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Curing Quality of Composites as Influenced by the Filler Content, Light Source and Curing Time

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Abstract

The quality of composite polymerization has been of great concern for researchers. Curing of nanocomposites under long distance (8-mm) and extended light exposure through conventional (halogen and LED) and argon laser lamps is unclear in the literature. This study evaluated the influence of curing modes and filler particle size on hardness and degree of conversion of dental composites photoactivated at an 8-mm distance. Light sources (LED 1100mW/cm2-Bluephase; LED 700mWcm2-Ultra-lume; halogen lamp 450mW/cm2-XL3000; and argon-laser 500mW/cm2-AccuCure), curing times (20 and 60 s), microhybrid (Filtek-Z250) and nanofilled (Filtek-Supreme) resins were investigated. Eighty samples (n=5) were made using Teflon molds. Hardness and degree of conversion were obtained for bottom/top surfaces of 2-mm increments. Data were submitted to ANOVA and Tukey tests (α=5%).Top surfaces showed similar hardness. A 60s exposure time increased bottom hardness and Filtek-Z250 showed higher hardness for curing units except Bluephase. Regarding degree of conversion, bottom/top surfaces showed similar means at 60s; at 20s, bottom/top surfaces revealed similar means only for Filtek-Z250 cured by Bluephase and Ultra-lume. High irradiance and extended exposure time can improve hardness and conversion on bottom surface. Microhybrid resin presented better conversion of monomers than the nanofilled composite under higher irradiance and extended exposure times.

Keywords: Degree of conversion; Hardness; Light emitting diode; Halogen lamp; Laser

Introduction

Since the introduction of light-cured resin-based composites, the quality of polymerization has been of concern for researchers worldwide [1]; its clinical success is directly related to its degree of cure [2]. Primary clinical manifestations of uncured composite are untoward symptoms when chewing. Inadequate polymerization may stimulate the growth of certain caries-related bacteria around restoration margins and cause adverse biological effects in mammalian cells [3]. Moreover, the majority of unreacted components may be released within the first few days and may enter human body via skin, oral and gastrointestinal mucosa, dentine and pulp [4]. The lower the degree of conversion (DC) of a composite resin, the higher its solubility [5]. Based on such findings, it would be wise to investigate curing protocols in an attempt to increase the DC of resin-based restorative materials.

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) [6]. One of the most used indirect methods to evaluate the degree of polymerization of the composite resins is the hardness test. Direct laboratory tests are more effective in measuring the DC than the indirect ones, the latter is influenced by the type of polymeric network formed after photocuring, with the cross-link chains providing higher hardness levels if compared to the linear ones [7]. However, the vibrational spectroscopy test, which is not influenced by the types of network formed during polymerization, is more effective in quantifying monomers converted to polymers [8]. Therefore, both tests should be used to evaluate the polymer structure of photocured composite resins, since they provide complementary results, allowing for a better understanding of the polymeric network of the resin-based materials.

The light-curing of composite resins depends on such factors as material translucency, filler particle size, curing time, incremental thickness, light unit irradiance, and the distance between the curing light tip and the sample increments [5-11]. The efficiency of the radiation source for photopolymerization of these materials has thus become increasingly important [12]. Class I and II cavities, in some cases, require distances such 8 mm between the tip of the curing-light and the resin surface increment [13], resulting in a decrease in light irradiance. In view of this fact, it must be investigated curing modes which can overcome the irradiance decrease in these conditions to provide a network polymer with satisfactory conversion of monomers.

The application of nanotechnology to resin composites has been one of the most important advances in this field in the last few years. Like the microhybrid filler based resins, the nanofilled ones have revealed satisfactory outcomes concerning tensile/compressive strength and resistance to fracture [10] and are recommended to be used in posterior restorations. However, a fewer studies have focused to investigate the influence of nanofillers on hardness and DC of composites cured under long distances by light sources with different irradiance levels and increased curing times. Although the argon laser has a narrow wavelength band that is optimally correlated to the absorption peak for initiating the polymerization of composite resins [14], its effectiveness to cure nanofilled composites with increased distance in comparison with conventional LED's and halogen lamp must also be investigated.

This study evaluated the influence of curing modes and filler particle size on hardness and degree of conversion (DC) of dental composites

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photoactivated at an 8-mm distance. The null hypotheses tested were that (1) there would be no statistically significant difference among composites with different filler particle sizes, curing units and times, and bottom and top surfaces concerning hardness values; and (2) there would be no statistically significant difference among composites with distinct filler particle sizes, several curing devices and curing times, and the bottom and top surfaces of the resin increments concerning DC values.

