



ORIGINAL ARTICLE

Effect of different light-curing techniques on hardness of a microhybrid dental composite resin

Efeito de diferentes técnicas de fotoativação na dureza de uma resina composta dental microhíbrida

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ABSTRACT

Objective: This study assessed the Vickers hardness (VHN) provided by two LCUs when using (1) direct and indirect light-curing techniques, (2) 40 and 60 s and (3) top and bottom surfaces. **Material & Methods:** One halogen Curing Light 2500 (3M Espe) and one LED (MM Optics) were used by direct and indirect (0, 1.0, 2.0 and 3.0 mm of dental structure) techniques during 40 and 60 s. The samples were made with FiltekTM Z250 in a metallic mould with a central orifice (4 mm in diameter, 2 mm in thickness). The samples were stored in dry mean by ± 24 h and the hardness measurements were performed in a testing machine (Buehler MMT-3 digital microhardness tester Lake Bluff, Illinois USA). A 50 gf load was used and the indenter with a dwell time of 30 s. The data were submitted to multiple ANOVA and Newman-Keuls's test ($p < 0.05$). **Results:** Halogen LCU exhibited higher Vickers hardness values than LED mainly because of the power density used. Hardness values were influenced by LCUs, light-curing techniques, irradiation times and surfaces. For both LCUs, hardness values were found to decrease with indirect light-curing technique, mainly for the bottom surface. Samples irradiated for 60 s exhibited higher hardness values when the halogen LCU was used. For 60 s, the VHN values were statistically significant greater than 40 s. Significant differences in top and bottom surfaces Vickers hardness number (VHN) values were observed among different LCUs used 40 and 60 s. **Conclusion:** The LCUs, light-curing techniques, variations of irradiation times, and surfaces (top and bottom) influence the composite resin hardness.

KEYWORDS

Composite resin, LED, Halogen lamp, Hardness, Photo-activation.

RESUMO

Objetivo: Este estudo avaliou a dureza Vickers (VHN) em função de duas fontes de luz quando utilizadas diferentes (1) técnicas de fotoativação, direta e indireta, (2) 40 e 60 s, e (3) superfícies de topo e base. **Material e Métodos:** Uma fonte de luz halógena Curing Light 2500 (3M Espe) e um LED (MM Optics) foram utilizados nas técnicas de fotoativação direta e indireta (0, 1,0, 2,0 e 3,0 milímetros de estrutura dental), durante 40 e 60 s. As amostras foram feitas utilizando-se FiltekTM Z250 em matriz metálica com orifício central (4 mm de diâmetro, com 2 mm de espessura). As amostras foram armazenadas em meio seco por ± 24 h e as medidas de dureza foram realizadas em microdurômetro (Buehler MMT-3 digital microhardness tester Lake Bluff, Illinois USA). Uma carga de 50 gf durante 30 s foi utilizada. Os dados foram submetidos à análise de variância múltipla e teste de Newman-Keuls ($p < 0,05$). **Resultados:** A fonte de luz halógena promoveu os maiores valores de dureza Vickers, principalmente, em função da densidade de potência utilizada. Os valores de dureza foram influenciados pelas fontes de luz, técnicas de fotoativação, tempos de irradiação e superfícies, topo e base. Para ambas as fontes de luz, os valores de dureza diminuíram com a técnica de fotoativação indireta, principalmente para a superfície de base. Amostras irradiadas por 60 s apresentaram valores de dureza maiores quando a fonte de luz halógena foi utilizada. Durante 60 s, os valores de VHN foram estatisticamente significativos maior do que 40 s. Diferenças significativas foram observadas nos valores dureza Vickers (VHN) para as superfícies de topo e base utilizando 40 e 60 s. **Conclusão:** As fontes de luz, técnicas de fotoativação direta e indireta, tempos de irradiação e superfícies (topo e base) influenciam na dureza da resina composta.

PALAVRAS-CHAVE

Resina composta, LED, Lâmpada halógena, Dureza, Fotoativação.

