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CURING EFFECTIVENESS OF NANOFILLED RESIN COMPOSITE USING DIFFERENT LED CURING INTENSITIES AT DIFFERENT IRRADIATION TIME

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ABSTRACT

Aim: evaluated the effectiveness of different LED curing light intensities at different irradiation time on the microhardness and depth of cure (hardness ratio) of nanofilled resin composite material.

Materials and methods: Nanofilled resin composite material (Filtex Z350XT, 3M, ESPE) and LED curing unit (Dr's light AT, Good doctors co.ltd. korea) with two different light intensities were used in this study. Two Circular Teflon moulds with two different thickness were used to prepare 25Resin composite discs specimen that were divided into five groups (5 discs each). For **group 1**, 2mm thick resin composite disc specimens where polymerized using standard light curing intensity (800 mW/cm^2) for 30 sec. while for **group 2**, 4mm thick resin composite disc specimens where polymerized using standard light curing intensity (800 mW/cm^2) for 30 sec. **group 3**, 2mm thick resin composite disc specimens where polymerized using high intensity light curing mode (1400 mW/cm^2) for 10 sec. **group 4**, 4mm thick resin composite disc specimens where polymerized using high intensity light curing mode (1400 mW/cm^2) for 10 sec. **Group 5**, 4mm thick resin composite disc specimens where polymerized using high intensity light curing mode (1400 mW/cm^2) for 20 sec. Microhardness testing was performed and the mean microhardness values and hardness ratio % of the specimens were calculated, tabulated and statistically analyzed using independent (unpaired) t test for 2 groups comparisons while one way analysis of variance (ANOVA) and Tukey post hoc tests were used to study the significance between more than 2 groups. The significance level was set at $p \leq 0.05$. Statistical analysis was performed with SPSS 19.0

Results: The highest mean value was recorded in **group 5:** 4mm thick disc specimens cured with high intensity for 20 sec (91.16%), whereas the lowest mean was recorded in **group 4:** 4mm disc specimens cured with high intensity for 10 sec (73.34%). A significant difference was found between both groups (p=0.032). However, no significant difference was detected between **group 3:** High 10 (2mm) that recorded 85.56% , **group 5:** High 20 (4mm) recording 91.16% and **group 1 and 2:** standard (2mm and 4mm respectively) recording 86.96% and 81.08% respectively.

Conclusions: 1. Depth of cure and microhardness values were greatly affected by the interaction between curing light intensities and irradiation time and material thickness as well. 2. Nanofilled resin composites irradiated with LED high light intensity (1400 mW/cm^2) for 20 sec. demonstrated a higher microhardness. 3. Composites might be cured in increments higher than 2 mm and up to 4mm when using LED high intensity light curing system with special caution to the irradiation time applied.

Key words: nanofilled composite, depth of cure, LED, light intensities, microhardness ratio

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INTRODUCTION

Since resin composites were developed, many efforts have been made to improve their clinical performance.[1] the organic and inorganic components of these materials greatly influence their clinical behaviour, however, the filler particles features and rate of curing are the most important factors responsible for the improvement in mechanical properties of the resin composites.[2,3] With the introduction of nanotechnology, nanofilled resin composite materials were presented with their excellent initial polishing ability together with superior polish and gloss retention. [3,4,5] Yet, the degree of curing provided by different light curing systems remain an important issue to be considered for the clinical success of these materials.[6] A curing light intensity output and the total energy determines the mechanical properties of the resin composites materials. Recently, high intensity light emitting diode units (LEDs) was introduced in the last few years with the aim of fast curing of resin composites and less heat generation.[7]

As the Physical properties of composite resin are dependent on the degree of conversion (DC) of the resin matrix. A positive correlation has been established between hardness and degree of conversion (DC). However, the calculation of the hardness bottom/top ratio and giving an arbitrary minimum value for this ratio can predict the degree of conversion and depth of cure of resin composite materials as well. [1,8] Therefore, the current study evaluated the effectiveness of different LED curing light intensities at different irradiation time on the microhardness and depth of cure (hardness ratio) of nanofilled resin composite material.

