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Influence of Light Guide Tip Used in the Photo-Activation on Degree of Conversion and Hardness of One Nanofilled Dental Composite[1]

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Abstract—The aim of this study was to evaluate the degree of conversion and hardness of a dental composite resin Filtek^{TM} Z-350 (3M ESPE, Dental Products St. Paul, MN) photo-activated for 20 s of irradiation time with two different light guide tips, metal and polymer, coupled on blue LED Ultraled LCU (Dabi Atlante, SP, Brazil). With the metal light tip, power density was of 352 and with the polymer was of 456 mW/cm^2, respectively. Five samples (4 mm in diameter and 2mm in thickness—ISO 4049), were made for each Group evaluated. The measurements for DC (%) were made in a Nexus-470 FT-IR, Thermo Nicolet, E.U.A. Spectroscopy (FTIR). Spectra for both uncured and cured samples were analyzed using an accessory of reflectance diffuse. The measurements were recorded in absorbance operating under the following conditions: 32 scans, 4 cm^{-1} resolution, 300–4000 cm^{-1} wavelength. The percentage of unreacted carbon double bonds (% C=C) was determined from the ratio of absorbance intensities of aliphatic C=C (peak at 1637 cm^{-1}) against internal standard before and after curing of the specimen: aromatic C–C (peak at 1610 cm^{-1}). The Vickers hardness measurements (top and bottom surfaces) were performed in a universal 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 the *test t Student* at significance level of 5%. The mean values of degree of conversion for the polymer and metal light guide tip no were statistically different ($p = 0.8389$). The hardness mean values were no statistically significant different among the light guide tips ($p = 0.6244$), however, there was difference between top and bottom surfaces ($p = 0.001$). The results show that so much the polymer light tip as the metal light tip can be used for the photo-activation, probably for the low quality of the light guide tip metal.

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1. INTRODUCTION

Dental composites are an important class of material widely employed in restorative procedures. In recent years, the popularity of aesthetic tooth-colored restorations has promoted a rapidly increasing use of composite resin [1].

Now, the generally preferred mode of cure in dental composite resins is photo-activation method [2]. The main advantage of photo-activation mode is the control that the operator has over the working time [3, 4].

Physical properties and better clinical performance are related with the photo-activation of dental composite resins and to improve them is necessary to understand the effect of several parameters involving the polymerization process of these materials [5].

Effectiveness of cure may be verified directly or indirectly. The direct methods include those that determine the degree of conversion of a composite material, like Fourier transformed infrared spectroscopy (FTIR) and Raman spectroscopy [6]. One of the most used indirect methods to evaluate the degree of polymerization of the composite resins is the hardness test [3, 7–11]. Many authors have related that the degree of conversion is an important meaning and an inadequate degree of conversion of the dental composite resins has been associated with lower physical properties, higher solubility, lower retention, adverse pulpal responses [3, 12], lower biocompatibility and excessive wear [13], that can affect the clinical performance of the restorative procedures [3, 14].

The polymerization of photo-activated composite resins depends mainly on the characteristics and type of the radiation source used, a way to achieve better properties of the end restoration cured is the improve-

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Table 1. Characteristics of the restorative material used in the study

Material	Manufacturer	Shade	Material type	Matrix	Filler size	Filler volume	Lote number
Filtek™ Z-350	3M/ESPE	A_2	Nanofilled composite	Bis-GMA, Bis-EMA, TEGDMA	Agglomerated/non-aggregated of 20 nm silica nanofiller anda loosely bound agglomerate silica nanocluster consisting of agglomerates of primary silica nanoparticles of 5 to 20 nm size fillers. The cluster size range is 0.6 to 1.4	59.5%	7HY/6HN

ment of the light-curing units. In this direction, different light-curing sources should be tested to verify their viability for clinical application [15].

Halogen lamp based light-curing units have become the method of curing dental composites in the clinical setting, however these light-curing units have some drawbacks. The power density of the halogen light-curing units decreases over time due to bulb and filter aging. For halogen lamps, up to 70% of the input power is converted to heat and only 10% is visible light. Of this visible light, a further 90% is lost due to the use of cutoff filters [16–19].