INTRODUCTION

The generally preferred mode of polymerization in dental composite resins is photo-activation method [1]. Effectiveness of the polymerization is one important meaning to obtain adequate physical properties.

One limitation of photo-activated dental composite resins is that a hard top surface is not an indication of adequate polymerization throughout the depth of restoration [2,3]. Poorly polymerized composite resin can lead to undesirable effects, such as: gap formation, marginal microleakage, recurrent caries, adverse pulpal effects and ultimate failure of restorative procedure [2,3]. Effective polymerization is important not only to ensure optimum physical-mechanical properties, however also to ensure that clinical problems do not arise due to the cytotoxicity of inadequately polymerized material [4, 5-7].

Many factors affect the polymerization effectiveness. These factors can be related to the material's composition, resin chemistry, shade, translucency, catalyst concentration, power density, spectral distribution of the light source, irradiation time, absorption coefficient and placement technique [8].

Now, light-curing units (LCUs) and light curing methods have been in constant evolution. Light curing of composite resins with blue light has proven to be the best photo-activation method and can be made with different light-curing sources [9]. Four light-curing sources have been clinically applied: quartz tungsten halogen (QTH) lamps, light emitting diodes (LED) units, plasma-arc lamps and argon-ion lasers [10,11]. However, halogen lamp and LED LCUs are overwhelmingly applied in daily clinical practice [10]. However, the most widely used light-curing units (LCUs), a low cost technology, are based on quartz tungsten halogen lamps (QTH) [12]. The main radiant output from a QTH LCU is infrared energy, which may be absorbed by dental composite resins and results in an

increased molecular vibration and consequently heat generation. Thus, the QTH LCUs need of use of filters to reduce the passage of infrared energy from the LCU to the tooth. However, the filter degrades over time due to high operating temperatures and significant heat produced during curing cycles [13]. Thus, unfiltered infrared energy can result in heat generation at the pulp chamber [14]. In addition, the halogen bulbs have a limited effective lifetime of about 40-100 h and reflectors too degrade over time. Then, the drawbacks of the halogen LCU will reduce the effectiveness of polymerization in composite restoratives.

LED (light-emitting diodes) light-curing units (LCUs) developed to overcome the problems inherent to halogen LCUs, use junctions of doped semi-conductors (p-n-junction) to generate light [15,16]. Under proper forward biased conditions, electrons and holes recombine at the LEDs p-n junction leading, in the case of gallium nitride LEDs, to the emission of blue light. A small polymer lens in front of the p-n junction partially collimates the light. As spectral output of gallium nitride blue LEDs falls within the absorption spectrum of the camphorquinone photo-initiator, no filters are required in LED LCUs. The absorption spectrum of camphorquinone lies in the 450-500 nm wavelength range, with peak absorption at 470 nm [17-19].

The basic composite technique insertion and photo-activation protocol usually recommends the use of increments not thicker than 2 mm to provide an effective polymerization. Further, the light guide should be as close as possible to the composite surface to guarantee the light will not be dissipated. However, some clinical situations present a real challenge to the utilization of these recommended polymerization techniques, such as accessing the floor of Class II proximal boxes where the distance between the light guide and the material surface is generally greater [20]. For such situations, the increase of the light-curing time and the use of photo-activation of

the composite resin through teeth have been strongly recommended [21,22].

It has been reported that the light is not transmitted well through composite resins and through teeth. The photo-activation of the composite resin through teeth which used transdental technique (TDT) or indirect techniques was introduced based on the common belief that the direction of the shrinkage vectors was towards the polymerization light, attempted to change the direction of the vectors towards the bonded walls [23]. However, it was further demonstrated that the shrinkage vectors actually develop toward the bonded walls, irrespective of the light position [24]. Nonetheless, the TDT could be effective in modifying the kinetics of polymerization, as a reduction in light intensity of up to 70 % may occur when light passes through the dental structure [25]. The photo-activation of the composite resins through the dental structure, enamel and/or dentine, is related to the curing depth of these materials and can promote a reduction in hardness values, depending yet on the dental structure thickness [26]. However, little is known about the influence of different light sources when using the TDT.

