Curing effectiveness of resin composites at different exposure times using LED and halogen units

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Abstract

Objective: To compare the polymerization effectiveness of two resin composites cured with a quartz tungsten halogen (QTH) lamp or a light emitting diodes (LED) unit. Study design: Filtek Z250 (3M ESPE) and Spectrum TPH (Dentsply DeTrey) resin composites were placed in 9 mm deep and 4 mm wide metallic molds and cured using the QTH light Hilux 200 (Benlioglu) or the LED unit Smartlite IQ (Dentsply DeTrey) for 20 or 40 s (three specimens per group). Measurement of depth of cure was carried out by means of a scraping technique, according to ISO 4049. The microhardness measurements were performed using a calibrated Vickers indenter (100 g load, 30 s) at depths of 0.5, 1.5, 2.5, 3.5, 4.5 and 5.5 mm from the top of the composite in the same specimens. Results were analyzed by ANOVA, Student´s t and Student-Newman-Keuls tests ($p<0.05$). Results: Filtek Z250 exhibited higher depth of cure and Vickers microhardness values than Spectrum TPH under each experimental condition evaluated. Depth of cure and microhardness were not affected by the curing light used. However, hardness values were influenced by the interaction between curing light and exposure time. Specimens irradiated for 20 s exhibited higher microhardness values when the LED curing light was used. Exposure time had no influence on the microhardness values for depths from 0.5 to 2.5 mm. At higher depths, irradiation for 40 s produced greater microhardness values. Conclusions: Curing effectiveness of resin composite is not only dependent on the curing light unit. Results vary greatly with composite brand, thickness of the resin composite and the duration of the exposure.

Key words: *Light curing, resin composite, microhardness, depth of cure, light emitting diode, tungsten halogen.*

Introduction

Nowadays, light-curing dental materials are extensively used in dentistry. Four types of polymerization sources have been developed and applied: quartz tungsten halogen (QTH) lamps, light emitting diodes (LED) units, plasma-arc lamps and argon-ion lasers (1, 2).

Halogen lights and LED units are overwhelmingly applied in daily clinical practice (1). Halogen lamps, a low cost technology, have been the most frequent source employed for polymerization of resin composite materials (3) as their broad emission spectrum allows the polymerization of all known resin composite materials available (2).

However, they have several drawbacks. Their efficiency in converting electronic energy into light is estimated to be low. Up to 70% is transformed to heat and only 10% is visible light, including the blue range desired for polymerization (4). Therefore, filters are required to reduce heat energy transferred to the oral structures and provide further restriction of visible light into the narrower spectrum of photoinitiators (2). Of the visible light, due to the use of cut-off filters, a further 90% is wasted. Therefore, the final blue light output is less than 1% of the total energy input (2). Moreover, light filters degrade with time due to the high operating temperatures and proximity to the halogen bulb (5). Several studies have pointed out that many halogen units used by clinicians do not reach the minimum power output specified by the manufacturers (6). A lack of maintenance, such as omitting to check the light curing units´ irradiance or to replace the halogen bulb from time to time, is the reason for this (5). The lifespan of a conventional quartz-tungsten-halogen lamp ranges between 30-50 hours (7). These shortcomings could result in inadequate curing which could negatively affect restoration long-time success (8).

With the objective of overcoming these limitations inherent to halogen lamps, in 2001, the first light emitting diode (LED) curing units were introduced into the dental market (9). LEDs use a combination of two different doped semiconductors instead of a hot filament (2, 5). The spectral output of gallium nitride blue LED conveniently falls within the absorption spectrum of camphoroquinone (1, 5, 10). Therefore, they do not require filters to produce blue light and they convert electricity into light more efficiently (3). They produce less heat so no cooling fan is required and they can be smaller and cordless (9). Moreover, LEDs can operate for thousands of hours with a constant light output in power and spectra (10).

