

# Effects of Preheating and Precooling on the Hardness and Shrinkage of a Composite Resin Cured with QTH and LED

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## Clinical Relevance

The composite hardness was not affected by different pretreatment temperatures, whereas the shrinkage was not affected by the temperatures only when the composite was cured with an LED-curing unit.

## SUMMARY

**The aim of this study was to evaluate *in vitro* the hardness and shrinkage of a pre-cooled or preheated hybrid composite resin cured by a quartz-tungsten-halogen light (QTH) and light-**

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emitting diode (LED) curing units. The temperature on the tip of the devices was also investigated. Specimens of Charisma resin composite were produced with a metal mold kept under 37°C. The syringes were submitted to 4°C, 23°C, and 60°C (n=20) before light-curing, which was carried out with the Optilux 501 VCL and Elipar FreeLight 2 units for 20 seconds. The specimens were kept under 37°C in a high humidity condition and darkness for 48 hours. The Knoop hardness test was carried out with a 50 gram-force (gf) load for 10 seconds, and the measurement of the shrinkage gap was carried out using an optical microscope. The data were subjected to analysis of variance and the Games-Howell test ( $\alpha=0.05$ ). The mean hardness of the groups were similar, irrespective of the temperatures ( $p>0.05$ ). For 4°C and 60°C, the top surface light-cured by LED presented significantly reduced shrinkage when compared with the bottom and to both surfaces cured by QTH

( $p < 0.05$ ). It was concluded that the hardness was not affected by pre-cooling or preheating. However, polymerization shrinkage was slightly affected by different pre-polymerization temperatures. The QTH-curing generated greater shrinkage than LED-curing only when the composite was preheated. Different temperatures did not affect the composite hardness and shrinkage when cured by a LED curing unit.

## INTRODUCTION

The mechanical properties of composite resins are fundamental for the longevity of the restorations. On the other hand, mechanics is not the only factor to be observed. Attention needs to be paid to the composite polymerization in order to minimize the effects of shrinkage, like gap formation.<sup>1</sup> Marginal gaps seem to predispose teeth to secondary caries by plaque accumulation, leading to the early loss of the restoration.<sup>2,3</sup>

The Knoop hardness test has been commonly used to evaluate the mechanical behavior of dental materials.<sup>4-10</sup> Hardness tests are the most frequently used method to evaluate the curing depth<sup>4,11</sup> and the polymer cross-linking of dental composites.<sup>12,2</sup> There are many ways to investigate the polymerization shrinkage, including the analysis of shrinkage gap formation between the resin-based material and a metallic ring.<sup>14</sup> Among the several factors that may impact the hardness and the polymerization shrinkage of the composite resin are pre-cooling, preheating, and the use of different light units for curing.<sup>8,15-18</sup>

Preheating has been used to improve the mechanical properties and the degree of conversion of resin-based materials.<sup>12,16,19,20</sup> Preheated composite resins have demonstrated higher marginal adaptation compared with a room-temperature material.<sup>12</sup> A previous study<sup>8</sup> demonstrated that cooling before light-curing did not result in differences in hardness for the composite resins after polymerization. In addition, it was observed that the pre-cooling of the composite resin might decrease the shrinkage.<sup>18</sup> Therefore, the lack of literature and the fact that manufacturers usually recommend keeping the composite syringes inside the refrigerator justifies further investigation on the effect of pre-cooling on the mechanical properties of composite resins.

Another important factor is the light source, where the quartz-tungsten-halogen light (QTH) and light-emitting diode (LED) are the most commonly used

devices. The QTH is composed of a tungsten filament that emits visible light and a great amount of infrared radiation. A filter is necessary to select only the wavelength indicated for the photo-initiator activation of the composite resins. These devices require a fan to minimize the high temperatures that are generated inside. In contrast, LED-curing units emit light through the applied voltage in a semiconductor system with gallium nitride. In this case, the light shows a specific wavelength controlled by the chemical composition of the semiconductor. Nevertheless, the wavelength<sup>21</sup> and the heat generated during polymerization<sup>10,22,23</sup> seem to be significant differences between the QTH and the LED.