Effectiveness of polymerization may be assessed directly and indirectly. Indirect techniques have included scraping, visual and surface hardness. However, incremental surface hardness has been used, because surface hardness shown to indicate the degree of conversion of the monomers. Direct methods have included the degree of conversion, such as infrared spectroscopy and laser Raman spectroscopy. However, these techniques are complex, expensive and time-consuming. Hardness testing appears to be the most popular technique for investigating factors that affect effectiveness of polymerization of dental composite resins [13,18,19,27,28].

Thus, the purpose of this study was to evaluate the effectiveness of polymerization of one microhybrid composite resin cured with a halogen lamp or a LED LCUs with two irradiation times by means of Vickers hardness testing when direct or indirect light-curing techniques were used.

MATERIAL & METHODS

Composite resin used

The microhybrid composite resin, Filtek™ Z250 (3M Espe Dental Products Division, St. Paul, MN 55144-1000, USA - batch n°: 1370 – 3WH) at the color A2 was used in the samples preparation. The material was based on bisphenol glycidyl methacrylate (BIS-GMA)/urethane dimethacrylate (UDMA)/bisphenol ethylene methacrylate (BIS-EMA) resin matrix, with camphorquinone as photoinitiator and 60 vol % inorganic filler content with the medium size of the 0,19 a 3,3 microns. The inorganic filler is based on zirconia/silica. This material is clinically indicated as a universal hybrid composite resin for anterior and posterior restorations.

Photo-activation of the samples

For the photo-activation procedure two different light-curing units (LCUs) were used. The Table 1 shows the LCUs used and their characteristics (Table 1).

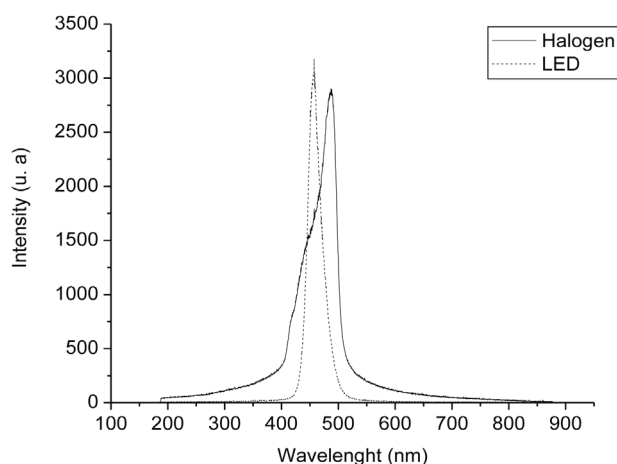
The power of the units was measured with a power meter (Fieldmaster, Coherent, model n° FM, set n° WX65, part n° 33-0506, USA) and then the power density was calculated by the equation:

$$I = \frac{P}{A}$$

Where: P = power (mW), A = area of the light tip (cm²).

Table 1 - Light-curing units, exposure times, power density and peak wavelength that were used in this study

Light-curing units	Manufactures	Power density (mW/cm ²)	Tip mm	Peak wavelength nm	Irradiation times s
Halogen Curing Light 2500	3M Espe, Dental Products Division, St. Paul, MN 55144 1000, model 5560AA, serial number 3000552	550	8	487	40 and 60
LED	MMOptics, São Carlos Brazil, model SPL 110F15-A/28	270	10	458	40 and 60

**Figure 1** - Spectral distribution emitted by the halogen and LED LCUs.

The Figure 1 shows the spectral range of the LCUs used, as well as its maximum emission peaks obtained by the spectrophotometer USB 2000 (Ocean Optics Inc.), which has a photosensitive cell where the light tip was positioned, registering, in that way, the emission spectrum (Figure 1).