Different light-curing units have been developed, with newer types of light curing-sources using other curing methods such as argon ion laser, xenon arc and light-emitting diodes (LED) based technologies. Argon ion laser and xenon arc curing units have the advantage of a reduced curing times, however, these light-curing units have a larger and more complicated construction, and are more costly than halogen and LED light-curing units. The use of laser is currently more concerned with the suppression of dental hypersensitivity, soft tissue surgeries, intracanal disinfection and caries removal [20, 21]. Currently, LEDs have been the light-curing source more used in the photoactivation. It has a working lifetime of over 10 000 h, while halogen bulbs have a limited effective lifetime of about 40–100 h. LEDs can have wavelength peaks of around 470 nm [1, 8, 22, 23], the ideal range for activating the most popular photo-initiator, camphorquinone (CQ), which has a peak wavelength of 468 nm, and probably present better results than halogen light due the LED has a more appropriate wavelength for the polymerization of the most of resin materials [24, 25]. In addition, the thermal emission of the LED light-curing units is significantly lower than that of halogen lamp light-curing units [26]. Besides the different light-curing sources, power density, wavelength and irradiation times are other factors than can influence the polymerization of dental composite resins. Another factor affecting the polymerization reaction is the light guide tip used for light transmission. Now, a wide variety of commercially available light guide tips claim to fit different operative procedures based on different clinical situations [27–29].

Technologies have been developed that enable production of the appropriate amount of light required for the efficient conversion of composite resins [10]. Light-curing units of the pistol type are the more used now and, in these models, the conductive system of the light is based on a rigid probe that it contains the fiber optic involved by a glass material covered for glass amber or metal [30].

The type of material of the light guide tips can hinder the light passage in her itinerary, increasing her dispersion. A wide variety of commercially light guide tips with variation of the material that it covers them, diameters, and forms, with the objective of facilitating the access to the different areas or cavities have been development. However, these differences can interfere in the power density values what would have direct repercussion in the polymerization process of the composite resins [31, 32].

Another problem that should be pointed out is that light guide tips which are available for LED LCUs, have a variety of diameters and materials, for example, polymer or fiber optic. The polymer tip scatters the guided light, thus reducing the power density at the end of the tip [32].

However, it should be considered the possibility of the material that it covers the light guide tips of the light-curing units promote the light dispersion in the itinerary of the light to the material, in spite of the works that approach this subject they be scarce in the literature. In this way, this study evaluated the influence of the light guide tips used in the polymerization by means of degree of conversion and Vickers hardness of a nanofilled composite resin.

2. METHODS AND MATERIALS

The Table 1 shows the main composition of the dental composite resin used. According to the manufacturer, the monomer matrix is composed of bisphenol glycidyl methacrylate (BIS-GMA)/bisphenol etylene methacrylate (BIS-EMA)/triethylene glycol dimethacrylate (TEG-DMA). The total content of inorganic fillers is 78.5 wt % or 59.5 vol %. The mean particle size is between 0.6 and 1.4 μm. Additional contents: catalysts, stabilizers, pigments, and camphorquinone.

Table 2. Characteristics of the light guide tip used in the study

Light-curing unit	Light guide tip	Diameter entry, mm	Diameter exit, mm	Geometry
Ultraled	Metal	11	8	Turbo
	Polymer	10	8	Turbo

2.1. Material

The dental nanofilled composite resin Filtek™ Z-350 (3M ESPE Dental Products Division, St. Paul, MN 55144-1000, United States, batch n° 1370) at color A_2 was used in this study.

2.2. Light-Curing Unit (LCU)

One blue LED LCU (Ultraled, Dabi Atlante, Ribeirão Preto, SP, Brazil, serial number: 002041) with two different light guide tips, metal and polymer was used in this study. The power output was measured using a Fieldmaster powermeter (Fieldmaster Power to Put, Coherent-model n° FM, set n° WX65, part n° 33-0506, USA). The values of power density (mW/cm^2) were computed as the ratio of the output power and the area of the tip with the following formula:

$$I = P/A,$$

where P is the power in milliwatts and A is the area of the light tip in squared centimeters. The LED LCU coupled with the metal light guide tip presented 352 mW/cm^2 and with the polymer was of 456 mW/cm^2. The characteristics of the light guide tips are shown in Table 2.

2.3. Samples Preparation

The samples preparation were made with a metallic mould with central orifice (4 mm in diameter and 2 mm in thickness) according to ISO 4049 [33]. The metallic mould was positioned in a 10 mm thick glass plate. The composite resin was packed in a single increment and the top and base surfaces were covered by a mylar strip. A glass sheet 1-mm thick was positioned and a 1-kg weight was used to pack the composite resin. Photo-activation was performed by positioning the light guide tip on the top surface of the composite resin samples. The samples were irradiated during 20 s. After photo-activation, the samples were removed from de mould and stored in a dry mean, in dark containers, at 37°C(±1°C) for 24 h.

For degree of conversion, after 24 h, the composite resin was pulverized into a fine powder. The pulverized composite resin was maintained in a dark room until the moment of the FT-IR analysis. Five milligrams (5 mg) of the ground powder were thoroughly mixed with 100 mg of the KBr powder salt. This mixture was placed into a pelleting device, and then pressed in a press with a load of 10 t over 1 min to obtain a pellet.

