



The Effect of Different Light Curing Modes on the Microhardness Value of Different Composite Resins

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Abstract

Background: The purpose of the study was to compare the microhardness values of composite resins polymerized with different modes of a third-generation light curing unit (LCU).

Methods: Three nanohybrid composites (Filtek Z550 3M ESPE MN USA, Spectra ST HV Konstanz GERMANY, Tetric N-Ceram Liechtenstein) and one microhybrid composite (Filtek Z250 3M ESPE MN USA) were used. Eighteen samples (8 × 2 mm) were prepared for each composite (n=18). Half of them were cured with light-emitting diode (LED) LCU's (VALO; Ultradent USA) standard mode, the other half were cured in Xtra power mode.

The Vickers microhardness test was performed at the polished surface for 3 times of each specimen.

Results: For each composite, no significant difference was detected between the cure modes. For standard mode, a statically significant difference was detected between all composites, except for Tetric N-Ceram and Spectra ST HV. For Xtra power mode, a significant difference was detected between all composites, except for Z550 and Z250.

Conclusion: Xtra power cure for a microhybrid composite was showed the highest microhardness value. When the composites were compared with each other, the effect of Xtra power on microhardness values varied depending on the composites. The modes used of the light device did not affect the microhardness values of the composite resins used in the study.

Keywords: Dental composite, light curing, microhardness

INTRODUCTION

For many years, resin composites have been the material of choice for dental fillings in clinical dentistry.¹ Composite resins have undergone many advances with the development of their various types.² And they still rank among the most popular restorative materials because of their excellent mechanical qualities, high function, and aesthetics.^{3,4} Microhybrid composites are composed of bigger filler particles with an average size of 0.01–0.1 µm mixed with microfill particles. These have the advantages of improved physical properties, high fill percentage, and enhanced aesthetics. Larger particle sizes indicate that the primary disadvantage of this group is the challenge of maintaining high polish over an extended period of time. To address this issue, nanohybrid composites were created, which contain nanomeric particles in a conventional resin matrix.⁵

For a restoration to be clinically successful, resin composites' surface microhardness is critical; thus, the restorative material's resistance to scratches and surface wear improves with increasing microhardness.⁶

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For the dental composite resin photopolymerization, several technologies, including laser, plasma-arc, quartz-tungsten halogen, and light-emitting diode (LED), have been suggested for use.⁷ Light-curing resin-based restorative materials can be polymerized with various light sources. The most commonly used of these light sources are quartz-tungsten halogen light sources. Although it has the advantage of being low in cost, it has some disadvantages as the curing depth is limited, the curing time is long, and the light intensity decreases over time. In order to eliminate these disadvantages, LED light devices have been introduced to the market.⁸ Because of its longer lifespan, cordless functioning, and lack of filter requirement, LED lights seem to provide the greatest technology available.⁹ The first- and second-generation LEDs have some deficiencies^{10–11} Manufacturers have claimed that they can reduce light exposure time by increasing the light output of the device. In other words, if the radiation increases, the polymerization time may be shortened. The first- and second-generation LED LCUs can polymerize resin-based composites (450–470 nm) containing camphorquinone as photoinitiator in 20–40 seconds. However, over time, resin-based composites containing different photoinitiators have been introduced to the market. To activate these alternative photoinitiators and achieve adequate polymerization, third-generation LED LCUs have been developed that emit polywave light and can provide sufficient polymerization with short-term curing.¹² To enable polymerization of restorative materials utilizing more than only CQ (camphorquinone) as initiator, producers of curing lights had to provide sets of LED chips that radiated many wavelengths. A third-generation LED curing device was recently introduced to the market. Depending on the mode selected, it can achieve irradiances of up to 3200

mW/cm² and deliver adequate polymerization in 3 seconds.¹³ Samples polymerized using high-intensity LED LCUs have been shown to have improved DC and microhardness values in a number of studies. Additionally, a logarithmic relation between the energy density of the light device and the hardness value of resin composites was found.^{14–17} An additional study revealed that the hardness value was unaffected by light intensities above 1000 mW/cm².¹⁸

The power of dental LED LCUs can affect the microhardness values of composite resins. The use of devices with high power can lead to better polymerization and therefore a better microhardness value. However, there are not many studies on the hardness of composite resins containing different photoinitiators.

As a result, this study examines the microhardness of various composite resins after polymerization using different modes of third-generation LED light curing unit (LCU)'s different modes.

