

Effect of Light Curing Modes and Light Curing Time on the Microhardness of a Hybrid Composite Resin

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Abstract

Aims: The aim of this *in vitro* study was to evaluate the influence of light curing modes and curing time on the microhardness of a hybrid composite resin.

Methods and Materials: Forty-five Z250 composite resin specimens (3M-ESPE Dental Products, St. Paul, MN, USA) were randomly divided into nine groups (n=5): three polymerization modes (*conventional* - 550 mW/cm²; *light-emitting diodes* (LED) - 360mW/cm², and *high intensity* - 1160 mW/cm²) and three light curing times (once, twice, and three times the manufacturer's recommendations). All samples were polymerized with the light tip 8 mm from the specimen. Knoop microhardness measurements were obtained on the top and bottom surfaces of the sample.

Results: Conventional and LED polymerization modes resulted in higher hardness means and were statistically different from the high intensity mode in almost all experimental conditions. Tripling manufacturers' recommended light curing times resulted in higher hardness means; this was statistically different from the other times for all polymerization modes in the bottom surface of specimens. This was also true of the top surface of specimens cured using the high intensity mode but not of conventional and LED modes using any of the chosen curing times. Top surfaces showed higher hardness than bottom surfaces.

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Conclusions: It is important to increase the light curing time and use appropriate light curing devices to polymerize resin composite in deep cavities to maximize the hardness of hybrid composite resins.

Keywords: Light-curing modes, composite resin, microhardness, LED

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Introduction

An important milestone in the history of modern restorative dentistry is the development of light-cured composite resins for direct procedures.¹ Improvements in the mechanical properties of composite resin and in the light curing devices used to polymerize them have permitted their use in posterior teeth with greater reliability than was the case some years ago.^{2,3} Polymerization of composite resin occurs through the conversion of resin monomer molecules into a polymer network accompanied by a closer packing of the molecules which causes contraction of the composite.^{4,5} When more intense light energy is used to cure a resin composite, more photons reach and activate camphorquinone (CQ) photoinitiator molecules within the resin and raise them to an excited state. The excited CQ molecules then 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.⁶

Adequate polymerization is a crucial factor in obtaining optimal physical performance to improve the clinical performance of resin composite materials.⁷ However, there are several variables affecting the amount of light energy delivered to



the top and bottom surfaces of a resin composite restoration. These include the following:^{6,8,9}

- Design and size of the light guide
- Distance of the light guide tip from the resin composite
- Power intensity
- Exposure duration
- Shade and opacity of the resin composite
- Increment thickness and material composition

If the resin composite does not receive sufficient total energy, various problems may occur with the final restoration such as the following:^{6,8,10}

- A reduction in the amount of monomer to polymer conversion
- An increased cytotoxicity of the restorative material
- A reduction in hardness of the restorative material
- An increased potential for staining
- A decreased dynamic elasticity modulus
- An increased wear of the restoration
- Increased marginal breakdown
- A weak bond between the tooth, adhesive, and the restoration

Clinically, deficient polymerization can happen in deeper Class I and Class II cavities due to the dispersion of light energy that occurs due to the distance between the tip of the light curing wand and the first resin composite increment.¹¹ In a deep Class II cavity the interface between the first increment of resin composite and the tooth structure may be under polymerized. The exposure of this interface to the oral environment can result in marginal discoloration, restoration fracture, as well as solubility of the resin composite and adhesive leading to microleakage and secondary caries.

Therefore, adequate polymerization is necessary to achieve the physical and mechanical properties of the material.¹² During the past few years widespread use of light curing techniques has given rise to the development of several types of light curing units.⁹ High intensity quartz tungsten halogen (QTH) and light-emitting diodes (LED) were recently introduced as options for polymerization. A strategy for overcoming the reduction in light intensity with distance is to use light curing units with higher light intensity.¹¹ Wang and Sang¹³ concluded a resin composite polymerized with a high intensity rate significantly increased the bottom surface hardness of resin composite. Curing with high intensity light units occurs very quickly. This is recommended because of the curing depth and favorable physical properties created when these systems are used. However, high light intensities do not allow enough flow for internal stress reduction in the restorative material, thus, contributing to high polymerization shrinkage.^{14,15}

LED units feature very narrow spectral ranges and are, therefore, highly efficient light sources.¹⁶ Operating around 470 nm, with a bandwidth of about 20 nm, blue LEDs have all the spectral purity for highly efficient curing of resin composites.¹⁷ Some studies have demonstrated good performance of these units in terms of an adequate depth of cure, flexural strength, and surface hardness.^{18,19} However, further studies are necessary in order for these light curing units to be used with safety when the light-curing tip is some distance from the filling material.

