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Influence of Pre-Heat Treatment and Different Light-Curing Units on Vickers Hardness of a Mcrohybrid Composite Resin[1]

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Abstract—The aim of this study was to evaluate the hardness of a dental composite resin submitted to temperature changes before photo-activation with two light-curing unite (LCUs). Five samples (4 mm in diameter and 2 mm in thickness) for each group (n = 30) were made with pre-cure temperatures of 37, 54, and 60°C. The samples were photo-activated with a conventional quartz-tungsten-halogen (QTH) and blue LED LCUs during 40 s. The hardness Vickers test (VHN) was performed on the top and bottom surfaces of the samples. According to the interaction between light-curing unit and different pre-heating temperatures of composite resin, only the light-curing unit provided influences on the mean values of initial Vickers hardness. The light-curing unit based on blue LED showed hardness mean values more homogeneous between the top and bottom surfaces. The hardness mean values were not statistically significant difference for the pre-cure temperature used. According to these results, the pre-heating of the composite resin provide no influence on Vickers hardness mean values, however the blue LED showed a cure more homogeneous than QTH LCU.

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1. INTRODUCTION

Since the introduction of composite resin formulation based on BIS-GMA by Bowen in 1963, which showed a significant advance in the mechanical properties of the composite resins, many efforts to improve their clinical performance have been done [1]. Now, dental composite resins typically contain organic resin matrix (polymer), inorganic filler particles, initiator system, stabilizers and pigments [2].

Firstly, the photo-activation systems of the composite resins used were based on ultraviolet light sources. These systems were replaced by the quartz-halogen tungsten (QTH) as an improvement over ultraviolet lights because the harmful effect to the human eyes and limitations of curing depth. Currently, different light-curing units such as: the high power plasma arc [3] and the argon laser (light amplification by stimulated emission of radiation) have been used [4]. However, the most commonly device used are conventional light of quartz-halogen tungsten (QTH) and more recently the blue light-emitting diodes (LED).

The mechanical properties of the dental composite resins are related with the quality of polymerization [5]. The curing depth of the composite resins depends on the exposure time to the light, the power density [6–10] and the amount of visible light transmitted through the material [11]. The LCUs used should emitting radiation with wavelength useful for the most commonly photo-initiator used in dental composites, the camphorquinone (CQ) [12], which absorbs blue light in 450 to 500 nm [9].

An inadequate curing depth of the dental composite resins has been associated with lower physical properties, higher solubility, lower retention, adverse puipal responses [13–15], lower biocompatibility and excessive wear [16], that can affect the clinical performance of the restorative procedures [15, 17]. Effectiveness of cure may be assessed directly or indirectly. One of the most used indirect methods to evaluate the degree of polymerization of the composite resins is the hardness test [18, 19]. According to some studies, there is a correlation between the degree of conversion and hardness test [16]. If the degree of conversion shows the higher values, the properties of the material will be better [20]. The degree of conversion is significantly influenced by the power densities and irradiation times of the LCUs [15].

The confection of the restoration in composite resin depends on the power density of the light, irradiation time, method of light application and presence of moisture in the restoration [21, 22]. Few attentions have been given to the properties of the composite resin at different thermal conditions [22].

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Experimental groups investigated in this study

Group	LCUs	Temperature of heating, °C
1	QTH	37
2	QTH	54
3	QTH	60
4	LED	37
5	LED	54
6	LED	60

The manufacturers have increased the concentration of load of the composites in order to improve the physical properties; however more quantity of load increases the viscosity, which become more difficult to marginal adaptation. A device called "Calset" was developed to heat the composite resin, reducing the viscosity of the composites. This procedure was called the technique of thermal photopolymerization. The principle of this technique is based on heating of composite resin to moderate temperatures of 37, 54, or 60°C before to photo-activation to improve the performance of manipulation and adaptation [21, 23]. The temperature has a significant effect on the immediate conversion obtained in dental light-cured composite resins [24]. Thus, the aim of this study was evaluate the effect of the pre-heating of a dental composite resin at three different temperatures and photo-activated with two different light-curing units by means of Vickers hardness test (VHN).

2. MATERIAL AND METHODS

The groups investigated in this study are showed in table.

One QTH LCU Translux CL (Heraeus-Kulzer, D-6393, serial number: 15/35890, 400 to 500 nm) and one LCU based on blue LED Bluephase (Ivoclar/Vivadent, serial number: 1666128, 380 to 515 nm) were used in this study. The QTH LCU was used in the conventional mode with 650 ± 10 mW/cm^2 for 40 s and blue LED LCU was used in the soft-start mode for 40 s (early with 600 ± 10 mW/cm^2 for 10 and 30 s remaining with 980 ± 10 mW/cm^2). The power density were checked using a powermeter (Fieldmaster, Coherent Commercial Products Division, model number FM, set n° WX65, part number 33-0506 in USA) and the irradiance were calculated with this formula:

$$I = P/A,$$

where: P is power in mW (milliwatts); A is area of the light tip in cm^2 (centimeter square).