Enamel and Dentin Specimens Preparation

For preparation of enamel and dentin specimens, three recently extracted and caries-free inferior lower third molars were selected (protocol number 38/04 Research Ethic 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 seven (7) days. The teeth had their coronary portions separated from their roots to the enamel-cement junction level by the use a diamond disk with 0.3 mm of thickness mounted in a cutting machine Isomet 1000 (Buehler Ltd, Lake Bluff, Illinois). The enamel and dentin specimens were obtained by the cut of the buccal face of the dental crowns. The enamel and dentin specimens (1.0; 2.0 and 3.0 mm) were flattened with wet 1200-grit silicon carbide paper (3M) mounted in a manual polishing machine.

To measure the thickness of enamel and dentin specimens, a digital caliper was used (Brown & Sharpe - model n°599-571-3).

Composite Resin Samples Preparation:

The samples (n=80) were made in a metallic mold, with central orifice (2 mm in thickness and 4 mm in diameter) according to ISO number 4049 [29].

The metallic mold was positioned on a glass plate with 10 mm of thickness where a mylar strip was taken place at the bottom surface of the metallic mold. The composite resin was packed in only increment and then covered with another mylar strip and then pressed with a glass slab to accommodate the material into the matrix and to guarantee the superficial smoothness of the composite for the hardness evaluation. Five samples (n = 5) were made for each Group. The samples were light-

cured by direct (control) and indirect techniques (enamel and dentin specimens) for 40 and 60 s of irradiation times.

Hardness Testing:

The samples were stored in a dark environment for 24 h in dry mean at 37 °C (\pm 1 °C). Following storage, the Vickers Hardness Number (VHN) was recorded in the top and bottom surfaces of the samples with a digital hardness tester (MMT-3 Microhardness Tester - Buehler Lake Bluff, Illinois USA). A 50 gf load was applied through the indenter with a dwell time of 30 s. In each top and bottom surfaces eight impressions were made according to Figure 2 (Figure 2).

Mean values and standard deviations of hardness were calculated for each Group. Statistical analysis was performed with a three-way analysis of variance (ANOVA) regarding light-curing units, power densities and irradiation times. Two-way analysis of variance (ANOVA) was applied regarding light-curing techniques and surfaces (top and bottom) in order to determine their influence. The tests were conducted at a significance level of 5%. In addition, Student-Newman-Keuls range test was used for further comparisons. Statistical significance was considered at the 95% confidence level.

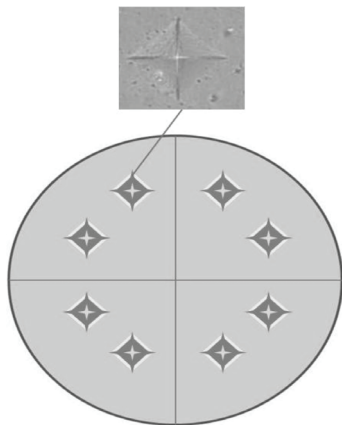


Figure 2 - Esquemático de distribuição das indentações na superfície superior e inferior das amostras.

RESULTS

The Figures 3 and 4 show the results obtained with the hardness test as a function of light-curing units, and indirect (Transdental, TDT) light-curing techniques (0, 1.0, 2.0 and 3.0 mm) and 40 and 60 s of irradiation times for the top and bottom surfaces (Figure 3 and 4).

ANOVA showed that hardness was influenced by light-curing units ($p < 0.001$), by light-curing techniques ($p < 0.001$) and by irradiation times (40 or 60 s) ($p < 0.001$). Halogen LCU exhibited a statistically higher hardness

Three-way ANOVA revealed significant interaction between direct and indirect light-curing techniques and top and bottom surfaces. Therefore, the effects of the LCUs on hardness were light curing techniques (direct and indirect), surfaces (top and bottom) and irradiation times dependent. At the top and bottom surfaces, VHN mean values after photo-activation with direct light-curing technique were significantly higher than indirect light-curing technique, independently of the irradiation times and LCUs used. There is, therefore, a significant decrease in the effectiveness of polymerization at the bottom surface.