Additionally, because the composite resin temperature increase may accelerate the conversion of the monomers,<sup>19</sup> both the preheating and the light-curing unit may induce a fast increase in the material toughness, leading to greater shrinkage stress.<sup>24</sup> Still, although the temperature before light-curing and the different light-curing units have been investigated, such studies have stabilized the temperature during light-curing,<sup>16,19,25</sup> which is not possible in a clinical situation. Other studies have applied only the halogen light among the variables.<sup>18,19</sup>

New studies are necessary to evaluate hardness and shrinkage gap formation of composite resins submitted to pretreatment by heating and cooling. It is also important to evaluate the composite behavior under a temperature that is close to body temperature. The impact of the QTH and LED units on preheated or pre-cooled composite resin remains unclear.

The aim of this study was to investigate hardness and shrinkage of a pre-cooled or preheated hybrid composite resin using QTH- and LED-curing units. Additionally, the temperature on the tips of the curing units was also measured. The evaluated null hypotheses were as follows: 1) temperature pretreatment of composite would not affect hardness or gap formation; and 2) the type of light-curing unit would not affect composite hardness or shrinkage gap.

## METHODS AND MATERIALS

A total of 120 samples of the composite resin Charisma (Heraeus-Kulzer, Hanau, Germany) were prepared at the temperatures of 4°C, 37°C, and 60°C ( $n=20$ ) and light-cured by a QTH or LED for 20 seconds. The QTH light-curing unit used was an Optilux VCL 501 (Demetron/Kerr, Danbury, CT, USA), and the LED light-curing unit used was an

Elipar FreeLight 2 (3M ESPE, St Paul, MN, USA). The irradiance of the light sources was measured by a radiometer coupled to the Optilux VCL 501. The QTH was used with an 11-mm tip diameter with 800 mW/cm<sup>2</sup>, and the LED was used with an 8-mm tip diameter with 1200 mW/cm<sup>2</sup>. Within 20 seconds, QTH generated 16 J/cm<sup>2</sup> energy density and the LED generated 24 J/cm<sup>2</sup>.

A heated platform (TE 0851, Tecnal, Piracicaba, Brazil) was used to simulate the oral temperature (37°C). This temperature was checked using a digital multimeter (DT-838, Impac, São Paulo, Brazil). A 7-mm-thick glass plate and a 1-mm-thick glass slide were placed on the heated platform, which was used as a base for the molds. The molds used—one for each specimen—were made of brass in a metallic ring shape with an external diameter of 12 mm, an internal diameter of 7 mm, and a height of 2 mm. The composite resin was inserted into the mold using a composite filling instrument (Goldstein Flexi-Thin 2, Hu-Friedy, Chicago, IL, USA). The total insertion time, from the material removal from the container to the insertion of the composite resin in the mold, was approximately 40 seconds. Another 1-mm-thick glass slide was placed on top of the assembly. Then the light-curing was carried out for 20 seconds, as recommended by the resin composite manufacturer, with the QTH and LED units divided into separate groups. All the samples were made inside a room with controlled temperature and humidity, at 23°C ± 1°C and 50% ± 10%, respectively.

A refrigerator (Consul CRC08, Consul, Joinville, Brazil) was used to obtain the temperature of 4°C for the composite resin. Six composite syringes were placed in the refrigerator for at least 30 minutes to stabilize the cooled temperature. Immediately after light-curing, the syringe was returned to the refrigerator and replaced by another one at the stabilized cooled temperature to make another sample.

In order to obtain the temperature of 37°C for the composite resin, the syringes were stored at room temperature (23°C ± 1°C). The composite resin was taken from the syringe and inserted into the mold that was kept at 37°C. The composite resin was light-cured only after reaching 37°C, which was checked by the multimeter and took about 40 seconds to occur. The samples were light-cured only after this period of time.

