Polymerization of Composite Resin Using Different Light-curing Units by Direct and Indirect Techniques

Alessandra N. S. Rastelli[a], Marcelo F. Andrade[b], Vanderlei S. Bagnato[c]

[a] *Researcher in Physics and Materials Science, Physics Institute of São Carlos, University of São Paulo, SP, Brazil.*
[b] *Assistant Professor, Department of Restorative Dentistry, Araraquara School of Dentistry, University of São Paulo State, UNESP, SP, Brazil.*
[c] *Titular Professor, Physics Institute of São Carlos, University of São Paulo, SP, Brazil.*

Purpose: The aim of this study was to evaluate the curing efficiency, through tooth slices (0, 1.2, 1.5 and 2.0 mm), of three different light sources.

Materials and Methods: Three caries-free mandibular 3rd molars were used. After the roots were cut from the coronal portions, slices of different thickness (1.2 mm, 1.5 mm, 2.0 mm) were made by cutting the buccal face of the dental crowns. Then, samples were made in a metallic mold (4 mm diameter and 2 mm thickness) with a microhybrid composite resin (Charisma; Heraeus-Kulzer). The samples (n = 140) were divided into 3 groups: group I – halogen LCU for 20 and 40 s; group II – argon laser LCU for 20 and 40 s; group III – LEDs LCU for 20, 40, and 60 s. The samples were cured either directly (no tooth slice intervening; control), or indirectly (through tooth slices of different thickness). The samples were stored dry at 37°C for 24 h, and then Vickers microhardness testing was performed.

Results: The data were submitted to ANOVA ($p < 0.05$). Standard deviation values from the microhardness measurements were less than 1% of the mean and showed a statistically significant difference among the light curing units and the cure through the tooth slices of different thicknesses.

Conclusion: In the indirect photoactivation technique, it was necessary to increase the exposure time independent of the LCUs used mainly for the bottom surface. Under these experimental conditions, the LED curing unit promoted the lower microhardness values when compared to the halogen and argon laser LCUs.

Keywords: composite resin, microhardness, light emitting diodes, halogen light curing unit, argon laser, curing depth.

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The constant development of composite resins used as esthetic restorative materials, both in anterior and increasingly in posterior teeth, has become one of the greatest forces for progress in esthetic dentistry.

Since the introduction of the monomer bis-GMA (bisphenol A-glycidyl methacrylate) system in 1962 by Bowen, the composite resins have undergone several changes in their compositions, as well as in the activation system of the curing reaction to improve their physical, chemical and biological properties, thus increasing the longevity of the restorations. Adequate curing is very important to obtain a restoration which provides better clinical performance.[1-3]

The curing reaction of the composite resins takes place by the activation of the photoinitiator component (camphorquinone), which needs enough luminous in-

Table 1 Light-curing units and technical parameters

Light-curing unit	Power (mW)	Light intensity (mW/cm^2)	Wavelength (nm)	Light tip (cm)	Exposure time (s)
Halogen KM-200R unit, DMC-São Carlos/SP, Brazil - serial number 1374	440	875.8	380-525	0.8	20-40
Argon laser Innova 100 unit, Coherent; Santa Clara, CA, USA, Model I 200, – serial number 3240	25	200	488	0.4	20, 40
LED LEC 470 I unit, MMOptics-São Carlos, SP, Brazil, serial number F 0545	40	80	470 (±20)	0.8	20, 40, 60

tensity to keep it in an excitement or triplet status. Therefore, only when the camphorquinone is in the excitement or triplet state, is it able to react with the reducing agent (amine-N), yielding free radicals, which will start and provide adequate curing.[4-5,8-13] However, the polymerization process only takes place if the light has a wavelength of 450 to 500 nm. According to Nomoto,[12] the absorption spectrum of this photoinitiator is achieved in the interval from 450 to 500 nm, with the most adequate wavelength situated between 450 and 490 nm, and the most efficient wavelength for the activation of the polymerization reaction situated between 468 and 470 nm. When the camphorquinone is excited, a chain reaction is established, giving the composite resins the desired mechanical characteristics for their clinical applications.[12]