Contrary to first generation LED curing lights, newer units deliver with a density power higher than 400 mW/cm^2, allowing a reduction of the exposure time recommended by composite manufacturers (11).

An adequate polymerization of resin composites is essential for the ultimate success of the restorations (12). The degree of cure of resin composite materials influences their mechanical properties, solubility, dimensional stability, color change and biocompatibility (13, 14). Depth of cure and microhardness testing have been widely used to assess the relative degree of cure of resins and, thus, the efficiency of light sources (15, 16).

The aim of this study was to compare the effectiveness of polymerization of two resin composites cured with a QHT lamp or a LED curing light unit with two polymerization times. Therefore, the hypothesis of this study is that a commercially available LED light can polymerize two extended used resin composites at irradiation times of 20 and 40 s better than a conventional QTH light curing unit.

Materials and Methods

Two hybrid resin composites, Filtek Z250 (3M ESPE Dental Products, St Paul, MN, USA) and Spectrum TPH (Dentsply DeTrey, Konstanz, Germany), of A3 shade were used for this study. Both resin composites contain only camphorquinone as photoinitiator. The two light curing units evaluated were the QTH lamp Hilux 200 (Benlioglu Dental, Ankara, Turkey) and the LED light Smartlite IQ (Dentsply DeTrey, Konstanz, Germany).

Depth of Cure

Measurement of depth of cure was carried out by means of a scraping technique, according to ISO 4049 (17). Cylindrical specimens of each composite were prepared with stainless steel molds of 9 mm of length and a diameter of 4 mm. The moulds were filled with each resin composite, over a piece of transparent film. A glass slide was placed over the composites and pressure was applied to extrude the excess material. Composites were irradiated through a polyester strip (50 µm thickness) using the LED unit and the halogen light for 20 or 40 s. For each light curing unit and for each exposure time three specimens were prepared.

The halogen light-curing output was checked (600 mW/cm^2) with a curing radiometer (Optilux, KerrHawe, Bioggio, Switzerland) after every five specimens. The resin composite was extracted from the mold 30 s after illumination and the non-cured material was gently removed with a plastic spatula. The height of the cured material was measured in three different places with a digital micrometer (Mitutoyo, Kanagawa, Japan) and the mean value divided into two was registered as the depth of cure.

Hardness testing

Prior to the hardness measurement, the previous samples were longitudinally polished using a sequence of 800-1200-4000 grit silicon carbide paper and alumina polishing paste (1µm). Microhardness test was performed 24 hours after specimen preparation and during this time they were kept in darkness at 37ºC.

Microhardness (Vickers Hardness Number, VHN) was determined using a digital microhardness tester (Buelher 2101, Lake Bluff, Illinois, USA) applying a 100 g load through a Vickers indenter with a dwell time of 30 s. Five indentations were made on each specimen at depths of 0.5, 1.5, 2.5, 3.5, 4.5 and 5.5 mm from the top of the composite.

Statistical analysis

Mean values and standard deviations of depth of cure and microhardness were calculated for each group of specimens. A multifactor ANOVA was performed for the dependent variables depth of cure and microhardness, considering as independent variables the light curing unit, the resin composite used, the exposure time applied and the depth of the measure, in order to determine their influence. In addition, Student-Newman-Keuls range test and Student´s t test were used for further comparisons. Statistical significance was considered at the 95% confidence level.

Results

Depth of cure

Table 1 shows the mean values of depth of cure obtained. ANOVA showed that depth of cure was influenced by resin composite (Filtek Z250 or Spectrum TPH) (F=24.737, p<0.001), by exposure time (20 or 40 s) (F=55.414, p<0.001) and not influenced by curing lights (QTH or LED) (F=1.219, p>0.05). Interactions were significant between resin composite and time exposure (F=14.554, p<0.01).

