

IMPACT OF THE LIGHT-CURING SOURCE AND CURING TIME ON THE DEGREE OF CONVERSION AND HARDNESS OF A COMPOSITE

ABSTRACT

Adequate physical properties of the resinous materials are related to clinical longevity of adhesive restorations. The aim of this investigation was to assess the impact of light-curing source and curing time on the degree of conversion (DC) and Knoop hardness number (KHN) of a composite resin. Circular specimens (5 x 2 mm) were carried out (n = 7) of the Filtek Z250 (3M ESPE) composite. The specimens were light-cured by quartz-halogen-tungsten (QTH) XL 3000 (3M ESPE, 450 mW/cm²) or light-emitting diode (LED) Bluephase 16i (Vivadent, 1390 mW/cm²) for 20, 40, or 60 s. After 24 h, absorption spectra of composite were obtained using Spectrum 100 Optica (Perkin Elmer) FT-IR spectrometer in order to calculate the DC and, KHN was performed in the HMV-2T (Shimadzu) microhardness tester under 50-g load for 15 s dwell time. DC and KHN data were subjected to 2-way ANOVA and Tukey's test at a pre-set alpha of 0.05. The LED showed highest DC and KHN values than QTH (p < 0.05). The increase of curing time improved the DC and KHN, all curing times with statistical difference (p < 0.05). The use of light-curing units with high irradiance and/or the time of cure increased may improve the physical properties of resin-based materials.

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INTRODUCTION

Since the arising of the light-cured composites, the polymerization quality of these materials has been studied. The surface hardness, as well as the degree of conversion (DC) are factors most commonly analyzed in order to qualify the polymerization process, which co-determines the clinical performance of resinous materials.¹⁻⁴ Thus, the higher amount of monomers converted into polymer during the cure, as well as high values of hardness of the composite are characteristics required after the polymerization reaction.

Several factors are involved in the composite polymerization quality, such as the irradiance of the curing unit, the wavelength of light emitted by this device, the distance from the light source tip, the irradiation time and; the amount, form, and size of filler particles of the material.^{3,5-7} While the quartz-tungsten-halogen (QTH) unit has been extensively used for a long time, its light intensity decreases over time by the lamp and filter degradation, resultant of the bulb overheating.⁸ Light-emitting diode (LED) technology presents less degradation over time, blue light emission without require filter, and narrow wavelength centered at 470 nm; close to maximum absorption peak of the camphorquinone (468 nm), photo-initiator used in most of light-cured materials; becoming popular in the dental practice.⁸⁻⁹

The restoration of deep cavity increases the distance between the light guide tip and resinous material surface, thus the irradiance that reaches the restorative material can be reduced, resulting in the formation of more linear polymers and/or lower monomer conversion into polymer network and, consequently inferior physical properties.¹⁰ It has been reported improvement of the physical properties with the increased curing time,^{5,11-12} which can increase the long-term durability of the adhesive restorations. The hardness of composite materials is dependent to the conversion rate and related to wear resistance.¹³

The spectroscopy is a direct method used to quantify the remaining C=C bonds of resin-based materials, the improvement of the mechanical behavior is related to highest conversion rate of monomer into polymer¹⁴⁻¹⁵ and more clinical durability. However, different carbon double bond concentrations coexist in the same polymer and the DC is not enough to characterize the 3-dimensional dental composite structure.¹⁵ Different linear polymer content, which is more susceptible to softening than a more cross-linked polymer, is found in the same DC value.¹⁶

Thus, the study of mechanical properties as Knoop hardness number (KHN), together with the DC is important to knowledge of the dental materials performance. The purpose of this study was to

evaluate the influence of the QTH and LED light-curing units, and the increased curing time. The null hypotheses were: 1) there would be no difference among the light sources, 2) as well as among the curing times on the KHN and DC values.

MATERIAL AND METHODS

In this investigation, one light-cured microhybrid methacrylate-based composite resin (Filtek Z250, shade A2, 3M ESPE, St. Paul, MN, USA) was used. Circular specimens (5 mm in diameter and 2 mm in thickness) were carried out ($n = 7$). The polytetrafluoroethylene mold was filled with the composite resin, and then covered with a Mylar strip, and a microscope slide. A 500-g load was used to compact, prevent bubble formation, and remove the excess of material.

Simulating the restorative procedure of a cavity with 8 mm of depth, the composite was light-cured at 6 mm of distance between the light guide tip and top surface of the composite (2 mm of thickness). A holder coupled to the light source was used to standardize the distance from the material surface, controlled by digital caliper (Mitutoyo Sul Americana, Suzano, SP, Brazil).

The curing times tested were 20, 40, or 60 s using the QTH XL 3000 (3M ESPE, St. Paul, MN, USA) or LED Bluephase 16i (Vivadent, Bürs, Austria) curing units with an output irradiance of 450 and 1390 mW/cm²,

respectively. The irradiances were monitored by radiometer (model 100 - Demetron/Kerr, Danbury, CT, USA). After light-curing, the specimens were removed from the matrices, dry stored in light proof containers at 37 °C for 24 h, and polished with 1200-grit silicon carbide (SiC) grinding paper (CarbiMet 2 Abrasive Discs, Buehler, Lake Bluff, IL, USA).

