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Effect of different curing times and distances on the microhardness of nanofilled resin-based composite restoration polymerized with high-intensity LED light curing units



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KEYWORDS

LED; High intensity; Microhardness; Composite; Polymerization; Efficiency Abstract *Purpose:* This study examined the effect of different distances and curing times on the microhardness (VHN) of nanofilled resin-based composite (RBC) restorations polymerized with high-intensity LED LCUs.

Materials and methods: Seventy-five RBC specimens (2 mm thickness and 5 mm diameter) were fabricated from Tetric-N-Ceram (Ivoclar Vivadent). Each of the 25 specimens was polymerized by means of one of three types of high-intensity LED LCUs: (B) Blue-Phase-G2 (polywave LED, Ivoclar Vivadent), (E) Elipar S10 TM (single-peak, 3 M ESPE), and (P) Planmica Lumion (single-peak, Mectron) at three different distances (0 mm, 2 mm, and 4 mm) at 20 sec, 40 s, and 60 sec. A micro-hardness tester (NOVA, Innovatest, The Netherlands) was used to measure the VHN from the top and bottom surfaces. Data for VHN were analyzed using mixed ANOVA, followed by post hoc analyses with p-values < 0.05.

Results: A significant difference was found in VHN between all three LED LCUs, where (B) specimens had the highest means, followed by (E) and (P). Bottom surface VHN values were reduced significantly (p < 0.05) compared to top surface values in all LCU types. With increasing distances up to 2 mm and 4 mm, VHN values with (E) and (P) were significantly reduced on the top

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and bottom surfaces (p < 0.05). When the curing times were increased for 40 and 60 sec, the VHN values were significantly improved (p < 0.05). Meanwhile, increasing the distance with (B) did not significantly reduce the VHN. Moreover, increasing the curing times did not significantly improve the VHN of the bottom surfaces.

Conclusion: High-intensity LCUs have variable effects on the surface (top/bottom) hardness of Tetric-N-Ceram nanofilled RBC restoration. With increasing distance, VHN was reduced; therefore, compensation with more curing time (2 mm/40 sec and 4 mm/60 sec) is highly recommended with Elipar S10 and Planmica Lumion LCUs to improve the material surface hardness.

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1. Introduction

Global interest in performing aesthetic restorations has led to an increase in the use of light-curing units for polymerizing tooth-colored restorative materials (Frederick Allen Rueggeberg et al., 2017). The physical and mechanical properties of photocured composites are directly influenced by the level of conversion achieved during polymerization (Ribeiro et al., 2012) Thus, a high degree of conversion enhances the restoration wear resistance, hardness and flexural strength (J. L. Ferracane et al., 1997). Nanotechnology in manufacturing RBC restorative materials is being applied to dentistry (Mitra et al., 2003). Due to their small size, it is possible to incorporate more filler content (Nitta, 2005), which results in better mechanical properties (Leprince et al., 2013; Mitra et al., 2003).

The optimal conversion of the monomer in the resin composite material is directly related to irradiance (Calheiros et al., 2006; F. A. Rueggeberg et al., 1994) and exposure time (Vargas et al., 1998). Thus, photopolymerization using lightcuring units with low irradiance presents poor physical properties such as increased wear and staining and a greater risk of pulp irritation and recurrent caries (Barghi et al., 2007; Maghaireh et al., 2013; Pilo et al., 1999; F. A. Rueggeberg et al., 1994) To obtain optimal physical properties and clinical performance of resin composite restorations, it is necessary to have sufficient irradiance energy for the monomer to be converted to a polymer during the polymerization reaction (Knežević et al., 2001; Yoon et al., 2002). This has led to the development of high-intensity light curing units, such as quartz-tungsten-halogen, plasma arc and light-emitting diode (LED) light (Barghi et al., 2007; Calheiros et al., 2006; Maghaireh et al., 2013). Nevertheless, LED sources have almost replaced other light curing systems (Jandt & Mills, 2013; Nomoto et al., 2009).

The manufacturers of the newly introduced high-intensity LED LCUs have claimed a sufficient depth of polymerization and superior physical properties of the RBC restoration along the whole depth of the 2 mm increment of composite restoration owing to their higher irradiance (Leprince et al., 2013).