Materials and Methods

Knoop Hardness Number (KHN) and Degree of Conversion (DC) of two commercially available composite resins (nanofilled – Filtek Supreme XT, 3M ESPE; and micro-hybrid – Filtek Z250, 3M ESPE) were tested (Table 1). Four light curing devices with different irradiance levels such as 2nd generation LED (Bluephase 16i - BP), 3rd generation LED (Ultra-Lume LED 5 - UL), halogen lamp (XL 3000 - XL), and argon laser (AccuCure 3000 - AC) (Table 2) were used to photo-cure the materials at two irradiation times (20 seconds and 60 seconds).

Specimens were assigned to sixteen groups based on the factorial product: composites (2) x light curing units (4) x times (2). Eighty test specimens (n=5) were made/fabricated using individual cylindrical Teflon molds 5 mm in diameter and 2 mm high. Each mold was placed onto a glass slide to obtain a smooth bottom surface of the composite resin, which was inserted in the mold in a single increment. A polyester strip was placed on top of the uncured material and a load of 500 grams was applied for 30 seconds to provide a smooth top surface. Samples were then photo-cured at a light source distance of 8 mm established by using a digital caliper [15]. The power output of all curing devices was measured with a radiometer (Demetron, Serial 105415, Research Corporation) at distances of 0 and 8 mm away from the radiometer reading area to monitor the decrease in light irradiance at 8 mm (Table 2).

After polymerization, the polyester strips were discarded and the samples were removed from the molds and stored dry in a light proof container at 37°C for 24 hours. Bottom and top surfaces of the specimens were then polished using 1200-grit abrasive paper attached to a polishing machine (APL-4, Arotec, São Paulo, SP, Brazil) under continuous water cooling to remove the resin rich surface layer. The samples were then washed, air-dried and stored dry again for 24 hours at 37°C. KHN and DC were measured considering the bottom and top surfaces of each specimen.

Composite	Manufacturer	Code	Classification/composition		Lot
Filtek Supreme XT (A2E shade)	3M ESPE (St Paul, MN, USA)	FS	Nanofilled Filler: 57.7% vol, 72.5% wt, SiO2 nanofiller (75 nm), ZrO2/SiO2 nanoclusters (0.6 - 1.4 µm) Polymeric matrix (42.3% vol, 27.5% wt): Bis-GMA, Bis-EMA, EDMA, TEGDMA		5BU
Filtek Z250 (A2 shade)	3M ESPE (St Paul, MN, USA)	FZ	Micro-hybrid Filler: 60% vol, 82% wt, ZrO2/SiO2, 0.6 µm mean size Polymeric matrix (40% vol, 18% wt): Bis-GMA, Bis-EMA,UDMA, TEGDMA		7LP
Curing device	**Manufacturer**	**Code**	**Radiometer* data 0mm**	**Radiometer* data 8mm**	**Serial**
Bluephase 16i	Vivadent	BP	1100	540	1633858
Ultra-Lume LED 5	Ultradent	UL	700	350	511372
XL 3000	3M ESPE	XL	450	250	208811
AccuCure 3000	LaserMed	AC	500	300	0404490

Bluephase 16i – Vivadent, Bürs, Austria; Ultra-Lume LED 5 – Ultradent, South Jordan, USA; XL 3000 – 3M ESPE, Grafenau, Germany; AccuCure – LaserMed, W Jordan, UT, USA.
*Demetron Research Corporation model 100, serial 105415 (Kerr Corporation - Orange).

Table 1: Composite resins and light curing devices used.

