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ORIGINAL ARTICLE

Effect of light curing protocol on degree of conversion of composites

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Abstract

Objective. To evaluate the degree of conversion (DC) of two light-cured composites with different protocols of light curing. ***Materials and methods.*** One hundred and ninety two specimens ($n = 8$) were prepared (5 mm × 2 mm) according to experimental groups: two composite resins (Filtek Supreme and four seasons); three light curing protocols [20 s with the tip of the light curing unit (LCU) device touching composite surface (C); 20 s with the tip of the LCU at 8 mm distant from composite surface (D); and tip of the LCU at 8 mm distant from composite surface and polymerization time required to obtain a radiant exposure of 16 J/cm^2 (DS)]. Four LCUs (Bluephase 16i, Ultralume LED 5, XL 3000 and Optilux 501C) were used. DC of the bottom and top surface of specimens were measured using a FTIR spectrometer. Data were statistically analyzed by 3-way split splot ANOVA and Tukey's test (alpha = 0.05). ***Results.*** The results showed that DC of the top surface was higher than the bottom at all experimental conditions ($p < 0.0001$). Overall, the curing at 8 mm of distance did not affect conversion rate on the top surface ($p > 0.05$), but bottom surfaces showed DC reduction ($p < 0.05$). The highest monomer conversion values were observed for C and DS situations. ***Conclusion.*** The distance between the LCU and material surface and radiant exposure can affect the DC. Polymerization at distance should be performed with curing units with higher light power and/or extended exposure time.

Key Words: *polymerization, composite resin, degree of conversion*

Introduction

Resin-based materials used in dental restorations have a chemical complex composition with a high diversity of monomers and additives that vary with type and concentration [1]. The most used monomers are BisGMA, UDMA, TEGDMA and more recently BisEMA [2]. Furthermore, others additives, like photoinitiators, co-initiators and inhibitors, are present in the composition of composites [3]. The microhybrid and nanofilled composites are materials commonly used in anterior and posterior dental restorations; nanocomposites had changes in inorganic content and are based on nanotechnology with nano-sized particles, showing similar mechanical properties of universal microhybrids and high aesthetic quality [4,5].

The polymerization of resin-based materials occurs through conversion of monomers into polymer with high cross-linked structure [6], widely influencing the clinical performance of these materials [7]. Incomplete polymerization affects physical properties [8], leading to elution of residual monomers [9]. The elution is caused by ingress of solvents on the matrix, expanding spaces among polymeric chains and releasing soluble substances, such as monomers [3]. This releasing may be cytotoxic, promoting allergic reactions, besides stimulating the increase of bacteria [10].

These light-activated materials can be cured with quartz-tungsten-halogen (QTH) and light-emitting diode (LED) curing units. The QTH device has been used for a long time in dentistry, but it shows irradiance reduction over time by the lamp and filter

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degradation, due to the bulb overheating [11]. LED showed less degradation, blue emission without filter. However, 1st and 2nd generation LED devices have a narrow wavelength spectrum, specific for a camphoroquinone photoinitiator, unlike QTH and 3rd LED generation curing units, which show a wide spectrum [11,12].

Recent studies show that the increase of distance between the tip of the light-curing unit (LCU) from the increment of composite may jeopardize the polymerization due to a decrease of irradiance [13] and dispersion and scattering of light by restorative material can hinder the monomer conversion on deeper surfaces [14], increasing the action of solvents and lixiviation process. Use of the LCU with higher irradiance has been recommended or increasing the radiant exposure, by the extended irradiation time [15], in these situations.

The aim of this study was to evaluate the degree of conversion (DC) of two composites with different light-curing protocols. The research hypotheses tested were: (1) the DC would decrease with the increase of distance of the tip of LCUs, (2) the higher irradiance would improve the DC and (3) there would be no difference between composites.