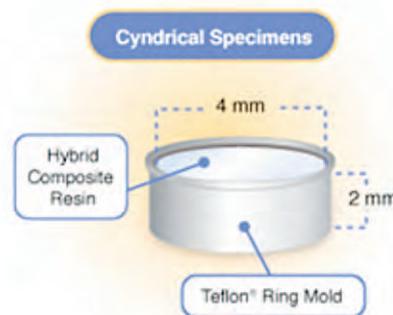
Another way of overcoming the reduction in light intensity due to distance may be to increase the light curing time. According to Sobrinho et al.⁹ the curing time recommended by manufacturers should be extended to cure the resin composite regardless of the restoration depth. However, according to Prati et al.,¹¹ the clinician should adjust the light curing time to the cavity depth and light curing unit intensity.

However, few studies have been done with the purpose of testing the depth of resin composite curing in situations where the light curing tip is distant from the filling material. It is important to evaluate the minimum light curing time required for correct polymerization for the light curing unit

used. With that in mind, the objective of this *in vitro* study was to evaluate the influence of the light curing time and the polymerization mode on the hardnesses of top and bottom resin composite surfaces in a clinical simulation when the light curing tip was a distance of 8 mm from the resin composite and the resin composite thickness was 2 mm.

Methods and Materials

Forty-five cylindrical specimens of Z250 hybrid composite resin (3M-ESPE Dental Products, St. Paul, MN, USA) were prepared in Teflon[®] ring molds. The size of the resin specimens were 4.0 mm in internal diameter and 2 mm in depth. The mold cavity was randomly filled in a single increment and polymerized according to criteria for nine experimental groups as shown in Table 1.



Five specimens ($n=5$) were assigned to each of the nine groups. Three polymerization modes were used as follows:

- **Conventional** - using an XL 3000 halogen curing light (3M-ESPE, Grafenau, Germany) at an intensity of 550 mW/cm².
- **LED** - using an Elipar Freelight(3M-ESPE, Grafenau, Germany) at an intensity of 360 mW/cm².
- **High intensity** - using an Optilux 501C high intensity halogen light (SDS Kerr/Demetron, Danbury, CT, USA) at an intensity of 1160 mW/cm².

The specimens were held between two glass slabs separated by Mylar matrix strips and compressed with a 500 g static load. Polymerization was performed with the curing light tip positioned 8 mm away from the top surface of the sample in a holding device

Table 1. Experimental groups.

Groups	Polymerization Mode	Curing Time (Seconds)	Intensity mW/cm ²	Light Curing Unit
1	Conventional	1X (20)	550	XL 3000
2	Conventional	2X (40)	550	XL 3000
3	Conventional	3X (60)	550	XL 3000
4	LED	1X (40)	360	Elipar Freelight
5	LED	2X (80)	360	Elipar Freelight
6	LED	3X (120)	360	Elipar Freelight
7	High Intensity	1X (10)	1160	Optilux 501C
8	High Intensity	2X (20)	1160	Optilux 501C
9	High Intensity	3X (30)	1160	Optilux 501C

Manufacturers:

1. XL 3000: 3M-ESPE, Grafenau, Germany
2. Elipar Freelight: 3M-ESPE, Grafenau, Germany
3. Optilux 501C: SDS Kerr/Demetron, Danbury, CT, USA

controlled by an electronic digital caliper. Three light curing times were used for each of the three polymerization modes as follows:

- 1X = the manufacturer’s recommended curing time
- 2X = twice the manufacturer’s recommended curing time
- 3X = three times the manufacturer’s recommended curing time

Each specimen was removed from its mold and stored in a lightproof container at 37°C with a relative humidity of 95% (± 5) for 24 hours. The samples were then washed and the hardness on the bottom and top of each specimen was tested using a Knoop hardness testing device (FM - Future Tech Corp., Japan) under a 25 g load for 10 seconds. Five measurements were taken at the approximate center of the specimen as was done by Price et al.⁶ The values obtained in micrometers were converted to a Knoop Hardness Number (KHN) using Excel for Windows® software (Microsoft, Inc., Redmond, WA, USA).