MATERIALS AND METHODS

Nanofilled resin composite material (Filtex Z350XT, 3M, ESPE, St Paul, MN, USA Batch#N731284) shade A3 and LED curing unit (Dr's light AT, Good doctors co.ltd. korea) with two different light intensities were used in this study. This nanofilled resin composite contains bis-GMA, UDMA, TEGDMA, and bis-EMA resins. PEGDMA has been substituted for a portion of the TEGDMA resin to moderate the shrinkage. The fillers are a combination of non-agglomerated/non-aggregated 20 nm silica filler, non-agglomerated/non-aggregated 4 to 11 nm zirconia filler, and aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4 to 11 nm zirconia particles). The inorganic filler loading is about 72.5% by weight (55.6% by volume).

Two Circular Teflon molds were prepared, one had (4mm internal diameter x 2mm thickness) while the other one was (4mm internal diameter x 4mm thickness). Each mold was placed over glass slab covered with transparent mylor strip. A total number of 25 resin composite disc specimens were prepared that were divided into five groups (5 specimens each). Specimens were prepared by packing nanofilled resin composite material (Filtex Z350XT, 3M, ESPE) inside the mold that was covered with mylor strip, then 1mm glass slide was placed gently over it to remove excess material. Specimens were then light cured with LED curing unit according to the group divisions. For **group 1**, 2mm thick resin composite disc specimens where polymerized using standard light curing intensity (800 mW/cm^2) for 30 sec according to the manufacturer instruction as a control. while for **group 2**, 4mm thick resin composite disc specimens where polymerized using standard light curing intensity (800 mW/cm^2) for 30 sec. **group 3**, 2mm thick resin composite disc specimens where polymerized using high intensity light curing mode (1400 mW/cm^2) for 10 sec. **group 4**, 4mm thick resin composite disc specimens where polymerized using high intensity light curing mode (1400 mW/cm^2) for 10 sec. However in **group 5**, 4mm thick resin composite disc specimens where polymerized using high intensity light curing mode (1400 mW/cm^2) for 20 sec. the light intensity was checked every time with radiometer. The curing tips were placed perpendicular to the surface at zero distance to the glass slide to ensure optimum curing.

Micro-hardness testing

Prior to the Micro-hardness measurement, the specimens were longitudinally polished using a sequence of 800-1200-4000 grit silicon carbide paper and alumina polishing paste (1μm). Surface Micro-hardness of the specimens was determined using Digital Display Vickers Micro-hardness Tester (Model HVS-50, Laizhou Huayin Testing Instrument Co., Ltd. China) with a Vickers diamond indenter and a 20X objective lens. A load of 100g was applied to the surface of the specimens for 20 seconds. Three indentations, which were equally placed over a circle and not closer than 0.5 mm to the adjacent indentations, were made on the surface of each specimen. The diagonals length of the indentations were measured by built in scaled microscope and Vickers values were converted into micro-hardness values.

Micro-hardness calculation:

Micro-hardness was obtained using the following equation:

$$HV=1.854\ P/d^2$$

where, **HV** is Vickers hardness in Kgf/mm^2, **P** is the load in Kgf and **d** is the length of the diagonals in mm.

Five VK readings were recorded for each sample surface (top and bottom). For a given specimen, the five hardness values for each surface were averaged and reported as a single value. The mean microhardness values and hardness ratio % of the specimens were calculated and tabulated using the formula:

Hardness ratio% = VK of bottom surface /VK of top surface X 100

Statistical analysis

Values were presented as mean and standard deviation (SD) values. Data were explored for normality using Kolmogorov-Smirnov test of normality. The results of Kolmogorov-Smirnov test indicated that most of data were normally distributed (parametric data), so independent (unpaired) t test was used for 2 groups comparisons and one way analysis of variance (ANOVA) and Tukey post hoc tests were used to study the significance between more than 2 groups.

