# EFFECT OF DIFFERENT LIGHT SOURCES ON DEGREE OF CONVERSION, HARDNESS AND PLASTICIZATION OF A COMPOSITE


## ABSTRACT

Clinical performance of composite resins depends largely on their mechanical properties, and those are influenced by several factors, such as the light-curing mode. The purpose of this study was to evaluate the influence of different light sources on degree of conversion (DC), Knoop hardness (KHN) and plasticization (P) of a composite resin. Disc-shaped specimens (5 x 2 mm) of Esthet-X (Dentsply) methacrylate-based microhybrid composite were light-cured using quartz-tungsten-halogen (QTH) Optilight Plus (Gnatus) or light-emitting diode (LED) Ultraled (Dabi Atlante) curing units at 400 and 340 $mW/cm^2$ of irradiance, respectively. After 24 h, absorption spectra of composite were obtained using Nexus 670 (Nicolet) FT-IR spectrometer in order to calculate the DC. The KHN was measured in the HMV-2000 (Shimadzu) microhardness tester under 50 g loads for 15 s, and P was evaluated by percentage reduction of hardness after 24 h of alcohol storage. Data were subjected to t-Student test (alpha=0.05). QTH device showed lower P and higher KHN than LED ($p<0.05$), and no difference between the light-curing units was found for DC ($p>0.05$). The halogen-curing unit with higher irradiance promoted higher KHN and lower softening in alcohol than LED.



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## KEYWORDS

Composite resins. Polymers. Hardness. Dental materials.



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## INTRODUCTION

Light-activated composite resins are widely used in dental practice to perform direct restorations, due to the increase in the aesthetics demand by patients, simplification of bonding procedures and improvement of these materials.[1] Degree of Conversion (DC), that is the number of double carbon links which are broken and transformed in single bonds transforming monomers in polymers, is related to physical properties of composite resin restorations.[2] This parameter is influenced by many factors, such as chemical structure of monomers, filler composition, curing time, light intensity and others.[3]

Quartz-Tungsten-Halogen (QTH) lamp has been extensively used as dental light-curing unit for a long period. However, the bulb overheating causes lamp and filter degradation, decreasing the light intensity over time.[4] Light-emitting diode (LED) technology has become popular in dental practice due to its benefits, such as narrow wavelength centered at 470 nm [close to the maximum absorption peak of the camphorquinone (468 nm)], less degradation over time and blue light emission without require filter.[4-5]

An adequate and uniform cure of resin-based restorative materials can improve mechanical and physics properties of restoration.[6] Spectroscopy is a direct method to measure the conversion rate of the monomer into polymers.[6-9] However, the DC by itself is not enough to characterize the three-dimensional dental composite structure, and different residual carbon double bonds coexist in the same polymer,[10] resulting in polymers more linear or crosslinked.[11]

The Knoop microhardness test (KHN) has been used to predict the wear resistance related the conversion of monomer into polymer network.[12] The plasticization (P) techniques consists in an indirect method to measure the crosslink density in polymers structure[13], recovering the hardness of material after 24h storage at 100% ethanol.

Thus, the aim of this study was to determine the effect of light cure unit in mechanical properties, such as the KHN and P, in addition with the DC to characterize the performance of dental materials. The null hypothesis tested was that QTH and LED light sources produce no influence on the KHN, P and DC of composite resin.

## MATERIAL AND METHODS

In this study, one light-cured microhybrid methacrylate-based composite resin (Esthet-X, shade A2, Dentsply, Milford, DE, USA) was used. Disc-shaped specimens (6mm in diameter and 2mm in thickness) were made (n=10). The polytetrafluoroethylene mold was filled with composite; then covered with a mylar strip (KDent, Joinville, SC, Brazil), and a microscope slide (Biosan, Belo Horizonte, MG, Brazil). A 500 g load was used to compress the material, preventing bubble formation, and the excess of material was removed. The composite resin was light-cured for 20 s, according to manufacturer's recommendation, using the QTH Optilight Plus (Gnatus, Ribeirão Preto, SP, Brazil) or LED Ultraled (Dabi Atlante, Ribeirão Preto, SP, Brazil) curing units with an output irradiance of 400 and 340 mW/cm$^2$, respectively.

Irradiances were monitored by specific radiometer to QTH light sources (model 100 - Demetron/Kerr, Danbury, CT, USA) and LED (SDI, Bayswater, Victoria, Australia). Specimens were dry stored in dark 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 absorption spectrum was obtained using Fourier transformed infrared spectroscopy (FT-IR) spectrometer (Nexus 670, Nicolet Instrument Corp. Madison, WI, USA), with 64 scans at a resolution of 4 cm$^{-1}$, within a wavelength of 4800-4500 cm$^{-1}$. Direct method of determining the conversion rate using the Near Infrared (NIR) region is based on the measurement of reduction in the absorption band intensity at 4743 cm$^{-1}$, assigned to the vibration of aliphatic double-bond (C=C), which after light curing is converted into single-bond during the monomer conversion into polymer.[7-8] This spectroscopic procedure depends on the absorption band presence that is not modified with polymerization, and serves as a normalization standard of monomer and polymer spectra.[7] For example, composites that have aromatic monomers, with band of absorption in 4623 cm$^{-1}$ can serve as internal standards of normalization.[8] This eliminates the necessity to consider the thickness of the specimen.[7] To calculate the DC, the ratio (R) between the peak heights of C=C aliphatic (4743 cm$^{-1}$) and aromatic (4623 cm$^{-1}$) for cured and uncured composite was used, according to the formula: DC (%) = [1 – (R polymer/R monomer)] x 100.[14]