Surface hardness testing has been shown in many studies to be a good indicator for the degree of conversion (DC) (Dionysopoulos et al., 2015); however, this is considered by others not a sufficient clinical indicator of the degree of conversion because a low-intensity light source also promotes superficial hardness, even when the deeper layers are not well polymerized. According to Morimoto et al., this was true at the top surface, which was not greatly affected by variations in irradiance (Morimoto et al., 2016). Several studies indicated a better DC and microhardness with high-intensity LED LCUs (Jandt & Mills, 2013). A logarithmic relationship was found between the hardness of dental composites and the received radiant exposure (energy density) (Davidson & de Gee, 2000; R. B. T. Price et al., 2004; Watanabe et al., 2015). Meanwhile, another study suggested that there is no benefit from increasing irradiance beyond approximately 1000 mW/cm² (Musanje & Darvell, 2003).

Microhardness values at the bottom surfaces of the restoration when there is a distance between the LC tip and the restoration, as in many clinical cases of deep class IIs, may still represent a challenging validity even for present high-intensity LCUs (Elkorashy et al., 2013; Shimokawa et al., 2017; Yoon et al., 2002). Several studies concluded a reduced polymerization at the bottom surface of the restoration, and when it is performed from a distance, the effect of light will be even less (R. B. T. Price et al., 2004). Therefore, compensating by increasing the curing time has been proposed, and some have recommended up to 60 s of curing (Xu et al., 2006). Thus, this study examined the effect of different distances and curing times on the microhardness (VHN) of nanofilled RBC restorations polymerized with high-intensity LED LCUs.

2. Materials and methods

2.1. Restorative material

Details of the dental restorative material evaluated in the study and the LED LCUs are shown in Table 1. A total of 75 RBC disc-shaped specimens were fabricated from a single restorative material (Tetric-N-Ceram, Ivoclar Vivadent). The RBC restoration specimen was placed in a silicon mold (2 mm deep \times 5 mm diameter) on a Mylar strip and a glass slide. Then, it was covered with another Mylar strip followed by a microscopic glass slide to prevent the formation of an airinhibited layer on the top surface and pressed to extrude excess material to obtain a smooth, standardized exterior surface.

2.2. Polymerization

Twenty-five specimens were polymerized with one of the three LED LCUs assigned for the study according to their manufacturer's instructions at an ambient temperature of $23^{\circ} \pm 1C^{\circ}$. The LCUs were calibrated to measure their irradiance value directly on a digital radiometer (with 0 mm distance) and from two different distances (2 mm and 4 mm) to evaluate the actual value that can reach the top of the specimen. The radiometer Table 1

Resin-based composite restor	rative material					
Composite (shade)	Туре	Recommended curing time 10–20 s with high irradiance LCUs (>1000 mW/cm ²)		Composition	Manufacturer	Lot No. Z017Y9
Tetric-N-Ceram	Nanofilled, Shade A ₂			Dimethacrylates, additives, catalysts, stabilizer sand pigments, barium glass, ytterbium trifluoride, mixed oxide and prepolymerized filler (prepolymers) (56% vol.)	Ivoclar-Vivadent, AG, 9494 Schaan/ Liechtenstein	
Light curing units (LCUs)						
(LCU)	Туре	Irradiance 0 mm	Irradiance 2 mm	Irradiance 4 mm	Manufacturer	Ser. No.
Blue phase G2	blue/violet polywave LED	1570 mW/ cm ²	1390 mW/ cm ²	1210 mW/cm^2	Ivoclar Vivadent AG FL-9494 Schaan/ Liechtenstein, Austria.	222,788
Elipar S10 TM	Blue/Single-peak LCU	1120 mW/ cm ²	1010 mW/ cm ²	890 mW/cm ²	3 M ESPE, D-82229 Seefeld, Germany.	939,123,009,611
Planmeca Lumion	Blue/Single-peak LCU	1220 mW/ cm ²	1070 mW/ cm ²	700 mW/cm^2	Mectron S.p.A., via Loreto 15'A, 16,042 Carasco-GE-Italy.	309PG403
Light curing radiometer						
Name	Description			Manufacturer		Ser. No.
Bluphase - meter	4,5 DC, 3x Battery LR6/AA/1,5 V DC			Ivoclar Vivadent AG FL-9494 Schaan/Lie	00,735	
Microhardness tester	, , ,					
Name	Description			Manufacturer		Ser. No.
NOVA	Weight: 37.5 kg Man. date: 2017 Power: 100 V-240 V/50–60 Hz/3A Model No.: 130			INNOVATEST, Europe BV, Borgharenw	13,002,178,648	

