

ORIGINAL ARTICLE

Influence of successive light-activation on degree of conversion and knoop hardness of the first layered composite increment

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Abstract

Purpose: To evaluate the influence of light-activation of second, third and fourth increments on degree of conversion (DC) and microhardness (KHN) of the top (T) and bottom (B) surface of the first increment. **Materials and methods.** Forty samples ($n = 5$) were prepared. In groups 1–4, after each increment light-activation (multiple irradiation), T and B of the first increment were measured in DC and KHN. In groups 5–8, only the first increment was made (single irradiation) and measurements of DC and KHN were taken at 15 min intervals. The light-activation modes were (XL) $500 \text{ mW/cm}^2 \times 38 \text{ s}$ (G1/G5); (S) $1000 \text{ mW/cm}^2 \times 19 \text{ s}$ (G2/G6), (HP) $1400 \text{ mW/cm}^2 \times 14 \text{ s}$ (G3/G7); (PE) $3200 \text{ mW/cm}^2 \times 6 \text{ s}$ (G4/G8). Data for DC and KHN were analyzed separately by using PROC MIXED for repeated measures and Tukey-Kramer test ($\alpha = 0.05$).

Results. For KHN, B showed lower values than T. PE resulted in lower values of KHN in B surface. For single and multiple irradiations, T and B of first measurement showed the lowest KHN and the fourth measurement showed the highest, with significant difference between them. For single irradiation, first and second increments presented similar KHN, different from the third and fourth increment, which did not differ between them. For multiple irradiations, the second light-activation resulted in KHN similar to first, third and fourth increments. For DC, except QTH, T presented higher DC than B. **Conclusion.** The light-activation of successive increments was not able to influence the KHN and DC of the first increment.

Key Words: Dental restorations, mechanical phenomena, spectroscopy

Introduction

Layering is the standard technique for placement of resin composite in cavity preparations that exceeds 2 mm and it is based on the desire to ensure a cure as complete as possible of the entire increment [1]. An effective polymerization is more easily achieved when the distance between light-curing unit and the resin composite is minimal and it has been related to better mechanical properties and to increased biocompatibility. However, complete polymerization is never achieved and, under normal curing conditions, they achieved ~ 45–70% conversion [2].

In clinical scenario, the distance between the light guide tip and the resin composite varies greatly and is

a condition difficult to control, since it depends on caries progression and cavity size [3]. In deep class II restorations, the distance between the gingival wall of the interproximal box and the light-curing tip could reach up to 7 mm depth [4] and the first layered increment might not receive proper irradiation. This distance will affect the degree of conversion of resin composite due the reduction in light intensity as a result of an increase in distance from the light source [5]. Besides that, during the light-activation process, the light is absorbed and scattered while it passes through the composite. Thus, the light is attenuated and its effectiveness is reduced as depth increases [6]. The clinical consequences of an inadequate polymerization are increased marginal breakdown

and debonding, resulting in post-operative sensitivity, increased cytotoxicity and secondary caries, all reducing the longevity of the restoration.

Light-activated resin composites are widely used in clinical Dentistry for both anterior and posterior tooth restoration [7]. This material contains photoinitiators, which start the polymerization reaction when exposed to proper light, usually blue light. Since the introduction of light-activated resin composites, light-curing units (LCUs) have become an important device, and the knowledge of the characteristics of resin composite and LCU are fundamental to success of tooth restoration and clinical longevity. The options of LCU include quartz-tungsten-halogen (QTH) lamps, plasma arc, argon laser and light-emitting diodes (LEDs) [8]. QTH is used since the beginning of visible light-activated resin composite, but some of the components deteriorate, decreasing significantly the power density over time, requiring constant monitoring and maintenance. The LED LCUs are increasingly incorporated into the arsenal of the dentist and are now available with high intensity power (above 1000 mW/cm²) and with a broadband emission spectrum (385–510 nm), combining power with increased compatibility to the absorption spectrum of resin composite [7]. They last significantly longer and their power of density does not decrease with time. Some high-powered LED LCU manufacturers suggested short polymerization times of up to 5 s [8,9].

Although composites are the material of choice for direct restorations and in spite of the improvement that has been submitted since its development, some limitations and disadvantages still exist and are related to the limited depth of cure and the poor distribution of degree of conversion [10,11]. However, during the placement of composite by incremental layering technique, the first layered increment might receive extra energy from the light-activation of the subsequent increment and this process might positively affect the overall polymerization of the first increment. The improvement of overall polymerization of the first increment might result in better polymer structure, favoring the sealing of this critical area.