The null hypothesis was that there wouldn't be any difference between the microhardness of composites cured with different modes of a third-generation LED LCU.

MATERIAL AND METHODS

Ethics committee approval was not needed because this study used only inanimate materials.

Preparation of Samples

Three nanohybrid composites (3M Filtek Z550, Spectra ST HV, Tetric N-Ceram), one microhybrid composite (3M Filtek Z250), and a third-generation LED LCU (VALO, Ultradent, USA) were used in this study (Table 1).

Table 1. Materials Used in the Study

Material	Organic Matrix	Filler Particles/Initiators	Company
3M Z250 (Shade A2) (microhybrid composite resin)	BisGMA, UDMA, Bis-EMA, fluorescent agents, pigments, stabilizers, and initiators	Zirconia/silica: 3 µm or less, zirconia/silica cluster, surface-treated silica: 20 nm, 78,5% (w) 60% (v)	3M/ESPE, St. Paul, MN, USA
3M Z550 (Shade A2) (nanohybrid composite resin)	BisGMA, UDMA, TEGDMA, BisEMA, PEGDMA	20 nm silica + 0.1–10 µm zirconia/silica 82% (w) 68% (v)	3M/ESPE, St. Paul, MN, USA
Spectra ST HV (Shade A2) (nanohybrid composite resin)	Dimethacrilate resin, ethyl-4-(dimethylamino)benzoate	SphereTEC® fillers (d3, 50≈15 µm); non-agglomerated barium glass and ytterbium fluoride; filler load (78–80 wt%) (60–62% v); highly dispersed, methacrylic polysiloxane nanoparticles, initiator: CQ, EDMAB	Dentsply Sirona Konstanz GERMANY
Tetric N-Ceram (Shade A2) (nanohybrid composite resin)	%19–%20 Bis GMA, UDMA, Dimethacrylates	Barium glass filler, Ba–Al fluorosilicate glass, Ytterbium trifluoride (0.7–1 µm mean filler size), mixed oxide, highly dispersed silica, prepolymers, additives, stabilizers and catalysts, pigments 80% (w) 55–57% (v)	Ivoclar vivadent AG, Liechtenstein
Light Curing Unit	Type	Irradiance/Recommended Curing Time	Manufacturer
VALO	LED third-generation polywave	Standard mode (1000 mW/cm ²) 20s – High-power mode (1400 mW/cm ²) 8s – Xtra power mode (3200 mW/cm ²) 3s	Ultradent Products Inc, South Jordan, UT, USA

For reducing the impact of colorants on polymerization, shade A2 was chosen, and 2 mm samples were used to ensure even polymerization.

Seventy-two disk-shaped samples made of restorative materials were placed in an 8 × 2 mm Teflon mold. Each composite included 18 samples. A Mylar strip was placed over the top and bottom of the mold, and the extra product was pushed out by pressing a glass slide up against the strip. After that, the glass plate was taken off, and the curing unit's light tip was placed concentrically within the mold's cavity, 0.5 mm above the sample.

For each composite, half of the samples were cured with the standard mode of LCU for 20 seconds, and the other half were cured with the Xtra power mode for 3 seconds. After curing, the cured surfaces of all the samples were polished for 10 seconds each with polishing discs (Shofu Super Snap Finishing&Polishing disk) with L506, L508, L501, and L502.

Microhardness Measurements

After each sample was kept in distilled water for 24 hours in the dark, microhardness of the polished surface of each sample was measured using a Vickers hardness tester. The surface hardness value of each sample was taken with a surface hardness device (Innovatest FALCON 300G2) using a 200 g load 15 seconds, and the measurements were recorded. The power of the light source was measured with a radiometer (Hilux Ledmax Light Curing Meter, Benlioglu Dental Inc., Ankara, Türkiye) after each sample was prepared. Three measurements were taken from the middle region of each sample, no closer than 1 mm to each other or to the edges, and the average of the 3 measurements was considered a single value for each sample.

Statistical Analysis

The Number Cruncher Statistical System (Utah, USA) package program was used for statistical analyses. After the normality test (Shapiro-Wilk), 2-way ANOVA was used to compare the composites within themselves and with each other for normally distributed variables. Subgroup comparisons were tested with Tukey's multiple comparison test, and intraclass correlation was used to determine microhardness measurement reliability. The coefficient and 95% CI were used. $P < .05$ is the level of statistical significance. (Table 2).

