ORIGINAL RESEARCH

Effect of LEDs with Different Wavelengths on the Microhardness and Nanohardness of Nanohybrid Composite Resins

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Abstract

Aim: The aim of this study is to compare the effectiveness of polymerization of nanohybrid composite resins with different colors and thicknesses, photocured by units of different wavelengths through Knoop microhardness (KHN) and Berkovich nanohardness (DUH).

Materials and methods: One hundred twenty specimens of Tetric N-Ceram (Ivoclar/Vivadent) were divided into groups ($n = 5$) according to the experimental test, the color of the composite resin (A2 and Bleach-M/BM), the light source: monowave (Elipar™ FreeLight DeepCure-3M/ESPE), dental products—1200 mW/cm^2/15 seconds (FL); or polywave (Bluephase-Ivoclar/Vivadent 1200 mW/cm^2/15 and 30 seconds (BP), and thickness (irradiated surface,1, 2, and 3 mm). The specimens were stored dry for 24 hours at 37°C and received five indentations on the top and button surfaces. Analysis of variance (ANOVA), Tukey, and Pearson correlation ($p < 0.01\%$) tests were used.

Results: A direct correlation was found between KHN and DUH. The higher values of KHN and DUH were observed with Elipar™ FreeLight DeepCure unit in irradiated surface and 1-mm-thick specimens at A2 color.

Conclusion: That Elipar™ FreeLight DeepCure unit showed better effectiveness in curing nanohybrid composite resins, used in this work, in different colors and depths as compared to Bluephase in both experimental tests, and that DUH can substitute KHN test when comparing the effectiveness of polymerization.

Clinical significance: The evaluation of the mechanical properties of composite resins is essential to verify their possible clinical performance.

Keywords: Composite resin, Microhardness, Nanohardness, Photocuring.

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Introduction

Composite resins are materials widely used in restorative dentistry and have evolved significantly since they were first introduced in the early 1960s.[1] Many research studies have been carried out in an attempt to improve clinical behaviors, mainly related to factors, such as polymerization. Although several aspects can influence the clinical performance of composite resins, their composition and cure rate are the most important in terms of improving the mechanical properties of these materials.[2,3]

Complete polymerization is essential to obtain the ideal physical properties of restorative materials.[4] An insufficient degree of cure is responsible for the excessive absorption of water, decreased wear resistance, and residual presence of uncured monomer with toxic effects.[5] In addition, they predispose the highest occurrence of gaps between the tooth and the composite, leading to complications, such as microleakage, postoperative sensitivity, recurrent caries, decreased mechanical properties, and dental fracture.[6] The photoinitiators present in the composites respond preferentially to light at specific wavelengths,[7] where a nonideal wavelength can result in incomplete or inconsistent polymerization. As a result, areas of reduced polymerization may appear, which may be negatively associated with the clinical longevity of the restorations.[8]

Camphorquinone (CQ) is the most widely used photoinitiator and has a sensitivity peak close to 470 nm in the blue range of the visible light spectrum. Due to the intense yellow color of the CQ, alternative lighter-colored initiators that disappear completely after photopolymerization were introduced in the market.[9] These include phenylpropanedione (PPD), acylphosphine oxide (APO), Lucirin, and Ivocerin. While the PPD absorption spectrum extends from the ultraviolet (UV) wavelength to approximately 490 nm, APO and Lucirin-trimethylbenzoyldiphenyl phosphine oxide (TPO) mainly absorb light in the UV range, with sensitivity peaks of approximately 370 nm and 420 nm, respectively.[10]

The presence of these photoinitiators can reduce the efficiency of photoactivation when a single-peak light-emitting diode (LED) (450–470 nm) is used. For this reason, photopolymerizing equipment capable of emitting blue and violet lights within the specific wavelength bands was introduced, known as "polywave," "multiple peak" or "multiple wave" lights.[11,12] Although monowave

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and polywave LEDs can produce similar power and provide the same power density (mW/cm^2) for the restoration, differences in their spectral outputs can have a significant effect on the photoinitiator system.[13] These devices are called third generation and are considered hybrid devices. They associate blue and violet LEDs and ensure the activation of photoinitiators, such as PPD, APO, Lucerin-TPO, and Ivocerin, present in some composites used currently.[14,15]