This way, the aim of this study was to evaluate the influence of light-activation of successive second, third and fourth increments on degree of conversion and microhardness of the top and bottom surface of the first increment.

Materials and methods

A microhybrid composite Filtek Z250 (3M ESPE, St Paul, MN) shade A3 and two LCUs (quartz-tungsten halogen XL 3000 3M ESPE, Grafenau, Germany and third LED generation Valo-Ultradent Products, South Jordan, UT) were selected for this study. The factors considered in the plot were: *light-activation*

protocols in four levels: (XL): 500 mW/cm² for 38 s; Standard (S): 1000 mW/cm² for 19 s; High Power (HP): 1400 mW/cm² for 14 s; Plasma Emulation (PE): 3200 mW/cm² for 6 s; and *exposure mode* in two levels (multiple or single irradiation). The *surface* (top and bottom) was considered as a split-plot and the factor *measurements* (four levels: first, second, third and fourth) were considered as repeated measures. This way, the study consists of eight experimental groups ($n = 5$).

Experimental groups

In groups 1–4 (multiple irradiation), the first increment was light activated according to the respective light-activation protocol (XL, S, HP and PE). After the light-activation, DC and KHN tests were made in the top and bottom surface. The subsequent increments were made in the same manner and after each light-activation of the recent layered resin composite; DC and KHN were measured in the top and bottom surface of the first increment. In groups 5–8 (single irradiation - control groups) one only increment was made according to the light-activation protocols (XL, S, HP and PE). The DC and KHN were taken in both surfaces. The second, third and fourth measurements of DC and KHN were made every 15 min without the sample receiving any light-activation.

System matrix

The samples were made in a bipartite Teflon mold containing a cylindrical cavity in the center, measuring 5 mm in diameter and 2 mm thick, allocated in an acrylic bracket. The acrylic bracket is 8 mm in height and three more identical Teflon molds were overlapped, each one with cylindrical cavities with the same measures describe previously (Figure 1).

Sample preparation

A transparent polyester strip was positioned on the bottom of the mold. The first cavity was filled in one increment and covered the top with another polyester strip (Figure 1F). A glass plate was positioned above the polyester strip for 10 s in order to provide a flat surface. The light-curing tip was positioned at a distance of 8 mm between the light guide tip and the bottom of the first increment, simulating the distance observed in the gingival wall of the proximal box in class II cavity. The sample was polymerized according to the light-activation protocols described above. In groups 1–4 (four increments each), before the insertion of the increment, a transparent polyester strip was positioned on the top of the previous increment in order to avoid adherence between them (Figures 1F–I).

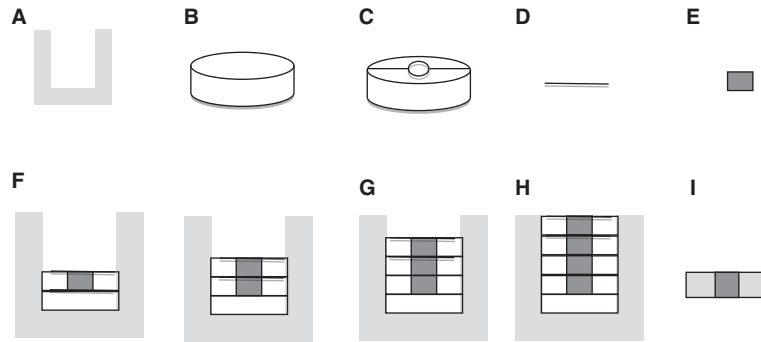


Figure 1. Schematic representation of System Matrix. (A) Acrylic bracket; (B) Teflon base; (C) Bipartite Teflon mold containing a cylindrical cavity in the center (5×2 mm); (D) Polyester Strip; (E) Composite; (F–I) Sample preparation of groups 1–4. (F) Sample preparation of groups 5–8.

Degree of conversion

The DC measurements were recorded in absorbance mode with a FTIR spectrometer (Spectrum 100 FTIR, PerkinElmer, Waltham, MA) coupled to a zinc selenide multiple (six) reflection Attenuated Total Reflection (ATR) accessory, with refraction index of 2.4 at 1000 cm^{-1} (Pick Technologies, Madison, WI) operating under the following conditions: $650\text{--}4000\text{ cm}^{-1}$ wavelength; 4 cm^{-1} resolution; 16 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}) for non-polymerized and polymerized resin composite. The non-polymerized resin composite was used as a reference. The degree of conversion was determined by the following formula:

$$\text{DC (\%)} = 100 * [1 - R_{\text{polymerized}} / R_{\text{non-polymerized}}]$$

where R = ratio by band height at 1638 cm^{-1} /band height at 1608 cm^{-1}

Microhardness test

KHN values of top and bottom surfaces of the first increment were obtained using a digital microhardness tester (HMV-2T E, Shimadzu Corporation, Tokyo, Japan). Five KHN measurements, under a load of 10 g for 10 s, were taken: one at the central portion, at which light was applied, and the other four $100\text{ }\mu\text{m}$ away from the central portion. The Knoop Hardness Number (KHN) was recorded as the average of the five indentations.