The significance level was set at $p \leq 0.05$. Statistical analysis was performed with SPSS 19.0 (Statistical Package for Scientific Studies, SPSS, Inc., Chicago, IL, USA) for Windows.

RESULTS

Microhardness ratio

a) Comparison of all groups

The highest mean value was recorded in **group 5:**4mm disc specimens cured with high intensity for 20 sec, whereas the lowest mean was recorded in **group 4:** 4mm disc specimens cured with high intensity for 10 sec. ANOVA test revealed a significant difference between groups (p=0.032). Tukey's post hoc test revealed no significant difference between **group 3:** High 10 (2mm), **group 5:** High 20 (4mm) and **group 1 and 2:** standard (2mm and 4mm respectively), (Table 1, Fig.1)

b) Comparison of high 10 groups

Comparing both high 10 groups revealed a higher mean value in High 10 (2mm) group. However this difference was not statistically significant (p=0.079) (Table 2)

c) Comparison of standard groups

Comparing both standard groups revealed a higher mean value in standard (2mm) group. However this difference was not statistically significant (p=0.265) (Table 3)

d) Comparison of 2mm groups

Comparing both 2mm groups revealed a higher mean value in standard (2mm) group. However this difference was not statistically significant (p=0.833) (Table 4)

TABLE (1) Comparison of microhardness ratio% in all groups (ANOVA test)

	Mean	Std. Dev	Std. Error	95% Confidence Interval for Mean		Min	Max
				Lower	Upper		
High 10 2mm	85.56 ^{a,b}	11.60	5.19	71.16	99.96	71.80	97.50
High 10 4mm	73.34^{b}	5.30	2.37	66.76	79.92	69.00	82.00
High 20 4mm	91.16^{a}	8.18	3.66	81.00	101.32	77.50	98.70
Standard 2mm	86.96 ^{a,b}	8.45	3.78	76.47	97.45	77.00	100.00
Standard 4mm	81.08 ^{a,b}	6.98	3.12	72.41	89.75	73.50	91.70
F	3.287						
P	0.032*						

Significance level P<0.05, *significant Tukey's post hoc test: means with different superscript letter are significantly different

TABLE (2) Comparison of microhardness ratio in High 10 groups (unpaired t test)

High10	Mean	Std. Dev	Std. Error Mean	Mean Difference	Std. Error Difference	95% C.I. difference		t	P value
						Lower	Upper		
2mm	85.56	11.6	5.19	12.22	5.703	-1.98	26.42	2.143	.079ns
4 mm	73.34	5.3	2.37						

Significance level P<0.05, ns=non-significant 95% C.I. difference = 95% Confidence Interval of the Difference

TABLE (3) Comparison of microhardness ratio in standard groups (unpaired t test)

Standard	Mean	Std. Dev	Std. Error Mean	Mean Difference	Std. Error Difference	95% C.I. difference		t	P value
						Lower	Upper		
2mm	86.96	8.45	3.78	5.88	4.9	-5.49	17.25	1.2	0.265ns
4 mm	81.08	6.98	3.12						

Significance level P<0.05, ns=non-significant

95% C.I. difference = 95% Confidence Interval of the Difference

TABLE (4) Comparison of microhardness ratio in 2mm groups (unpaired t test)

2mm	Mean	Std. Dev	Std. Error Mean	Mean Difference	Std. Error Difference	95% C.I. difference		t	P value
						Lower	Upper		
High 10	85.56	11.60	5.19	-1.4	6.42	-16.44	13.64	-0.218	0.833 ns
Standard	86.96	8.45	3.78						

Significance level P<0.05, ns=non-significant 95% C.I. difference = 95% Confidence Interval of the Difference

e) Comparison of 4mm groups

Comparing 4mm groups revealed the highest mean value in high 20 (4mm) group, whereas the lowest mean value was recorded in high 10 (4mm) group. ANOVA test revealed that this difference was statistically significant (p=0.005). Tukey's post hoc test revealed no significant difference between standard and each of the high 10 and high 20 groups (Table 5)

