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Degree of Conversion and Contraction Stress Development of a Resin Composite Irradiated Using Halogen and LED at Two C-factor Levels

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Clinical Relevance

Curing methods using lower irradiance levels were shown to be effective in reducing the rate of stress generation without compromising the conversion of the restorative composite. Higher C-factor was shown to enhance the stress rate and amount of stress generated.

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SUMMARY

This study verified the influence of curing methods and light sources on contraction stress, stress rate and degree of conversion (DC) of a restorative composite at two C-factor (CF) levels. For the stress test, composite (0.84 mm thick) was applied between two glass rods 5-mm in diameter mounted in a servohydraulic testing machine. Stress rates were calculated as the change in stress vs time at each second. DC was measured by micro-FTIR. Five curing methods were tested at two C-factor levels (1.5 and 3.0): High Intensity LED (LED HI), Continuous Light (QTH CL), Medium Intensity LED (LED MI), Low Intensity LED (LED LI) and Pulse Delay (QTH PD). The results were analyzed by ANOVA and Tukey's test

(α=0.05). For the stress test at CF 1.5, QTH PD presented lower values than LED HI, QTH CL and LED LI. At CF 3.0, no difference was observed among the curing methods. For all curing methods, stress values at CF 3.0 were statistically higher than those at CF 1.5. LED HI presented the highest maximum stress rate, followed by QTH CL, LED MI, LED LI and QTH PD for both C-factors. In the DC test, no difference was observed among the methods and between the C-factor levels.

INTRODUCTION

Clinical failure of composite restorations is often the result of an incomplete sealing of the tooth/restoration interface.[1] The role of composite polymerization stress as one of the main causes of marginal integrity loss and consequent post-operative occurrences, such as hypersensitivity, microleakage and secondary caries, has been described in some studies.[2-3]

Stress magnitude is related to the restorative technique employed, as well as to the composite composition and degree of conversion. One of the factors associated with the restorative procedure is the restoration's bonded-to-unbonded surface area (cavity configuration factor or C-factor).[4] The confinement of the composite would hinder the volume reduction compensation by viscous flow from the free surface, which may occur before and during gelation of the material.[5]

It has been proposed that the method by which light energy is delivered to the composite is capable of reducing the rate of stress development,[6-7] and this is associated with a delay in the acquisition of elastic modulus, allowing polymeric chains to re-arrange microscopically and macroscopically.[8] The use of low irradiances has become common in clinical practice, as several studies have shown that the use of continuous low intensity curing routines and those characterized by reduced irradiance during the initial seconds, may lead to significant reductions in microleakage and gap formation in composite restorations.[9-10]

Different light sources may also influence the development of physical properties. Blue light-emitting diode (LED) has the advantage of a narrower spectral range than the quartz-tungsten-halogen (QTH) light and a better match of light emitted with the absorption spectrum of the photoinitiator camphorquinone.[11-13] Manufacturers claimed that this could increase conversion efficiency and reduce the required exposure time.[14-15] In addition, LED units do not use filters, which are required with halogen units for wavelength selection. From this viewpoint, LED units represent an improvement over halogen lamps. However, there is no evidence that, for a given radiant exposure, LED units provide better results compared with QTH units.[16-17]

Therefore, the current study evaluated the effect of different irradiance levels and light sources (QTH and LED) on the stress generated, stress rate and degree of conversion of a resin composite at two C-factor levels. The light-curing methods using reduced irradiance levels were hypothesized to promote a reduction in contraction stress and the stress rate, with no reduction in degree of conversion of the restorative composite. A higher C-factor level was hypothesized to generate an increase in the stress level and stress rate, with no influence on the degree of conversion values.