The results of the top and bottom surface Knoop hardness tests were submitted to subdivided parcels analysis of variance (ANOVA) (Split Plot) test (p=0.05) and a Tukey test at the 5%



significance level. The factors of light curing modes and light curing times were considered in the parcels, and the surface factor (top and bottom surfaces) was considered in the sub-factor.

Results

The microhardness test results are charted in Figure 1.

The ANOVA revealed significant differences among the light curing mode, light curing time, and surface and a triple interaction between the light curing mode, light curing time, and surface. The

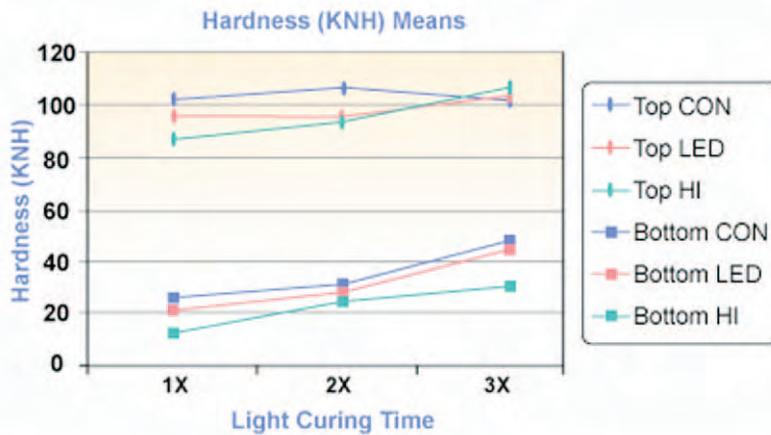


Figure 1. Microhardness test results.

Table 2. Hardness media (KHN) for the top surface.

Mean values with the same letter were not statistically different ($p < 0.05$). (Values with the same lower case letter were not statistically different for comparison among different light curing modes, and those with the same upper case letter were not statistically different for comparison among different light curing times).

Light Curing Time			
Curing Mode	1X	2X	3X
Conventional	102.31 (8.76) Aa	105.68 (4.52) Aa	101.69 (9.57) Aa
High Intensity	87.01 (3.82) Bb	92.64 (6.07) Bb	106.70 (8.75) Aa
LED	95.95 (4.40) Aa	95.63 (5.35) Ab	103.61 (9.95) Aa

1X = Manufacturer's recommended curing time.
 2X = Twice the manufacturer's recommended curing time.
 3X = Three times the manufacturer's recommended curing time.
 () = \pm Standard deviation

Tukey test was applied to individual comparisons ($p = 0.05$).

As shown in Table 2, the top surfaces showed no statistically significant differences among the light curing times for conventional and LED polymerization modes. For the high intensity mode, the 3X light curing time showed higher hardness means and were statistically different from the 1X and 2X curing times. Considering only the light curing mode factor, the conventional and LED polymerization modes showed higher hardness means and were statistically different from the high intensity mode for 1X and 2X light curing times and for 3X light curing time. There were no statistical differences among the three polymerization modes.

As shown in Table 3, the bottom surface presented higher hardness means at a 3X

curing time that were statistically different from 2X and 1X curing times for conventional and LED polymerization modes. In the high intensity group 3X and 2X curing times showed the highest means and were statistically different from the 1X curing time. The conventional polymerization mode was found to be significantly higher than the high intensity mode using 1X and 3X light curing times. The LED showed no statistical differences from any polymerization mode using 1X and 2X curing times. For all experimental conditions, the top surface showed higher hardness than the bottom surface.