The evaluation of the mechanical properties of restorative materials is essential to verify their properties and possible clinical performance. In this context, microhardness analysis is an indirect evaluator used to verify the degree of polymerization of composite resins. However, the nanohardness technique allows investigations under various loading regimens, based on load displacement data with subchronic scale teeth.[16] These characteristics suggest that the nanohardness test has an advantage over microhardness due to a high-strength resolution and precise positioning.[17]

For teeth which underwent bleaching treatment, it is necessary to use extremely light-colored resins to combine with the color of the teeth. The color of the composites is a highly relevant factor for cosmetic dentistry, where it is necessary to change the amount or type of photoinitiator in its composition. The light source must also act on all photoinitiators present in the composite, regardless of whether they are light or dark in color.[14] In addition, the depth of cure in deep restorations is a discussion in the literature. The photocuring quality is closely related to the potential of the light-curing equipment and the characteristics of the composite.[18]

The objective of this study was to compare the microhardness and nanohardness of Tetric N-Ceram (Ivoclar-Vivadent/Germany) nanohybrid composite resin of different thicknesses (1, 2, and 3 mm) in A2 and bleach colors, using light-curing units (LCUs) with different wavelengths: LED blue (monowave) and LED blue/violet (polywave).

Materials and Methods

This *in vitro* study was carried out in the Dental Materials Laboratory of the Faculty of Dentistry, University of São Paulo (USP, Brazil). One hundred twenty specimens were made with the nanohybrid composite resin (Tetric N-Ceram/Ivoclar-Vivadent), in colors A2 and Bleach-M. Each composite resin color was distributed into two main groups of LEDs: one that used Elipar™ FreeLight 2 (3M/ESPE), LED Azul, which has a wavelength of 430 to 480 nm and another that used Bluephase (Ivoclar/Vivadent), LED Azul/violet, which has a wavelength of 380 to 515 nm. Three thicknesses (1, 2, and 3 mm) were analyzed compared with the irradiated surface (0 mm). The specimens were divided into groups ($n = 5$): Knoop—KHN microhardness, DUH—ultra-microhardness and ME elasticity module, and color (A2 and Bleach-M [BM], LED blue photoactivator (Elipar FreeLight 2/3M-15 seconds) [FL]), and LED blue/violet (Bluephase/Ivoclar/Vivadent 15 seconds [B15] and 30 seconds [B30]).

Split black polypropylene matrices (5 mm internal diameter) surrounded by fixed aluminum rings were used in the tests. The specimens with different thicknesses (1, 2, and 3 mm) were cleaned and supported by a glass slab. The glass slab was placed on a black cardboard to avoid light reflection during photoactivation.

The matrix was positioned over a polyester strip fixed on the glass slab. The nanohybrid composite resin (Tetric N-Ceram, Ivoclar/Vivadent, Schaan, Liechtenstein, Germany) (Bleach-M: BM, L65229 or A2, L58656) was introduced into the matrix in a single amount using an nº 12 Thompson spatula (Cosmedent, Chicago, USA). A second polyester strip was then placed on the resin, and a glass slide was used to cover it. To ensure a level plane on the top and bottom surfaces, a load of 3.5 kg was applied to the glass slide for 10 seconds using standard equipment (handmade by Dental Materials Laboratory, USP, SP, Brazil).

Light curing was performed by touching the LCU to the polyester strip after the glass was removed. The tip of the LCU monowave Elipar FreeLight DeepCure/3M-ESPE (St. Paul, MN, EUA) 1200 mW/cm^2/430–480 nm for 15 seconds (FL) or polywave Bluephase/Ivoclar-Vivadent (Schaan, Liechtenstein, Germany) 1200 mW/cm^2/380–515 nm for 15 (B15) or 30 seconds (B30) occupied the entire area of the composite resin, ensuring that light output was generated along the entire length of the material. The power output of the light sources was measured using a radiometer to verify whether the sources were working properly (SDI Limited, Bayswater, Australia).