METHODS AND MATERIALS

Light curing was performed with two light-curing units. The first curing unit was the VIP (Variable Intensity Polymerizer, BISCO, Schaumburg, IL, USA), a halogen light unit that provides different levels of irradiance used in a continuous exposure (QTH CL) and a modulated exposure (QTH PD). The second curing unit was the Free-Light II (3M-ESPE, St Paul, MN, USA), an LED light source of high irradiance used in three different irradiances: high (LED HI), medium (LED MI) and low (LED LI). The protocols of the curing methods evaluated are listed in Table 1. For the modulated curing method QTH PD, photoactivation was started by an initial short pulse of light using low irradiance (5 seconds at 150 mW/cm^2), followed by a waiting time of three minutes (no light exposure) before the final light exposure was performed in higher irradiance (550 mW/cm^2). In order to accomplish the irradiances used in the protocols evaluated in all of the following tests, neutral density filters were placed between the light source and guide for both light-curing units. In addition, for VIP, a Turbo tip was used. Filtek Z250 (shade A2, 3M-ESPE, batch #5JH) composite was used for all experiments.

Table 1: *Light-curing Methods Evaluated with Their Outputs and Respective Energy Density*

Method	Irradiation Protocol	Light Source	Radiant Exposure
High Intensity LED (LED HI)	19 seconds at 850 mW/cm^2	LED	16 J/cm^2
Continuous light (QTH CL)	30 seconds at 550 mW/cm^2	Halogen	16 J/cm^2
Medium Intensity LED (LED MI)	30 seconds at 550 mW/cm^2	LED	16 J/cm^2
Low Intensity LED (LED LI)	80 seconds at 200 mW/cm^2	LED	16 J/cm^2
Pulse Delay (QTH PD)	5 seconds at 150 mW/cm^2 + 3 minutes + 28 seconds at 550 mW/cm^2	Halogen	16 J/cm^2

Polymerization Contraction Stress and Stress Rate

The polymerization contraction stress test was performed with a closed loop servohydraulic, low compliance testing instrument (MTS 858, MTS Systems, Eden Prairie, MN, USA). Two borosilicate glass rods 5 mm in diameter were sandblasted (50 µm Al_2O_3) and treated with silane (Silane ceramic primer, 3M-ESPE, batch #5WJ) and light-cured adhesive resin (Scotchbond MP, 3M-ESPE, batch #0MA). One of the glass rods (upper rod, 12 mm in height) was wrapped with eight layers of adhesive cellophane tape and attached with four set screws to a metallic fixture connected to the actuator, with a slot through which the light curing guide was kept in contact with the opposing side of the rod. The other rod (lower rod, 10 mm in height) was bonded with dental composite to a steel fixture attached to the load cell. Contraction stress was measured by placing two different thicknesses of composite between the two rods: 1.66 mm (C-factor = 1.5) and 0.84 mm (C-factor = 3.0). A near zero compliance system was set up by using an eddy current feedback system (Kaman Instruments, Colorado Springs, CO, USA) that worked with the actuator to keep the distance between the rods constant. A light-curing guide was directed down through the upper rod, and the contraction forces were recorded for 10 minutes from the initiation of light activation. The maximum force was divided by the cross-sectional area to calculate the axial stress. Five samples of each curing method were tested. The stress rates were calculated as the change in stress vs time at each second during the measurement period.

Prior to testing, the light intensity at the top of the specimen (bottom of the upper glass rod) was measured using a power meter (Power Maximum 5200, Molectron, Portland, OR, USA) for both light-curing units. For the VIP unit, when the irradiance at the end of the Turbo light guide was 780 and 220 mW/cm^2, the irradiance at the surface of the composite was approximately 550 and 150 mW/cm^2, respectively, demonstrating a 30% reduction in light intensity when the light passed through the glass rod. The same reduction was observed for the Free-Light II unit. Therefore, irradiances of 1200, 780, and 290 mW/cm^2 at the end of the light guide corresponded approximately to 850, 550 and 200 mW/cm^2 at the top surface of the composite.