Materials and methods

In this study, a nanofilled (Filtek Supreme, shade A2; 3M ESPE, St. Paul, MN) and a microhybrid (4 Seasons, shade A2; Ivoclair Vivadent, Bürs, Austria) composite resin were used. One hundred and ninety-two cylindrical specimens ($n = 8$) were prepared in circular Teflon molds for each composite, with 5 mm as the internal diameter and 2 mm thickness, held between two glass slabs, separated by Mylar matrix strips and then pressed with a 500 g load. The cavity was filled with one increment and randomly polymerized according to experimental groups: two composite resins (Filtek Supreme; four seasons); three light curing protocols [20 s with the tip of LCU touching the composite surface (C, control); 20 s with the tip of the LCU at 8 mm distant from composite surface (D, at distance); tip of the LCU at 8 mm distant from composite surface and polymerization time required to obtain a radiant exposure of 16 J/cm^2 (DS, at distance and standardized 16 J/cm^2)] and four LCUs (Bluephase 16i; Ultralume LED 5; XL 3000; and Optilux 501C). Polymerization was performed with the tip of the light source positioned in a device, the light-curing tip being 0 mm or 8 mm distant from the top surface of the sample (controlled by an electronic digital caliper). The LCUs used irradiance and radiant exposure considering the time of 20 s and polymerization time required to obtain a radiant exposure of 16 J/cm^2 are presented in Table I.

Each specimen was removed of the mold and dry stored in a lightproof vial at 37°C for 24 h. After this period, the top and bottom surfaces of each specimen were polished with aluminum oxide discs (Sof-Lex Pop-On, 3M ESPE, St. Paul, MN) during 30 s each grit-disc (medium, fine and superfine) at low speed, discarded each second specimen.

The degree of conversion of top and bottom surfaces were measured using Fourier Transform infrared spectroscopy–FTIR (Spectrum 100 Optica; PerkinElmer, Waltham, MA), equipped with an universal attenuated total reflectance (ATR) device with a horizontal ZnSe crystal (Pike Technologies, Madison, WI). A preliminary reading for the uncured material was recorded under the following conditions: 1665–1580 cm^{-1} frequency range at 4 cm^{-1} of resolution in absorbance mode. Additional FTIR spectra of cured samples were obtained at 24 h. DC was calculated using a baseline technique [16] based on band ratios of 1638 cm^{-1} (aliphatic carbon-to-carbon double bond) and 1608 cm^{-1} (aromatic component group) as an internal standard between the cured and uncured samples.

The results of DC on the top and bottom surface were submitted to three-way split-splot ANOVA and Tukey's test at the pre-set alpha of 0.05. The factors light curing protocols, light-curing units and composite resins were considered in the parcels and the factor surface (top and bottom surfaces) was considered in the sub-factor.

Table I. Light-curing units used, irradiance, radiant exposure considering the time of 20 s and polymerization time required to obtain a radiant exposure of 16 J/cm^2.

Light-curing unit		Irradiance (mW/cm^2) at 0 mm	Irradiance (mW/cm^2) at 8 mm	Radiant exposure (J/cm^2) at 0 mm (C)	Radiant exposure (J/cm^2) at 8 mm (D)	Necessary time (s) to reach 16 J/cm^2 (DS)
Bluephase 16i	LED 2nd generation	1100	400	22	8	40
Ultralume 5	LED 3rd generation	700	280	14	5.6	57
XL 3000	Halogen 1	450	200	9	4	80
Optilux 501C	Halogen 2	600	290	12	5.8	55

Bluephase 16i: Vivadent, Bürs, Austria; Ultralume LED 5: Ultradent, South Jordan, UT; XL 3000: 3M/ESPE, Grafenau, Germany; Optilux 501C: Demetron/Kerr, Danbury, CT.
Irradiance was monitored by a radiometer (model 100; Demetron/Kerr, Danbury, CT).

Table II. Degree of conversion (%) of the top surface (standard deviation), according to composite resin, light-curing unit and photoactivation protocol.