The DC assessment was recorded in the absorbance mode using a Fourier Transform Infrared (FT-IR) spectrometer (Spectrum 100 Optica, Perkin Elmer, MA, USA), equipped with an attenuated total reflectance (ATR) device with a horizontal ZnSe crystal (Pike Technologies, Madison, WI, USA). Absorption spectra of the cured and uncured composites were obtained on the top surface with 32 scans at 4 cm⁻¹ of resolution in the region between 1400-1800 cm⁻¹. To calculate the DC the ratio (R) between the peak heights of the C=C aliphatic (1638 cm⁻¹) and aromatic (1608 cm⁻¹) band absorptions for cured and uncured composite were used, respectively. According to the formula: DC (%) = [1 - (R polymer/R monomer)] x 100.

The KHN was measured on the top surface of each specimen using a microhardness tester (HMV-2T, Shimadzu, Tokyo, Japan) with a Knoop diamond indenter under 50-g load for 15 s dwell time. Five indentations were made, one at the center and others 4 at a distance of 100 μm from the

central location. The average of the five KHN values was calculated for each specimen.

The experimental design of this study was constituted of 2 factors (light-curing source in 2 levels: QTH and LED units and, time of cure in 3 levels: 20, 40, and 60 s). DC and KHN data were subjected to two-way Analysis of Variance (ANOVA) and Tukey's test at a pre-set of 0.05.

RESULTS

Table 1 shows the DC values. The LED Bluephase 16i unit exhibited higher conversion rate compared to QTH XL 3000 ($p < 0.001$). The increase of curing time also improved the DC, all the times of cure with statistical difference ($p < 0.001$).

The KHN was influenced by the light-curing unit ($p < 0.001$) and time of cure ($p < 0.001$), as well as by the interaction of both factors studied ($p = 0.0201$). The LED device and the increase of curing time showed highest hardness than QTH, except for KHN at 60 s of light curing ($p < 0.05$); which no difference was found ($p > 0.05$) (Table 2).

DISCUSSION

Several clinical studies have reported long-term longevity of composite resin restorations.¹⁷⁻¹⁹ This investigation assessed the influence of light sources and curing time

on the DC and KHN of a microhybrid methacrylate-based composite resin.

The first null hypothesis was rejected, since the light-curing units showed distinct behavior on the physical properties tested. In general, LED promoted higher DC and KHN compared to QTH (Tables 1 and 2). The greater irradiance of the LED unit can promote multitude of growth centers and tendency to form a branched polymer,²⁰ which are less susceptible to the softening action of food substance and to enzymatic attack,¹⁶ and higher monomer conversion rate.^{4,14} The improvement of the mechanical properties has been associated to increase of the conversion of the monomer into polymer.^{5,11-12} Thus, the greater monomer conversion probably resulted in the higher superficial hardness of the composite resin.

However, during the restorative procedure is common that the light guide tip is far of the first increment of composite, reducing the irradiance, which could affect the polymerization effectiveness.¹⁰ Only 1 mm of air interposed between the tip of the light-curing device and the restorative material decreases the irradiance in approximately 10 %.²¹

Previous studies have related the improvement of the physical properties of resin-based materials due to the extended curing time.^{5,11-12} These findings are in agreement with the results of the present

study, which presented better DC and KHN values with the increase of curing time, and therefore the second null hypothesis also was rejected.

The increased curing time promotes more light energy available to increase of monomer conversion into polymer.^{5,12} Once, at

60 s of curing time similar KHN was found for both light sources. This improvement on the DC suggests higher mechanical properties and long-term clinical durability.

Table 1. Degree of conversion [DC (%)] of the composite resin according to light-curing unit and curing time.

Light-curing unit	Curing time			
	20 s	40 s	60 s	
Bluephase 16i	52.82 (4.14)	65.99 (3.60)	72.86 (4.89)	a
XL 3000	31.51 (9.15)	43.62 (4.94)	55.88 (4.45)	b
	C	B	A	

Distinct letters (lower and capital comparing light-curing units and curing times, respectively) are statistically different ($p < 0.05$).

Table 2. Knoop microhardness number [KHN (Kgf/mm²)] of the composite resin according to light-curing unit and curing time.

Light-curing unit	Curing time		
	20 s	40 s	60 s
Bluephase 16i	38.23 (1.51) aC	50.40 (5.23) aB	60.31 (4.65) aA
XL 3000	29.55 (5.07) bC	37.86 (4.90) bB	57.50 (3.80) aA

Distinct letters (lower in the column and capital in the row) are statistically different ($p < 0.05$).

A previous retrospective longitudinal study after 22 years of posterior composite restorations suggested that the physical properties of the material might have some impact on the restoration longevity.¹⁷ Thereby, little difference on the physical properties can exhibit similar clinical performance in short-term, but not over extended period of time.

CONCLUSION

The use of light-curing units with high irradiance and/or the time of cure increased

may result in better physical properties of resin-based materials, improving the clinical longevity of adhesive restorations.

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