Degree of conversion and hardness of resin composite using various light curing units

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Abstract

Objective*: To evaluate the effect of various quartz tungsten halogen (QTH) and light-emitting diode (LED) light curing units on degree of conversion (DC) and hardness of resin composite cured at polymerized times used.*

Material and methods*: One hundred and eighty cylindrical specimens (4.0 mm in diameter and 4 mm in thickness) of Premise shade A2 resin composite were prepared. Three QTH: Elipar 2500 (EL2500); Sprectrum 800 (SP); and Demetron LC (DELC), and three LEDs: Elipar S10 (ELS10); BlueShot (BS); and Demi (DELED) were used. Resin composite was cured for 20, 40, and 60 seconds and 10, 20, and 40 seconds for QTH and LED groups, respectively. The DC and surface hardness of specimens were measured at top and bottom surface hardness. Data were analyzed using two-way ANOVA, Tukey's test and pair t-test (α=0.05).*

Results*: Surface hardness values were affected by the light intensities (different QTHs or LEDs) (p<0.01), and polymerized times used (10, 20, or 40 seconds for LED, and 20, 40, or 60 seconds for QTH) (p<0.01). DC was found to be no significant differences of all QTH and LED groups (p>0.05). Overall, LED groups provided hardness values greater than QTH groups (p<0.05).*

Conclusion*: Intensities of light-curing units and polymerized times used affected hardness values. However, these effects have no influence on DC values.*

Keywords*: Light curing units; Light-emitting diode; Quartz tungsten halogen; Resin composite*

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Introduction

Demand for esthetic in dentistry has presently increased. One of the esthetic restorative materials used is light cured resin composite which has been widely applied in clinical dentistry. This also results in a rapid increase development in the number of light curing units to polymerize light-activate resin composites.

Photocuring of resin composites by light curing unit is initiated by electromagnetic wavelengths between 400 and 500 nm. This

blue light activates camphoquinone and resin, and raises them to an excited state. The excited camphorquinone molecules collide with amine molecules forming free radicals which in turn react with the carbon to carbon double bond (C=C) of a monomer molecule and initiate the polymerization process.[1] Resin composite polymerization occurs through conversion of monomer molecules into polymer network accompanied by a closer packing of the molecule which causes contraction of the resin composite.[2,3] However, the dimethacrylate monomers used in resin composite exhibit a considerable residual unsaturation in the final material with a degree of conversion ranging from 55-75% under a conventional cure condition.[4,5]

Quart tungsten halogen (QTH) light curing units are the most commonly employed light-activation units in dentistry. Minimal intensity (400 mW/cm²) in the proper spectral distribution is necessary for complete polymerization of light cured resin composite of 2 mm in depth.[6] The use of QTH curing units to polymerize resin composite has several drawbacks despite their popularity. The halogen bulbs have a limited effective lifetime of about 40 to 100 hours. The reflector and filter degrade overtime due to high operating temperatures and the large quantity of heat produced during the curing cycles.[6] To overcome the several drawbacks of QTH curing light units, blue light-emitting diodes (LED) have been developed for polymerization of light-activated resin composite. LED curing units feature very narrow spectral ranges around 470 nm and bandwidth of about 20 nm.[7] They have lifetime of more than 10,000 hours and undergo little degradation of light output overtime. They use junctions of doped semiconductors (p-n

junctions) to generate light and require no filters to produce light. LED curing units are also resistant to shock and vibration. Their relatively low power consumption makes them suitable for portable use. Previous studies have demonstrated good performance of LED light-curing unit in terms of adequate depth of cure, flexural strength, and surface hardness.[8,9]

The degree of conversion is an important factor that affects clinical performance of resin composite restorations.[10-12] It can be correlated with composition of monomers and oligomers used in the material, which is the number of ethylene double carbon bonds converting into single bonds and provides the degree of conversion (in per cent) of a resin composite. Several methods have been used to determine the degree of conversion of resin composite. Fourier transformation infrared spectroscopy (FTIR) has been widely used as a reliable method for examining the degree of conversion. It detects the C=C stretching vibrations directly before and after curing of materials.[13] FTIR spectra of both uncured and cured samples were analyzed using an accessory of the reflectance diffusion. However, to measure the degree of conversion of bulk resin composite by FTIR, the procedure is time consuming as the polymerized specimens need to be pulverized.