Results for DC and KHN were analyzed separately. For both tests, the data were analyzed by using PROC MIXED for repeated measurements and Tukey-Kramer test ($\alpha = 0.05$).

Results

The sample size was calculated by Gpower Software (<http://www.ats.ucla.edu/stat/gpower/>) in order to

detect the effect size ($\alpha = 5\%$ and power test $\geq 80\%$ for factors and interactions), resulting in $n = 5$.

For microhardness test, PROC MIXED for repeated measures showed a significant effect for *light-activation protocol*, *surface* and *measurement* factors and a significant effect for interaction between *light-activation protocol* \times *surface* ($p = 0.0005$) as well as *exposure mode* \times *measurement* ($p = 0.0383$). In all experimental conditions, the top surface showed higher means of KHN, with a significant difference to the bottom surface. For the bottom surface, the light-activation protocol PE resulted in lower values, with a significant difference to the other light-activation protocols. For the single exposure mode, there was no difference between the first and second measurement, followed by a significant increase in KHN in the third measurement, which showed no difference to fourth measurement. When multiple exposure modes were used, higher values were obtained in the fourth measurement, which did not differ from the second and third measurement; and lower values were noticed in the first measurement, with a significant difference in the fourth. The second measurement did not differ from the others (Table I).

For the DC test, PROC MIXED for repeated measures showed a significant effect for *surface* factor ($p \leq 0.0001$). There was a significant interaction between *light-activation protocol* and *surface* ($p = 0.0272$). The top surface presented higher DC with a significant difference to the bottom surface in all experimental conditions, except for the XL light-activation protocol. There was no significant effect for the light-activation protocol ($p = 0.2200$), exposure mode ($p = 0.0638$) and measurement ($p = 0.8629$) (Table II).

Discussion

The esthetic restoration is a routine procedure in everyday practice. In class II cavities restored with composite resins, the interface between tooth/gingival wall of the interproximal box is the most critical area due to the difficulty to effectively cure the material

Table I. Knoop hardness (SD) results.

Exposure mode	Surface	Protocol	Measurement			
			1	2	3	4
Single	Top	XL	65.46* (10.16) ^a	61.11* (5.66) ^a	68.82* (4.83) ^a	73.84* (21.53) ^a
		S	61.39* (4.25) ^a	61.93* (2.96) ^a	70.36* (5.16) ^a	69.01* (7.11) ^a
		HP	61.57* (2.81) ^a	61.68* (5.32) ^a	66.31* (2.93) ^a	66.54* (4.08) ^a
		PE	58.57* (3.41) ^a	63.25* (3.25) ^a	66.25* (5.66) ^a	65.88* (5.73) ^a
Single	Bottom	XL	45.78 (3.55) ^a	46.44 (4.94) ^a	51.13 (2.16) ^a	58.38 (15.42) ^a
		S	45.38 (3.23) ^a	46.71 (3.88) ^a	51.35 (7.59) ^a	48.76 (3.4) ^a
		HP	43.00 (4.64) ^a	43.44 (5.07) ^a	47.71 (4.22) ^a	44.96 (5.73) ^a
		PE	36.45 (5.71) ^b	39.42 (4.44) ^b	39.33 (4.67) ^b	41.72 (7.56) ^b
	Tukey		B	B	A	A
Multiple	Top	XL	59.75* (7.47) ^a	66.01* (5.81) ^a	65.168 (5.16) ^a	68.4* (9.65) ^a
		S	58.89* (2.28) ^a	65.91* (4.12) ^a	69.96* (4.46) ^a	63.75* (7.46) ^a
		HP	61.58* (3.76) ^a	65.77* (1.72) ^a	65.59* (1.97) ^a	66.42* (6.18) ^a
		PE	61.88* (3.81) ^a	61.49* (4.36) ^a	66.86* (5.10) ^a	64.41* (6.73) ^a
Multiple	Bottom	XL	47.35 (3.86) ^a	47.54 (4.41) ^a	50.08 (4.86) ^a	49.91 (3.87) ^a
		S	45.51 (2.08) ^a	47.95 (3.73) ^a	50.08 (3.69) ^a	50.36 (5.67) ^a
		HP	48.15 (1.47) ^a	49.23 (3.01) ^a	50.14 (4.22) ^a	51.29 (4.25) ^a
		PE	39.42 (4.92) ^b	38.4 (4.53) ^b	39.92 (4.91) ^b	40.27 (4.13) ^b
	Tukey		B	AB	A	A

There was no difference for exposure mode ($p = 0.8678$).