After the polymerization step, the specimens were marked with a pen on their top surfaces to differentiate it from the bottom surfaces. The specimens were then removed from their matrices and stored dry at 37°C in dark conditions for 24 hours. Next, the specimens were finished/polished in a Politriz EcoMet/AutoMet 2000 (Buehler, Illinois, USA), using a sequence of 400, 600, 1200, 2400, and 4000 grit silicon carbide paper (Buehler, Illinois, USA) under continuous water cooling. Felt cloths (Buehler, Illinois, USA) were also used along with Buehler polishing slurry (diamond polishing compound) of 6 and 3 μm without refrigeration. The specimens were marked slightly on their central axes using a scalpel blade to discriminate between the microindentation and the nanoindentation sides. The specimens were then ultrasonically cleaned in distilled water for 8 min (digital ultrasonic cleaner, Kondortech, São Carlos, SP, Brazil). After being washed, the samples were dried on an absorbent paper, while taking care not to touch the surface before the microhardness and nanohardness tests.

Microindentation measurements were performed on the bottom surfaces at thicknesses of 1, 2, and 3 mm. The control group corresponded to the top surface of 1-mm samples, where 25 g of load was applied for 40 seconds at five points using a HMV-2 microhardness tester (Shimadzu, Tokyo, Japan) controlled by CAMS-WIN software for Windows. The location of the indentations used to obtain the readings was determined at 100 μm from the central marking with 100 μm spacing between indentations along a straight line.

The nanoindentation measurements were performed at 23°C using a DUH-211S nanoindenter (Shimadzu, Tokyo, Japan) equipped with a Berkovich three-sided pyramidal diamond tip. Five indentations for each loading/unloading rate were performed on selected areas of each specimen. The indentations were located 100 μm from the central mark with 20 μm spacing. The maximum load applied by the nanoindenter was 10 mN with no hold time, in accordance with ISO 14577.

A one-way ANOVA was used to analyze the data at a significance level of p-value ≤ 0.01. The average of the five measurements from the Knoop and Berkovich indentations for each sample was used. Tukey test was applied when significant differences were detected by ANOVA. Pearson's correlation was performed to analyze the relationship between microhardness and nanohardness averages.

Results

Microhardness

ANOVA demonstrated a statistically significant difference at the level of 1% between thicknesses ($F = 44.43$), between sources

($F = 125.45$), between resins ($F = 151.71$), and in the resin interaction versus source versus thickness ($F = 5.08$). For the source factor, the Tukey value ($T = 3.66$) indicated a statistically significant difference between the sources FL (monowave Elipar FreeLight DeepCure), B15 (polywave Bluephase 15 seconds), and B30 (polywave Bluephase 30 seconds). The FL source promoted higher hardness values than the B30 source, which exhibited higher values than the B15 source ($F >$ B30 $>$ B15).

The irradiated surface BM (Bleach-M) color resin did not show significant differences between the light sources tested ($p = 0.952$). However, 1-mm-thick resin base showed a significant difference between FL and B15 ($p = 0.021$) and B30 ($p = 0.011$). The 2-mm-thick BM resin had a higher hardness value with the FL unit, which was statistically higher than the B15 ($p = 0.019$) and B30 ($p = 0.026$) sources. The 3-mm-thick BM resin showed a statistically significant difference between FL ($p = 0.001$) and B15 ($p = 0.0385$) sources. No significant difference was detected between the B30 source and the FL source ($p = 0.834$).

For resin A2 with the FL source, a significant difference in hardness was observed between the irradiated surface, 1, 2, and 3 mm thick ($p < 0.05$). For light sources B15 and B30, no significant differences in microhardness were found between the different thicknesses ($p = 0.014$). However, for B30, the thickness of 3 mm showed significantly less hardness when compared to others ($p < 0.05$). The FL light source showed significantly greater hardness when compared to B15 and B30 ($p < 0.05$) for the irradiated surfaces, 2 and 3 mm (Table 1).