Student t test revealed that the depth of cure of Filtek Z250 was significantly higher than the one obtained with Spectrum TPH (0.19, CL 95%: 0.03 to 0.35). Regarding exposure time, the depth of cured obtained for Filtek Z250 was independent of time of exposure (p>0.05) while Spectrum TPH exhibited a significantly higher depth of cure at 40 s (0.42, CL 95%: 0.55 to 0.30). For irradiation time of 40 s, depth of cure was similar for both materials (p>0.05).

Hardness testing

Table 2 shows the results obtained with the microhardness test as a function of resin composite, time of cure, light curing unit and depth of the measurement.

ANOVA showed that microhardness was influenced by resin composite (Filtek Z250 or Spectrum TPH) (F=801.217, p<0.001), by exposure time (20 or 40 s) (F=173.303, p<0.001), depth (from 0.5 to 5.5 mm) (F=260.478, p<0.001) and not influenced by curing lights used (QTH or LED) (F=0.765, p>0.05).

Filtek Z250 specimens exhibited a statistically higher micro-

Table 1. Mean depth of cure in mm and standard deviation (SD) of composite resins in function of light curing units used and time of exposure.

	Filtek Z250				**Spectrum TPH**			
	20 s		**40 s**		**20 s**		**40 s**	
	Smarlite	**Hilux**	**Smarlite**	**Hilux**	**Smarlite**	**Hilux**	**Smarlite**	**Hilux**
DEPTH OF CURE	2.8 (0.14)	3.0 (0.01)	3.0 (0.04)	3.1 (0.04)	2.6 (0.03)	2.6 (0.12)	3.0 (0.05)	3.0 (0.16)

Table 2. Mean microhardness (VHN) values and standard deviation (SD) of composite resins in function of light curing units used, time of exposure and different depths.

DEPTH	**Filtek Z250**				**Spectrum TPH**			
	20 s		**40 s**		**20 s**		**40 s**	
	Smarlite	**Hilux**	**Smarlite**	**Hilux**	**Smarlite**	**Hilux**	**Smarlite**	**Hilux**
0.5 mm	74.6 (9.7) A,2	72.1(10.7) A,2	84.2 (8.2) A,1	80.8 (8.6) A,1	61.1 (3.7) A,3	61.5 (4.6) A,3	59.7(2.0) A,3	59.4 (3.4) A,3
1.5 mm	75.0 (8.5) A,1	70.5 (8.2) A,2	77.4 (4.2) B,1	78.2 (4.3) A,B,1	62.1(4.2) A,3	62.3 (4.2) A,3	59.4(1.9) A,3	60.7 (5.0) A,3
2.5 mm	72.6 (4.6) A,1,2	69.8 (5.6) A,2	73.5 (3.1) C,1,2	74.9 (2.8) B,C,1	61.3(4.6) A,3	56.0 (7.4) B,4	57.5(2.5) A,B,3,4	58.5 (2.1) A,B,3,4
3.5 mm	61.6 (10.5) B,1,2	45.6 (12.2) B,2	68.1 (3.3) D,1,2	70.9 (4.2) C,1	46.1(7.4) B,3	43.3 (7.7) C,4	55.5(2.3) B,3,4	56.4 (1.8) B,3,4
4.5 mm	46.1 (6.0) C,2	45.6 (12.2) C,2	59.5(3.9) E,1	61.8 (3.9) D,1	-	20.0 (3.3) D,3	50.8(3.1) C,4	49.3 (2.8) C,4
5.5 mm	-	-	42.8(3.7) F,2	56.6 (1.8) E,1	-	-	35.7(6.6) D,3	41.0 (4.4) D,2

For each column, means with the same letter are statistically similar after Student-Newman-Keuls multiple comparisons (p>0.05). For each row, means with the same number are statistically similar after Student-Newman-Keuls test (p>0.05).

Table 3. Mean microhardness (VHN) values and standard deviation (SD) obtained with each resin composite and each exposure times at different depths, regardless the curing unit used.