The curing times, correct wavelength of the light source, and material compositions strongly influence the degree of conversion.[14,15] Previous studies have reported that high intensity light provide higher values for degree of conversion[8,16-18] and demonstrated that LED curing units provide deeper depth of polymerization than QTH lamps.[8] However, LEDs also produce higher contraction strains during resin composite polymerization.[8] The

degree of conversion of light-cured resin composites is proportional to the amount of light to which they are exposed. At the upper surface of a restoration, where no overlying resin composite interferes with light transmission, it has been found that even a curing source with relatively low intensity can cure the resin matrix to an extent almost equal to that when high intensity lights are used. However, as light passes through the bulk of the restorative material, its intensity is decreases greatly; thus decreasing the potential for polymerization. This decrease results in a gradation of the cure such that it decreases from the top surface inward. This decrement in cure has been termed "depth of cure" and has significant influence on both physical and biological properties of the restoration.[19] Addition hardness tests of resin composite at different depths or thicknesses are commonly method of measuring the degree of conversion. They correlated well with the degree of conversion test.

However, little is known about the relationship between minimal light curing time required for proper polymerization and various QTH and LED light curing units which have different light intensities. Therefore, the aims of this study was to evaluate degree of conversion and hardness of resin composite polymerization by various QTH and LED curing units at different polymerized times used. The null hypothesis was that there would be no effect of various light sources or intensities on degree of conversion and hardness of resin composite at polymerized times used.

Materials and methods

Specimen preparations

One hundred and eighty cylindrical specimens of one brand of resin composite (Premise shade A2, Kerr Corp., Orange, CA, USA) were prepared in polytetramethyl fluoroethylene ring molds, 4.0 mm in internal diameter and 4 mm in thickness. To minimize the effects of colorants on the light penetration, the vita shade A2 of resin composite was selected.[20] The mold cavity was then filled with resin composite in a single increment on a glass plate and covered with a mylar matrix strip and a second glass plate over a mylar strip. A static load of approximately 500 g was applied to this plate for 30 seconds to extrude excess resin composite and to obtain a flat surface that would facilitate hardness testing. Subsequently, the glass plate was removed from the top of the mold. The curing-light tip was then placed in contact with the top surface of the specimens. Three QTH light curing units: Elipar 2500 (EL2500) (3M ESPE, Grafenau, Germany); Sprectrum 800 (SP) (Dentsply DeTrey GmbH, Konstanz, Germany); and Demetron LC (DELC) (SDS Kerr, Danbury, CT, USA), and three LED light curing units: Elipar S10 (ELS10) (3M ESPE, Grafenau, Germany); BlueShot (BS) (Shofu, Kyoto, Japan); and Demi (DELED) (SDS Kerr, Danbury, CT, USA) were used in this present study. All LED light curing units were used in the continuous mode. The intensity of all the curing light units was checked with a radiometer (Cure Rite model 8000, EFOS Inc., Mississauga, Ontario, Canada) prior to use to ensure consistency in intensity output from the light source. The light-curing times used were 10, 20, and 40 seconds for the LED curing unit groups, while the light-curing times of 20, 40, and 60 seconds were used for QTH groups.

After polymerization, the mylar strip on the top and glass plate on the bottom of the mold were removed. Subsequently, the specimen was removed from a ring mold. Ten specimens were assigned to each of eighteen groups according to Tables 1 and 2.

Surface hardness measurement

Five specimens (n=5) of each group were tested using a Vickers hardness testing device (Micromet II, Buehler Ltd., Lake Bluff, IL, USA). Hardness measurement was taken under a 100 g load for 10 seconds. Five hardness indentations were made on the top surface (near the light source) and five hardness indentations were made on the bottom surface (away from the light source) of each specimen of 4 mm in thickness. The mean of the five hardness measurement on each specimen was recorded as the hardness value of that surface of the specimen (top and bottom). The hardness ratio were also calculated from dividing hardness values of top surface by hardness values of bottom surface for each curing time. This value should be greater than 0.8.[21]