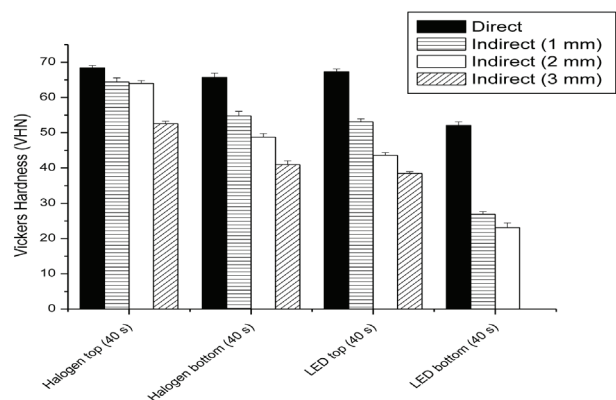


Figure 3 - VHN mean values for halogen and LED LCUs used during 40 s of irradiation time for top and bottom surfaces when direct and indirect (Transdental, TDT) light-curing techniques were used.

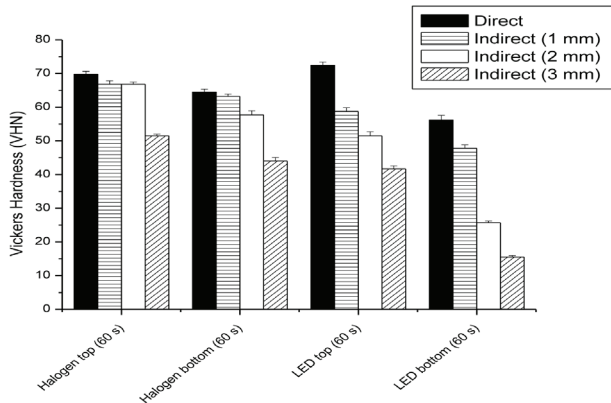


Figure 4 - VHN mean values for halogen and LED LCUs used during 60 s of irradiation time for top and bottom surfaces when direct and indirect (Transdental, TDT) light-curing techniques were used.

DISCUSSION

The polymerization effectiveness can play an important role on physical, chemistry and biological properties [4-6,30]. Problems associated with inadequate polymerization has been associated to the inferior physical properties, solubility in the oral environment, increased microleakage and adverse pulpal response to the free monomers [18,22].

Polymerization effectiveness has been assessed directly or indirectly [31-36]. Direct methods check the degree of conversion, like Fourier transformed infrared spectroscopy (FTIR) and Raman spectroscopy; however, this method is complex, expensive and time-consuming [10,24]. Moreover, the indirect methods, which include the hardness test, have been show to be an indicator of the degree of conversion and there seems to be a good correlation between Vickers hardness and infrared spectroscopy [23,37,38]. The hardness testing appears to be the most popular method for investigating factors that influence the effectiveness of polymerization with relative simplicity. The purpose of micro indentation 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 [25,37]. In a way of determining degree of polymerization, several studies have compared hardness of dental composite resins [37,39-41].

Now, clinicians face a dilemma when selecting a protocol for light-curing techniques of light-cured dental composite resins. Contemporary choices of LCUs range from conventional, continuous output quartz-tungsten to blue LED. Many authors and manufactures have stated that LED LCUs have similar polymerization effectiveness when compared with QTH LCUs and the advantage of reducing overheating [23].

Adequate physical properties of light-cured dental composite resins are achieved when the LCUs deliver enough light at the appropriate wavelength of the respective photoinitiator systems in dental composite resins. Unlike halogen LCUs, LED emission spectrum is narrow and located close to the absorption maximum of camphorquinone. The polymerization of light-cured dental composite resins depends not only on the power density, but also on its wavelength. In this sense, LCUs based on LED seems to be the best option [30].

LCUs based on blue LED produce light by electroluminescence, while the halogen LCUs produce light by incandescence, when a tungsten filament is heated, causing excitation of atoms over a wide range of energy levels that produce a very broad spectrum. Therefore, a filter is required to restrict the emitted light to the blue region of the spectrum required for curing.