DEPTH /Resin composite	Filtek Z250		Spectrum TPH	
	Exposure time			
	20 s	40 s	20 s	40 s
0.5 mm	73.4 (10.1) A,2	82.5 (8.4) A,1	61.3 (4.1) A,3	59.6 (2.8) A,3
1.5 mm	72.7 (8.5) A,2	77.8 (4.2) B,1	62.2 (4.1) A,3	60.1 (3.8) A,3
2.5 mm	71.3 (5.2) A,2	74.2 (3.0) C,1	58.6 (6.6) A,3	58.0 (2.4) A,3
3.5 mm	61.8 (8.0) B,2	69.5 (4.0) D,1	44.7 (7.5) B,3	55.9 (2.1) B,2
4.5 mm	45.8 (10.0) C,3	60.7 (4.0) E,1	20.0 (3.3) C,4	51.1 (3.0) C,2
5.5 mm	-	46.6 (7.1) F	-	

For each column, means with the same letter are statistically similar after Student-Newman-Keuls multiple comparisons (p>0.05). For each row, means with the same number are statistically similar after Student-Newman-Keuls test (p>0.05).

hardness when compared to Spectrum TPH ones and higher hardness were attributed to 40 s irradiation times. Regarding the depth of the hardness measurement, values decreased significantly with depth, except for hardness at 1.5 mm that showed values intermediate and statistically similar to those obtained at 0.5 and 2.5 mm.

A significant interaction between curing lights and exposure time was evidenced (F=6.891, p=0.009), and also between resin composite and depth (F=2.443, p=0.033), between curing lights and depth (F=4.481, p<0.001), between exposure time and depth (F=30.906, p<0.0001) and among resin composite, exposure time and depth (F=11.424, p<0.0001).

Using the halogen curing light, an exposure time of 40 s produced higher hardness than 20 s irradiation time (1.5, CL 95%: -7.5 to -1.5), while microhardness was statistically similar at 20 and 40 s when Smartlite IQ LED unit was evaluated. And for a curing time of 20 s, the hardness obtained with Smartlite IQ was significantly higher. However, when specimens were irradiated for 40 s similar VHNs were achieved with both curing units.

Similar VHNs were obtained when Hilux 200 was used at 0.5, 1.5 and 2.5 mm, decreasing significantly at 3.5, 4.5 and 5.5 mm, without differences between the last two depths. Similar results were observed with the LED unit, with significant differences also being observed between 4.5 and 5.5 mm depths. For each depth, similar hardness was determined with both curing units except at 5.5 mm, where specimens irradiated with the halogen light were significantly harder (p<0.01).

Interactions were also significant among resin composite, exposure time and depth (F=11.424, p<0.0001) (table 3). Hardness values were statistically similar for each resin composite irradiated during 20 or 40 s for depths between 0.5 and 2.5 mm, exception being Filtek Z250 when photopolymerized for 40 s. In this case, a gradual significant decrease of hardness with depth was observed. Nonetheless, this material showed a higher hardness for each experimental condition.

Discussion

In the present work curing effectiveness was measured using indirect methods such as scraping and hardness testing. Direct methods that assess the degree of conversion, like infrared spectroscopy and laser Raman spectroscopy are complex, expensive and time-consuming (18). These techniques are also more qualitative than quantitative in nature (4). In contrast, indirect methods are relatively easier to perform (19).

Depth of cure and microhardness are considered essential physical properties of composite resin materials, relevant to the clinical technique of incremental packing and curing (20). In the present study, depth of cure was evaluated according to the ISO standard scrape test (17). However, overestimation of the depth of cure and low sensitivity are drawbacks associated to this method (21). This was the reason why hardness was also assessed by a digital microhardness tester as it exhibits a good correlation with degree of conversion (21, 22). The same resin composite shade was selected (A3) in order to reduce the possible effect of colorants on photopolymerization (1, 23).