Many factors influence the light curing process, such as the light-curing unit used, light intensity emitted by the unit, wavelength, exposure time, post-activation period, color and thickness of the composite resins, as well as external factors such as the presence of dental tissue.[4,13-17]

The development of techniques and units for the photoactivation process has greatly influenced the curing process of the composite resins. For instance, manufacturers have developed new light-curing units which present additional characteristics when compared to the halogen light curing sources. Argon laser light curing units are coherent and photoactivate composite resins at 10 s or less.[18-20] It has been proposed that, because of the monochromacity of the argon laser set at 468 nm and a much greater intensity of irradiation, less light attenuation may occur due to saturation in the depths of composite resins, as compared to halogen light curing units with much lower intensities, thus resulting in a greater extent of polymerization.

Recently, light emitting diodes (LEDs) that emit visible blue light in a wavelength around 470 nm (± 20 nm) have been investigated. The curing efficiency of LEDs is explained by the better match of their emission spectra with the standard photoinitiator camphorquinone (CQ) than the broad spectra of halogen light curing units (LCUs).[30-34]

Photoactivated composite resins require an adequate amount of light energy to polymerize. If the curing light emits a low intensity light or wavelengths outside the curing range of the photoinitiator, the composite resin will not adequately polymerize. Inadequate curing may increase the wear, cytotoxicity, and marginal breakdown, and may result in an uncured layer of composite resin that is soft and inadequately bonded to the tooth.[24]

It has been reported that the light is not transmitted well through composite resins and through teeth. Thus, the photoactivation of the composite resins through dental tissue, enamel and/or dentin, is related to the

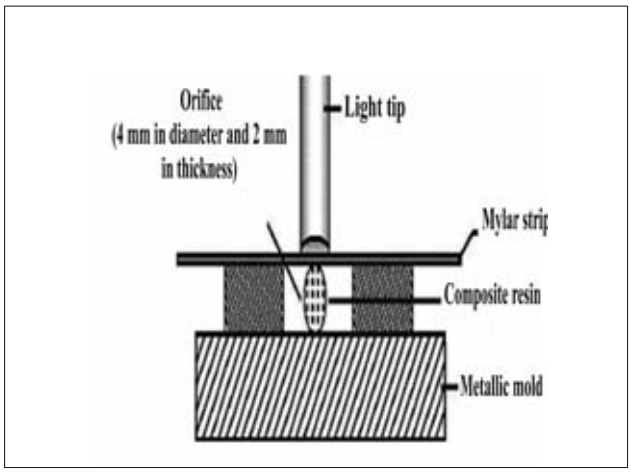

Fig 1 Sample preparation without the tooth slice (control groups).

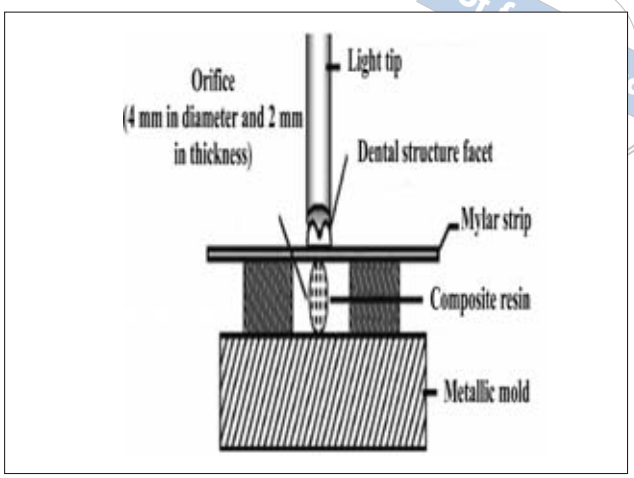

Fig 2 Sample preparation with the tooth slice (labelled "dental structure facet" in the figure) (1.2, 1.5, or 2.0 mm thickness).

curing depth of these materials and promotes a reduction in the microhardness values, depending on the thickness of the dental tissues.[6,8,21-23]

Thus, the purpose of this study was to evaluate the polymerization by microhardness measurements of the microhybrid composite resin photoactivated directly (no dental tissue) and indirectly through tooth slices, with the use of halogen, argon laser, and LEDs light curing units used for different exposure times.