Degree of Conversion

The degree of conversion of the resin composite for the five curing methods tested was determined using a micro-Fourier Transform Infrared (FTIR) spectrometer (DS20/XAD, Analect Instruments, Irvine, CA, USA). Glass rings of two different heights (0.5 and 2.0 mm), both being 4 mm in diameter, and glass slides, were sandblasted and silanated (Silane ceramic primer, batch #5WJ). The rings were bonded to the slide using a thin coat of adhesive (Scotchbond MP, batch #2MT), resulting in two different glass cavities of bonded-to-unbonded area ratio of 1.5 (4 mm in diameter and 0.5 mm deep) and 3.0 (4 mm in diameter and 2 mm deep). The glass cavity was bulk filled with the restorative composite. A clear matrix strip and glass slide was pressed over the glass cavity to force the composite to adapt to the cavity walls and extrude the excess material. The composite was then light activated through the glass slide at the bottom of the cavity. For the VIP unit, when the irradiance at the end of the Turbo light guide was 610, 170 and 90 mW/cm^2, the irradiance at the bottom surface of the composite was 550, 150 and 80 mW/cm^2, respectively, considering a 10% reduction in light intensity when the light passed through the sandblasted glass slide. The same reduction was observed for the Free-Light unit. Therefore, irradiances of 950, 610 and 220 mW/cm^2 at the end of the light guide corresponded approximately to 850, 550 and 200 mW/cm^2 at the surface of the composite. Three samples for each experimental condition were prepared and stored dry for 24 hours at room temperature. Chips of composite, approximately 50 x 100 µm, were removed with a scalpel from the specimen's top surface (opposite surface from the light exposure) under safe yellow light and subsequently analyzed in transmission FTIR at 8 cm^{-1} resolution. Three spectra were analyzed per specimen. The ratio between the intensities of aliphatic C=C (at 1637.3 cm^{-1}) and aromatic C=C (at 1608.3 cm^{-1})

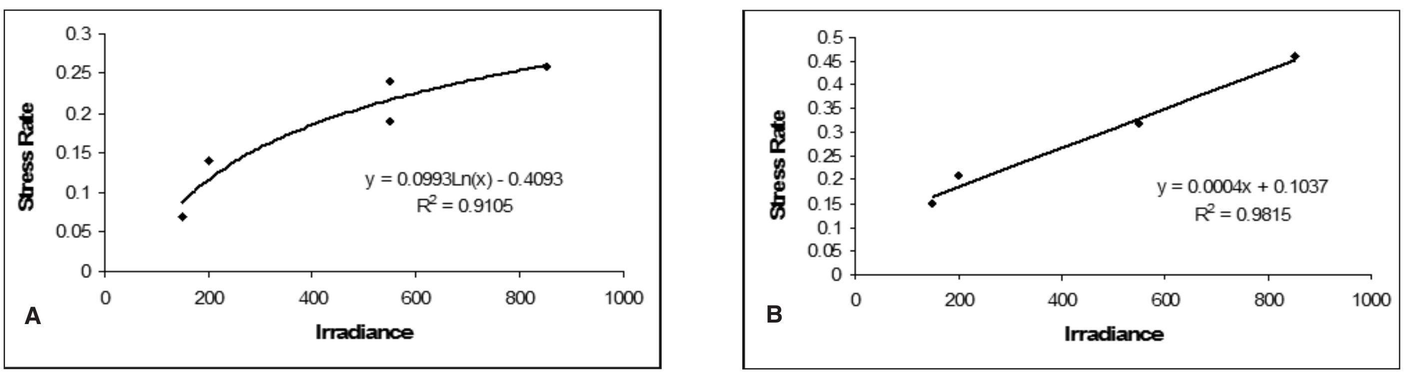


Figure 1. *Correlation curves of irradiance vs stress rate at C-factor 1.5 (A) and at C-factor 3.0 (B).*

peaks for cured and uncured samples was used to calculate the degree of conversion, according to the following equation:

$$DC = \left[1 - \left(\frac{[\text{Abs (C=C aliph)/Abs)C}\cdots\text{ arom)] cured resin}}{[\text{Abs (C=C aliph)/Abs (C}\cdots\text{ arom)] uncured resin}}\right)\right] \times 100$$

where DC is the degree of conversion, *Abs (C=C)* arom is the height of the benzene ring peak and *Abs (C=C) aliph* is the height of the aliphatic C=C bond peak, for both cured and uncured composites.

Statistical Analysis

Maximum contraction stress and degree of conversion values were analyzed by one-way ANOVA and Tukey's test at a significance level of 5%. The correlation between initial irradiance and stress rate was also performed for C-factor 1.5 (Figure 1A) and for C-factor 3.0 (Figure 1B).

