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Microhardness of composite resin cured through different primary tooth thicknesses with different light intensities and curing times: *In vitro* study

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ABSTRACT

Objective: The aim of this study was to evaluate the effect of increased exposure time and light intensity on microhardness of cured composite through different thicknesses of tooth structure in primary teeth. **Materials and Methods:** One hundred and seventy cylindrical resin composite specimens were prepared. All specimens were divided into 17 experimental and control groups. "Light-emitting diode" light curing unit (LCU) applied directly or through 1, 2, and 3 mm thicknesses tooth slices for experimental groups. The irradiation protocols were 25 and 50 s at 650 mW/cm^2 and 15 and 30 s at 1100 mW/cm^2. The "quartz-tungsten-halogen" LCU (400 mW/cm^2) for 40 s was used in control group. Microhardness was measured by the Vickers hardness test. **Results:** Indirectly cured specimens and those cured through a 1 mm thick tooth structure, an increase in intensity caused hardness drop. In the specimens cured through 2 and 3 mm thick tooth structures, increased intensity and/or exposure time did not show any appropriate changes on microhardness. **Conclusion:** Irradiation through a 1.0 mm thick tooth slice resulted in reduced microhardness although it was still within the clinically acceptable level. The hardness values of the specimens cured through 2 or 3 mm thick tooth slices fell below the clinically acceptable level even after doubling the exposure time and/or light intensity.

Key words: Composite resin, curing time, light intensity, microhardness, primary tooth

INTRODUCTION

In light-activated resin composites, photopolymerization is of fundamental importance. Adequate polymerization is a crucial factor in obtaining the optimal physical performance of resin composite.[1,2]

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The polymerization depends on chemical composition filler content, photoinitiators, shade, and thickness[3]

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of composites; it also correlated with the irradiation time and intensity, as well as the distance of the light tip from the tooth-restorative material.[4]

Depth of cure is getting importance as the composite cure through the tooth structure in many clinical cases. The intensity of the curing light is attenuated by the tooth structure through which the light passes.[5] Inadequate polymerization results in poor resistance to wear and color stability, increased rates of water sorption and solubility, decreased dynamic elasticity modulus, as well as early restoration failure.[1,6,7] Furthermore, there may be greater deterioration at the margins of restoration, decreased bond strength between the tooth and the restoration, greater cytotoxicity and pulpal irritation, postoperative sensitivity, and reduced hardness.[1,2,6,7] Further, the unreacted monomers could be eluted from restoration due to hydrolysis over time which in turn leads to the decline in the physical properties.[8,9]

Several studies demonstrated these poor mechanical properties of composite while being cured through the tooth structure.[5,10-13] Subsequently, some researchers have suggested increasing the light intensity[14,15] and exposure time[13,16,17] to compensate for reduced light intensity in these situations. However, the results have been controversial and the threshold thickness of tooth substance, in which a reduction in intensity becomes clinically important, remains unclear.

To the best of our knowledge, there has not been a study done on irradiation through tooth structure on primary teeth. With regard to structural differences of primary and permanent teeth[18,19] and existing controversial results in studies[1,7,20,21] done on permanent teeth, the aim of this study was to assess the effect of increased exposure time and light intensity on microhardness of cured composite through different thicknesses of tooth structures in primary teeth.

MATERIALS AND METHODS

This study was approved by the Research and Ethics Committee, School of Dentistry, Mashhad University of Medical Science, Mashhad, Iran, and all experiments were carried out at the Pediatric Department of the Mashhad Dental School. In this study, intact extracted human primary second molars, free of any type of decay, cracks, hypocalcification, fractures, abrasions, previous restorations, or structural deformities were used to prepare different thicknesses of sound tooth structure slices (1, 2, and 3 mm). Written informed consent was obtained from the parent of each patient to sanction the use of the extracted teeth for the abovementioned purpose. The teeth were disinfected with thymol 0.1% immediately after extraction, cleaned with slurry of pumice, and stored in normal saline before the experiment. Different slices were prepared using a grinder (METASERV 2000 grinder/polisher, Buehler UK Ltd., Coventry, England) with 240, 400, and 600 grit Sic papers. Next, the teeth slices were measured at the center with an orthometer gauge. They were prepared from lingual surfaces of the teeth (along the mesiodistal axis); they had 1 mm enamel and zero, 1, and 2 mm dentin thicknesses, respectively. The slices were stored in distilled water before and during the experiment to prevent desiccation. They were dried with air before light application and put back into distilled water after testing.