2 The samples were made with a compule of hybrid composite resin Tetric Ceram (Ivoclar/Vivadent, Schaan, Liechtenstein, shade A_3, lote J03862). According to the manufacturer, the monomer matrix is composed of bisphenol glycidyl methacrylate (Bis-GMA), urethane dimethacrylate (UDMA), and triethylene glycol dimethacrylate (TEGDMA)—20.2% weight. The total content of inorganic fillers is 79 wt % or 60 vol %. The mean particle size is 0.7 μm. Additional contents: catalysts, stabilizers, and pigments (0.8 wt %).

The composite resin was pre-heated with the Calset device (AdDent Inc. P/N: GS-817. Model: DA-20 to 20). This unit has three different temperatures, which are indicated by a LED, where: green is 37°C, orange is 54°C and red is 60°C. A thermocouple was used to analyze the time required for the device and composite resin achieve the desired temperature, which was about 15 min (Fig. 1).

For the samples preparation, it was used a metallic mold with central orifice (4 mm in diameter and 2 mm in thickness) according to the ISO 4049 [25]. The composite resin was packed into the mold and a polyethylene film covered each side of the sample and a glass slide was placed on the top surface of the samples. The top surface was standardized with a circular weight (1 kg). The light tip of the LCUs was positioned on the glass slide, leaving the 0.55 mm of the composite resin. The samples were photo-activated for 40 s. The Vickers hardness test was performed in a hardness testing machine, MMT-3 Microhardness Tester (Buehler Lake Bluff, Ilinois USA) equipped with Vickers diamond (VHN), which has a format of pyramid of 136° where the two diagonals are measured [9, 26] using load of 50 gf (gram force) for 30 s. In each surface, the top 3 (turned to the light source) and bottom (opposite to the light source) took place an impression for quadrant. The hardness means values were calculated for each surface.

The data were submitted to the Analysis of Variance with two fixed criteria (two-way-ANOVA). The tests were performed at the level of 5%.

3. RESULTS

Figure 2 shows the VHN mean values (Kgf/mm^2) for each Group. The hardness mean values at the top surface of the samples photo-activated with QTH showed the highest values when compared with the mean values for LED. However, the differences were no evident. The hardness mean values for the bottom surface showed lower values for the samples photo-activated with QTH with the same temperatures when compared with blue LED. The samples photo-activated by blue LED have a systematic increase of the hardness mean values with the increase of temperature, which cannot be observed in the samples photo-activated with QTH.

The curing depth of the samples was take place through the proportion between top and bottom surfaces. Figure 3 shows the bottom/top ratio values for each Group. The mean hardness values at the top and

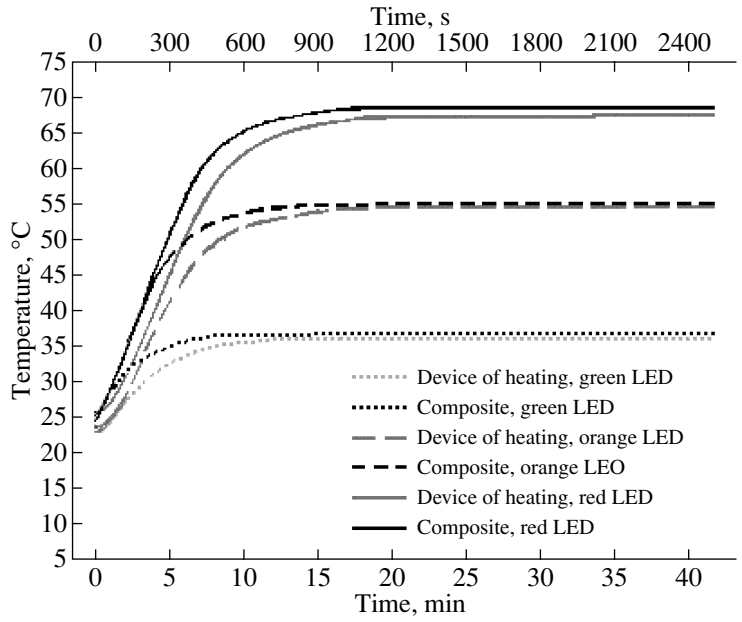

Fig. 1. Description of the time needs to reach the temperature.

bottom surfaces were very close for the LED, while the QTH were around 25% lower for bottom.

The results of analysis of variance showed that the LCU factor was statistically significant ($p < 0.0001$). The temperature of heating factor ($p = 0.8445$) and the interaction between the two factors ($p = 0.8493$) showed no statistically significant differences.