A water bath (TE 054 Mag, Tecnal, Piracicaba, Brazil) was used to obtain the temperature of 60°C for the composite resin. A waterproof plastic bag

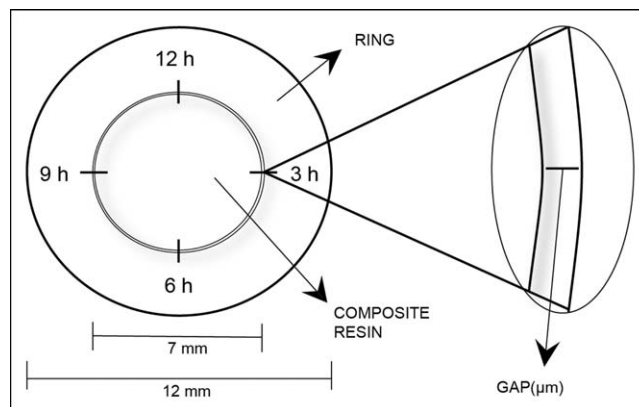


Figure 1. Measurement of the shrinkage gap formation in micrometers.

protected the syringes during heating. The syringes remained immersed in water for 15 minutes so that the temperature of 60°C would be reached.

All the samples were stored under high-humidity conditions for 48 hours in an oven at 37°C. After this period, the Knoop hardness test and the shrinkage gap formation test were carried out.

### Knoop Hardness

A microdurometer (HMV 2, Shimadzu Corporation, Tokyo, Japan) was used for the Knoop hardness test with an applied load of 50 gram-force (gf) for 10 seconds. Five indentations were made on the top and the bottom of each sample. The distance between each indentation was at least four times the length of the larger diagonal of the Knoop indentation tip. The five indentation values were averaged at the top and at the bottom sites (n=20).

### Shrinkage Gap Formation

After the Knoop hardness was checked, the samples were polished on the top and bottom with #500, #600, and #1200 silicon carbide sandpaper in order to remove composite excess from the interface of the composite resin/ring.

The shrinkage gap formation analysis was carried out at four sites on the ring (3 hours, 6 hours, 9 hours, and 12 hours) (Figure 1) using a 20× magnification optical microscope (BX60 F5, Olympus Optical Limited, Tokyo, Japan) and an image analyzer software (OMNIMET Express, Buehler Ltd, Lake Bluff, IL, USA). An average of four values in micrometers for both the top and bottom surfaces (n=20) was obtained according to Obici and others.<sup>14</sup>

### Temperature on the Tips of the Devices

A digital multimeter and a digital video camera (Nikon D90, Nikon Corporation, Tokyo, Japan) were used to check the temperature emitted by the tips of the light-curing unit. The tip of the multimeter was placed in contact with the center of the light-curing unit tip, and the video camera was positioned where it focused on the screen of the digital multimeter. Ten videos were made of the multimeter screen during the 20 seconds that the QTH and LED units were activated (five videos for each light-curing unit). The temperatures at two, five, 10, 15, and 20 seconds were checked in each video. The average of the temperatures was obtained for each time verified and for both light-curing units.

### Statistical Analysis

The Knoop hardness values and the shrinkage gap formation values were subjected to the Kolmogorov-Smirnov test to check the normality in distribution, the full factorial model analysis of variance (ANOVA) with three factors, the Games-Howell test ( $\alpha=0.05$ ), and the Pearson correlation test.

## RESULTS

### Knoop Hardness Results

The ANOVA results showed that there were significant differences ( $p=0.000$ ) between QTH (29.40) and LED (30.48), regardless of the temperature prior to light-curing and location of verification (bottom or top). There were also significant differences ( $p=0.002$ ) between the top (31.52) and bottom (28.35) regardless of light source and temperature. Among the prepolymerized composite resin temperatures of 4°C, 37°C, and 60°C, there were no significant differences ( $p=0.054$ ) regardless of light source and location of verification (bottom or top).

As the Games-Howell test (Table 1) indicated, there were no significant differences in hardness among the experimental groups submitted to different temperatures ( $p>0.05$ ). For the temperatures of 4°C and 37°C, a significant reduction in hardness was found at the bottom of QTH-cured composite ( $p<0.05$ ). The same reduction was found for the bottom surfaces cured by QTH and LED with the 60°C preheating, without significant differences from each other. For the temperature of 37°C, the bottom light-cured by QTH presented the smallest values ( $p<0.05$ ). For the temperature of 60°C, the top light-cured by QTH presented the highest values ( $p<0.05$ ).