	LED 2	LED 3	Halogen 1	Halogen 2
Nanofilled composite				
Control (C)	50.09 (3.64)^{ABa}	46.15 (2.78)^{ABa}	44.69 (3.54)$^{\star Bb}$	51.45 (3.52)^{Aa}
Distanced (D)	43.83 (2.71)$^{\star Bb}$	49.09 (2.94)^{ABa}	46.73 (4.29)^{Bb}	54.11 (3.05)^{Aa}
Distanced with 16 J/cm^2 (DS)	45.14 (1.99)$^{\star Bab}$	46.86 (3.28)$^{\star ABa}$	51.90 (3.13)$^{\star Aa}$	51.16 (3.81)$^{\star Aa}$
Microhybrid composite				
Control (C)	50.28 (3.97)^{Aa}	49.18 (3.30)^{Ab}	50.63 (3.78)^{Ab}	54.55 (3.71)^{Aa}
Distanced (D)	54.39 (4.71)^{Aba}	49.62 (3.71)^{BCb}	47.09 (4.37)^{Cb}	56.29 (2.67)^{Aa}
Distanced with 16 J/cm^2 (DS)	55.38 (4.85)^{ABa}	54.68 (4.22)^{Ba}	60.16 (2.24)^{Aa}	55.28 (3.77)^{ABa}

Distinct letters (capital in the row and lower in the column within of each composite) are statistically different ($p \leq 0.05$).
*Differences of microhybrid composite resin ($p \leq 0.05$).

Results

The DC results of the top and bottom surfaces are presented in Tables II and III, respectively. For all experimental conditions, the top surface showed higher DC than the bottom surface ($p < 0.0001$).

On the top surface, analyzing the nanofilled composite, Halogen 2 showed higher DC than Halogen 1 in control situation (C), Halogen 1 and LED 2 (2nd generation) with the light-curing tip at 8 mm distant from composite (D) and LED 2 at 8 mm distance and radiant exposure standardized at 16 J/cm^2 (DS). Comparing the light curing protocols, there were no statistical differences between them for the groups LED 3 (3rd generation) and Halogen 2. For Halogen 1, there was no difference between C and D, but DC values were significant lower than the DS situation. For LED 2, the highest the groups cured for Halogen 2 showed the highest DC values, without statistical differences for LED 2 in all situations tested DC was obtained in C situation, with statistical difference in D.

For the top surface of microhybrid composite, in C situation, there was no difference between the polymerization modes tested. In D situation, Halogen 1 showed the lowest DC without difference for LED 3 and Halogen 2 presented the highest conversion rate. In DS situation, Halogen 1 showed higher DC than LED 3. Comparing the composite resins, microhybrid composite showed higher DC than nanofilled in the DS situation for all LCUs, for Halogen 1 in C and for LED 2 in D situation.

For the bottom surface, the D situation had lower DC than C and DS for both composite resins, except group microhybrid composite cured with Halogen 2. Comparing the LCUS, Halogen 1 showed significantly lower DC than for other groups in C and D situations for nanofilled and in the C situation for microhybrid material. Nanofilled composite showed lower DC than microhybrid only in the situation D light-cured with Halogen 1 device.

Discussion

The radiant exposure may be directly proportional to higher monomer conversion into polymer of the composite resin [15]; it is affected by the curing time and

Table III. Degree of conversion (%) of the bottom surface (standard deviation), according to composite resin, light-curing unit and photoactivation protocol.