4. DISCUSSION

The hardness values and degree of conversion are inversely proportional to increase of the depth of the composite resin [27]. Increasing the power density provides highest degree of conversion and therefore better mechanical properties were found [28]. An increase in the rate of conversion of carbon double bonds is associated with higher values of hardness [29],

The maximum hardness obtained on the surface of composites is directly related to the power density and distance of the light curing tip from the surface of the material [7–9, 30]. The depth of cure of visible-light activated composite resins is affected by factors such as material's filler composition and resin chemistry [30, 31], thickness [10, 27], irradiation time, power density, spectral distribution [10, 16, 28, 30, 31] and distance of the light tip of the LCUs [10, 30, 32]. The depth of cure of composite resins is limited due to the attenuation of irradiation through the systems and structures adjacent to the teeth [6]. The type of monomer compositions can influence the transmittance of light. If the quantity of light at the base of the composite resin is high, the depth of cure and conversion of polymerization will be high [11].

The QTH light has been used [33] for more than two decades, however the blue LED is an alternative method used since 1995. The QTH LCUs have as disadvantage short lifetime and heat generation [34]. Currently, the LED has been regarded as a LCU able to cure the composites with properties similar to that obtained by the QTH light that has low-cost technology and a long-life [31] and less heat is generated. Significant temperature increases occur during the visible light-curing process of composite resin using different LCUs [35–37]. The temperature increase during the composite light curing with halogen LCUs was higher than the argon laser and blue LED [37]. The QTH provided highest increase in the temperature of the tooth than the LED [36], and when used the blue LED and plasma arc the temperature increase was lower [34].

The QTH LCUs have shown difference between the degree of polymerization obtained on the surface irradiated, near to the light and deeper layers [18]. The QTH LCU with under performance can cure the top surface of the composite resin as efficient as a LED LCU [38]. In this investigation the QTH LCU showed hardness mean values at the top surface slightly higher than the mean values for LED LCU, however the mean values for the bottom surface were 25% lower, which the QTH even with low irradiance presented greater superficial hardness, however hardness mean values showed low in bottom surface.

The new generation of LEDs has shown a sufficient capacity to light-cure the composites in similar time when compared with QTH [31]. When comparing the monomers conversion of a composite at different depths polymerized by different light-curing units

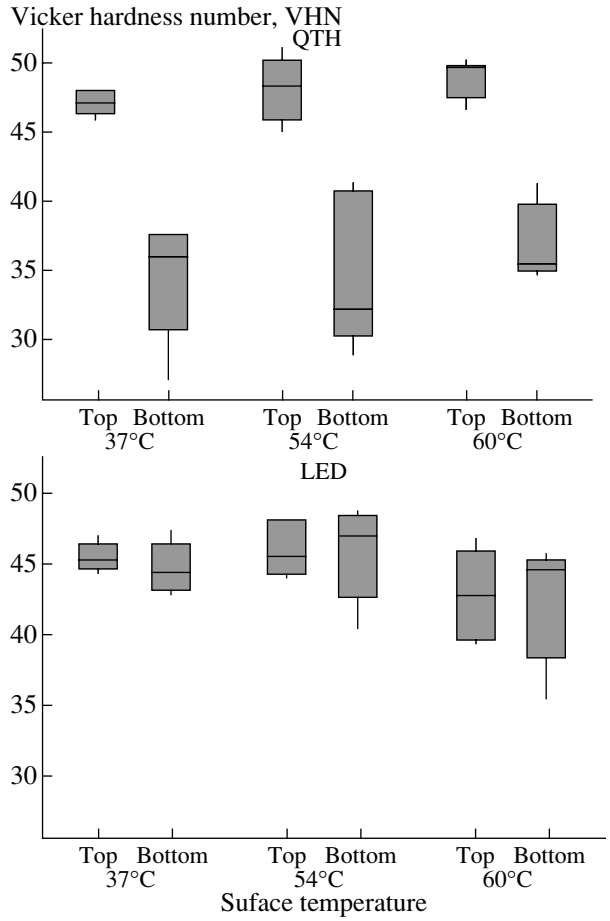

Fig. 2. VHN mean values for halogen and LED LCUs used during 40 s of irradiation time for top and bottom surfaces when pre-heating of composite resin was used.