MATERIALS AND METHODS

Three different light sources were used in this study (Table 1) for direct and indirect light-curing techniques. All LCUs (light curing units) were used in the continuous modes (Table 1).

For preparation of the tooth slices, three recently extracted, caries-free mandibular third molars, were selected (protocol number 61/01 Research Ethics Committee, Araraquara School of Dentistry, UNESP/SP, Brazil). After the extraction, the teeth were stored in 0.5% chloramine solution for 24 h. After they were rinsed and gross debris was removed, the teeth were again stored in chloramine solution at 0.5% for 7 days. The coronal portions of the teeth were separated from their roots at the cementoenamel junction level using a 3-mm-thick diamond disk mounted in a cutting machine (Isomet 1000, Buehler; Lake Bluff, IL, USA). The tooth slices were obtained by cutting the buccal face of the dental crowns. The tooth slices (1.2 mm, 1.5 mm, and 2.0 mm) were ground flat with wet 1200-grit silicon carbide paper (3M; St Paul, MN, USA) mounted in a manual polishing machine.

To measure the thickness of the tooth slices, a digital caliper was used (Brown & Sharpe, model no 599-571-3; USA). The tooth slices remained stored in distilled water until sample preparation.

The samples were made in a circular metallic mold. The central orifice measuring 2 mm thickness and 4 mm diameter (ISO 4049)[24] was packed with the Charisma (Heraeus-Kulzer, batch number 010040; Hanau, Germany) composite resin, shade A2. According to the manufacturer, Charisma microhybrid composite resin is a radiopaque microglass with Ba-Al-F glass particles (0.02 to 2 μm), silicon dioxide highly dispersed (0.02 to 0.07 μm). The average size of the particles is 0.7 μm.

A mylar strip covered the top surface of the composite resin, without the interposition of the tooth slice (control groups, direct light-curing technique, Fig 1) or with the tooth slice (other groups, indirect light-curing technique, 1.2, 1.5, and 2.0 mm thickness, Fig 2). Five (n = 5) samples were made for each group. After photoactivation, the samples were stored in dry conditions at 37°C for 24 h.

Microhardness measurements were performed 24 h after the photoactivation of the samples. The samples were divided into 4 equal quadrants. The microhardness was determined on the top and the bottom surfaces. The microhardness tester used was the MHT-10 Microhardness Tester Anton Paar (Paar Physica, USA;

Table 2 Vickers microhardness means as function of the tooth slice thickness, LCUs and exposure times for the top surface

Light-curing sources	Exposure times (s)	Thickness (mm) 0.0	1.2	1.5	2.0
Halogen	20	128.1	116.8	96.2	78.9
	40	142.3	134.7	128.1	116.3
Argon laser	20	125.4	119.7	93.9	79.5
	40	141.3	134.9	127.6	113.7
LEDs	20	110.3	83.4	47.4	36.7
	40	135.4	124.8	68.9	58.2
	60	148.2	135.3	124.9	87.7

Table 3 Vickers microhardness means as function of the tooth slice thickness, LCUs and exposure times for the bottom surface

Light-curing sources	Exposure times	Thickness (mm) 0.0	1.2	1.5	2.0
Halogen	20	90.9	81.6	72.6	53.3
	40	122.0	113.2	100.6	85.6
Argon laser	20	70.3	42.2	39.4	28.9
	40	101.7	83.5	68.3	58.4
LEDs	20	53.4	29.1	18.8	11.2
	40	88.5	60.7	43.5	36.2
	60	113.5	82.3	72.8	54.5