The hardness measurements was used in this study In order to analyze the polymerization effectiveness of halogen and LED LCUs by the direct and indirect (Transdental, TDT) light-curing techniques.. The Figures 2 and 3 show the VHN mean values obtained with halogen and LED LCUs, respectively for microhybrid dental composite resin (top and bottom surfaces) by direct and indirect (TDT) light-curing techniques

during 40 and 60 s of irradiation times. In this study, halogen LCU showed the higher VHN mean values. The amount of energy may be explaining these findings.

The results show that for the direct light-curing technique, the top surface was not as susceptible to the effects of power densities as the bottom surface.

(In the ideal situation, the hardness:thickness ratio of dental composite resins should be equal or very close to 1:1 or very close. However, as light passes through the bulk of the restorative material, its power density is greatly decreased due to the light absorption and scattering by dental composite resins, thus, decreasing the potential for polymerization [42,43].

This scattering of light accounts for the minor differences in hardness between the top and bottom surfaces observed in this study.

In this study, this fact may be also associated with the use of indirect light-curing technique. According to Dietschi et al. and Price et al. the hardness means values decrease when the thickness of dental structure was increased in the indirect technique (TDT) [42,44]. The results observed in this study may be explained by the fact that the light emitted by the LCUs was not well transmitted through the dental structure, mainly through dentine.

In this sense, the exponential decrease in the power density plays an important role on decrease of VHN mean values, mainly for the bottom surface. In general, when the thickness of dental structure was increased a decrease in the VHN mean values was observed [39,45,46]. Frequently, for the top surface, the VHN mean values were equivalent. However, for the bottom surface, the decrease may be high when the LED LCU was used.

Many authors have shown that the presence of external interferences, such as the dental structure during the photo-activation of

dental composite resins may influence on the polymerization process [26]. When compared to the other techniques, the radiant exposure for TDT was noticeably lower, as a function of the significant reduction in irradiance for the light transmitted through enamel and dentin, probably leading to a lower degree of conversion [23].

According to Price et al. clinically, the photo-activation of dental composite resin through dental structure, enamel and or dentin, with 2 mm of thickness or plus, produces inadequate polymerization and then, inferior mechanical properties reducing in this way the lifetime of restorative procedure [44]. The reduction in hardness was verified for TDT when compared to the direct light-curing technique. An attempt to explain this outcome considers that the initial exposure at low power density for TDT might result in the formation of short, low-molecular weight polymer chains, with less cross-linking interfering with the mechanical properties of the composite [47,48].

Many authors have shown that the polymerization effectiveness cannot be assessed by top surface hardness alone. According to Rueggeberg et al. for the top surface, only irradiation time is a significant factor that contributes to monomer conversion [24].

The results of this study show that 60 s of irradiation time provided VHN mean values higher than 40 s mainly for the indirect light-curing technique. In general, an increase in VHN mean values was noted with increased irradiation time for both direct and indirect technique.

Despite the marked increase in availability of LED dental LCUs, research comparing composite polymerization associated with halogen LCU and LED LCUs is generally limited. Thus, the polymerization effectiveness using different LCUs by direct and indirect (Transdental, TDT) techniques warrants further investigations. In addition, it is important to

highlight the fact that high-power density lights should be used, as irradiance through the dental structure would be markedly reduced.

CONCLUSION

1. The bottom surface resulted in a significant decrease in hardness mean values for both LCUs used, and direct and indirect techniques.

2. Increased irradiation time resulted in an increase in hardness mean values for both LCUs used, surfaces and direct and indirect techniques.

3. The different light sources (halogen and LED) showed significant influence on the hardness mean values, while the halogen unit yielded greater hardness than LED.

4. The indirect light-curing technique significantly interfering the hardness mean values, regardless of the light-curing units, the irradiation times used and the thickness of dental structure (enamel/dentine) for both, top and bottom surfaces.

5. Maybe when indirect light-curing technique is used will be necessary to change the parameters used during photoactivation and decrease the thickness of composite resin incremente.

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