Table 1: Mean Knoop Hardness (SD) for the Composite Resin, Cured by QTH and LED Under Different Pretreatment Temperatures on the Top and Bottom Surfaces

Device/ Surface	Temperature		
	4°C	37°C	60°C
QTH, top	32.47 (2.58) Aa <sup>a</sup>	31.95 (2.23) Aa	33.55 (2.03) Aa
QTH, bottom	25.68 (1.97) Ab	25.79 (2.22) Ab	26.93 (4.60) Ac
LED, top	30.31 (2.48) Aa	29.65 (2.04) Aa	31.18 (1.85) Ab
LED, bottom	32.57 (2.48) Aa	29.97 (2.78) Aa	29.18 (3.63) Abc
Abbreviations: LED, light-emitting diode; QTH, quartz-tungsten-halogen. <sup>a</sup> Means followed by the same uppercase letter indicates no significant statistical difference in the row and the same lowercase letter indicates no significant statistical difference in the column ( $p>0.05$ )			

### Shrinkage Gap Formation Results

The ANOVA results showed that there were significant differences in the shrinkage gap ( $p=0.000$ ) between QTH (29.57) and LED (25.21) regardless of the temperature prior to light curing and location of verification (bottom or top). There were also significant differences ( $p=0.000$ ) between the top (25.06) and bottom (29.72) regardless of light source and temperature. Among the composite resin temperatures of 4°C, 37°C, and 60°C, there were significant differences in shrinkage ( $p=0.000$ ) regardless of light source and location of verification (bottom or top).

As the Games-Howell test (Table 2) indicated, for 4°C and 60°C, the top surface light-cured by LED presented significantly reduced shrinkage when compared with the bottom and to both surfaces cured by QTH ( $p<0.05$ ). For top and bottom surfaces cured by LED there were no significant differences in the composite hardness despite the temperature.

The Pearson analysis showed that there was no correlation between hardness and shrinkage ( $r=0.0040$  and  $p=0.95$ ).

### Temperature on the Tips of the Devices

Figures 2 and 3 show the average temperature in Celsius emitted by the tips of the devices at two, five,

Table 2: Mean Values ( $\mu\text{m}$ ) for the Shrinkage Gap (SD) for the Composite Resin, Cured by QTH and LED Under Different Pretreatment Temperatures on the Top and Bottom Surfaces

Device/ Surface	Temperature		
	4°C	37°C	60°C
QTH, top	29.34 (10.07) ABab <sup>a</sup>	25.16 (8.83) Bab	36.11 (9.51) Aa
QTH, bottom	24.73 (4.08) Bb	27.45 (6.25) ABa	34.65 (9.30) Aa
LED, top	17.33 (6.02)	18.88 (7.97) Ab	23.57 (6.89) Ab
LED, bottom	30.77 (4.08) Aa	29.66 (7.50) Aa	31.08 (6.67) Aa

<sup>a</sup> Means followed by the same uppercase letter indicates no significant statistical difference in the row and the same lowercase letter indicates no significant statistical difference in the column ( $p>0.05$ )

10, 15, and 20 seconds. The average temperature was 40.24°C on the QTH device and 37.88°C on the LED unit.

DISCUSSION

Previous studies demonstrated that the higher the composite resin temperature is before light curing, the higher the hardness and the degree of conversion up to five minutes after polymerization.<sup>8,26</sup> The viscosity of the composite resin decreases with preheating, providing more mobility for the free radicals and an increase in the collision frequency of the nonreactive groups.<sup>26</sup> The present study showed that pre-cooling or preheating did not affect the

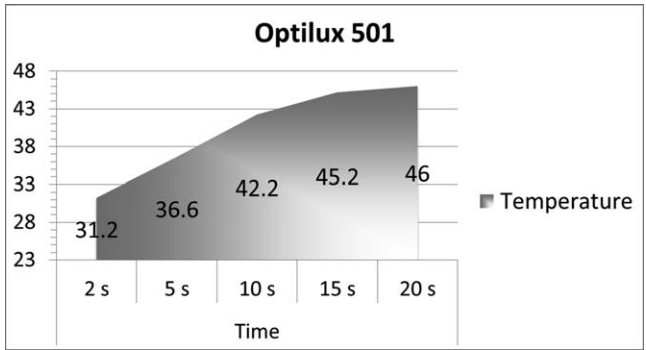


Figure 2. Temperature values in Celsius for the tip of the Optilux 501 curing unit.