One hundred-seventy specimens of the hybrid resin composite A_2 Z-250 (3M Dental Products, St. Paul, MN, USA) were prepared (3 mm in diameter and 2 mm in thickness) using Teflon molds. The mold was filled in a single increment while covered by Mylar strips (KerrHawe Striproll 8 mm/0.05 mm, Switzerland) at the top and bottom using glass slabs and finger pressure to remove any excess material. The light guide tip of a light-emitting diode (LED) light curing unit (LCU) (Bluephase Style, Ivoclar Vivadent, Schaan, Liechtenstein) was positioned on the surface of the composite specimen with the light passing through the Mylar strip (direct light curing) or tooth slices (indirect light curing: 1, 2, and 3 mm thicknesses). Specimens were located on a vertically adjustable holding device to obtain an accurate distance and position in relation to light guide tip. A dentinal table was used under the specimens to simulate the light reflection. Before light application to each specimen, the intensity of LCU was calibrated with a radiometer (Coltolux Light Meter, Model No. C7900, Coltene Whaledent Inc., Switzerland). The irradiation protocols are shown in Table 1. All specimens were divided into 16 experimental subgroups (n = 10) according to light intensity and curing time and

Table 1: Irradiation protocols used in the study

Protocols	Intensities (mW/cm²)	Light curing times (s)	Energy density (J/cm²)
1	Low (650)	25*	16.25
2	Low (650)	50**	32.50
3	High (1100)	15*	16.50
4	High (1100)	30**	33.00

*The manufacturer's recommended time (regarding low intensity, the recommended time is 20, but it was justified to 25 to produce a nearly equivalent energy density with high intensity), **Twice the manufacturer's recommended time the thickness of the tooth slices and one control group (n = 10) (400 mW/cm^2 intensity and 40 s of exposure time using a quartz-tungsten-halogen [QTH] LCU [Astralis 7, Ivoclar Vivadent, Schaan, Liechtenstein] applied in control group).

All specimens were stored in 100% humidity in a light proof container at 37°C for 24 h, followed by a careful polish of the bottom and top surfaces of the each specimen with 600, 1000, 2000, and 2500 grits SiC papers. The Vickers hardness test under a 300 g load for 20 s (300 g/20 s) was carried out on the top and bottom surfaces of each specimen with a microhardness tester (Matsuzawa Seiki Co., Ltd., MH2, Japan, 1990). The hardness was tested at one central and four radial indentations. The average of these values was considered the mean Vickers hardness number (VHN) of the specimen's surface. The hardness ratio of the specimens was then calculated using the formula:

$$\text{Hardness ratio} = \frac{\text{VHN of top or bottom surface of experimental specimen}}{\text{VHN of top surface of control specimen}}$$

If this ratio at the both, top and bottom, surfaces of the specimen was at least 90%, that specimen was considered clinically acceptable.[22] Statistical analysis was performed with ANOVA and independent t-test at a significance level of 5%.

RESULTS

The absolute frequency distributions of composite specimens according to intensity, curing time, and method of light curing (direct or indirect) are shown in Figure 1.

A multifactor ANOVA revealed double and triple interactions between independent variables (P = 0.001) of the tooth structure thicknesses, the exposure time and light intensity; therefore, the variables were analyzed distinctly.

Regarding indirect curing (curing through tooth structure), as the tooth slices increased in thickness, the VHN of the cured resins decreased in all groups [Figure 2].