2.4. Degree of Conversion Measurements (% DC)

To measure the degree of conversion, the pellet was then placed into a holder attachment into the spectrophotometer Nexus-470 FT-IR (Thermo Nicolet, EUA). The Fourier transform infrared spectroscopy (FT-IR) spectra for both uncured and cured samples were analyzed using an accessory of the diffuse reflectance. The measurements were recorded in the absorbance operating under the following conditions: 32 scans, a 4 cm^{-1} resolution, and a 300 to 4000 cm^{-1} wavelength. The percentage of unreacted carbon–carbon Double bonds (% C=C) was determined from the ratio of the absorbance intensities of aliphatic C=C (peak at 1637 cm^{-1}) against an internal standard before and after the curing of the specimen: aromatic C–C (peak at 1610 cm^{-1}). This experiment was carried out in triplicate. The degree of conversion was determined by subtracting the % C=C from 100%, according to the formula:

$$DC(\%) = 1 - \frac{(1637\ \text{cm}^{-1}/1610\ \text{cm}^{-1})_{\text{cured}}}{(1637\ \text{cm}^{-1}/1610\ \text{cm}^{-1})_{\text{uncured}}}.$$

The percentage of unreacted carbon–carbon double bonds (% C=C) was determined from the ratio of absorbance intensities of aliphatic C=C (peak at 1637 cm^{-1}) against.

2.5. Vickers Hardness Measurements

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 pyramidal diamond microindentor of 136°, where the two diagonals are measured [9, 26] using a load of 50 gf (gram force) during 30 s. Each surface of the sample was divided in 4 equal quadrants. On each surface, the top (turned to the light source) and bottom (opposite to the light source) surfaces took place as an impression for each quadrant. The hardness mean values were calculated for each surface.

2.6. Statistical Analysis

The data were submitted to statistical analysis by the test *t Student* at 5% significance level.

Table 3. Mean values and standard deviation (±sd) for degree of conversion of the dental composite photo-activated with different light tips

	Metal light tip	Polymer light tip
Mean values	71.4 ± 6.62	72.3 ± 6.93

3. RESULTS

3.1. Degree of Conversion

The Table 3 shows the degree of conversion mean values for the all groups investigated in this study.

It can be observed that the degree of conversion mean values for the polymer and metal light guide tip was 72.3 and 71.4%, respectively. The Test *t Student* showed that the results no were statistically different ($p = 0.8389$), and these values might have been obtained because the low power density provided with the metal light tip coupled on LED.

3.2. Vickers Hardness

Regarding the hardness, as it is shown in Table 4, there was no statistical significant differences among the light guide tips ($p = 0.6244$), however, there was difference between top and bottom surfaces ($p = 0.001$). The top surface showed the higher mean values than the bottom surface as it can be seen too in Table 4.

4. DISCUSSION

Since the introduction of photo-activated resin-based composites, the quality of polymerization has now become one of the great concerns of researchers. Therefore, new technologies have been developed that enable production of the appropriate amount of light required for the efficient conversion of composite resin, resulting in the enhancement of their physical-mechanical properties [10].

The use of LED as the light source to light-cure the polymerization reaction of the composite resins was proposed in 1995 and, since then, many changes of the parameters have been made for an appropriate polymerization [23]. A number of studies have addressed the application of blue LED technology to cure dental materials. LEDs have an expected lifetime of more than 10000 h without significant degradation of light intensity over time. Also, they do not require filters to produce blue light and have low power consumption [32]. In this study, it was used one LED with power density of 352 mW/cm^2 when coupled with metal and of 456 mW/cm^2, with the polymer light tip. Even with lower power density, photo-activated samples with both metal and polymer light tip promoted good values for degree of conversion and hardness. Different light-curing sources, increment thickness [34, 35], irradiation times, power density, spectral distribution [13, 35–38], the geometry and the material of the light guide tip can influence the physical properties of the composite restorative materials [31, 32, 35, 36, 39].

The properties of photo-activated composite resins can be analyzed and studied by several means, such as degree of conversion and hardness tests [10]. The term "degree of conversion" applied to composite refers to the conversion of carbon–carbon double bonds monomeric carbonic to carbonic simple polymer [40, 41]. The degree of conversion can be analyzed by mechanical (dilatometric), calorimetric and spectroscopy. In principle, the latter method provides more reliable results. Spectroscopy methods provide direct measurements of DC values, because specific vibrational bands can be used as internal standards [32]. One of the most frequently used indirect methods for verifying the degree of resin composite polymerization is the microhardness test [10].

In this study, two light guide tips were used in the photo-activation of composite resin with different power densities and then, the degree of conversion (%) and hardness measurements were determined. This investigation is important to the dental practitioners because a higher degree (%) of resin polymerization has been associated with improved clinical performance of these materials [39, 42].