Table 2. Statistical Comparison of Materials and Cure Modes

Microhardness (MPa)	Type III Sum of Squares	df	Mean Square	F	P
Intercept	258746.20	1	258746.20	5657.17	.0001
LCU	66.23	1	66.23	1.45	.233
Material	17021.58	3	5673.86	124.05	.0001
LCU * Material	1229.70	3	409.90	8.96	.0001

LCU, light curing unit.

Table 3. Mean Vickers Microhardness Values of Composite Materials for Standard and Xtra Power Modes of LED LCU

Material Group	Standard	Xtra Power
Z550	66.33±8.20 A a	80.58 ± 6.83 A a
Z250	77.52±5.47 A b	76.52 ± 6.40 A a
Tetric N-Ceram	47.56±6.66 A c	38.92 ± 7.70 A b
Spectra ST HV	44.54±7.79 A c	47.60 ± 4.13 A c

Means followed by distinct capital letters represent statistically significant differences in each row ($P < .05$).

Means followed by distinct lowercase letters represent statistically significant differences in each column ($P < .05$).

RESULTS

In standard mode, a difference was observed between the Z550, Z250, Tetric N-Ceram, and Spectra ST HV composite groups ($P=.0001$). The microhardness values of Z250 were found to be statistically significantly higher than the microhardness values of Z550, Tetric N-Ceram, and Spectra ST HV ($P=.011$, $P=.0001$). The microhardness values of Z550 was found to be statistically significantly higher than those of Tetric N-Ceram and Spectra ST HV ($P=.0001$). No statistically significant difference was observed between Tetric N-Ceram and Spectra ST HV ($P=.804$).

In Xtra Power mode, a statistically significant difference was observed between Z550, Z250, Tetric N-Ceram, and Spectra ST HV ($P=.0001$). While the microhardness values of Z550 and Z250 were seen to be statistically significantly higher individually than Tetric N-Ceram and Spectra ST HV ($P=.0001$), no significant difference was observed when compared to each other ($P=.542$).

When the 2 cure modes were compared for each composite used in the study, no significant difference was found between the microhardness values ($P=.233$). (Table 3).

DISCUSSION

One of the most important parameters that can be evaluated to obtain information about the physical properties of dental materials is microhardness. Hardness is typically associated with resistance to intraoral softening, mechanical strength, and rigidity. The microhardness value is also related to the polymerization of the material and therefore may vary depending on the polymerization device used. In our study, the standard mode and Xtra power mode of the VALO LED LCU were used in the polymerization of different composite groups, and the microhardness values of the samples were measured.

In our study, microhardness evaluations were made 24 hours after light curing. In many similar studies, microhardness measurements were made after 24 hours to ensure that the samples reached maximum hardness.^{19,20}

As a result of polymerization under standard mode and Xtra power mode, there was no significant difference between the microhardness values of each composite. Therefore, the null hypothesis that there would not be any difference between the microhardness of composites cured with different modes of a third-generation LED LCU was accepted. As a result of polymerization under standard mode, the microhardness values of Z250 were found to be significantly higher than the microhardness values of Z550, Tetric N-Ceram, and Spectra ST HV. In accordance with our study, Mobarak et al.²¹ also showed in their study that the microhardness values of microhybrid composites polymerized with an LED LCU and containing camphorquinone were higher than other composites. In another study, microhybrid Filtek Z250 and a BisGMA-free nanohybrid Purefill composite group were compared with each other, and the microhardness values of Z250 were higher than Purefill. They explained that the surface hardness of composite resins is affected by the organic structure of the material and the ratio and size of inorganic fillers.²² They stated that high BisGMA content increased the microhardness values. In our study, Z250 and Z550 showed the highest microhardness values. Z250 and Z550 have a high molecular weight monomer structure such as BisGMA and Bis EMA. Since these monomers with high molecular weight form more intense cross-links, higher microhardness values may have been observed in the Z250 and Z550. Also, it was reported that the zirconia content in Z550 may have been a factor in the high surface hardness.²³

In contrast to our findings, Jafarzadeh et al.²⁴ found the highest microhardness value for Tetric N-Ceram, a microhybrid composite, and the lowest microhardness value was recorded for Ceram-X Mono, a nanohybrid composite. They also revealed, consistent with some previous studies. Jafarzadeh et al. also revealed that there was no significant difference in the microhardness values of composite resins when QTH or LED LCEs were used for polymerization, consistent with some previous studies.²⁴ There was no significant difference in the microhardness values of composite resins when QTH or LED LCUs were used for polymerization. In our study, Tetric N-Ceram showed lower microhardness values than other composites. Besides the filler type and density, the hardness of composites may also depend on other factors such as the size of the filler particles and the volume of content.²⁵ In our study, Tetric N-Ceram had the lowest filler density by volume. Therefore, it can be thought that the low microhardness value may be due to this.