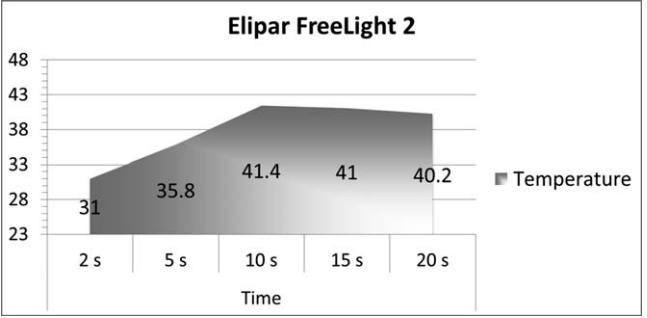


Figure 3. Temperature values in Celsius for the tip of the Elipar FreeLight 2 curing unit.

composite hardness 24 hours after light irradiation. Thus, the time period in which the hardness is measured seems to play an important role in determining the effects of temperature pretreatment for hybrid composites.

Another important factor to be considered is the residual stress that can be generated when temperature is an issue. This stress is a form of concentrated energy in the material bulk without the application of an external load.<sup>27</sup> When the composite resin restoration is submitted to occlusal load, there is a decrease in wear resistance and an increase in bonding failure.<sup>28</sup> It was already demonstrated that residual stress is greatly increased with increases in temperature.<sup>27</sup> Because increased residual stress is also associated with an increased monomer conversion, higher hardness values should be expected when the test is performed immediately after light-curing.<sup>27</sup> In the present study, after a 48-hour pre-polymerization period, this stress was probably released.

Another factor that should be observed is the temperature stabilization process during light-curing. Although the composite resin temperature was strictly standardized in this study, the insertion time of the composite resin to the ring system (40 seconds) and the exposure time (20 seconds) may have decreased the 60°C temperature and increased the 4°C temperature. Moreover, the bottom temperature of the specimens at 37°C and the temperature on the tip of the light-curing unit may have favored the temperature increase of the cooled composite resin. Thus, the real difference among the temperatures may not have had a significant impact on the composite hardness.

Regarding the comparison between the top and bottom, there were significant differences when the composite resin was light-cured by the QTH unit. The QTH unit presented higher temperature values on the tip, with the highest temperature of 46°C for all times observed up to 20 seconds. This heat may

have helped to accelerate the surface polymerization on the top, having facilitated the polymerization kinetics.<sup>26</sup> However, the bottom of the specimens that were light-cured by the QTH unit may not have received all this energy, mainly because the quick formation of cross-bonding on the superficial layer inhibits the light transmission to the inner layers of the mass.<sup>29</sup> However, the halogen light may have generated a higher loss of energy through the 2 mm of the composite resin layer to provide faster polymerization on the top.

The fact that there were no differences in hardness between the bottom and the top of the composite resin light-cured by the LED unit verified a distinction between this unit and the halogen light. The temperature on the tip of the LED unit (maximum of 4.6°C below the QTH temperature) may have resulted in slower superficial polymerization, generating less heat and presenting less monomer mobility on this surface.<sup>26</sup> The bottom light-cured by the LED may have received effective luminous energy for a longer time, possibly as a result of slower polymerization on top that did not block the correct dissipation of the light through the 2 mm of the specimen in the early moments of light-curing.<sup>29</sup> The QTH and LED units used in this study presented different irradiances, which may have affected the bottom hardness. Previous studies<sup>5,29</sup> indicated that the higher the energy density is, the better the polymerization depth will be. In this study, after 20 seconds of light curing, the QTH with 800 mW/cm<sup>2</sup> generated an energy density of 16 J/cm<sup>2</sup> for the composite resin, whereas the LED with 1200 mW/cm<sup>2</sup> generated 24 J/cm<sup>2</sup>. However, it would not be correct to attribute hardness differences only to the irradiance difference because it can be argued that the thickness of the material and the light-application period are the most important variables to be considered, whereas the material and the irradiance are the least important.<sup>30</sup> Because the thickness and the light-application period were standardized, the wavelength is another important factor. The LED wavelength is narrower than that of the halogen light, and it has an emission peak at >468 nm.<sup>31</sup> The short LED spectrum seems to be better for the absorption of the camphorquinone compared with the wide spectrum of the halogen light, which is the principle underlying the deeper polymerization of the LED compared with that of the halogen light.<sup>32-34</sup>