Determination of the degree of conversion

Five specimens (n=5) of each group were tested using FTIR spectrometer (model EQUINOX 55, Bruker Optics Inc., Billerica, MA, USA). Uncured paste of resin composite was smeared onto a potassium bromide disc and the absorbance peaks before curing were obtained by transmission mode of FTIR. The polymerized specimen was pulverized into fine powder with a hard tissue-grinding machine (model MA590, Marconi, Piracicaba, São Paulo, Brazil) immediately after curing. The absorbance peaks were then recorded using the diffuse-reflection mode of FTIR. The percentage of unreacted carbon-carbon double bonds (%C=C) was examined from the ratio of absorbance intensities of aliphatic C=C (peak at 1635 cm^{-1}) against internal standard before and after curing of the specimen and the aromatic C-C (peak at 1614 cm^{-1}). The degree of conversion was calculated by subtracting the %C=C from 100% according to the following formula[13]:

$$\text{Degree of conversion (\%)} = 1 - \left[\frac{\left(1635 \text{ cm}^{-1}/1614 \text{ cm}^{-1}\right)\text{cured}}{\left(1635 \text{ cm}^{-1}/1614 \text{ cm}^{-1}\right)\text{uncured}} \right] \times 100$$

Statistical analysis

A two-way analysis of variance (ANOVA) and Tukey Honestly Significant Difference (HSD) test were applied to test the effect of light-curing units (or intensities) and polymerized times on degree of conversion and hardness of the resin composite. Pair-samples t-test was used to test these effect on the top and bottom surface hardness of the resin composite (α=0.05).

Results

The results of two-way ANOVA presented that hardness values were affected by the light intensities (different QTHs or LEDs) (p<0.01) and polymerized times used (10, 20, or 40 seconds for LED, and 20, 40, or 60 seconds for QTH) (p<0.01). Significant interactions were presented in hardness values between the intensities and polymerized times used (p=0.002). However, for degree of conversion, the results of two-way ANOVA showed that degree of conversion values were not affected

by the light intensities (p=0.491) and polymerized times used (p=0.715). Significant interactions were not also presented in degree of conversion values between the intensities and polymerized times used (p=0.276).

The microhardness, hardness ratio, and degree of conversion values of the resin composite after polymerization by QTH and LED light curing units at different time are shown in Tables 1 and 2, respectively. For Table 1 of QTH groups, no statistically significant difference was found in degree of conversion of resin composite among three QTHs at different times (p>0.05). However, significant differences were noted in microhardness values of resin composite when regarding to polymerized times or intensities of light-curing units used (p<0.05). Similarly, for Table 2 of LED groups, the result of degree of conversion values demonstrated that there was no statistically significant difference among three LEDs at different times (p>0.05). On the contrary, significant differences were presented in microhardness values of resin composite when regarding to polymerized times or intensities of light-curing units used (p<0.05).

Comparing between the top and bottom surface hardness, for QTH groups, there were significant differences in DELC, and SP groups at 4 mm for 20 and 40 seconds (p<0.01 for all comparisons). For EL2500 group, significant differences were found when polymerized 4-

mm resin composite for 20 and 40 seconds (p=0.002 and p=0.001, respectively). Whilst for LED groups, the top and bottom surface hardness were found to be significant differences in DELED, ELS10, and BS groups at 4-mm thickness for 10 and 20 polymerized times used (p<0.01 for all comparisons).

As the polymerized time increase, for QTH groups, significant increase in hardness values were recorded for all groups (p<0.01 for all comparisons). The highest top and bottom surface hardness value was found in EL2500 groups (89.26±2.21 and 84.42±3.39, respectively) which has the highest intensity, for 60 seconds times used (p<0.01 for both comparisons). Similar results were found for LED groups. As the polymerized time increase, significant increase in hardness values were noted for all groups (p<0.01 for all comparisons). The highest hardness value was found in BS groups (84.93±4.09 and 84.50±3.48, respectively) which has the highest intensity, for 40 seconds times used (p<0.01). The hardness ratio values which lower than 0.8 were found on 4 mm. thickness of DELC group for 20 seconds, SP group for 20 and 40 seconds (QTH groups) , DELED group for 10 and 20 seconds, and ELS10 for 10 seconds (LED groups).