Discussion

Adequate polymerization is a crucial factor in obtaining the optimal physical performance of resin composite materials.⁷ Several studies²⁰ have been performed with the intent of evaluating a method of polymerization, a light curing device,

Table 3. Hardness media (KHN) for the bottom surface.

Mean values with the same letter were not statistically different ($p < 0.05$). (Values with the same lower case letter were not statistically different for comparison among different light curing modes, and those with the same upper case letter were not statistically different for comparison among different light curing times).

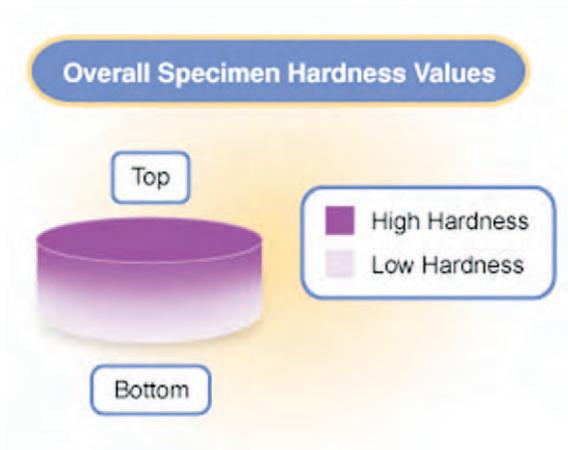
Light Curing Time			
Curing Mode	1X	2X	3X
Conventional	25.86 (1.83) Ba	31.23 (5.41) Ba	48.56 (6.26) Aa
LED	21.71 (1.88) Bab	28.46 (4.35) Ba	45.27 (1.21) Aa
High Intensity	13.57 (2.45) Bb	24.41 (1.23) Aa	30.50 (3.61) Ab

1X = Manufacturer's recommended curing time.

2X = Twice the manufacturer's recommended curing time.

3X = Three times the manufacturer's recommended curing time.

() = \pm Standard deviation



or to determine if adequate polymerization of a restorative material occurred under specific conditions.

The effectiveness of composite polymerization may be assessed by a direct or an indirect method. Direct methods, such as laser Raman and infrared spectroscopy, are not used routinely as they are complex, expensive, and time consuming.^{20,21} Indirect methods including scraping, visual inspection, and surface hardness evaluation are more commonly employed.²⁰⁻²² Incremental surface hardness has been shown to be an indicator of the degree of conversion,²⁰⁻²³ and it has correlated well with the infrared spectroscopy.^{24,25} Therefore, this method was used in this study to evaluate the influence of light curing time and polymerization mode on the hardness of top and bottom surfaces of resin composite in a clinical simulation when the light curing tip was 8 mm away from the resin composite during use.

Using the method described in the present study, the results showed the time recommended by the manufacturers of light curing devices and resin composites was insufficient for optimum polymerization, mainly on the bottom surface of standardized specimens. The resin composite on the bottom surface disperses the light of the light curing unit. As a result, when the light passes through the bulk of the composite, the light intensity is reduced due to the scattering of light by filler particles and the resin matrix.^{9,11,20,26} On the top surface, the light intensity is usually sufficient for adequate polymerization.²⁰ The results of the present study showed the top surface had higher hardness values than the bottom surface in all experimental conditions.

The bottom surface has been shown to be problematic with regard to the degree of polymerization because the thicker the resin composite increment, the worse the polymerization degree will be.^{22,27} In this study an increment of 2 mm thickness was used for it was determined to be an adequate depth based on previous studies,^{8,28} but these studies were done with the light curing tip almost in contact with the top surface of the composite resin. The present study showed an increased distance from the tip of the light-curing unit to the top surface of the composite resin was detrimental to adequate polymerization. The ratio between the bottom and top hardness for all experimental groups (Table 4) was much lower than considered ideal (0.8 or greater²⁰) even when the curing time was three times that recommended by manufacturers. The distance between the light curing tip and the resin

Table 4. Hardness ratios between bottom and top surfaces.