			Curing time	
Surface	Light source	Composite	20 s	60 s
Top	BP	FS	96.0 (13.8)* Aa	97.81 (12.84)* Aa
	BP	FZ	105.4 (13.4)* Aa	113.16 (32.6)* Aa
	UL	FS	95.8 (13.2)* Aa	103.1 (28.2)* Aa
	UL	FZ	93.8 (17.7)* Aa	98.9 (15.2)* Aa
	XL	FS	85.5 (11.0)* Aa	120.3 (40.4)* Aa
	XL	FZ	118.1 (32.5)* Aa	99.9 (21.6)* Aa
	AC	FS	93.6 (12.8)* Aa	99.5 (13.4)* Aa
	AC	FZ	89.6 (15.6)* Aa	109.1 (16.2)* Aa
Bottom	BP	FS	54.2 (5,1)Ba	64.1 (5,1)Aa
	BP	FZ	55.9 (3,2)Ba	68.9 (4,1)Aa
	UL	FS	43.0 (5,7)Bb	56.1 (9,4)Ab
	UL	FZ	51.7 (10,8)Ba	69.0 (7,2)Aa
	XL	FS	34.5 (4,4)Bb	49.8 (4,1)Ab
	XL	FZ	41.0 (3,92)Ba	57.3(10,1)Aa
	AC	FS	47.8 (7,2)Bb	55.1 (6,9)Ab
	AC	FZ	50.6 (4,5)Ba	62.0 (6,8)Aa

*Different from the bottom by Analysis of Variance (ANOVA) ($p<0.05$).
Mean values with the same letter were not statistically different by Tukey's test ($p>0.05$). (Lowercase letters for vertical and capital letters for horizontal).

Table 2: KHN means (standard deviations) considering composite types, light sources, curing times and surface of samples.

Knoop hardness test

KHN values were obtained using a digital microhardness tester (HMV-2T E, Shimadzu Corporation, Tokyo, Japan). Five KHN measurements, under a load of 25 grams for 10 seconds, were taken: one at the central portion, at which light was applied, and the other four 100μm away from the central portion.

Degree of conversion (DC)

After polymerization, the specimens were removed from the matrices and stored dry in light-proof containers at 37ºC during 24 hours. The DC measurements were recorded in absorbance mode with FTIR spectrometer (Spectrum 100 FTIR, PerkinElmer, São Paulo, SP, Brazil) coupled to a zinc selenide multiple (six) reflection Attenuated Total Reflection (ATR) accessory, refraction index of 2.4 at 1000 cm^{-1} (PerkinElmer, São Paulo, SP, Brazil), operating under the following conditions [16]: 650-4000 cm^{-1} wavelength; 4 cm^{-1} resolution; 32 scans. The percentage of unreacted carbon-carbon double bonds (C=C) was determined from the ratio of absorbance intensities of aliphatic C=C (peak at 1638 cm^{-1}) against the internal standard (aromatic C-C, peak at 1608 cm^{-1}) before and after curing the specimen. The degree of conversion was determined by subtracting the % C=C from 100%.

Statistical analysis

Statistical analysis was performed using SAS (Statistical Analysis System 8.2) software at a significance level of 5%. After verifying normal distribution of errors and the homogeneity of variance using Shapiro-Wilk's test and Levene's test, respectively, variables concerning KHN and DC were analyzed separately using analysis of variance (subdivided parcels). The parcels represented the factorial: composite resins (2) x light curing units (4) x curing times (2); while the subparcels were assigned to the bottom and top surfaces. Tukey's test was used to make multiple comparisons among the groups (α=0.05).

Results

Knoop hardness number

Results concerning the microhardness test are shown in Table 3. Analysis of Variance (ANOVA) showed that there was a statistically significant difference between top and bottom surfaces of the samples ($p<0.001$), and towards the interaction between light curing unit and resin composite ($p=0.04$). Tukey test showed higher KHN values for the top surface considering all experimental conditions. No statistically significant difference was found among the times and resin composites tested concerning the top surface. Higher KHN values were observed for the bottom surface at 60 seconds. The microhybrid resin (FZ) photocured with UL, XL and AC showed HKN values higher than those observed for the nanofilled one (FS). BP revealed no significant difference between the composite resins tested.

Degree of conversion

Results of DC are listed in Tables 3 and 4. Analysis of Variance (ANOVA) showed statistical differences towards the interactions among surface versus composite resin versus curing time ($p=0.04$); between light curing unit versus composite resin ($p<0.001$); and between light curing unit versus surface ($p< 0.001$). Tukey test revealed no statistically significant difference among DC values concerning top and bottom surfaces at 60 seconds. The top surface showed higher DC values at 20 seconds, except FZ samples photocured with BP and UL.

No statistically significant difference was found among top surface DC values at 20 seconds and 60 seconds, except for FS increments photocured with UL, showing higher DC values at 60 seconds, when compared to those obtained at 20 seconds. However, samples cured at 60 s showed higher DC values on the bottom surface, except for FZ samples photocured with BP. FZ showed higher DC values when cured with BP (on both surfaces cured at 20 seconds and 60 seconds) or with XL (on top surface at 60 seconds). No statistically significant difference was found between the resin composites tested (Table 4), regarding the other experimental conditions.

