Evaluation of degree of conversion and hardness of dental composites photo-activated with different light guide tips

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ABSTRACT

Objective: The aim of this study was to evaluate the degree of conversion and hardness of different composite resins, photo-activated for 40 s with two different light guide tips, fiber optic and polymer.

Methods: Five specimens were made for each group evaluated. 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 were performed in a universal testing machine. A 50 gf load was used and the indenter with a dwell time of 30 seconds. The degree of conversion and hardness mean values were analyzed separately by ANOVA and Tukey's test, with a significance level set at 5%.

Results: The mean values of degree of conversion for the polymer and fiber optic light guide tip were statistically different (P<.001). The hardness mean values were statistically different among the light guide tips (P<.001), but also there was difference between top and bottom surfaces (P<.001).

Conclusions: The results showed that the resins photo-activated with the fiber optic light guide tip promoted higher values for degree of conversion and hardness. (Eur J Dent 2013;7:86-93)

Key words: Composite resins; hardness; polymerization

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INTRODUCTION

Light-cured composite resins are widely used in dental restorations, as they are mercury-free and esthetically pleasing to the patient.[1]

The introduction of the visible light system for the photo-activation of composite resins had its beginning in 1970 with the use of ultraviolet light. However, due to the adverse effects caused by this light system, it was substituted quickly by the halogen light system.[2]

Previously, the halogen lamp was the most common light source used for composite photo-activation. However, heat generation is the major disadvantage of these LCUs (Light Curing Unit).[3-7] Moreover, the bulb, reflector and filter can degrade over time due to high operating temperatures caused by a large quantity of heat, which is produced during cycles.[3]

In recent years, light-emitting diodes (LEDs) have been used to create compact, cordless LCUs.[8] They have a working lifetime of over 10,000 h, can have wavelength peaks of around 470 nm, it is not necessary to use filters and can be portable. In addition, the thermal emission of the LED LCUs is significantly lower than of halogen lamp LCUs. Studies using dental resins irradiated with blue light LEDs have been reported to have a higher degree of polymerization, a more stable three-dimensional structure, and a significantly greater curing depth than those cured with conventional QTH (Quartz tungsten-halogen) lights.[1,2,9,10]

The quality of the polymerization has been one of the most studied since the development of composite resins polymerized by light. Thus, there is the need for light sources that promote an appropriate conversion of monomers in polymers, so that the restoration has appropriate physical, chemical and mechanical properties.[10-16]

The study of some properties can be made by the degree of conversion and hardness tests. Degree of conversion (DC) is an important parameter in determining the final physical, mechanical and biological properties of photo-activated composite resins.[17] The DC is determined by the proportion of the remaining concentration of the aliphatic C=C double bonds in a cured sample relative to the total number of C=C bonds in the uncured material. Fourier Transform Infra-red Spectroscopy (FT-IR) is one of the most widely used techniques for measurement of DC in dental composites.[13,18]

Several factors can influence the DC such as light source used, power density, wavelength, irradiation time, light-tip size, photo-activation method, distribution, quantity of inorganic fillers, the type and quantity of the photoinitiator, and color also strongly affect the DC of the composite resins.[13]

Vickers hardness measurement is one of the most important to compare restorative materials, and is defined as the resistance to indenter penetration or standing on the surface. It is a mechanical property that should always be taken into account, especially when they are faced with large areas of masticatory effort.[19-21]

Technologies have been developed that enable production of the appropriate amount of light required for the efficient conversion of composite resins. Now, light-curing units have used different kinds of conductive systems based on a rigid probe that it contains the fiber optic involved by a glass material covered for glass amber or metal.[2,22-25]

The type of material of the light guide tips can hinder the light passage in its itinerary, increasing her dispersion. A wide variety of commercial light guide tips with variation of the material that covers them, diameters, and shape, with the objective of facilitating the access to the different areas or cavities have been developed.[3,4,9,24] These differences on light guide tips can provide changes in the power density values and then compromising the polymerization of the composite resins.[3]

It has been hypothesized that the material that covers the light guide tips of the light-curing units promote the light dispersion in the itinerary of the light. In this way, the aim this study was evaluated the influence of the light guide tips used in the photo-activation of dental composites by means of degree of conversion and Vickers hardness.