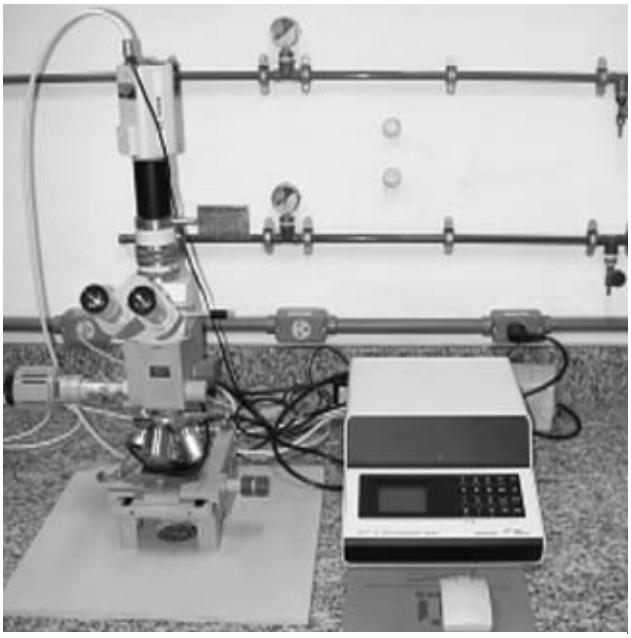

Fig 3 Microhardness testing machine (MHT-10 Microhardness Tester Anton Paar - Paar Physica, USA).

Fig 3) equipped with diamond Vickers, where 50 gf load (gram force) was used for 30 s. On both top and bottom surfaces, three impressions per quadrant were made.

The microhardness value means were calculated and the data were compared using ANOVA ($p < 0.05$).

RESULTS

Tables 2 and 3 show the Vickers mean microhardness values for the different LCUs (halogen, argon laser and LEDs) used in the photoactivation of the microhybrid composite resin by the direct (no tooth slice) and indirect techniques with different thicknesses of tooth slice (1.2, 1.5, and 2.0 mm) and exposure times (20, 40, and 60 s) for the top and bottom surfaces, respectively.

The mean microhardness values obtained after halogen LCU polymerization (Tables 2 and 3) were significantly greater than those obtained by the argon laser

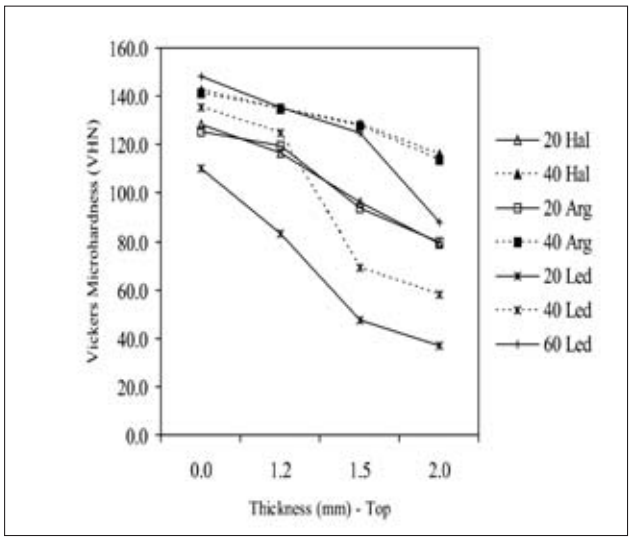

Fig 4 Vickers mean microhardness values (VHN) as function of the thickness of the tooth slices for the different LCUs (Hal = halogen, Arg = argon laser) and exposure times, for the top surface.

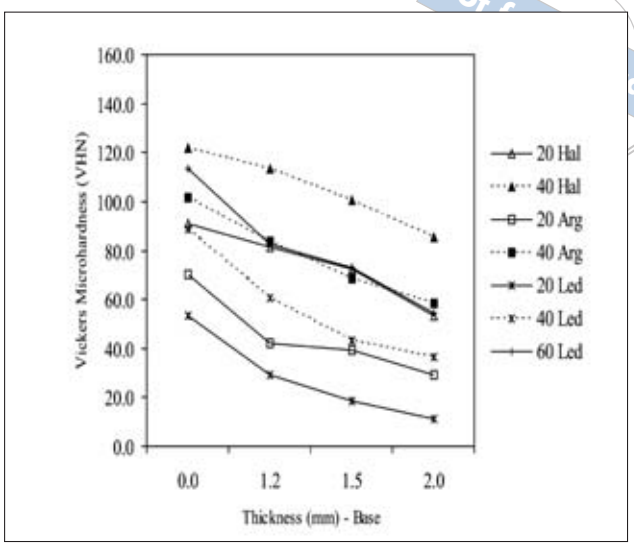

Fig 5 Vickers mean microhardness values (VHN) as function of the thickness of the tooth slices for the different LCUs (Hal = halogen, Arg = argon laser) and exposure times, for the bottom surface.

and LED LCUs at a 0.05 level of significance. Three-way ANOVA revealed a significant difference when different LCUs were used for polymerization through different thicknesses of tooth slices (indirect technique) and exposure times for the top and bottom surfaces.