Nanohardness

The ANOVA test showed significant differences at a level of 1% in nanohardness for the various light sources ($F = 48.66$), thicknesses ($F = 17.29$), and resins ($F = 25.48$). Additionally, no interaction was observed with respect to resin type versus source versus thickness ($F = 6.28$). To compare the mean values, Tukey statistics were calculated. According to the Tukey test ($T = 5.83$), a significant difference was observed between the three light sources. The FL source had a higher hardness value than the B30 source, which was greater than the B15 source ($F >$ B30 $>$ B15).

For the thickness factor, the Tukey test demonstrated that no significant difference existed between the 1- and 2-mm resins or between the 2- and 3-mm resins. The irradiated surface exhibited nanohardness values that were significantly different from those of other resin thicknesses.

The irradiated surface and 1-mm BM resin thicknesses were not statistically different, concerning the three light sources ($p < 0.05$). No differences were detected for the FL and B30 light sources for any of the resin thicknesses ($p < 0.05$). In the case of B15 source, the 2-mm-thick and 3-mm-thick resins decreased in terms of hardness compared to the irradiated surface.

The nanohardness values for the A2 resin were not significantly different among the 1-, 2-, and 3-mm-thick resins ($p > 0.05$) (Table 2).

Correlation between Microhardness and Nanohardness

For both types of composite, a direct correlation was found between the microhardness and the nanohardness. For the BM resin, the r was 0.7435 and the p was less than 0.0001. For the A2 resin, the r was 0.7410 and the p was less than 0.0001.

The graphs in Figures 1 and 2 illustrate the dispersion values for the correlation between the microhardness and nanohardness for the Bleach-M and A2 resins.

Discussion

The indentation used in the tests simulated the condition found in the oral cavity during the chewing process in which the restorative material is penetrated by the surface of the antagonistic tooth, which can cause greater or lesser deformation depending on the applied force and the composition/resistance of this material.[19] In this study, the composite resin Tetric N-Ceram (Ivoclar-Vivadent) was used, which contains 80% of load and can be used in the anterior and posterior regions of the oral cavity. In addition, this

Table 1: Mean and standard deviations of the resin versus source versus thickness interaction—Knoop microhardness

	Bleach-M			*A2*		
	FreeLight 25s	*Bluephase 15s*	*Bluephase 30s*	*FreeLight 25s*	*Bluephase 15s*	*Bluephase 30s*
0 mm	60.92 ± 7.82^{Aa}	52.68 ± 4.99^{Aa}	61.48 ± 4.73^{Aa}	77.00 ± 3.62^{Aa}	55.66 ± 2.75^{Ab}	59.08 ± 2.04^{Ab}
1 mm	65.94 ± 5.21^{Aa}	44.12 ± 6.35^{Ab}	48.88 ± 3.99^{ABb}	69.46 ± 2.44^{ABa}	59.76 ± 2.27^{Aa}	57.96 ± 5.64^{Aa}
2 mm	64.34 ± 2.31^{Aa}	26.88 ± 6.87^{Bc}	43.64 ± 4.01^{Bb}	72.64 ± 1.26^{ABa}	55.16 ± 3.50^{Ab}	59.16 ± 2.70^{Ab}
3 mm	47.06 ± 8.15^{Aa}	25.28 ± 5.02^{Bb}	39.4 ± 3.69^{Ba}	63.68 ± 6.51^{Ba}	49.60 ± 2.62^{Ab}	40.02 ± 12.88^{Bb}

Different capital letters indicate a statistically significant difference in the vertical ($p < 0.05$); Different lower case letters indicate a statistically significant difference in the horizontal ($p < 0.05$)

Table 2: Mean and standard deviations of the resin versus source versus thickness interaction—nanohardness