MATERIAL AND METHODS

One blue LED LCU (Ultrablue IS, DMC, São Carlos, SP, Brazil, serial number: 002041) with two different light guide tips, fiber optic and polymeric was used in this study. Prior to the curing procedures, the power output of the LCU was measured with a calibrated power meter (Fieldmaster Power Meter, Coherent-model n° FM, set n° WX65, part n° 33-0506, USA) and the diameter of the light guide tip was measured with a digital caliper (Mitutoyo, Tokyo, Japan). Power density (mW/cm^2) was computed as the ratio of the power output and the area of the tip with the following formula: $I = P/A$, where P is the power in (mW/cm^2, milliwatts per centimeter square) and A is the area of the light tip in centimeters square.

The LED LCU coupled with the fiber optic light guide tip presented 653 mW/cm^2 and with the polymeric 596 mW/cm^2. The characteristics of the light guide tips are shown in Table 1.

Experiments were performed with two restorative systems: Filtek™ Z 250 (3M Espe Dental Products Division, St. Paul, MN 55144-1000, USA), a universal microhybrid and Filtek™ Supreme XT (3M Espe Dental Products Division, St. Paul, MN 55144-1000, USA), a nanofilled.

The specimens were made using a metallic mould with a central orifice (4 mm in diameter and 2 mm in thickness) according to ISO 4049.[26] The metallic mould was positioned on 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 thickness was positioned and a mass of 1 kg 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 specimens. The specimens were irradiated during 40 s. After photo-activation, the specimens were removed from the mould and stored in a dry mean, in dark containers, at 37º C (±1°C) for 24 hours.

Degree of Conversion Measurements (DC%)

For this technique, five specimens were made for each investigated Group (n=20) and 24 h after photo-activation, the specimens were 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 tons over 1 min to obtain a pellet.

To measure the degree of conversion, the pellet was then placed into a holder attachment into the spectrophotometer Nexus-470 FT-IR (Thermo Nicolet, Vernon Hills, Illinois, USA). The Fourier transform infrared spectroscopy (FT-IR) spectra for both uncured and cured specimens 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:

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 internal reference aromatic C=C (peak at 1610 cm^{-1}) before and after curing of the specimens.

Vickers Hardness Measurements

For this technique, five specimens for each investigated Group were made (n=20) and then the Vickers hardness was measured on the top and the bottom surfaces of the specimens. The Vickers hardness test was performed in a hardness testing machine, MMT-3 Hardness 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 using a load of 50 gf (gram force) during 30 s. Each surface of the specimen was divided into 4 equal quadrants. On each surface, the top (turned to the light source) and bottom (opposite to the light source) surfaces, one indentation took place for each quadrant. Eight indentations were taken from each specimen (4 to the top and 4 to the bottom). The hardness mean values were calculated for each surface.

The data for degree of conversion and hardness were statistically analyzed by Analysis of Variance (ANOVA) using a confidence interval of 95% and Tukey's test.

RESULTS

Degree of Conversion

The Table 2 shows the degree of conversion (DC%) mean values obtained from different dental composites and different light guide tips. The degree of conversion values varied from 67.99% (±1.00) to 55.63% (±2.27) for nanofilled resin photo-activated by fiber optic and polymer light guide tips, respectively. For microhybrid resin, the de-

Table 1. Characteristics of the light guide tip used in the study.

Light-Curing Unit	Light Guide Tip	Diameter entry	Diameter exit	Geometry
Ultrablue IS	Fiber Optic	11mm	8mm	Turbo
	Polymer	10mm	8mm	Turbo

gree of conversion values varied from 68.37% (±1.02) to 55.71% (±2.54) when fiber optic and polymer light guide tips were used, respectively.

ANOVA showed that the degree of conversion was influenced by light guide tips (P<.001), however differences were not observed for different dental composites. According to the results presented, the fiber optic light guide tip presented higher values for DC% regardless the type of dental composite used.

After 24 hours, using the irradiation time recommended by the manufacturers (20 seconds), DC% of microhybrid resin and nanofilled resin, were not statistically different (P=0.988) when the different light guide tips were used (P=1). Therefore, the results suggested that the light guide tips used had a significant (P<.001) impact on the DC%, whereas the type of resin did not influence DC%.