	LED 2	LED 3	Halogen 1	Halogen 2
Nanofilled composite				
Control (C)	39.73 (4.06)^{Aa}	39.34 (3.57)^{Aa}	33.25 (2.69)^{Ba}	37.78 (2.28)^{Aa}
Distanced (D)	33.43 (2.67)^{Ab}	30.47 (3.25)^{Ab}	25.75 (1.64)$^{\star Bb}$	32.07 (1.59)^{Ab}
Distanced with 16 J/cm^2 (DS)	40.21 (3.69)^{Aa}	36.44 (3.47)^{Aa}	36.92 (3.36)^{Aa}	39.31 (2.62)^{Aa}
Microhybrid composite				
Control (C)	37.87 (1.20)^{Aa}	40.96 (1.85)^{Aa}	31.89 (3.57)^{Bb}	38.28 (2.05)^{Aa}
Distanced (D)	31.57 (4.61)^{Ab}	31.88 (3.39)^{Ab}	31.68 (2.73)^{Ab}	35.06 (3.41)^{Aa}
Distanced with 16 J/cm^2 (DS)	39.28 (3.37)^{Aa}	38.50 (4.41)^{Aa}	38.82 (3.55)^{Aa}	34.88 (1.56)^{Aa}

Distinct letters (capital in the row and lower in the column within of each composite) are statistically different ($p \leq 0.05$).
*Differences of microhybrid composite resin ($p \leq 0.05$).

irradiance and influences the formation of polymer chains. The increase of exposure time causes an increase in the energy dose, even without changes in the irradiance [15]; this fact is important principally during light curing at distance with devices of lower power density during the clinical procedure. In this present study, the highest radiant exposure was due to an increase of the light curing time with different LCUs. However, a high initial irradiance causes more growth centers of polymers, increasing cross-linked structure [17], but the fast vitrification may limit the polymerization process [18].

The increase of the distance to the LCUs tip did not affect the DC of the composite top surface, except for nanofilled composite resin cured with LED 2. The same radiant exposure for all curing devices did not increase the DC at the top surface, except for Halogen 1 for both composites. The highest curing time promotes an increase of radiant exposure, which allows a higher monomer conversion rate [19]. Thus, for LCUs with lower irradiance or cured at a distance the radiant exposure can be increased by the increase of curing time. However, with the same curing time at distance, it is better to opt for LCUs with higher irradiance because, in this situation, there is attenuation of irradiance. The halogen 2 unit with wide wavelength spectrum combined with a sufficient irradiance resulted in adequate DC [11,12].

The bottom surface presented lower DC in all situations. The dispersion and scattering of light by the matrix and fillers of composite decrease the irradiance and can be attributed to this result [20]. Furthermore, only 1 mm of air interposed between tip of the LCU and composite resin decreases the light irradiance in ~10% [21]. Reduced DC values compromise mechanical properties, such as microhardness, as observed in previous studies [19,22].

Overall, same radiant exposure for all light-curing devices did not differ from the control situation (C), but were higher than distanced (D), demonstrating a significant influence of the distance of the light source tip from the composite surface on the DC, principally for the bottom surface. Comparing the LCUs, halogen units with low intensity showed the lowest DC values. Lower irradiance excites less photoinitiators, decreasing the free radicals formation and reducing the monomer conversion rate [23]. In this case, it is recommended to increase the curing time, activating more photoinitiators and, consequently, increasing the degree of C=C conversion.

In some situations, microhybrid composite resin presented higher DC than nanofilled material, principally on the top surface. The composition of both composites tested is similar, based on BisGMA, TEGDMA and UDMA. However, there is an addition of BisEMA in nanofilled, decreasing the others components; this fact could explain the results observed. The monomer BisEMA is less effective on the polymerization process than TEGDMA, reaching a lower monomer conversion; this last monomer has lower molecular weight and allows a polymerization reaction in three points of ether bonds of glycol groups and two carbonyls of methacrylate groups [24]. In contrast, a previous study observed improvement on the DC of mixtures with higher content of BisEMA replacing BisGMA monomer [25]. BisEMA has both group's ether and carbonyl, but these groups are less reactive because high molecular weight leads to difficult polymerization compared to diluent monomers, such as TEGDMA and UDMA, but lower than BisGMA [24,25].

Based on the results of this study, the conclusion is that the DC can be affected by the distance of the LCU tip from composite and by the radiant exposure. Therefore, LCUs with higher irradiance should be preferred and, in special situations, such as polymerization at distance, can increase the exposure time.

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