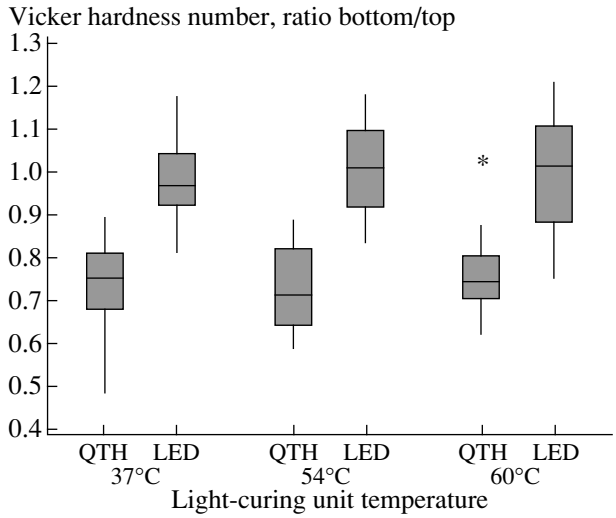

Fig. 3. VHN mean values of the bottom/top ratio for each Group.

(QTH, LED or plasma arc), the LED showed little reduction on degree of conversion, when located more deeply, indicating a cure more homogeneous [39]. The samples cured by LED LCUs showed the same hardness mean values or higher than the QTH LCUs, however the samples cured by LED showed bottom/top ratio more homogeneous than halogen [40], so the energy of the blue LED is almost totally absorbed by material along the photo-activation, producing a more uniform cure without compromising the chemical and mechanical properties of the material over time [41]. The LED LCUs used for polymerization achieved greater depth of cure than the leading halogen light [42]. In this study, the LED LCU provided higher hardness mean values at the bottom surface than QTH LCU. The LED LCU provide higher power density, higher penetration depth of the light, being more absorbed which promotes more homogenous cure of the composite resin.

The low hardness mean values observed for the bottom surface can be explained by the decrease of power density. As light passes through the bulk of the restorative material, its power density is greatly reduced by the light scattering within the composite. The mean ratio between bottom and top surfaces of 0.92 was adequate for the hardening of the bottom surface [16]. In this study the bottom/top ratio was more than 0.92 for the Groups with LED.

Adequate polymerization is a fundamental factor in obtaining optimal physical properties and the clinical performance of composite resin restorative materials [5, 11, 15]. The degree of monomer conversion in light-cured composite resins depends on many factors. One of them is the quantity of free radicals formed during the irradiation. Thus, the energy delivered to the system and, consequently, the irradiation time and the power density are important factors [16].

The Bis-GMA increases the density of cross-links of the resinous polymers therefore best mechanical properties are obtained when comparing with the flexible monomers [43]. The high-viscosity occurs due the higher quantity of load, which become more difficult marginal adaptation [21–23, 44, 45]. The flowable composite resin has been used in posterior restorations to improve the marginal adaptation. However, this technique compromises the physical and mechanical properties of the restoration, due the low-viscosity of the composite [21, 46].

The pre-heating of the composite resin reduces the viscosity, improves the flow of material allowing a better adjustment in the marginal areas of the cavities without changing the composition, physical and mechanical properties [21–23, 44, 45]. The pre-heating of the composites slightly above the temperature of the body can improve the depth of cure of the material, improve the conversion rate and reduce the time of healing by 50% [21, 46]. The increase of the temperature of the composite resins has significant effect on the polymerization kinetics. The direct heating effect in the polymerization and the viscosity of the material may

contribute to the higher conversion rate [45]. The temperature increase of the composite before photo-activation provides higher mobility of the radicals due the reduction of the viscosity of the system providing an increase conversion rate of the monomers [47]. The increase on conversion rate provides more cross-links therefore will be better mechanical properties [14, 20, 45]. However, the mechanical properties of materials are dependent on the characteristics of polymer network, as the density of cross-links, and these are not equivalent to conversion.

An improvement in the viscosity of the material occurs with the temperature increase. In this study, the composite resin at temperatures of 54 and 60°C were manipulated more easily, providing better flowing and adaptation in the matrix. However, the increase in temperature had no significant interference in the results of Vickers hardness numbers (VHN). The LED LCU showed a low increase in hardness mean values with the temperature increase, however no significant difference was observed ($p = 0.05$).

The LCU showed a statistical difference in the results of VHN when evaluated the interaction between the LCU and pre-heating of the composite resin. The light source and power density of light influence the depth of cure of the material. The LED LCU results were more homogeneous because the differences in hardness of the bottom and top were low. The LED was able to promote a more homogeneous photopolimerization, avoiding discrepancies between the areas of bottom and top. The hardness mean values were not statistically significant different for the temperature increase.

5. CONCLUSIONS

Intense research is directed to improve the properties of dental composite resins because the material is widely used in Dentistry. Currently, the LED LCU is widely used and shows good results in polymerization of the dental composites. In addition, the LED light showed a homogeneous polymerization across the surface of the composite resin and good values of Vickers hardness. The pre-heating of composite resins prior to polymerization showed no influence in the hardness mean values, however more studies should be directed to assess the effect of pre-heat treatment in order to improve the physical and mechanical properties of composite resins.

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SPELL: 1. puipal, 2. compule, 3. gf, 4. flowable