	Bleach-M			*A2*		
	FreeLight 25s	*Bluephase 15s*	*Bluephase 30s*	*FreeLight 25s*	*Bluephase 15s*	*Bluephase 30s*
0 mm	69.72 ± 10.61^{Aa}	66.12 ± 23.44^{Aa}	67.90 ± 3.19^{Aa}	80.56 ± 12.90^{Aa}	58.42 ± 5.70^{Aa}	65.29 ± 8.27^{Aa}
1 mm	66.86 ± 4.98^{Aa}	61.45 ± 10.90^{Aa}	52.50 ± 9.10^{Aa}	68.98 ± 9.53^{Aa}	60.52 ± 1.30^{Aa}	63.81 ± 5.66^{Aa}
2 mm	63.17 ± 4.77^{Aa}	33.71 ± 5.35^{BCb}	59.28 ± 6.61^{Aa}	70.96 ± 5.58^{Aa}	50.39 ± 2.89^{Aa}	61.51 ± 5.16^{Aa}
3 mm	54.79 ± 14.39^{Aa}	20.21 ± 1.60^{Cb}	68.26 ± 5.06^{Aa}	69.32 ± 8.21^{Aa}	53.61 ± 5.46^{Aa}	54.02 ± 6.06^{Aa}

Different capital letters indicate a statistically significant difference in the vertical ($p < 0.05$); Different lower case letters indicate a statistically significant difference in the horizontal ($p < 0.05$).

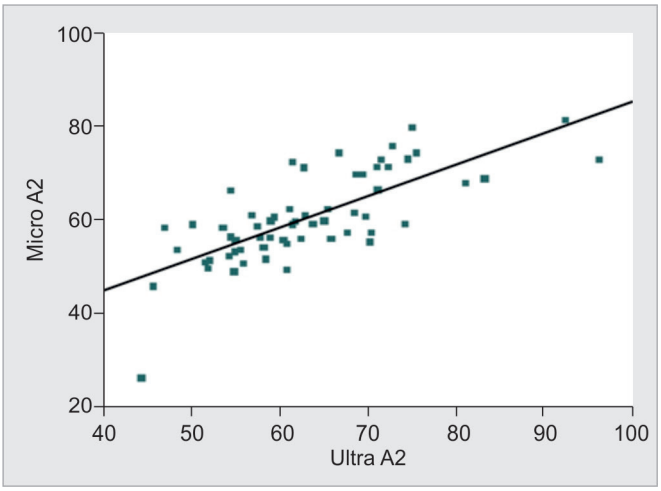

Fig. 1: Dispersion between the microhardness and the nanohardness for the A2 resin

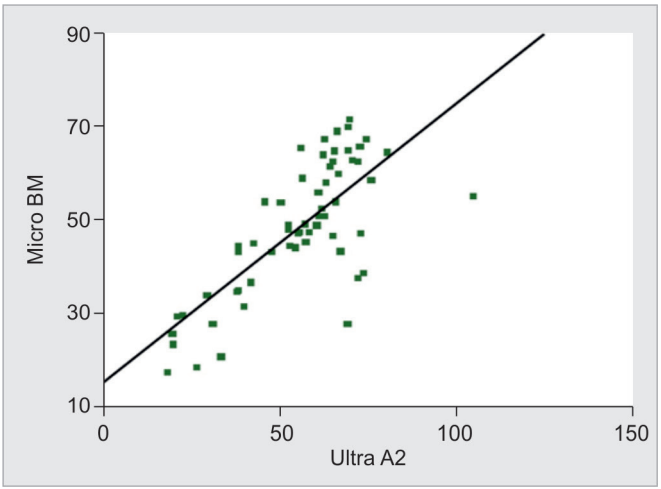

Fig. 2: Dispersion between the microhardness and nanohardness for the Bleach-M resin

composite contains the photoinitiator Lucerin-TPO, which gives the composites a lighter color.

Light-colored composite resins need to decrease the amount of CQ and choose other photoinitiators, such as Lucerin-TPO. A disadvantage of TPO and other alternative initiators is the shifted light absorption spectra toward the UV range which are mismatched with the emission spectra of monowave LED LCUs.[14,20,21] Thus, it is necessary to use polywave-curing units, which act on all photoinitiators present in the composite. However, in the present research, the monowave FL-curing unit showed the highest microhardness results for thicknesses of 2 and 3 mm in both resin colors studied. That is, in general, the FL-curing unit showed the best results when compared to Bluephase, regardless of the curing time. This can be explained by the chemical composition of the investigated composite resin since in addition to Lucerin-TPO it contains CQ in its composition. The CQ in conjunction with a tertiary amine, traditionally used in both dental adhesives and composite resins, is properly photopolymerized by monowave-curing units.[22] In addition, the manufacturer does not specify in detail the amount of Lucerin-TPO and CQ in the composite.