Initial microhardness (KHNi) reading was measured on the top surface of each specimen by program C.A.M.S. (New Age Industries, USA) using a hardness tester (HMV-2000, Shimadzu Corporation, Kyoto, Japan). Specimens were individually fixed in a clamping apparatus and positioned in such a way held perpendicular to the Knoop diamond indenter, under 50 g static load with dwell time of 15 s.[1,14] Three indentations were made in each surface of specimen, one at the center and others 2 at 100 μm of distance to right and left from the central location. The average of 3 KHN (Knoop hardness number, Kg/mm$^2$) values was calculated for each specimen.

Plasticization (P) analysis was evaluated by percentage reduction of microhardness (%KMHred) after alcohol storage. After KMHi assessments, all specimens were immersed in 75% ethanol-water solution at 37ºC for 24h.[15] Following this period, a second microhardness measurement (KMHf) was made as previously described. The same operator carried out the KHN test, before and after alcohol storage. The results were tabulated, and P was calculated using the following equation: %KMHred = 100 – [(KMHf X 100) / KMHi].[13,14]

The homogeneity of variance and normality of collected data of DC, KHN, and P were tested using Levene and Shapiro-Wilk tests, respectively. After that, data were submitted to t-Student test using Stat View Software (SAS Institute Inc., Cary, NC, USA) at a pre-set alpha of 0.05.

## RESULTS

The DC, KHN, and P values are presented in Table 1. T-Student test showed significant difference for KHN and P ($p < 0.05$). QTH unit resulted higher KHN and lower P than LED device ($p < 0.001$ and $p = 0.01$, respectively).The DC was not influenced by light sources ($p = 0.10$).

Table 1: Means (Standard deviation) of Degree of Conversion (DC), Knoop Hardness Number (KHN) and Plasticization (P).

| | DC | KHN | P |
|---|---|---|---|
| QTH | 60.7 (0.6) a | 56.6 (4.9) a | 31.8 (8.9) a |
| LED | 60.1 (1.0) a | 47.2 (5.7) b | 41.0 (6.0) b |

QTH - quartz-tungsten-halogen; LED - light-emitting diode. Distinct letters in the same column are statistically different means ($p < 0.05$).

## DISCUSSION

Several clinical studies have reported an adequate durability of composite resin restorations even after an extended long term evaluation.[16-18], This clinical performance may be influenced by many factors, such as light-curing method.[3] This study compared the polymerization effectiveness of halogen and LED light-curing units on the DC, KHN, and P of a microhybrid methacrylate-based composite resin, and the null hypothesis was partially rejected. The composite resin KHN and P was influenced by light sources tested. Fourier transformed spectroscopy has been used to measure the DC, indicating the amount of carbon double bonds that remain after light polymerization.[1,6-10,14] Similar conversion rate was obtained for both curing units (Table 1). The similarity of irradiance recorded for both light curing units (400 $mW/cm^2$ for QTH, and 340 $mW/cm^2$ LED) may explain this result.

A previous investigation was performed to evaluate the influence of light energy density on cure rate of composite by hardness test.[19] The authors observed similar KHN for the top surface at lower intensities as 200 or 300 $mW/cm^2$; however, adequate bottom surface cure needs a higher light irradiance or increasing of curing time. Furthermore, a minimum irradiance of 400 mW/cm$^2$, during 60 s associated with increments no larger than 2 mm, has been recommended for effective polymerization.[20]

The resinous materials softening in aqueous environmental, by swelling of polymer network and reduction of frictional forces between polymer chains, results in lower hardness.[10] Moreover, the insufficient cross-linked polymer is more susceptible to plasticization effect by chemical substances that enter during eating and drinking.[21]The hardness analysis after alcohol storage has been used to evaluate indirectly the polymer cross-linked structure.[11,13-15] Despite of similar DC values for both light-curing units evaluated, QTH unit with higher irradiance showed higher hardness and lower plasticization than LED (Table 1).

After light-curing, the polymer chains exhibit residual carbon double bond concentrations and modification on linear polymers content, which are more susceptible to softening than cross-linked polymer.[11] Thus, the monomer conversion rate does not necessarily provide complete representation of polymer structure quality. Probably, the higher irradiance of QTH unit promoted more growth centers, increasing the tendency to form a branched polymer, with superior cross-link density.[11]

Longitudinal study showed that mechanical properties of material may have some impact on restoration longevity.[16] Therefore, small differences in the physics properties could result in identical clinical performance after short-term, but not over extended period of time.

The recommendations of manufactures about the curing of composite resins may have to be made regarding to the total dose of energy (irradiance x time of exposure), instead of only to the irradiance.

## CONCLUSION

Within the limitations of this in vitro study, the following conclusions were drawn: the Quartz-Tungsten-Halogen (QTH) and Light-Emitting Diode (LED) curing units showed different behavior on properties tested: (1) Degree of Conversion was similar between the light-curing devices; (2) QTH exhibited higher Knoop Hardness value, and (3) lower Plasticization values than LED unit.

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