Table 4 shows DC values according to the light curing units. There were no statistically significant differences among DC values obtained for FS at 20 seconds and 60 seconds and among DC values for FZ at 20 seconds concerning the top surface. Moreover, FZ samples photocured with XL at 60 seconds showed lower DC values when compared to those photocured with BP, UL and AC. FS showed no statistically significant difference among DC values of samples cured at 60 seconds regarding

			Curing time	
Surface	Light source	Composite	20 s	60 s
Top	BP	FS	35.3 (1,3)*Aa	34.9 (2,0)Aa
	BP	FZ	35.3 (2,7)Aa	36.4 (1,1)Aa
	UL	FS	33.5 (1,8)*Bb	35.2 (2,0)Ab
	UL	FZ	37.2 (3,8)Aa	37.4 (3,0)Aa
	XL	FS	34.1 (2,6)*Aa	35.2 (2,9)Aa
	XL	FZ	34.1 (2,4)*Aa	31.4 (1,3)Ab
	AC	FS	35.7 (2,9)*Aa	34.8 (2,3)Aa
	AC	FZ	36.1 (0,6)*Aa	36.7 (0,8)Aa
Bottom	BP	FS	32.4 (1,0)Ba	35.5 (1,4)Aa
	BP	FZ	35.5 (2,5)Aa	36.3 (1,3)Aa
	UL	FS	28.1 (2,1)Bb	33.8 (1,3)Ab
	UL	FZ	36.0 (1,2)Ba	37.9 (2,5)Aa
	XL	FS	26.4 (3,9)Ba	32.5 (2,5)Aa
	XL	FZ	29.3 (3,3)Ba	31.4 (1,3)Aa
	AC	FS	31.6 (2,9)Ba	35.4 (1,8)Aa
	AC	FZ	33.2 (1,5)Ba	34.5 (2,5)Aa

*Different from the bottom by Analysis of Variance (ANOVA) ($p<0.05$).
Mean values with the same letter were not statistically different by Tukey's test ($p>0.05$). (Lowercase letters for vertical and capital letters for horizontal).

Table 3: Degree of conversion means (standard deviations) considering composite types, light sources, curing times and surface of samples.

			Curing time	
Surface	Composite	Light source	20 s	60 s
Top	FS	BP	35.3 (1,3)a	34.9 (2,0)a
		UL	33.5 (1,8)a	35.2 (2,1)a
		XL	34.1 (2,6)a	35.2 (2,9)a
		AC	35.7 (2,9)a	34.8 (2,3)a
	FZ	BP	35.3 (2,7)a	36.4 (1,2)a
		UL	37.2 (3,8)a	37.4 (3,0)a
		XL	34.1 (2,4)a	31.4 (1,3)b
		AC	36.1 (0,6)a	36.7 (0,8)a
Bottom	FS	BP	32.4 (1,0)a	35.5 (1,4)a
		UL	28.1 (2,1)ab	33.8 (1,3)a
		XL	26.4 (3,9)b	32.5 (2,5)a
		AC	31.6 (2,9)a	35.4 (1,8)a
	FZ	BP	35.5 (2,5)a	36.3 (1,3)a
		UL	36.0 (1,2)a	37.9 (2,5)a
		XL	29.3 (3,3)b	31.4 (1,3)b
		AC	33.2 (1,5)ab	34.5 (2,5)ab

*Different from the bottom by Analysis of Variance (ANOVA) ($p<0.05$).
Mean values with the same letter were not statistically different by Tukey's test ($p>0.05$). (Lowercase letters: vertical analysis).

Table 4: Degree of conversion means (standard deviations) comparing the light sources at each composite type, curing time and surface of samples.

the bottom surface. Nevertheless, FS increments photocured with XL at 20 seconds showed lower DC values than those polymerized with BP and AC. FZ samples photocured with XL showed lower DC values than those photocured with BP and UL at 20 seconds and 60 seconds.

Discussion

The first null hypothesis tested in the present study was rejected. Top surfaces showed higher KHN values concerning all types of photocuring devices, resin and time (Table 3). This finding might be due to a low light transmittance reaching the bottom surface of each specimen [17], interfering with the DC of the monomers [15].