was a Bluephase meter from Ivoclar Vivadent, which has a wavelength sensitivity of 400-510 nm and measures the irradiance from 100 to 1999 mW/cm². The irradiance values were obtained by means of three measurements of the LCUs.

During polymerization, three distances and three curing times were followed for each type of LCU: 0 mm distance for 20 sec, n = 5; 2 mm distance for 20 sec, n = 5; 2 mm distance for 20 sec, n = 5; a mm distance for 20 sec, n = 5; and 4 mm distance for 60 sec, n = 5 to simulate the clinical situations of deep proximal class II restorations. Table 1 shows the irradiance of each LCU that was applied for each distance. For the groups that received longer curing times, after each 20 sec treatment, a break of 4 sec was implemented to allow for heat dissipation. The specimens were then incubated in dark vials filled with distilled water for 24 h at 37° to ensure complete polymerization.

2.3. Microhardness testing

A microhardness tester was used to measure the Vickers hardness number (VHN) from five indentations on the top and five on the bottom surfaces. The VHN was determined for each specimen using an INOVATEST microhardness tester device, as shown in Table 1. A diamond indenter was used to apply a load of 300 gm for 15 s. Five values were recorded from each surface tested. In total, 75 specimens were made (one RBC × three lights × three distances), and 750 microhardness readings were obtained (75 specimens × 5 VHN measurements × 2 surfaces × 1 time).

2.4. Statistical analysis

Data that were obtained from the microhardness tester were analyzed using the statistical software IBM SPSS version 27 (Armonk, NY: IBM Corp., 2020). Mixed ANOVA (combination of within-subject and between-subject factors) was used to address the main effect of LED LCU type, distance/polymerization time, the interaction term between LED LCU type and distance/polymerization time. Statistically significant interactions and main effects were followed up with post hoc analyses (Tukey's HSD and simple effects). All inferential statistical analyses were conducted using a 0.05 level of statistical significance, with p-values < 0.05 interpreted as statistically significant.

3. Results

Descriptive statistical analysis shows that VHN values range from 32.1 to 63.7, with a mean of 49.65, a median of 51.60 and a standard deviation of 7.60. The histogram and boxwhisker plot suggest a slightly skewed distribution but can be assumed to be approximately normal (Fig. 1).

The results showed statistically significant differences between the three LED LCU types (p-values < 0.001). Blue-Phase-G2 specimens had the highest VHN values (M = 55.18), Planmica Lumion had the lowest VHN values (M = 40.66), and Elipar S10 had the lowest VHN values (M = 52.05).

Distance and polymerization time have statistically significant impacts on VHN for each LCU. Table 2 and Fig. 2 sum-



Fig. 1 The histogram suggests an approximately normal statistical distribution of the VHN values.

Table 2	Mean	and	standard	deviation	of	Vickers	hardness
number V	HN (s.	d.) of	f the top/b	ottom surf	ace	s for eac	h LCU at
different of	listance	es/cu	ring times	•			