Table 1 Mean hardness and degree of conversion and standard deviations (SD) of resin composites at polymerized times using QTH light curing units

Group and intensity (mW/cm²)	Time (second)	Mean hardness (kg/mm²) ± SD		Hardness ratio	Degree of conversion (%) ± SD
		Top	Bottom		
DELC	20	65.18 ± 3.89[a,C]	49.67 ± 3.97[*,b,C]	0.77 ± 0.09	69.08 ± 0.04

402.00 ± 7.21	40	66.36 ± 3.47[a,C]	56.40 ± 2.93[*,a,C]	0.85 ± 0.08	69.12 ± 0.03
	60	68.54 ± 6.37[a,C]	63.92 ± 8.22[a,B]	0.93 ± 0.06	69.12 ± 0.02
SP	20	70.72 ± 5.37[a,C]	50.02 ± 2.36[*,b,C]	0.71 ± 0.07	69.08 ± 0.08
450.67 ± 4.04	40	72.92 ± 6.77[a,B]	58.34 ± 4.86[*,a,C]	0.80 ± 0.11	69.11 ± 0.01
	60	73.59 ± 6.44[a,B]	64.81 ± 6.96[a,B]	0.88 ± 0.05	69.12 ± 0.02
EL2500	20	62.20 ± 3.64[b,C]	52.78 ± 4.22[*,b,C]	0.85 ± 0.06	69.12 ± 0.02
555.33 ± 11.02	40	71.38 ± 7.22[a,B]	58.94 ± 4.86[*,b,C]	0.83 ± 0.07	69.00 ± 0.16
	60	81.18 ± 7.42[a,A]	72.03 ± 4.80[a,A]	0.89 ± 0.06	69.13 ± 0.03

* indicates significant difference (in row) between top and bottom surface hardness of each time according to paired-samples *t*-test (p<0.05)

[a,b,c] indicate significant difference (in column) among different times and intensity according to Tukey HSD test (p<0.05)

[A,B,C] indicate significant difference (in column) among different times and intensities according to Tukey HSD test (p<0.05)

Table 2 Mean hardness and degree of conversion and standard deviations (SD) of resin composites at polymerized times using LED light curing units

| Group and intensity (mW/cm²) | Time (second) | Mean hardness (kg/mm²) ± SD | | Hardness ratio | Degree of conversion (%) ± SD |
		Top	Bottom		
DELED	10	66.03 ± 6.95[a,C]	37.77 ± 6.99[*,a,C]	0.57 ± 0.08	69.10 ± 0.04
1076.33 ± 24.09	20	70.56 ± 9.55[a,B]	54.94 ± 6.39[*,a,B]	0.79 ± 0.13	69.11 ± 0.05
	40	76.99 ± 5.46[a,A]	65.43 ± 6.61[a,A]	0.86 ± 0.11	69.14 ± 0.02
ELS10	10	69.56 ± 6.17[a,C]	42.53 ± 3.17[*,b,C]	0.62 ± 0.07	69.12 ± 0.02
1417.00 ± 14.93	20	70.87 ± 2.82[a,B]	57.72 ± 2.15[*,a,B]	0.82 ± 0.05	69.12 ± 0.02
	40	75.22 ± 9.27[a,A]	62.03 ± 8.69[a,A]	0.82 ± 0.04	69.12 ± 0.01
BS	10	68.33 ± 3.56[a,C]	56.83 ± 6.18[*,b,C]	0.83 ± 0.09	69.10 ± 0.02
1911.67 ± 4.72	20	74.06 ± 4.01[a,B]	64.43 ± 4.87[*,a,B]	0.87 ± 0.04	69.12 ± 0.01
	40	76.51 ± 8.96[a,A]	70.83 ± 7.11[a,A]	0.93 ± 0.03	69.12 ± 0.05

* indicates significant difference (in row) between top and bottom surface hardness of each time according to paired-samples *t*-test (p<0.05)

a,b,c indicate significant difference (in column) among different times and intensity according to Tukey HSD test (p<0.05)

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Discussions

The results of this present study support rejection of the null hypothesis since intensities of light-curing units and polymerized times used affected hardness values. However, these effects have no influence on degree of conversion values. The need for an adequate polymerization of the resin composite resulting in good physical and mechanical properties of the materials created for clinicians concerning the selection of the appropriate light-curing unit.