The different thicknesses of the tooth slices decreased the mean microhardness values for the top and bottom surfaces. Significantly lower values were obtained for the LED LCU through the 2.0-mm tooth slice on the bottom surface.

The greater exposure times to the different LCUs provided an increase in the mean microhardness values (Figs 4 and 5). The combination of mean values of the LCUs and exposure times were significantly different, except for the halogen and argon laser, both with 40 s of exposure time and with the tooth slices 1, 2 and 1.5 mm thick, for the top surface.

In almost all comparisons, the probability values obtained were much smaller than 0.05, indicating great statistical significance. It happened because the standard deviation of the microhardness measurements in almost all groups was under 1% of the average. In this way, the residual variation of the ANOVA was also very small. However, it became evident that in each combination of LCU, thickness of the tooth slice, and exposure time, the microhardness values were lower for the bottom than the top surface (Figs 4 and 5).

DISCUSSION

Adequate polymerization of the composite resins is related to its clinical performance. The clinical survival of the composite fillings is influenced by many factors, several are related to the photoactivation procedure. One limitation of the photoactivated composite resins is that a hard top surface is not an indication of adequate polymerization throughout the depth of restoration.

The polymerization depends on various parameters, such as light intensity, power, wavelength, exposure time, distance between the LCU and the composite resin, type of base resin and filler particles, quantity of diluent TEG-DMA and camphorquinone, transmission and absorption coefficient, and placement technique. Inadequate polymerization can lead to undesirable effects such as gap formation, marginal leakage, recurrent caries, adverse pulpal effects, and ultimately, failure of the restoration. Effective polymerization is important not only to ensure optimum physicomechanical properties, but also to ensure that clinical problems do not arise due to the cytotoxicity of inadequately polymerized material. Greater polymerization eliminates the need to refill a cavity preparation with several layers of composite resin.

Polymerization effectiveness may be assessed directly or indirectly. Microhardness testing is an indirect and important tool for characterizing the near-surface characteristics of composite resins. This testing appears to be the most popular method for investigating factors that influence polymerization effectiveness with relative simplicity. The purpose of microindentation hardness testing is to obtain a numerical value that distinguishes between the relative ability of materials to resist controlled penetration by a specified type of indenter which is generally much harder than the material being tested.

Tables 2 and 3 show the Vickers mean microhardness values for the top and bottom surfaces. Considering that the satisfactory microhardness value for the composite resin used in this study is around 100 Vickers, the mean values decreased with the increasing thickness of the tooth slice, independent of the LCU used. Figures 4 and 5 show these results. In agreement with our results, Dietschi et al,[7] Vogel and Saltz,[20] and Price et al,[21] found the mean microhardness values to decrease when the thickness of the tooth slice was increased. The results observed in this study can be explained by the fact that the light emitted by the LCUs was not well transmitted through the tooth slices.

The thickness of the composite resin and the distance of the LCUs are factors which reduce the light intensity. According to Atmadja and Bryant[13] and Weaver et al,[15] when the composite resins were photoactivated through the dental structure, they presented microhardness values inversely proportional to the thickness of the composite resin and the dental structure. The interposition of the enamel facets between the light tip and the restorative material promotes a reduction of the curing depth and the microhardness values of the composite resins. The greater thickness of the dental structures between the first layer of composite resin and the light tip decrease the polymerization of the composite resins.

The results obtained in this study showed that higher microhardness values were obtained for the top surface than the bottom (Fig 4). For the top surface, the Vickers microhardness values were greater using the halogen LCU and argon laser than using the LED. This fact was explained by the light intensity values used in this study.

If a photoactivated composite resin does not receive a sufficient number of photons at the correct wavelengths, the amount of polymerization and degree of conversion will be inadequate.