Distance/curing time	LCU	VHN (top) kg/ mm ²	VHN (bottom) kg/ mm ²
0 mm/20 sec	В	61.26 ^A (1.63)	51.76 ^D (0.93)
	Е	55.81 ^B (2.19)	52.62° (1.43)
	Р	45.75 ^E (1.06)	36.30 ^F (1.10)
2 mm/20 sec	В	59.45 ^A (1.01)	54.39 ^B (1.61)
	Е	52.37 ^C (1.12)	45.13 ^D (1.00)
	Р	42.46 ^E (2.73)	37.04 ^F (1.35)
2 mm/40 sec	В	56.85 ^A (2.75)	51.66 ^C (0.93)
	Е	55.73 ^A (1.50)	52.81 ^B (1.44)
	Р	45.56 ^D (1.46)	36.46 ^E (0.94)
4 mm/20 sec	В	58.47 ^A (0.75)	48.08 ^C (2.39)
	Е	52.01 ^B (0.85)	$43.08^{D} (0.58)$
	Р	39.70 ^E (1.90)	37.17 ^F (2.02)
4 mm/60 sec	В	61.11 ^A (1.10)	51.58° (0.83)
	E	54.99 ^B (1.55)	52.68° (1.60)
	Р	46.71 ^D (1.55)	39.90 ^E (2.16)

B: Bluephase GC; E: Elipar S10; P: Planmica Lumion. Means followed by the same letter indicate no significant difference at the 95% confidence level (p < 0.05).

marize the statistically significant interactions that were found among the variables.

The top and bottom surfaces of Planmica reported the highest remarkable reduction in VHN values with increasing distances. At 2 mm/20 sec and 4 mm/20 sec, the VHNs were M = 42.46 and 39.70 for top surfaces and M = 37.04 and 37.17 for bottom surfaces, respectively. Even after increasing the curing time to 40 and 60 sec, the VHN of Planmica was still statistically significant, with the lowest values for both the top and bottom surfaces among all other groups (M = 45.56 and 46.71 for top surfaces, M = 36.46 and 39.90 for bottom surfaces).

On the other hand, the Blue-phase reported significantly the highest VHN compared to the other LCU types (p < 0.05),



Fig. 2 The chart represents cluster interaction between all the variables (LCUs; curing time 20, 40, 60 sec; distances 0 mm, 2 mm, 4 mm).

and its performances were not affected by increasing distance (0 mm, 2 mm, 4 mm) on the top surfaces (M = 61.26, 59.45, 58.47, respectively) (p > 0.05). However, the bottom hardness was significantly reduced compared to the VHN values of the top at 0 mm, 2 mm, and 4 mm distances with a standard 20 sec curing time (M = 51.76, 54.39, 48.08, respectively). Interestingly, increasing the curing time with Blue-phase LCU did not significantly improve the VHN values, and it may actually have an inverse effect (p > 0.05).

The VHN values of Elipar S10 were intermediate compared to those of the other two LCUs. Its curing performance was significantly inferior to that of the Blue-phase and superior to that of Planmica. Its irradiance was affected by increasing the distance. A statistically significant reduction (p < 0.05) was shown in the results of VHN on the bottom (M = 52.62) surfaces compared to the top (M = 55.81). With distances of 2 mm and 4 mm, the bottom was (M = 45.13, 43.08) compared to the top (M = 52.37, 52.01). Upon increasing the curing time to compensate for the 2 mm/40 sec and 4 mm/60 sec distances, the VHN increased significantly (p < 0.05) on the top (M = 52.37, 61.11) and bottom (M = 52.81, 52.68) surfaces.

4. Discussion

Microhardness is an indirect measure of the degree of conversion of a material. It provides useful information on the depth of polymerization (curing) when measured on the top and bottom surfaces of a specimen (Aravamudhan et al., 2006; Jack L. Ferracane, 1985; Soh et al., 2003). Several studies have reported reduced DC and microhardness on the bottom side of the restoration. Additionally, increasing the distance could result in a significantly lower irradiance that can reach the surface of the resin in the tooth, which is often 2–8 mm away from the light tip (Corciolani et al., 2008; R. B. Price et al., 2000; 2011; Xu et al., 2006). For that reason, compensation was recommended by increasing the curing time from 20 to 60 sec to ensure sufficient polymerization (XU et al., 2006). Accordingly, in the present study, an assumption was estimated for the curing time of the 2 mm distance groups (40 sec) and the 4 mm groups (60 sec) to compare between the tested highintensity LCUs in their efficiency to polymerize the composite restoration from distance. The results showed that Blue-phase LCUs proved to have the highest performance among the other LCUs; even with distance, the readings of the bottom hardness were significantly higher than their counterpart LCUs. Its superior efficiency may be related to two factors: first due to its higher irradiance (up to 1750 mW/cm^2) and second due to its spectral emission. The Blue-phase is a polywave LCU that can polymerize different photoinitiators other than camphorquinone. Blue light polymerizes camphorquinone at a wavelength of 570 nm (Leprince et al., 2013), while violet light is capable of polymerizing ivocerin-dibenzoyl germanium derivative (Moszner et al., 2008) photoinitiators. Ivocerin has been incorporated in a Tetric-N-line composite material (Ivoclar Vivadent) for the purpose of enhancing their quantum efficiency and esthetic appearance by reducing the yellowish effect of the amine group that is incorporated as a coinitiator with camphorquinone. Moreover, when the manufacturer of a specific RBC restoration produces an LCU, it is usually considered more compatible with the restoration than other LC devices.