The degree of conversion is commonly measured by Fourier transform infrared reflectance spectroscopy (FTIR) which has been reported to produce highly reliable results.[22] However, one of the most frequently used indirect methods for verifying the degree of resin composite polymerization is the microhardness test[23] that indicates the strength under compressive loading. In this present study, the results presented that polymerized time used affected hardness values but no influence on degree of conversion values. In fact, as light passed through the bulk of a resin composite, its intensity is greatly decreased due to the absorption and scattering of light by filler particles and resin matrix. This decreasing results in a gradation of cure such that it decreases from top surface inward. This then accounted for the difference between top surface hardness and bottom surface hardness of all specimens cured with each light source[26]

which different from FTIR technique. To measure the degree of conversion of bulk resin composite by FTIR, polymerized specimens need to be pulverized into fine powder. So accounted of degree of conversion of specimens were average of bulk resin composite.

In the present study, for all light-curing units, microhardness values were high at the top surface which can be attributed to the relationship between irradiation distance and effectiveness of polymerization. In particular, the hardness values of the bottom surface should be close to the hardness values of the top surface, resulting in hardness ratio greater than 0.8.[20] In this present study, effective hardness ratios were achieved with the most light curing unit groups. The plausible reason for this outcome could be related to the light intensities and duration of the curing time used. The greater intensity of the light energy is sufficient to excite the camphoquinone in the resin composite material. As shown in the present study, increasing the duration of irradiation time provided significantly more polymerized than short irradiation time at a same thickness specimen, accompanying with Rueggeberg et al.[24]

It must be noted that there are some limitations to this study. The role of saliva has not been taken into consideration. The oral cavity presents a different testing environment due to the presence of water, temperature

change, and pH level. Therefore, the oral cavity may considerably affect these results. More in vivo studies are needed to assess the effects of different light intensities of light curing units and time on microhardness and degree of conversion.

Conclusions

Within the limitations of this study, the following conclusions could be drawn: Intensities of light-curing units and polymerized times used affected hardness values. However, these effects have no influence on degree of conversion values.

Acknowledgements

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ระดับขั้นการแปลงผันและความแข็งของเรซินคอมโพสิตเมื่อใช้เครื่องฉายแสงต่างกัน

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บทคัดย่อ

วัตถุประสงค์: เพื่อศึกษาผลของเครื่องฉายแสงฮาโลเจนและไดโอดที่ต่างกันต่อระดับขั้นการแปลงผันและความแข็งของเรซินคอมโพสิต

วัสดุและวิธีการ: เตรียมแผ่นกลมเรซินคอมโพสิตสีเอ 2 เส้นผ่านศูนย์กลาง 4 มม. หนา 4 มม. จำนวน 180 ชิ้น ฉายด้วยเครื่องฉายแสงฮาโลเจน 3 ผลิตภัณฑ์ เป็นเวลา 20 40 และ 60 วินาที และฉายด้วยเครื่องฉายแสงไดโอด 3 ผลิตภัณฑ์เป็นเวลา 10 20 และ 40 วินาที แล้ววัดระดับขั้นการแปลงผันและความแข็งด้านบนและด้านล่าง จากนั้นบันทึกข้อมูลและวิเคราะห์สถิติด้วยการทดสอบความแปรปรวนแบบสองทาง การทดสอบทูกีย์และทีที่ระดับนัยสำคัญ 0.05

ผล: ชนิดของเครื่องฉายแสง (ฮาโลเจนและไดโอด) $(p<0.01)$ และเวลาที่ใช้ $(p<0.01)$ มีผลต่อความแข็งอย่างมีนัยสำคัญทางสถิติ ส่วนระดับขั้นการแปลงผัน ไม่มีความแตกต่างกันอย่างมีนัยสำคัญทางสถิติ $(p>0.05)$ โดยภาพรวมพบว่า เครื่องฉายแสงไดโอดให้ค่าความแข็งมากกว่าฮาโลเจนอย่างมีนัยสำคัญทางสถิติ $(p<0.05)$

สรุปผล: ความเข้มของแสงและเวลาที่ใช้มีผลต่อความแข็ง แต่ไม่มีผลต่อระดับขั้นการแปลงผัน

คำสำคัญ: เครื่องฉายแสง; ไดโอด; เรซินคอมโพสิต; ฮาโลเจน

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