Hardness

The Tables 3 and 4 shows the VHN mean values (Kgf/mm^2) for the top and bottom surfaces for each Group measured. The ANOVA showed that the hardness values was influenced by light guide tips (P<.001) and was also observed for dental composites (P<.001).

The hardness mean values for the top surface varied from 67.72 (±0.68) to 51.58 (±1.39) for nanofilled resin photo-activated by fiber optic and polymer light guide tips, respectively. For microhybrid resin, the hardness mean values for the top surface varied from 72.01 (±0.71) to 61.72 (±1.34) when fiber optic and polymer light guide tips were used.

As can be seen in Table 5, there was statistical significant differences between top and bottom surfaces (P<.001). The top surface showed the higher mean values than the bottom surface.

The hardness mean values of the specimens photo-activated with fiber optic light guide tip showed highest mean values when compared with the mean values for polymer light guide tip. The differences were statistically significant (P<.001).

Table 2. Mean, Standard Deviation (±SD) and P value for degree of conversion.

Light Guide Tip	Dental Composite	Mean	SD	*	P value
Fiber Optic	Nanofilled Resin	67,99	1,00	a	0,988
	Microhybrid resin	68,37	1,02	a	
Polymer	Nanofilled Resin	55,63	2,27	b	1
	Microhybrid resin	55,71	2,54	b	

* Different letters denote significant difference (P<.001).

Table 3. Hardness mean values, Standard Deviation (±SD) and and P value for the top surfaces of the dental composite resin photo-activated with different light guides tips.

Light Guide Tip	Dental Composite	Mean	SD	*	P value
Fiber Optic	Microhybrid resin	72,01	0,71	a	<.001
	Nanofilled resin	67,72	0,68	b	
Polymer	Microhybrid Resin	61,72	1,34	c	
	Nanofilled resin	51,58	1,39	d	

* Different letters denote significant difference (P<.001).

Table 4. Hardness mean values, Standard Deviation (±SD) and P value for the bottom surfaces of the dental composite resin photo-activated with different light guides tips.

Light Guide Tip	Dental Composite	Mean	SD	*	P value
Fiber Optic	Microhybrid Resin	61,77	0,40	a	<.001
	Nanofilled resin	52,04	1,59	c	
Polymer	Microhybrid Resin	56,03	1,81	b	
	Nanofilled resin	42,51	1,12	d	

* Different letters denote significant difference (P<.001).

Table 5. Hardness mean values for the top and bottom surfaces and corresponding B/T ratio of the dental composite resin photo-activated with different light guides tips.

Light Guide Tip	Dental Composite	Top Surface	Bottom Surface	B/T%
Fiber Optic	Nanofilled Resin	67,72	52,04	82,75
	Microhybrid resin	72,01	61,77	85,75
Polymer	Nanofilled Resin	51,58	42,51	82,41
	Microhybrid resin	61,72	56,03	84,3

DISCUSSION

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, reactive sites (double bonds) are susceptible to hydrolization or oxidation and, thereby, lead to a degradation of the material.[27,28]

Then, the degree of conversion is an important tool to determine the final physical, mechanical, and biological properties of composite resins, since it has been showed that composite properties tend to improve as the degree of conversion attained during photo-polymerization is increased.[15] In addition, increased cure may result in a lower amount of uncured, potentially leachable monomer, leading to a more biocompatible restoration.[29] Moreover, uncured functional groups can act as plasticizers, reducing the mechanical properties.[30]

The minimum DC% for a clinically satisfactory restoration has not been precisely established. Nevertheless, a negative correlation of in vivo abrasive wear depth with DC has been found for values in the range of 55-65%. This suggests that, at least for occlusal restorative layers, DC values below 55% may be contraindicated.[3] According to some authors 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.[21-33]

In this our experiment, the DC mean values ranged from 67.99 to 68.37 % for fiber optic and 55.63 to 55.71% for polymer light tip, and according to these results presented on Table 3 there was statistical difference in DC (%) mean values between the light guide tips. These findings showing that the two light guide tips were able to light-cure microhybrid as nanofilled composite resins with 2 mm thickness. However, the degree of conversion of composites photo-activated with the fiber optic light guide tip was statistically higher than those observed with polymer light guide tip. This fact can be explained by the material of the tip. Polymer materials provide the dispersion of the light in its itinerary decreasing power density.[3]

In a previous study, Soares et al[3] reported that the type of light guide tip material did not present a statistical significant difference on the final DC (%) of dental composite. However, this result can be explained by the low power density delivered by the light-curing units, which was around 130/140 mW/cm^2. As shown by Galvão et al[24] it was not observed statistical significant difference for degree of conversion of dental composites photo-activated with the different light guide tips. However, this result may be explained by the low quality of the fiber optic light guide tip used.

