wavelength as can be observed in Figure 5(a). The combination of Heliomolar/Optilux 501 did not reach a plateau in hardness values either at the top or bottom surfaces even after 100 seconds of photoactivation time. On the other hand, the LED units operating in higher wavelengths reached a plateau in hardness values for the Heliomolar material in 80 seconds photoactivation time both at top and bottom surfaces.

Interestingly, in the case of the Herculite XRV/Optilux 501 combination a plateau was reached after 80 seconds. This result can be attributed to less scattering in the Herculite XRV compared with Heliomolar resin-composite. According to a quadratic regression, 80 seconds of photoactivation time produced a plateau in hardness values both at top and bottom surfaces for all LCUs tested for Herculite XRV. SEM images [Figure 5(a) and (b)] are in agreement with the composition data (Table 1) showing Herculite XRV has a higher filler size than Heliomolar, and this size corresponds to more then one-half of the LCUs' wavelength used. Furthermore, a long photoactivation time can be uncomfortable for both dentists and patients and might increase the cost of the treatment. It is generally believed that 80 seconds is not a clinically acceptable photoactivation time. On the other hand, a reduction in the manufacturer's recommended exposure will save the clinician and the patient's time but it may compromise the long-term mechanical properties of the material. The aim of this study was not to create a new photoactivation protocol but to monitor the behavior of the tested materials along the different time intervals.

Conclusion

Therefore, one can conclude there is a specific, but not clinically acceptable, photoactivation time at which KHN at the top and bottom surfaces might reach a plateau. However, this time is LCU and resin-composite formulation dependent. The overall performance of LED LCUs was better than the QTH LCU for both resin composites tested.

Clinical Significance

The LCUs and the resin-composite formulation affected the exposure time required to stabilize hardness values. The overall performance of LED LCUs was better than the QTH LCU regardless of the material evaluated.

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