In this study, the polymerization shrinkage was affected by different prepolymerization temperatures only when a QTH unit was used, given that preheating the composite resin at 60°C generated

higher shrinkage than with the other temperatures. Walter and others<sup>18</sup> stated that the preheating of a microfilled composite resin increased the volumetric shrinkage. In another study, a composite resin was preheated to 54°C, the material was inserted in a class II cavity, and the light-curing was carried out immediately or with a 15-second delay. The results obtained were favorable for marginal adaptation only when the material was preheated and light-cured immediately.<sup>12</sup> Preheated composite resin becomes less viscous so its adaptation to the cavity walls is increased, although more shrinkage is generated.<sup>18</sup> In the present study, the 40 seconds used to insert the composite resin into the mold may have reduced the adaptability of the preheated composite resin.

According to the present study, only the QTH led to higher shrinkage values at 60°C. In a previous study, at a body temperature of 37°C, there was found a volumetric shrinkage of the preheated composite resin mass and a volume expansion of the pre-cooled mass.<sup>35</sup> Using a curing unit in which the temperature on the tip is higher than the body temperature, preheating could be more effective due to a minimum loss of temperature by the composite.

The LED unit did not result in significant differences in shrinkage among the tested temperatures at the top or at the bottom. The differences in shrinkage typically found for the tested temperatures in previous studies were verified when the halogen light was used<sup>18,19</sup> or when the temperature was stabilized before polymerization.<sup>25</sup> The heat generated by the halogen light may have caused a significant change in the dynamics of the monomer conversion during the light-curing process through the temperature transmission to the sample,<sup>16</sup> which did not happen to the LED polymerized specimens in such a high level. The average temperature generated on the tip of this device was 37.88°C, which is very close to the simulated body temperature of 37°C. It is possible that the temperature on the tip of the LED device may not have greatly impacted the composite resin compared with the QTH device.

Shrinkage differences were found only between the top and the bottom in the samples light-cured by the LED unit. The top that was light-cured by the LED exhibited less shrinkage than the bottom, which should be expected also for the specimens light-cured by the halogen light, because previous results<sup>14,36,37</sup> have shown, in accordance with the theory, that the composite shrinks freely from the deeper areas toward the superficial regions. It is likely that the emission temperature of the halogen light device increased the shrinkage at the top of the samples,

generating faster polymerization, which may have decreased the pre-gel phase and caused the shrinkage at the top to be similar to that at the bottom. Another important factor was the smaller shrinkage values found at the top of the specimens polymerized by the LED, which suggests a capacity for producing adequate hardness without compromising the shrinkage of the material with this device.

According to the limitations of this study, the association of pre-cooled composite resin and the use of the LED could be recommended in order to minimize shrinkage without affecting composite hardness. Particularly regarding the cooled composite, there is an apparent difficulty in inserting the material due to the increase in viscosity.<sup>18</sup> However, if the incremental technique for insertion in clinical situations were used, it is likely that the adaptation difficulty would decrease, provided that the average time of 40 seconds for insertion was respected for the composite resin to heat when in contact with the dental structure.

## CONCLUSIONS

In this study, hardness was not affected by precooling or preheating. However, polymerization shrinkage was slightly affected by different prepolymerization temperatures. The QTH-curing generated greater shrinkage than LED-curing only when the composite was pre-warmed. Different temperatures did not affect the composite hardness and shrinkage when cured by a LED unit.

## Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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