In Xtra power mode, the microhardness values of the Z550 composite were found to be higher than the microhardness values of the Tetric N-Ceram and Spectra ST HV, and

no statistically significant difference was observed in the microhardness values of the Z550 and Z250. Gonulol et al.¹⁹ observed the highest microhardness value in the Z550 composite group for the samples they polymerized with the VALO LED LCU for standard and Xtra power mode. Although there is no statistically significant difference between Spectra ST HV and Tetric N-Ceram, significantly lower microhardness values than Z250 and Z550 were observed. Graf et al.²⁶ associated spherical filler geometry and reduced average filler size with lower mechanical properties, higher sensitivity to aging, and reduced reliability. Spectra ST HV included the pre-polymerized fillers in spherical form. It was reported that the pre-polymerized filler content added to the resin also affects microhardness. Pre-polymerized fillers are heat-cured and do not form covalent chemical bonds with the polymerization matrix due to the absence of methacrylate groups on their surfaces. This can reduce the resistance of the material.²⁷ In our study, Spectra ST HV was significantly lower for Xtra power mode and standard mode. The fact that the prepolymerized fillers contained in Spectra ST HV were placed in the material in a fragmented manner and were weakly bonded to the matrix may have affected the low surface hardness.

In our study, the fact that the Z550 composite group has a higher microhardness value than the Tetric N-Ceram and Spectra ST HV composites can be explained by the TEGDMA monomer it contains. The properties of the resin matrix affect the hardness and general mechanical properties of the composite. Studies have shown that TEGDMA forms a much denser network than BisGMA.²⁸ In newly formulated composite resins, the TEGDMA content is increased and a more reactive diluent monomer, carboxylic anhydrides, and diketones are added; all of these lead to increased cross-linking in the polymerized matrix, resulting in improved mechanical properties.²⁹ Pieniak et al.³⁰ also compared nanohybrid composites with bulk fill composites in their study and stated that the highest microhardness value they recorded belonged to the Z550.

In this present study, there was no significant difference between the microhardness values of the samples polymerized with standard and Xtra power modes. However, Gonulol et al.¹⁹ found a significant decrease in the microhardness values of samples polymerized with VALO LED LCU's Xtra power mode compared to the microhardness values of samples polymerized with Elipar and VALO LED LCU's standard mode and VALO high-power mode. Bakkal et al.⁸ also compared VALO LED LCU with a second-generation LED LCU, and they found the microhardness values of the samples polymerized with VALO LED LCU to be significantly lower than the other groups. While Optima and Demi Ultra, which are second-generation LED LCUs, cure for 20 seconds, VALO cures for 6 seconds. Also, in their study wavelengths were measured as 420–480 nm for Optima, 450–480 nm for Demi Ultra, and 395–480 nm for VALO. As a result, they stated that optimum curing requires sufficient polymerization time and

light output. Szalewski et al.³¹ studied different cure modes as well and concluded that the lowest microhardness value was measured for the 3-second fast-cure mode. In accordance with our findings, Lindberg et al.³² determined that there was no significant difference between the microhardness values of composite samples polymerized for different periods of time.

CONCLUSION

It could be concluded that a microhybrid composite has the highest microhardness values. Besides, different cure modes for VALO LED LCU did not affect the microhardness of each composite. However, when the composites were compared with each other, the effect of Xtra power on microhardness values varied depending on the composite. The null hypothesis that the third-generation LED LCU would show microhardness values consistent in all tested materials even with shorter curing times was accepted to verify Xtra power's effect on microhardness of composites. Using only one LED LCU and not using composite materials containing more photoinitiators are the main two limitations of this study. Within the limitations of the study, it could be concluded that further studies are needed.

Ethics Committee Approval: Ethics committee approval was not needed because this study used only inanimate materials.

Informed Consent: Written informed consent was not obtained since no one participated in this study.

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