Curing Time	1X			2X			3X		
Curing Mode	Top	Bottom	Ratio	Top	Bottom	Ratio	Top	Bottom	Ratio
Conventional	102.31	25.86	0.2528	105.68	31.23	0.2955	101.69	48.56	0.4775
High Intensity	87.01	13.57	0.1560	92.64	28.46	0.3072	106.70	30.50	0.2858
LED	95.95	21.71	0.2263	95.63	24.41	0.2553	103.61	45.27	0.4369

Note: Curing time is expressed in terms of the multiples of the times recommended by manufacturers.

composite is a factor difficult to control clinically, due to variations in caries progression and cavity preparation depth. It is possible that placement and light curing of thin increments of resin composite can lead to improved polymerization. Increments thinner than 2 mm have been recommended by Atmadja and Bryant²² and Rueggeberg et al.²⁷ The disadvantages of thin increments are the long cure times, which are inconvenient for the patient, impractical with children, inconvenient for the dentist, and more expensive due to the increased clinic time required.²⁹

Atmadja and Bryant²² and Prati et al.¹¹ recommended increasing the light curing time when the cavity is deep. The results of the present study showed an improvement of hardness means with an increase of the light curing time, mainly on the bottom surface. Three times the manufacturers recommended curing time showed significantly higher hardness means than twice the recommended time for all light curing modes on the bottom surface of the specimens. Increasing the light curing time means increasing the total energy delivered to the resin composite increment. This increase may partly compensate for the energy loss due to dispersion of light resulting from an increase in distance between the resin composite and the tip of the light curing unit.

For the high intensity group only, tripling the curing times recommended by manufacturers showed significantly higher hardness means on the top surface than either doubling or using the manufacturers' recommended times. This supports the concept of the top surface hardness of composites being less dependent on light intensity than the bottom surfaces.²⁰

When light curing modes were compared, the conventional mode showed higher hardness

means. Statistically significant differences in the high intensity mode were found using the manufacturer's recommended curing time (1X) and three times the manufacturer's recommended curing time (3X) on the bottom surface. This was also for the top surface using 1X and twice the manufacturer's recommended curing time (2X).

The high intensity mode provides an intensity of 1160 mW/cm², along with a manufacturer's low recommended curing time of ten seconds resulting in a total energy of 11600 J/cm². The conventional mode provides an intensity of 550 mW/cm² and a recommended curing time of 20 seconds resulting in a total energy of 11000 J/cm². The total energy is nearly identical for both light-curing times used in this study. However, lower hardness means for the high intensity mode may be due to the following factors:³⁰

1. Dispersion of the light intensity because the standardized distance leveled the intensity to that of the conventional mode; so the light curing time was different for both conventional and high intensity modes.
2. The high intensity mode leads to rapid polymerization resulting in short chain lengths in the polymer reducing the modulus of elasticity modulus and decreasing the hardness of the composite resin.

The former explanation seems to be more plausible in this study when considering the large distance between the resin composite and the light-curing tip which resulted in the reduction of the intensity for the above-mentioned light curing modes.

The LED mode showed results similar to the conventional mode for almost all groups except for 2X curing times on the top surface. On

the bottom surface the LED mode did not differ statistically from the conventional mode for any curing time, and it differed from the high intensity mode in the 3X curing time group. The LED has a narrow spectral range with a peak around 470 nm which matches the optimum absorption wavelength for the activation of the CQ photoinitiator.^{28,31} While the LED mode usually presents a lower intensity than the other light curing modes, it provides a favorable degree of conversion due to the high degree of overlap within the absorption spectrum of CQ.³² In spite of the experimental distance between the tip of the curing light and the lowest intensity of all of the experimental modes of this study, it is possible the LED mode showed hardness values similar to the conventional mode because of the similar spectral range of CQ and the light curing time recommended by the manufacturer (40 seconds).

Conclusion

Within the limits of this study, the following conclusions can be made:

1. Resin composite has the capacity of reducing light penetration resulting in a decrease of the light intensity and, consequently, the polymerization effectiveness of the bottom surface of the composite specimens.
2. In deep cavity preparations it is important to increase the light curing time at least three times to improve the polymerization in the bottom surface of the first increments.
3. It is important to select a light curing unit and an adequate time for satisfactory polymerization of the hybrid resin composite especially for restorations in deep cavity preparations.

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