Low irradiance levels might lead to a greater number of linear than cross link chains8. Linear chains show lower hardness than cross link ones, the latter of which were possibly present on the top surface of each specimen due to higher irradiance levels [18]. Moreover, the constant light energy reaching the top surface may justify the fact that there was no significant difference among KHN values on the top surface considering different curing times and resins. Notwithstanding, increasing curing time had great influence on KHN values on the bottom surface, where light irradiance is naturally attenuated during polymerization [19-20].

Only when cured with BP, emitting the highest irradiance levels, the nanofilled composite resin (FS) showed KHN bottom surface values similar to those observed for the microhybrid one (FZ). Metal ZiO2 nanofiller present in FS might decrease light transmittance through the resin increment due to light scattering [2-21]. Hence, one may assume that FS requires high irradiance levels to polymerize bottom surfaces similarly to FZ.

The second null hypothesis tested can also be rejected. BP and UL light sources showed statistically similar DC values concerning top and bottom surfaces only for FZ cured for 20 seconds. However, the 60 seconds curing time revealed similar DC values for top and bottom surfaces regardless of the light sources and composite resins tested. A larger light exposure time to the FS top surface could improve light transmittance to its bottom surface, leading to higher DC values. This finding is crucial for better clinical outcomes concerning class I and II restorations using resin-based materials, whose first increment may be photocured under an 8-mm distance and lies in contact with the dentin. Consequently, if low DC values are obtained, composites might release nonreacted monomers causing pulpal injuries [22]. The higher the DC, the lower will be the percentage of non-cured monomers; pulp injuries will consequently be less likely to occur.

The fact that UL revealed lower DC values for FS top surface, when compared to that of FZ, might be due to the great DC provided by this light device in FZ accounting for the statistically significant differences found for these composites regarding DC. UL is a third generation LED that emits light at a wavelength ranging from 380 to 500 nm with peaks of 400 and 455 nm. The other light sources tested in this study (BP, UL and AC) emit light with a peak of about 455 nm. Photo-initiators other than camphorquinone such as Lucirin, phenylpropadione-PPD, acylphosphine oxide-PDB, bisacylphosphine BAPO-oxide may be contained in these composite resins which can be excited through lights with wavelengths lower than 455 nm. Therefore, a greater amount of photoinitiator could have been photoactivated in FZ, presenting less light attenuation and scattering than FS [2]. Further studies are needed to investigate the presence of other photoinitiators in FZ and FS in an attempt to clarify the above hypothesis.

Heat generated by halogen lamps, with the light emitted in the same direction [23], should be taken into account. At 60 seconds, temperature is increased [24] and more monomers can be converted into polymer [25]. Moreover, exothermic reaction during polymerization are also related to the amount of inorganic content in the composite resin. The lower the inorganic content, the higher the organic one and the greater the exothermic reaction [26]. FS has a higher content of organic components when compared to FZ (Table 1). Thus, the fact that FS showed higher DC values than FZ for the top surface at 60 seconds when cured by XL may be explained by the greater amount of heat reaching the top surface of the specimens. Although heat generation may have favored DC on the top surface of FS polymerized with XL, it might have adverse effects on dental pulp and gingival tissue [22].

The high light energy level emitted by BP and the low light scattering in FZ may have favored the fact that BP was the only light

device to provide similar DC values for the bottom surface of FZ at 20 seconds and 60 seconds. The greater amount of photoinitiator excited by UL in FZ, presenting less light attenuation and scattering than FS on the bottom surface [2] can explain the differences between FZ and FS concerning DC values on the bottom surface. Irradiance levels of curing devices tested in this study were crucial in determining DC values of both composites tested. Differences between XL and the other photo-curing devices (Table 4) observed in the present study may be justified due to the lowest irradiance level emitted by XL (250 mW/cm^2). Probably, if the argon laser presented radiance levels similar to the BP LED, comparable effectiveness of cure would be obtained by both these light-curing units, as observed elsewhere [14].

Within the limitations of this study, the null hypotheses tested were rejected. Filler particle size, curing device and photoactivation time influenced the DC and surface hardness especially on the bottom surface of the increments. The high irradiance devices tested, particularly the LED Bluephase 16i, provided better physical properties on the bottom surface. The microhybrid composite showed better properties than the nanofilled one; photocuring at 60 s can increase DC and surface hardness values on the bottom surface of resin composite increments cured by lower irradiance levels light sources. The argon laser did not provided improved polymerization than other light-sources tested.

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