RESULTS

Effect on Polymerization Contraction Stress and Stress Rate

As shown in Figure 2, a sharp increase in contraction stress was observed immediately following light activation for all curing methods and both C-factors, except for QTH PD, where a lower rate of stress development was observed. A continuous increase in stress values was observed when the light was turned off for all methods and both C-factors. Figure 3 shows both the stress rate in the first 20 seconds of light exposure for the curing methods and the influence of the C-factor on the stress rate. The maximum stress rate is listed in Table 2. At C-factor 1.5, LED HI presented the highest stress rate (0.26 MPa/s), reached at 3.3 seconds, followed by QTH CL, with a maximum stress rate of 0.24

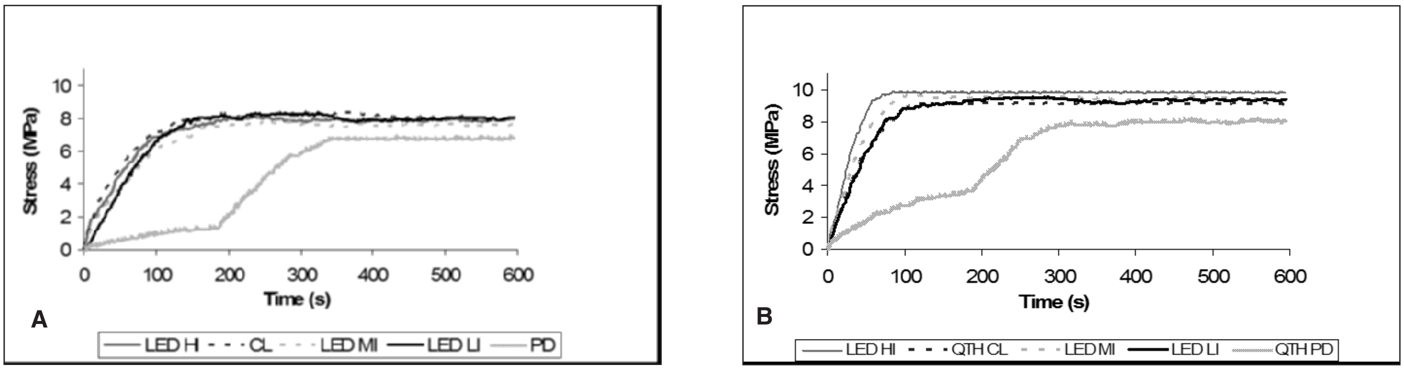
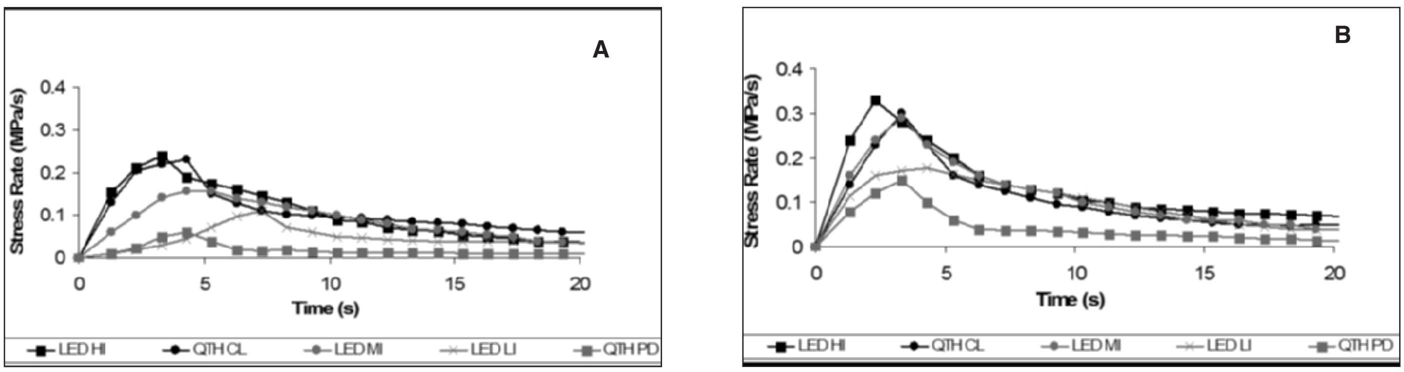