The mean microhardness has been found to be inversely proportional to the thickness of the composite resin, and also to the distance of the light source to the restorative material.[6] As the thickness increases, the number of photons available to transform camphorquinone to its activated state in a light-cured resin is reduced as the photons are absorbed and scattered. The amount of light energy present at the base of the composite resin restoration is important, because if insufficient camphorquinone molecules are being raised to the triplet state, the composite resin is not adequately polymerized and the microhardness is reduced.

The polymerization of the composite resin is considered an indication of the clinical success of a composite resin restoration.[22] The polymerization of the photoactivated composite resins decreased when the depth of the restoration was increased; however, it is important to achieve adequate polymerization at all depths of the restorative material.[23]

For the factor exposure time, it is evident that the increase in the exposure time provides higher microhardness values. The results of this study showed that the higher exposure times provided a significant increase in the mean microhardness values, for both the top and bottom surfaces, independent of the LCU used (halogen, argon laser and LED). The exposure time plays a fundamental role in the polymerization process of composite resins. The superficial microhardness is significantly increased when exposed to light for longer periods.[13-14]

According to other studies, when an argon laser LCU was used, the reduced exposure time did not seem to affect the mechanical properties of the composite resins.[25-28] When the composite resins were exposed to the argon laser LCU for 20 s, the microhardness values were equal or even superior to those of resins photoactivated by the halogen LCU for 40 s, depending on the parameters used for both LCUs.[19,25-28] However, the main drawback of the use of argon laser LCUs for light polymerization is related to the high cost when compared to conventional halogen LCUs. However, in this study, the best results were not obtained with the use of the argon laser, due to the parameters used for this LCU.

The use of blue LEDs to promote the polymerization of the composite resins has been investigated.[29-33] In this study, the LCU based on LEDs had an array with 6 units of LEDs. Table 2 and Fig 4 show that, compared to the other two LCUs, only the 60-s exposure time with the LED LCU provided adequate polymerization for the top surface, except with the 2.0-mm-thick tooth slice.

Nowadays, several of the LCUs used in dental practices have a halogen light source. However, the light

emitted by conventional units is not as effective in promoting the polymerization of the composite resins as is the light emitted by argon laser and blue LED units, in terms of fluency and light selectivity.

The halogen LCU used in this study emits radiation at wavelengths which are ineffective in promoting photopolymerization. These should be removed by more selective filtration. However, this component can be degraded with use. The smallest luminous selectivity verified on the conventional LCU can lead to greater heating of both the restorative material and of dental structures, irreversible alteration of the pulp tissue, and higher polymerization shrinkage of the composite resin.

In contrast, the emission spectrum of the investigated LEDs is strongly correlated to the absorption spectrum of the camphorquinone photosensitizer. The LED LCU has advantages over the halogen LCU. The non-induction of thermal change of the composite resin and of the dental structure, greater light selectivity, longer lifetime and a more favorable cost:benefit ratio.[29-33]

On the other hand, using the LED LCU to photoactivate 2-mm-thick composite samples through 2-mm-thick enamel/dentin tooth slices simulates the clinical situation in complex direct restorative procedures, resulting in strongly reduced microhardness at the base of the samples. The dentin greatly attenuates the visible light. Our results showed that when the composite resin was photoactivated by the indirect technique through a 2-mm-thick tooth slice, the microhardness values were not adequate.

CONCLUSION

In the indirect photoactivation technique, it was necessary to increase the exposure time independent of the LCU used, mainly for the bottom surface. The LED LCU, with the parameters used in this study, promoted lower microhardness values than did the halogen and argon laser LCU, but it may still be a viable LCU for the photoactivation process. Rather, meticulous guidelines with respect to exposure times must be established for each single clinical indication and specific brand to ensure the proper photoactivation of restorations.

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Contact address: Prof. Dra. Alessandra Nara de Souza Rastelli, Physics Institute of São Carlos, University of São Paulo, São Carlos, SP, Brazil, Trabalhador São Carlense, 400, Mailbox: 369, Zip code: 13566-590. Tel: +55-(016)-3371-2012, Fax: +55-(016)-3373-9811. e-mail: alerastelli@yahoo.com.br