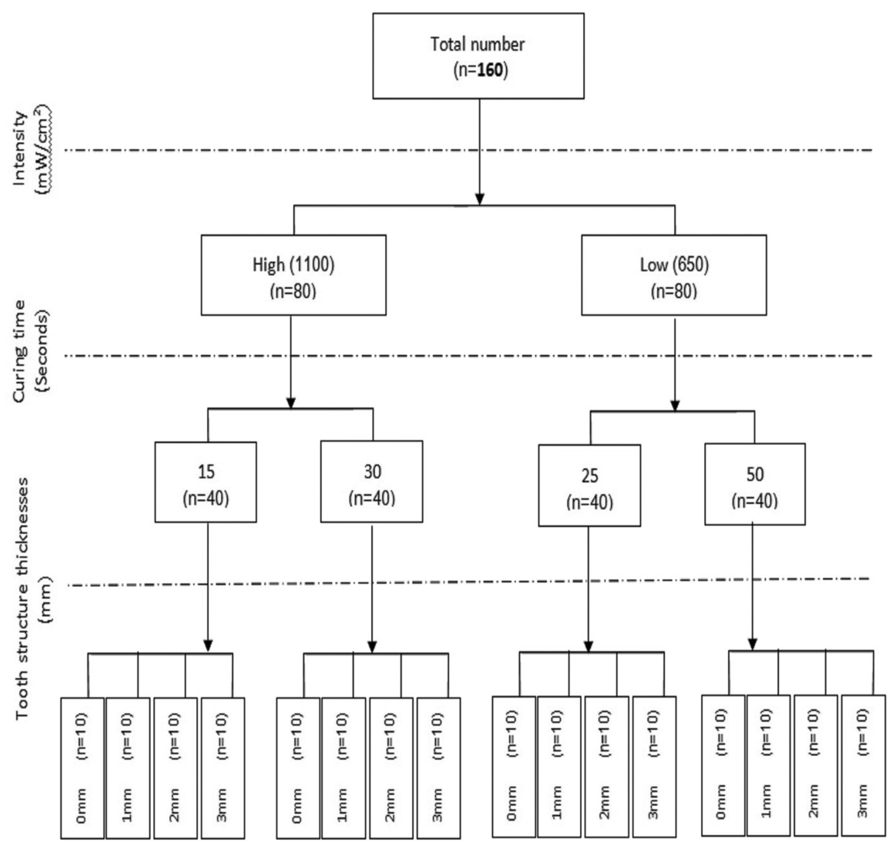

Figure 1: Absolute frequency distribution of composite specimens in experimental groups according to intensity, curing time, and direct or indirect light curing through different tooth structure thicknesses

The means and standard deviations of the VHN of the specimens which polymerized directly or through tooth structure under the experimental conditions used in this study are presented in Tables 2 and 3.

As shown in Table 2, with increasing light curing intensity, the means of the VHN of directly cured specimens and those that were cured through 1.0 mm thick tooth structure were significantly decreased ($P < 0.05$), whereas the means of the VHN of just the top surfaces of the specimens that were cured through 2.0 mm thick tooth structure were statistically increased ($P < 0.05$). Furthermore, the obtained results showed that there were no statistical differences for microhardness while the composite specimens cured through 3 mm of tooth structure thickness.

Table 3 shows the results after increasing the exposure times. Indirectly cured specimens and those that were cured through 1.0 mm thick tooth structure, increasing curing times along with high intensity resulted in significantly improved hardness. Whereas, the values of the specimens that were cured through 2 and 3 mm thick tooth structures were mostly significantly higher only at the top surfaces.

Only in the specimens of the directly cured group and the groups that were cured through 1 mm thickness of primary tooth structure, the microhardness of the top and the bottom surfaces was near clinically acceptable microhardness [Figure 3].

DISCUSSION

Due to the structural differences between permanent and primary teeth, the results of the former cannot generalized to the latter. Studies have shown that the density and diameter of dentinal tubules[19] and also mineral contents[18] in primary teeth are less than permanent teeth. All of these factors could affect the light distribution during passing through tooth structure,[23] the primary teeth.

In the present study, microhardness of the composite resin was significantly influenced by irradiation through tooth structure. As the thickness of tooth structure was increased, more reduction in microhardness was observed. However, the hardness of the specimens cured through 1 mm tooth thickness was still in a clinically acceptable limit. It has been demonstrated that the light-attenuating effect of enamel and dentin reduces the degree of polymerization, resulting in poor mechanical properties.[10,13,24]

Table 2: Mean (±standard deviation) of Vickers hardness number of the specimens polymerized directly or through tooth structure: Effect of increased intensity

Intensity	Thickness							
	Direct light cure (mm)		Indirect light cure (mm)					
	0		1		2		3	
	Top surface	Bottom surface	Top surface	Bottom surface	Top surface	Bottom surface	Top surface	Bottom surface
Low (650)*	86.49 (1.42)	85.08 (0.70)	85.87 (1.20)	83.69 (1.20)	81.64 (0.82)	77.95 (1.72)	78.83 (3.11)	75.87 (4.58)
High (1100)**	84.72 (0.87)	82.96 (0.66)	84.77 (1.09)	82.33 (0.67)	84.27 (0.93)	76.94 (1.05)	76.52 (1.33)	74.33 (1.11)
P	0	0	0.04	0	0	0.13	0.05	0.32

*Exposure time is 25 s, **Exposure time is 30 s

Table 3: Mean (±standard deviation) of Vickers hardness number of the samples polymerized directly or through tooth structure: Effect of increased exposure time