Figure 2. *Stress values (MPa) for each light-curing as a function of time at C-factor 1.5 (Figure 2A) and 3.0 (Figure 2B).*

Figure 3. *Stress rate (MPa/s) during the first 20 seconds of curing for each light-curing method. (Figure 3A) at C-factor 1.5; (Figure 3B) at C-factor 3.0; (Figure 3C) influence of the C-factor, independent of the light-curing method.*



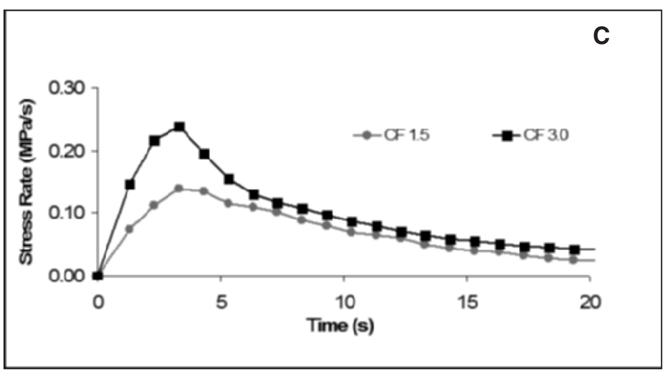


Table 2: *Contraction Stress (MPa), Maximum Stress Rate (MPa/s) and Degree of Conversion Generated by the Light-curing Methods at Each C-factor Level*

	Contraction Stress		Stress Rate		Degree of Conversion	
	CF 1.5	**CF 3.0**	**CF 1.5**	**CF 3.0**	**CF 1.5**	**CF 3.0**
LED HI	8.6 (0.6) a, B	10.5 (1.2) a, A	0.26	0.46	53.0 (2.2) a, A	52.5 (1.2) a, A
QTH CL	8.6 (0.5) a, B	10.3 (1.1) a, A	0.24	0.32	54.6 (0.9) a, A	54.5 (0.5) a, A
LED MI	8.0 (1.2) ab,B	10.4 (1.2) a, A	0.19	0.32	52.6 (2.1) a, A	53.1 (1.8) a, A
LED LI	8.7 (0.8) a, B	10.3 (0.6) a, A	0.14	0.21	54.1 (2.5) a, A	54.6 (1.9) a, A
QTH PD	7.3 (0.6) b, B	9.3 (1.3) a, A	0.07/0.12 [1]	0.15/0.15 [1]	53.6 (1.3) a, A	54.2 (2.6) a, A

[1]First value is maximum stress rate during primary step of pulse-delay cure, while second value corresponds to maximum stress rate during cure after the delay.
Mean values followed by different small letters in the column and capital letters in the row differ statistically among themselves for the Tukey test at the level of 5%. ( )—Standard Deviation

MPa/s, reached at 4.3 seconds. LED MI presented an intermediate value of the maximum stress rate 0.19 MPa/s at 4.3 seconds. The LED LI and QTH PD methods presented the lowest maximum stress rates: 0.14 MPa/s for LED LI at 7.3 seconds and 0.07 MPa/s for QTH PD at 4.3 seconds. At C-factor 3.0, the same order was observed: LED HI showed the highest maximum stress rate (0.46 MPa/s at 2.3 seconds), followed by QTH CL and LED MI (0.32 MPa/seconds reached at 3.3 seconds for both methods), LED LI (0.21 MPa/s at 4.3 seconds) and QTH PD (0.15 MPa/s at 3.3 seconds). Table 2 lists the mean maximum contraction stress values and standard deviations for all curing methods at the different C-factor levels. At C-factor 1.5, the LED HI (8.6 MPa), QTH CL (8.6 MPa) and LED LI (8.7 MPa) methods were responsible for the highest mean values, not differing statistically among themselves but being greater than the QTH PD method (7.3 MPa). The curing method LED MI (8.0 MPa) presented an intermediate mean value, equivalent to all other methods evaluated. At C-factor 3.0, no statistical difference was observed among the curing methods. For all curing methods, the mean maximum stress values reached at C-factor 3.0 were statistically superior to those reached at C-factor 1.5.