According to our results, Filtek Z250 exhibited higher depth of cure and VHNs than Spectrum TPH under each experimental condition evaluated, agreeing to previously reported (24). Hardness of a composite resin is influenced by the type and composition of the resin matrix, filler type and filler load (19, 24, 25). Differences in the organic matrix and the greater filler loading of Filtek Z250 might be responsible for these results (24).

Contrary to the hypothesis of this study, depth of cure and microhardness were not affected by the curing light used (QTH or LED). It has been reported that LED technology polymerizes resin composites as well or better than some QTH lights (5, 19, 25, 26). However, interactions between light curing source and exposure time and between light curing unit and depth significantly influenced microhardness results. Specimens irradiated for 20 s with the Smartlite IQ LED unit exhibited statistically higher hardness values than when photopolymerized under the halogen lamp Hilux 200. When specimens were irradiated for 40 s both curing units showed a similar performance. Thus, light emitted by LED lamps allows a reduction of the exposure time from that recommended by composite manufacturers for QTH curing lights, in accordance to previous authors (3, 4). Regarding the effect of light curing source and depth, both lamps achieved similar microhardness values at 0.5, 1.5 and 2.5 mm depths, as previously stated by other authors (27). As light passes through the mass of the resin composite its intensity is greatly decreased due to light absorption and scattering by restorative material attenuating its potential to cure (28). This is in consistency with the significant and gradual reduction in hardness observed for depths higher than 3.5 mm irrespectively of the curing light evaluated. Thus, the resin composites evaluated should not be cured in 3.5 mm or higher increments using Hilux 200 or Smarlite IQ. Both light curing units sufficiently polymerized composite to a depth of 2 mm which is the value acceptable for clinical application (11).

In the present study, microhardness values were significantly influenced by the interaction among resin composite, exposure time and depth. Filtek Z250 and Spectrum TPH VHNs were statistically similar for depths between 0.5 and 2.5 mm, after irradiation periods of 20 or 40 s. An exception was specimens of Filtek Z250 after photopolymerization for 40 s as their hardness decreased at each depth evaluated. No relevance is attributed to this circumstance since the mean hardness values were higher for this experimental group when compared with all the others. Both resin composites photopolymerized for 20 or 40 s exhibited a significant decrease in hardness values at 3.5 mm or higher depths.

At 0.5, 1.5 and 2.5 mm depths, an increase in irradiation time from 20 to 40 s produced a statistically significant increase in VHNs of Filtek Z250 specimens. Irradiation time has been pointed out to be a significant factor that contributes to monomer conversion at top surface (29). Although, manufacturer´s instructions for this material recommends to cure it in increments less than 2.5 mm for 20 s a better quality procedure seems to be related to longer irradiation times. Spectrum TPH specimens showed lower hardness values without differences between 20 and 40 s irradiation time. For higher depths, a relevant effect of irradiation time was detected as both resin composites exhibited higher microhardness when they were irradiated for 40 s. At 3.5 mm depth, Spectrum TPH specimens irradiated for 40 s were as hard as Filtek Z250 ones photopolymerized for 20 s and even harder at 4.5 mm depth. According to our results, the depth or thickness of the resin composite followed by the duration of the exposure are the main factors influencing microhardness in composites (29) and, therefore, in the degree of cure (18, 21, 30). The higher the degree of conversion, the better the mechanical properties, biocompatibility, water sorption, color stability and wear resistance of the resin composites (13, 14).

In conclusion, and under the conditions of this in vitro study:

1. Depth of cure and microhardness values were not influenced by the curing light used. However, microhardness results were significantly affected by interaction between curing light and exposure time and also by interaction between curing light and depth.
2. Resin composites irradiated for 20 s exhibited a higher microhardness when the LED unit was employed. Similar hardness values were achieved with both curing lights when composites were irradiated for 40 s.
3. Filtek Z250 exhibited a higher depth of cure and hardness compared to Spectrum TPH.
4. Composites should not be cured in increments higher than 2.5 mm regardless of the curing light used or the irradiation time applied.

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