Intensity (mW/cm²) Time (s)	Thickness															
	Direct light cure (mm)				Indirect light cure (mm)											
	0				1				2				3			
	Low (650)		High (1100)		Low (650)		High (1100)		Low (650)		High (1100)		Low (650)		High (1100)	
	T#	B##	T	B	T	B	T	B	T	B	T	B	T	B	T	B
1×*	86.49 (1.14)	85.08 (0.7)	83.35 (0.76)	82 (0.71)	85.87 (1.20)	83.69 (1.02)	83.86 (0.45)	81.45 (0.71)	81.64 (0.82)	77.95 (1.72)	80.38 (1.60)	75.78 (2.38)	78.83 (3.11)	75.87 (4.54)	74.30 (3.80)	68.05 (4.69)
2×**	86.03 (0.64)	84.62 (0.7)	84.72 (0.87)	82.96 (0.66)	86.1 (0.76)	83.75 (1.17)	84.77 (1.09)	82.33 (0.67)	83.09 (0.80)	78.80 (1.32)	84.27 (0.93)	76.94 (1.05)	81.91 (0.46)	78.27 (0.55)	76.52 (1.33)	74.33 (1.11)
P	0.28	0.16	0.002	0.006	0.9	0.03	0.03	0.01	0.001	0.2	0.00	0.18	0.01	0.13	0.1	0.002

#T: Top surface, ##B: Bottom surface, *1× (irradiation time): In low intensity was 25 s and in high intensity was 15 s, **2× (twice the above times): In low intensity was 50 s and in high intensity was 30 s

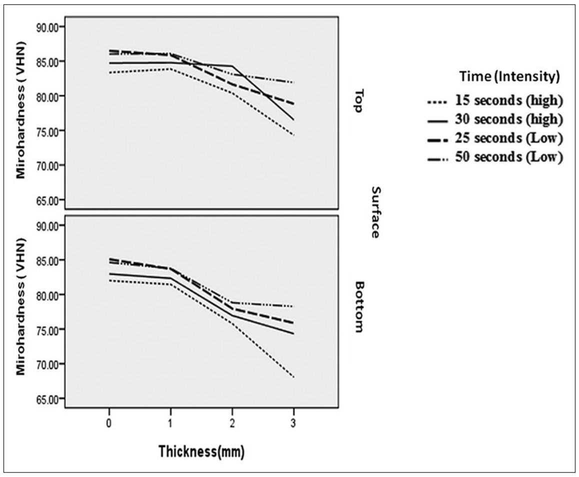

Figure 2: Mean Vickers hardness number of composite resins polymerized through different tooth thicknesses in studied subgroups

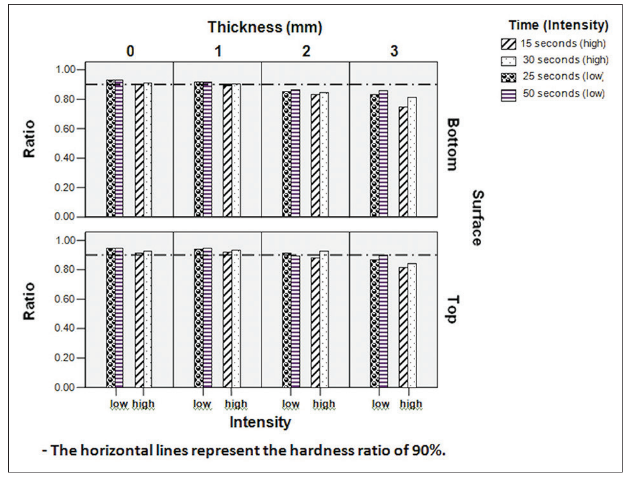

Figure 3: Comparison of clinically acceptable hardness ratio among different studied groups

To compensate for light energy loss while passing through the tooth structure, the light intensity and curing time were increased. Increasing the light intensity resulted in decreased hardness in specimens cured directly. This was consistent with the results of other studies.[1,7] Some researchers have reported that the use of high-intensity LCUs negatively affects the integrity of restoration-cavity interface; increases the incidence of restorative margin fracture, enamel margin fracture, and marginal openings; increases shrinkage stresses; and results in higher microleakage values.[25]

High intensity may cause rapid formation of a highly crosslinked polymeric network in surface layers of composite resin;[1] subsequently, the extent of the light passing through the bulk of composite may be reduced. Thus, high intensity can lead to rapid polymerization and the formation of a low polymeric chain and frequency of crosslinking, reducing the modulus of elasticity, and decreasing the hardness of composite resin.[1] The mechanical properties of material and frequency of crosslinking are of the opposite direction.[26,27] Hence, while light curing is applying directly, the use of high-intensity LCUs is not recommended. The data were not in agreement with the results of da Silva *et al*.[20] and Yap *et al*.[21] The two studies indicated that using higher intensities would improve the curing efficiency of composite resins. These contradictory findings might be explained by differences among studies in LCUs, wavelengths, characteristics of composite resins, and light intensities they used.