A study conducted by Santini et al.[14] showed that the use of polywave LEDs significantly improves the microhardness of materials containing TPO as a photoinitiator. Another study by Derchi et al.[23] showed that polywave LEDs work better than monowave, but not in all investigated composites. Although most studies show a better potential for polywave LEDs when compared to monowave for composites with different photoinitiators,[14,23–25] the proper cure of composite resins depends on several factors, such as LED tip diameter and cure distance.[26] In polywave LEDs, photons of violet light are delivered to all possible types of photoinitiators present in the composites. However, the blue emission is reduced compared to that of a monowave LED. This causes less activation of CQ at greater depths, which could justify the microhardness results of the present study.[26]

The nanohardness results showed that there was no significant difference between the FL and B30 curing units for any of the thicknesses and colors of composite resin. The microhardness was correlated to the degree of conversion previously, i.e., an increase in hardness corresponds to an increase in the degree of conversion during the setting of the composite resin. However, an absolute hardness value cannot be used to predict the degree of conversion when comparing different resins.[27] In the present study, a single composite resin was studied. Although, in general, there was no difference between the FL and Bluephase-curing units, the thicknesses of 2 and 3 mm of the Bleach-M resin showed nanohardness values significantly lower than the others, like what happened for the microhardness. This can be explained due to the deficiency of CQ activation at greater depths,[26] although resins for bleached teeth contain less CQ.

It is essential to understand the properties and composition of restorative materials, as these are generally related to the clinical behavior of restorations.[28] The composite resins investigated in the present research have the same internal arrangement and quantity of charged particles. On the other hand, the amount of photoinitiators is different. Resins for bleached teeth contain less CQ and tertiary amine and more light-colored photoinitiators, such as Lucerin-TPO.[29] The Bleach-M and A2 color resins of thickness 2 and 3 mm showed the lowest microhardness cured by polywave LEDs when compared to monowave. This shows that for this composite the amount and type of photoinitiators were not decisive for the quality of polymerization but the depth of the restoration. A study by Jeong et al.[30] suggested that the degree of polymerization of the composite resins evaluated was minimally affected by the color of the resin. The shorter wavelength of Bluephase to activate resins containing alternative photoinitiators may not be sufficient to activate Lucerin properly. In addition, this polymerization difficulty may be related to the lower degree of monomer conversion of Lucerin.[31] Therefore, one of the main limitations of this study was a failure to measure the amount of photoinitiators in the studied resin.

In this study, the Knoop microhardness and the Berkovich nanohardness results were compared and a direct correlation between the two measurements was noted. However, any comparison of the results between nanoindentation and microindentation should be made with caution because during microindentation testing markings need to be displayed to allow the size of the indentation to be measured. This process is time consuming and introduces errors due to the inaccurate measurements of the lengths of the diagonals.[32] With the

nanoindentation test, the indentation does not need to be viewed after the test. The depth of the indentation versus the applied force is monitored in real time. Thus, information about the elastic and plastic properties of the material can be obtained.[33,34]

Thus, it is recommended to continue studies on this important issue by evaluating other photopolymerization systems and composites containing photoinitiators that absorb energy at a wavelength below that used by CQ, which is the most used photoinitiator. We encourage studies with experimental resins containing specific amounts of photoinitiators as commercial products have an unclear and imprecise amount of photoinitiators, making it difficult to conclude about the polymerization quality.

Conclusion

Monowave unit showed better effectiveness in curing nanohybrid composite resins in different colors and depths as compared to polywave in both experimental tests. In addition, a positive correlation was found between the microhardness and the nanohardness tests.

Clinical Significance

Different LCUs can influence the properties of resin composites and compromise its mechanical performance. Comprehension of how these factors can affect the quality and behavior of restorative material may help the dental professional in choosing the best-activating source for a specific clinical utilization.

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