Factors such as light source, irradiation times, power density, correct wavelength of the light source, light-tip size, distribution, light guide tip and material's composition can influence the degree of conversion (%) and, then, the final characteristics of the dental composite resin.[13] All these factors strongly influence the degree of conversion (%), which is a number of ethylene double carbon bonds that are converted into single bonds of the composite resin obtaining optimal chemical-physical and clinical performance. Therefore, it plays an important role in determining the ultimate success of the restoration.[34-36]

Hardness evaluation is a widely used test to examine composite curing and, as a consequence, the efficiency of the light source.[37] It is applied especially to restorative materials that are used where high bite forces and stresses can exacerbate inherent material defects, resulting in inadequate fracture resistance of the materials.[38,39]

According to some authors,[40] there is still no consensus for the Vickers hardness be considered optimal. Some authors believe that for composite resins, a hardness values can exceeding 50 (VHN) to be considered ideal.[41] In this investigation, nanofilled and microhybrid dental composite resins photo-activated with the fiber optic light tip showed hardness mean values at the top surface above 50 VHN. At the bottom surface, only one Group did not reach 50 VHN when polymer tip was used.

Johnston et al[42] believe that the curing efficiency could be measured by the ratio between bottom and top surface hardness (B/T), which should be 90%, however according to some authors,[43,44] the bottom surface of the specimens can be at least 80% of the hardness of top surface, which is consistent with our findings which showed a ratio of 82.41% and 85.75% between top and bottom surfaces of the specimens cured with fiber optic and polymer light tips, respectively, as shown in Table 5.

As shown in Table 3 and 4 statistical significant differences among the light guide tips (P<.001) and dental composites (P<.001) were observed. Statistical differences were also found when comparing top and bottom surfaces, for both the light guide tips and for the dental composites. On the top surface, the power density is usually sufficient for adequate polymerization, however, on the bottom surface the light of the light-curing unit frequently disperses, and then the polymerization can be compromised. 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.[5,27,45] The results obtained in this study, showed statistical significant differences among the dental composites, demonstrating that the type of resin used can also influence the results of hardness obtained. In this study a microhybrid dental composite showed higher hardness mean values than the nanofilled for both, top and the bottom surfaces.

According to Wu et al[46] the composite resins based on nanotechnology describe research or products where critical component dimensions are in the range of 0.1 to 100 nanomers (nanometric scale), through several physical and chemical methods. In 2003, the first composite resin with these characteristics was introduced in Dentistry. The goal was to use nanotechnology to create a composite that offers the polish retention of a microfill with the strength of a hybrid composite.[47,48] However, in our study it was observed that the nanofilled composite presented hardness values lower than those microhybrid, for the top and bottom surfaces especially photo-activated with the polymer light guide tip of.

This can be primarily explained by the difference in the composition of the resin matrix, filler size, filler volume, and filler type of the materials. Although the organic phase of composite resins evaluated in this study are similar, there are differences in the inorganic phase (size, shape, and volume filler content). The filler volume% of the microhybrid composite and nanofilled composite are also similar around of 60%, however the microhybrid filler size has an average medium size of 0.6µm and nanofilled nanoparticles of 20nm size fillers.[17,49] This statement may explain the results found in this study.

In this study, it was observed that there were difference in the degree of conversion and hardness of composite resins photo-activated with fiber optic and polymer light tips, showing that the materials of the light guide tips used may have direct impact on the power density, which would have great influence on the physical, chemical and mechanical and properties of composite resins.

CONCLUSIONS

The results obtained in this study indicated that the light guide tips used in the photo-activation (fiber optic and polymer) promoted differences in the degree and conversion, regardless of the type of dental composite. The fiber optic light guide tip provided higher degree of conversion. However, hardness was influenced by light guide tip, but also by the type of dental composite. The microhybrid dental composite photo-activated by fiber optic light guide tip provided the highest values for hardness, either top and bottom surfaces.

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