*Differs from bottom surface ($p < 0.05$).

Means followed by different letters (capital letters in row and lower letters in column comparing light-activation protocol inside surface and exposure mode) differ between them ($p \leq 0.05$).

Table II. DC (%) results and standard deviation (SD).

Surface	Exposure mode	Light-activation protocol	Measurement			
			1	2	3	4
Top	Single	XL	49.28 (2.52)	48.29 (2.08)	48.18 (2.99)	47.67 (2.15)
		S	46.56 [#] (1.68)	48.27 [#] (2.66)	46.76 [#] (2.36)	44.46 [#] (8.58)
		HP	51.52 [#] (2.41)	47.45 [#] (3.6)	50.32 [#] (2.97)	51.31 [#] (4.77)
		PE	48.84 [#] (3.58)	48.04 [#] (4.08)	48.72 [#] (2.74)	48.45 [#] (2.31)
	Multiple	XL	44.93 (3.81)	45.86 (4.63)	45.36 (6.23)	45.66 (6.86)
		S	54.25 [#] (2.79)	53.17 [#] (4.33)	51.44 [#] (6.95)	53.92 [#] (7.06)
		HP	50.59 [#] (2.84)	52.79 [#] (3.98)	50.28 [#] (6.12)	51.16 [#] (4.89)
		PE	54.01 [#] (4.92)	50.93 [#] (2.94)	53.99 [#] (5.15)	52.03 [#] (5.75)
Bottom	Single	XL	46.36 (1.92)	46.62 (2.92)	47.74 (3.79)	47.12 (3.5)
		S	42.04 (5.33)	44.05 (4.09)	45.22 (4.5)	43.89 (6.82)
		HP	45.78 (3.02)	47.6 (2.58)	48.13 (2.95)	48.49 (3.05)
		PE	42.58 (3.56)	44.35 (1.49)	43.05 (3.25)	43.83 (3.84)
	Multiple	XL	43.96 (4.2)	41.98 (7.82)	42.09 (9.99)	48.12 (3.94)
		S	49.39 (2.39)	49.3 (6.05)	48.71 (7.22)	49.34 (8.41)
		HP	46.92 (3.09)	46 (4.82)	47.34 (4.14)	48.08 (5.18)
		PE	46.17 (4.51)	48.59 (6.42)	45.97 (6.61)	49.84 (5.25)

[#]Differs from bottom at the same exposure mode, light-activation protocol and measurement ($p \leq 0.05$); There was no significant difference for light-activation protocols ($p = 0.2200$), exposure mode ($p = 0.0638$) and measurement ($p = 0.8629$).

deposited there. The need to overcome this difficulty resulted in some clinical strategies, such as decreasing the resin composite thickness [3], increasing the time of light-activation [12] and the use of lighter shade resin composite in deeper portions of the cavity [13]. The incremental insertion technique (increment no more than 2 mm thick) favors the preservation of the tooth/restoration interface [14] and requires the light-activation of a subsequent layered increment. It is known that the distance between LCU and material surface and radiant exposure can affect the properties of the material [11] but it is unknown if the successive light-activation of the increments is capable of improving the properties of the material applied in the first increment. The present study was designed to answer this question and aimed the evaluation of the influence of light-activation of successive increments on microhardness and degree of conversion of the top and bottom surface of the first layered increment.

For the KHN test, in all experimental conditions, the top surface showed a higher means of KHN, with a significant difference to the bottom surface. The optimum cure is often reached slightly below the surface layer where the light transmission is high and there is a significant heat build-up [15]. Thus, because it is an area easily reached by the irradiation, the top surface is not a good indicator for polymerization effectiveness. In this sense, the bottom surface should receive more attention [16]. The fact that the bottom surface showed a lower KHN value is a consequence of light passing through composite. The light emitted by a LCU suffers absorption and scattering, resulting in a decrease in light intensity and reducing its effectiveness. Besides that, part of the irradiation light that illuminates the composite surface is reflected and another part that penetrates the composite has the function of exciting the photosensitizer to start the polymerization process [17].