The correlation of initial irradiance vs stress rate at C-factor 1.5 showed a reasonable fit with a logarithmic function (Figure 1A). The stress rate demonstrated an increase with increasing initial irradiance values but tended to level off at the higher energy levels. At C-factor 3.0, the correlation of initial irradiance vs stress rate was best described by a linear function (Figure 1B).

Effect on Degree of Conversion

As can be seen in Table 2, no statistical difference (p>0.05) was observed for the curing methods tested for both C-factors evaluated. At C-factor 1.5, mean values ranged from 52.6 % (LED MI) to 54.6 % (QTH CL). To the C-factor 3.0, mean values ranged between 52.5 % (LED HI) and 54.6 % (LED LI). No influence of the C-factor levels was observed on the mean values of degree of conversion for either curing method.

DISCUSSION

No statistical difference was observed among the curing methods for the maximum stress values at C-factor 3.0. At C-factor 1.5, QTH PD showed a lower mean stress value when compared to the other curing methods, except LED MI. However, it was possible to observe a significant influence of curing method on the contraction stress rate. The correlation analysis between irradiance and stress rate at C-factor 1.5 (r^2 = 0.91) and at C-factor 3.0 (r^2=0.98) showed a logarithmic and linear relation, respectively. Indeed, the method that used the highest initial irradiance (LED HI) was associated with the highest stress rate when compared to lower initial irradiance curing methods, such as LED LI and QTH PD. In fact, QTH PD led to the lowest stress rates (0.07 and 0.15 MPa/s—at C-factors 1.5 and 3.0, respectively) in this study, being 73% lower at C-factor 1.5 and 67% lower at C-factor 3.0 than the highest stress rates found in this study (LED HI—0.26 and 0.46 MPa/s at C-factors 1.5 and 3.0, respectively). At C-factor 1.5, the slower polymerization reaction increased the probability of partial stress release. Indeed, the 73% reduction in the stress rate observed for QTH PD could be responsible for a significant reduction in the final stress generated, resulting in a statistically inferior mean value of contraction stress to QTH PD when compared to LED HI.

However, the same situation was not observed at C-factor 3.0. Though the stress rate of QTH PD was significantly reduced (67% lower), it did not result in a significant decrease in final contraction stress. Findings of previous studies[18-20] suggest that the polymerization rate must be reduced below a certain threshold in order to significantly reduce final contraction stress. This may be due to the gelation of methacrylates that occurs at very low degrees of conversion[21] and, once the reaction has started, an auto acceleration phenomena is observed and stresses are expected to increase drastically because of the accompanying increase in stiffness produced by crosslinking.[22] Therefore, the period allowed for the material to flow is very restricted, and a substantial decrease in curing rate is required to significantly affect contraction stress development. This situation is even worse at a high C-factor level, wherein the faster polymerization reaction decreases the probability of partial stress release. However, it is also important to note that materials with distinct compositions may show different behaviors when submitted to the same curing methods. A study evaluating the effect of low curing rates on contraction stress development of three commercial materials found stress reductions between 19% and 30%.[23]

One possible explanation for the similar final contraction stress values found for all experimental groups at C-factor 3.0 is that the final radiant exposure employed for all was similar (16 J/cm^2). According to previous findings,[24] this would lead to the same degree of conversion, as was also observed in the current study. Silikas and others[25] and Vandewalle and others[26] found a high correlation (r^2>0.99) between the degree of conversion and stress generation. Therefore, if reduction in the stress rate caused by the modulated curing methods was not sufficient to significantly decrease the final stress of the composite, as observed for the high C-factor level groups, then the degree of conversion becomes the most important factor affecting the development of final contraction stress in dental composites.[27] Thus, the statistical equivalent in the degree of conversion for the curing methods could explain the results found in the current study for the maximum stress mean values at C-factor 3.0.