A lower degree of conversion could affect the longevity of the composite restoration, because an incomplete conversion may result in unreacted monomers, which might dissolve in a wet environment. In addition, lead to a degradation of the material. Therefore, this fact could directly affect the bio compatibility of the restoration, since increasing the DC number of methacrylate pendant groups available for hydrolytic degradation decreases. Hydrolytic degradation and oxidation of composites may lead to the leaching of different degradation products from composite resin. Formaldehyde has been identified as one of the degradation products. Methacrylic acid has also been identified as an eluted species that can cause irritation of the mucosa membrane and is cytotoxic [38, 43–45].

Table 4. Hardness mean values, standard deviation (±sd) for the top and bottom surfaces and Corresponding B/T Ratio of the dental composite resin photo-activated with different light guides tips

	Top surface	Bottom surface	B/T, %
Metal light guide tip	44.423 ± 2.95	35.675 ± 3.10	80.3
Polymer light guide tip	44.077 ± 2.10	35.565 ± 3.26	80.6

Ideally, the dental composite resin would have all of its monomer converted to polymer during the polymerization reaction [45]. However, the dimethacrylate monomers used in restorative materials exhibit considerable residual unsaturation in the final material, with a degree of conversion (%) ranging from 55 to 75% under conventional irradiation conditions [46–48].

From our experiment, the DC mean values ranged from 64.8 to 78.0% for metal and 65.4 to 79.2% for polymer light tip and according to the results presented on Table 3 there was no statistical difference in DC (%) mean values between the light guide tips. This findings showing that the two light guide tips were able to light-cure Filtek™ Z-350 composite with 2 mm thickness. However, although not statistically different, the light guide tip metal showed lower values of power density coupled LED. It was believed that the polymer light tip could scattered more light, presenting like this a low power density, however it presented a greater power density than the metal, what can be due to low quality of the metal light guide tip. Therefore, new studies should be accomplished with light guide tips of better quality.

The results are in agreement with Soares et al. [32] that showed that the influence of light guide tip material on DC was not statistically significant ($p > 0.05$) between the polymer and fiber optic. Others studies that investigated the influence of the geometry of the light guide tip, showed that the geometry of the tip significantly influences the curing depth [31, 49].

Mechanical properties including hardness as a function of material's thickness is an important factor to be studied. This applies especially to restorative materials that are used where high biting forces and stresses can exacerbate inherent material defects, resulting in inadequate fracture resistance of the materials [50].

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 [16, 18, 30, 51]. The depth of cure of visible-light activated composite resins is affected by factors such as material's filler composition and resin chemistry [30, 32], thickness [7, 24], irradiation time, power density, spectral distribution [11, 24, 30, 32, 52] and distance of the light tip of the LCUs [24, 30, 31]. The depth of cure of composite resins is limited due to the attenuation of irradiation through the light-curing units and structures adjacent to the teeth [17].

According with Craig and Powers (2004) [53] composite resins must have hardness values exceeding 50 (KHN) to be considered ideal. However, some authors believe that there is still no consensus on a Vickers hardness value considered optimal for that change in accordance with the type of resin used, mainly due to the amount and size of inorganic particles, among other factors [54]. In this investigation, the samples photo-activated with the metal light tip showed hardness mean values at the top surface of 44.423 (±2.95) and photo-activated with the polymer showed mean values of 44.077 (±2.10). At the bottom surface, the hardness mean values were 35.675 (±3.10) to metal and 35.565 (±3.26) to polymer light tips.

As it is shown in Table 4 there was no statistical significant differences among the light guide tips ($p = 0.6244$), however, there was difference between top and bottom surfaces ($p = 0.001$). On the top surface, the light intensity is usually sufficient for adequate polymerization [55]. The composite resin on the bottom surface disperses the light of the light curing unit. As a result, when the light passes through the bulk of the composite, its power density is greatly reduced due to the scattering of light by filler particles and the resin matrix [55–57]. Johnston et al. [58] believes that the curing efficiency could be measured by the ratio between bottom and top surface hardness (B/T), which should not be 90%, however according to some authors, the bottom surface of the samples must be at least 80% of the surface hardness of the top, which is consistent with our findings which showed a ratio of 80.3 and 80.6% between the top and bottom surfaces of the samples cured with metal and polymer light tips, respectively, as shown in Table 4.

There are many factors in relation to the tips of light-curing unit that has been studied as the diameter, distance from light source to the restorative material and geometry. The materials of the light guide tips may have direct impact on the power density, which would have great influence on the physical, mechanical and biological properties of composite resins. In our study, no difference in the degree of conversion and hardness of composite resins photo-activated with both metal and polymer light tips, however it is important to emphasize that further studies must be conducted in a way that actually will show that there is no influence of type of light guide tip used in light-curing on the properties of composite resins.

5. CONCLUSIONS

Based on these results, it was ended that:

So much the polymer as the metal light tip can be used for the photo-activation. It is important to emphasize that this is probably due to the low quality of the metal light tip that offered a low power density.

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