Manufacturers of Blue-phase LCUs have recommended a curing time of 10–20 sec, but in the present study, a 20 sec curing time was followed as a standard time for all the tested LCUs. When polymerizing from distance, increasing the curing time had no beneficial effect in increasing the bottom surface hardness with Blue-phase LCU. However, this was not the case with Elipar S10 and Planmica Lumion LCUs. Increasing the curing time (2 mm for 40 sec and 4 mm for 60 sec) contributed to significantly enhancing the top and bottom surface hardness. This could most likely be explained by the more time that was given to compensate for the distance, which allowed more of the irradiance to penetrate the whole thickness of the restoration and polymerize not only camphorquinone, which is com-

patible with their single peak spectrum (Shimokawa et al., 2017) but also has sufficient time to polymerize more of the other photoinitiators at the lower irradiance spectrum (370–460 nm wavelength) (Leprince et al., 2013).

A similar study was conducted by Shafadella et al. (Shafadilla et al., 2017), who concluded that curing distance and time both significantly affect the surface hardness of nanofilled composite resin. Their surface hardness results ranged from 72.40 kg/mm² to 80.33 kg/mm². They considered that the range of hardnesses was acceptable since they were comparable to that of dentin, which has a surface hardness of 57-60 kg/mm² (Segal et al., 2015). Therefore, the hardness results obtained in the present study for Elipar S10 and Planmica were not considered to be acceptable clinically, since the hardness values ranged from 40.66 to 52.05 kg/mm², which was less than that of dentin and far less than that of enamel. In clinical applications, when a short curing distance is not possible, the curing time and the intensity of the light from the light-curing unit must be increased to achieve maximum surface hardness. However, the curing distance must still be no > 4-5 mm (Segal et al., 2015; Shafadilla et al., 2017). Another important factor that should not be overlooked during polymerization with high-intensity LCUs is heat production. It is evident (Hori et al., 2019; Mouhat et al., 2017; Weerakoon et al., 2002) that this can produce a harmful effect on the pulp. Thus, a lag break of 4 sec was performed in the present study between every 20 sec of polymerization to reduce the heat effect.

The status of the curing unit should be evaluated periodically to ensure that it is still in optimal condition. Several studies have revealed that light-curing units used in dental practices do not emit sufficient radiation to achieve maximum photopolymerization (Maghaireh et al., 2013; Strassler & Price, 2014). Accordingly, composite resin restorations may not experience sufficient polymerization, causing them to be resistant to low abrasion (Maghaireh et al., 2013). Therefore, to compensate, the light energy emitted by the light-curing unit must be increased (Krämer et al., 2008).

5. Conclusion

High-intensity LCUs have variable effects on the surface (top/ bottom) hardness of Tetric-N-Ceram nanofilled RBC restorations. To obtain clinically acceptable hardness for RBC restoration, the choice of the LCU that matches the wavelength of the photoinitiators present in the restoration is the key factor for its success. The closer the tip of the LED light-curing unit is to the surface of the nanofilled composite resin restoration, the greater the surface hardness of the restoration. When that is not possible clinically, compensation with a longer curing time (2 mm/40 sec and 4 mm/60 sec) is highly recommended with Elipar S10 and Planmica Lumion LCUs to improve the material surface hardness. Further studies are indicated with other types of RBC restorative materials.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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