For all curing methods, the final stress reached at C-factor 1.5 was statistically inferior to that reached at C-factor 3.0. Since composite flow is more likely to occur from the free surfaces of the specimen, a lower level of C-factor will indicate a higher proportion of free composite surface, causing a smaller restriction to shrinkage, thereby reducing stress[28] and, consequently, reducing the probability of gap formation.[29] In the current study, a mean reduction of 20% in the maximum stress was found at C-factor 1.5 when compared to the values reached at C-factor 3.0. In addition, influence of the C-factor level was not related to only the amount of stress generated, but it also had a significant influence on the stress rate. As can be seen in the mean values of stress rate in Table 2 and Figure 3, the maximum stress rate found at C-factor 1.5, independent of the curing method, was around 35% lower than that related to C-factor 3.0.

The stress rate is dependent on irradiance and not on the radiant exposure.[30] The stress rate values reported here were an instantaneous rate, as opposed to the final stress/total time, as has been reported by others.[31] Therefore, it was possible to determine that the maximum stress rate occurred early in the curing cycle. Moreover, after the stress rate peak was reached, it declined, regardless of the irradiance and duration of exposure for all curing methods. Interestingly, the method that presented the highest stress rates (LED HI) also showed the most premature maximum stress rate development, 3.3 seconds and 2.3 seconds, at C-factors 1.5 and 3.0, respectively. In addition, the C-factor level had a significant influence on stress rate development. For all curing methods, the maximum stress rate was reached faster at C-factor 3.0, when compared to the values within the same method at C-factor 1.5.

The LED LI and QTH PD methods used similar low irradiances (200 and 150 mW/cm^2, respectively) but in continuous mode vs pulse-delay mode. Based on the stress rate values, QTH PD was more efficient at reducing the stress rates, perhaps because the first exposure used not only low irradiance but was performed for a short time. Since conversion was expected to continue increasing during the dark period, when the second, higher irradiance exposure was applied, there were less carbon double bonds remaining to be used in the growing chains. Therefore, the QTH PD method resulted in lower stress rates during the second cycle of light exposure (0.12 and 0.15 MPa/s at C-factors 1.5 and 3.0, respectively), even using the same irradiance as the QTH CL or LED MI methods.

Another interesting comparison that can be made from the results of the current study is between the QTH CL and LED MI methods. Some manufacturers claim that LED units can reach a higher degree of conversion compared with halogen units that use shorter exposure times, which would be clinically advantageous.[14-15] This is attributed to the narrow light spectrum of LED units, with wavelengths between 438 and 501 nm and peak intensity at 465 nm,[11-13] which is coincident with the absorption peak of camphorquinone, between 465 and 470 nm,[32] theoretically making it more efficient in activating the camphorquinone. In the current study, comparing the curing methods LED MI and QTH CL, both using the same irradiance of 550 mW/cm^2 and the same radiant exposure (16 J/cm^2), no statistical difference was observed in the degree of conversion and maximum contraction stress at both C-factor levels. At least for the degree of conversion, this fact corroborates the results of other studies,[16-17] where no difference was observed in the development of this property when LED and QTH units were compared.

The tested hypothesis that curing methods using reduced irradiance or modulation of light exposure would provide a significant reduction in contraction stress was partially validated by the results. Despite the lack of statistical difference among the LED methods and QTH CL at C-factor 1.5 and also for all methods at C-factor 3.0 as to the maximum stress generation, a significant reduction in stress rate values was observed for the curing methods using reduced irradiance levels. This was reached at no expense to the degree of conversion. The hypothesis regarding the C-factor was validated by the results. The higher the C-factor level, the higher the amount of stress generated and the faster the generation of stresses. However, the C-factor was proven to have no effect on the degree of conversion of the restorative composite.

CONCLUSIONS

Within the limits of the current study, the following conclusions were drawn. No difference was observed for all curing methods evaluated in this study as to the maximum stress generation when the C-factor was 3.0. However, a significant reduction in stress rate values was observed for the curing methods using reduced irradiance levels. This was reached at no expense to the degree of conversion.

The C-factor level was proven to negatively affect the stress generation. The higher the C-factor level, the higher the amount of stress generated and the faster the generation of stresses. However, no influence in the C-factor was observed in relation to the degree of conversion of the restorative composite.

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