The *exposure mode* was not statistically significant; however, the interaction between *exposure mode* and *measurement* was significant. In single irradiated samples, the determination of DC and KHN was done 15 min after light exposure. This time interval was set to correspond to the time required for preparation and evaluation of multiple irradiated samples. Four measurements were taken for both tests. For the single irradiation as for multiple irradiations, lower hardness values were observed in the first measurement and the higher values in the fourth and they were statistically different. For multiple irradiation, the light-activation of the second increment did not result in a statistically significant increase in KHN of the first increment and this fact shows that the photons emitted by the light-curing unit was not capable to excite some of the photoinitiators available in the first increment. For both single and multiple irradiation, it was possible to notice a hardness increase from the third measurement and there was no difference between

the third and fourth measurement. The hardness was increased, probably due to the post-cure hardening process that occurs with resin composite materials and not due to the light exposure of the subsequent increment. Microhardness increased with time, as the increase rate was substantial over the first hour and tended towards a maximum after 1 week [15]. So, the expected improvement in hardness of the first layered increment by multiple irradiations of subsequent increments was not noticed.

The interaction between *light-activation protocol* and *surface* was significant. The light-activation protocol PE showed the lowest values of hardness in the bottom surface in all experimental groups, with statistical difference from the others. The probable explanation is that, despite the PE protocol delivering an extremely high energy (3200 mW/cm^2), it promoted a rapid initiation of polymerization, inducing to shorter chain lengths and, hence, leading to a lower molecular weight, compromising the hardness of the bottom surface. This behavior is similar to observe for plasma arc curing units [18] and for short polymerization times of 5 or 10 s, even for high-powered LED LCUs [8].

For DC test, there was a significant effect for *surface* factor, presenting a highly significant difference ($p < 0.001$). The top surface presented higher DC with a significant difference to the bottom in all experimental conditions, except for the XL light-activation protocol. As previously stated, the top surface is most easily reached by the light and this favors a higher activation of the photoinitiator molecules and polymerization reaction. For XL, there was no difference in the DC between the top and bottom surfaces. This fact can be explained by the longer irradiation time used associated to relative lower light intensity ($500 \text{ mW/cm}^2 \times 38 \text{ s}$), which probably gave a longer time for molecular re-arrangement, allowing a higher homogeneity of the formed polymer.

There was no statistical effect for *light-activation protocol* in DC test. In the present study, two LCUs (QTH and LED) were analyzed. Both exposure time and light intensity were applied in different combinations and all delivered approximately the same density energy (19 J/cm^2). This absence of statistical effect for light-activation protocol is in agreement with other studies and it suggests that an equivalent DC may be achieved by applying lower intensity light for a longer time or by using variable light intensities over a given time, since the polymerization process depends on total energy rather than on light intensity [19]. Despite the similarity found in DC of different light-activation protocols, other factors need to be evaluated to characterize more fully the advantages, disadvantages and consequences of each protocol. For example, the PE protocol delivered $3200 \text{ mW/cm}^2 \times 6 \text{ s}$, an extremely high power density, and questions could be made concerning heat generation

and the effects on rapid polymerization shrinkage, as described for PAC LCUs [20,21]. Besides these concerns, the lack of difference in DC presented by PE protocol is a finding that contrasts with the fact that it showed the lowest values of hardness rate and lower values of hardness in the bottom surface.

Still regarding the DC test, there was no statistical effect for *measurement* or for *exposure mode* factors. Concerning the degree of conversion, no positive effect was observed in the first increment when it was exposed to light-activation of the later increments. For the single exposure mode, this result is in accordance with results of other studies that used different curing routines with similar energy density [22,23]. For multiple exposure modes, the photons emitted by the light-curing unit and which were able to excite photoinitiators available in the first increment were not enough to significantly increase the DC.

According to the study results, the light-activation of successive increments is not able to improve the properties of the first layered increment. The first increment layered in a proximal box of a class II cavity is difficult to be cured, especially due to the long distance between the light-curing tip and the material, even though the light-activation was made by a powerful LCU. A clinical possibility to contour this situation is the use of a lighter shade resin composite [13] and a composite thickness thinner than 1 mm [3].

Conclusions

Incremental restorative technique implies multiple light exposure of the former inserted increments. This successive exposure did not influence the microhardness and degree of conversion of the first increment in both the top and bottom surfaces. A light-activation protocol that delivered 3200 mW/cm² in 6 s results in lower microhardness in the bottom surface than the other light-activation protocols.

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