In the cases that light curing was carried out through the tooth structure, there were no significant differences in microhardness as light intensity increased. The exception was in the specimens cured through 1 mm depth, in which the microhardness decreased as the light intensity increased. In these cases, it seems that despite the decrease in power density during passage through the teeth, it is still high enough to cause a drop in the microhardness of the composite for reasons mentioned earlier. However, in thicker slices (2 and 3 mm) which consist of dentin besides enamel, it is assumed that the light absorption and scattering are so great that even doubling the power density cannot improve the hardness of composite resin except for the top surfaces in the 2 mm thickness group. With regard to the fact that surface hardness is not an adequate indicator for complete material polymerization and the hardness of the bottom surface should be also closed to the hardness of the top surface, it seems that further increasing in intensity might be required.

In this study, the results for the exposure time revealed that in specimens cured directly or through 1 mm thickness slices, increasing the exposure time in cases with high intensities, resulted in higher values of hardness; however, at low intensities in most cases, it did not cause a change in microhardness. Perhaps, this is because differences in exposure time do not result in differences in degree of conversion of optimally cured resin composite material (at low intensities); however, at high intensities that existing hardness has been decreased, increasing exposure time improved hardness significantly but not at the clinically acceptable limit.

As the thickness of the tooth which the light passes through increases, the numbers of the photons available to raise camphorquinone to an activated state is limited by absorption and scattering factors. This reduces the probability of collision of camphorquinone with amine.[28] To compensate for this decreased light intensity, the exposure time can be increased, providing enhanced opportunity for an excited camphorquinone molecule to collide with an amine, thus creating a free radical.[28] It seems to be the reason for increasing hardness in high-intensity groups after increasing exposure time. Several studies[1,13,17] were in agreement with ours.

Increasing exposure time had a significant effect just on the top surfaces of the specimens cured through 2 or 3 mm thicknesses; therefore, it is not a clinically helpful result.

An optimal clinically acceptable microhardness for composite resins has not yet been determined.[29] Consequently, in previous researches on adequate polymerization, the ratio between microhardness of the bottom and top surfaces is designated as a standard. Some researchers[22,30] advise that the optimal ratio should be 90% while others[31,32] recommend that it should be 80%. The composite resins can be polymerized almost ideally *in vitro*, in the clinical situations; however, in most of the time, it is not possible to polymerize them ideally. Hence, to generalize the results with more confidence, level of 90% was selected at the present study.

However, it must be taken into consideration that the results not to be misinterpreted. In studies which aimed at assessing new LCUs or irradiation modes, there is a possibility of specimens being poorly cured throughout and if the bottom surface is compared to the top surface of the same specimen, the ratio still could exceed 80 or 90% and be misinterpreted as a sufficiently cured material. On this note, the hardness of both surfaces must be compared to standard cured surface (control). In this study, the VHN values obtained with 40 s curing at 400 mW/cm^2 using a QTH LCU were used as the control.[15,22,32]

The microhardness ratio reached 90% in approximately all the specimens that were directly light cured and in the groups that the specimens were light cured through 1 mm thick tooth structure. Accordingly, when there were 2 or 3 mm tooth thicknesses between the light guide and composite resin, the used LED-LCU (Bluephase) could not establish the acceptable clinical microhardness, even after doubling the manufacturer's recommended exposure times or power densities or both.

The further studies will be needed to determine the effect of other LCUs such as plasma arc and laser systems on microhardness of the composites cured through thicker tooth structure, as well as similar clinical investigations should be performed to confirm this *in vitro* results.

CONCLUSION

With the limitations of this study, we concluded that the presence of different tooth thicknesses of primary teeth between the light guide and composite resin could cause reduction of microhardness of cured composite. With the LED device, "Bluephase" is applied for curing composite with the light guide most possible close to the resin or 1.0 mm thickness of tooth between them, increasing light intensity leads to reduction of microhardness, so this is not recommended. However, doubling the exposure time makes the microhardness significantly higher in these situations. When there are 2 or 3 mm thicknesses of tooth, increasing light intensity or curing time (doubling) caused no helpful changes in microhardness. Perhaps, because it may require a greater increase in power density and exposure time to increase the hardness to the acceptable clinical level.

Just in the cases of direct light curing or curing through 1 mm tooth thicknesses, the microhardness of the specimens was at the clinically acceptable rate.

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Conflicts of interest
There are no conflicts of interest.

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