TABLE (5) Comparison of microhardness ratio in 4 mm groups (ANOVA test)

4mm	Mean	Std. Dev	Std. Error	95% Confidence Interval for Mean		Min	Max
				Lower	Upper		
High 10	73.34^{b}	5.3	2.37	66.76	79.92	69.00	82.00
High 20	91.16^{a}	8.18	3.66	81.00	101.32	77.50	98.70
Standard	$81.08^{a,b}$	6.98	3.12	72.41	89.75	73.50	91.70
F	8.33						
P	0.005*						

Significance level P<0.05, *significant

Tukey's post hoc test: means with different superscript letter are significantly different

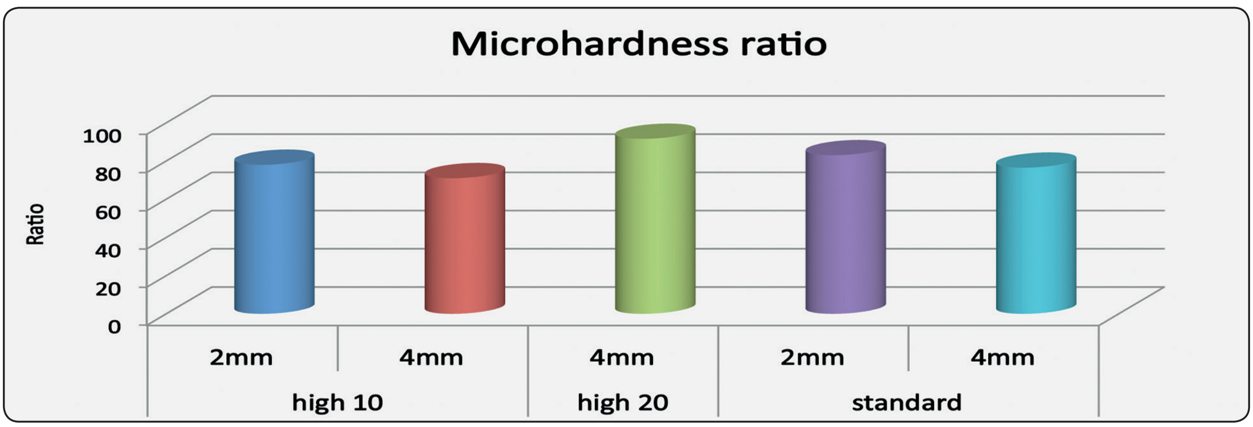

Fig. (1) Column chart showing mean microhardness ratio in all groups

DISCUSSION

Resin composites are polymeric materials that create a three-dimensional polymer network when polymerized. Their mechanical properties and resistance to functional stresses is very critical.[3,4] Over the past few years, nanofilled resin composites materials with better physical and mechanical properties have been developed. One of the critical parameters deciding the resin composites' resistance to stress is the depth of cure. Moreover, effective cure of resin composites is important for color stability as well as prevention of cytotoxicity of inadequately polymerized material. [9]The effectiveness of cure depends not only on the filler particle type, size, and quantity but also on the type, intensity, time and polymerization modes of the light source. [10]

Researchers [11,12] had found that with high intensity light-curing units, irradiation times of 10sec per 2 mm increment can be adequate to obtain a high degree of conversion. These data propose that a 2 mm buildup layering technique may not cause enough curing of the bottom layer for resin composite materials and that manufacturers need to provide quantitative information about the degree of conversion at specific activation light intensities and times that will ensure optimum cure of the bulk of a restoration. However, other studies have shown that 2-mm increments were well polymerized.[12,13]

In the current study curing effectiveness was measured using microhardness testing as indirect methods. As some authors [8] could demonstrate good correlation between increasing hardness and increasing degree of conversion. They concluded that the bottom-to-top surface microhardness ratios of a composite resin could be accurate reflection of degree of conversion. For light activated composites to be adequately polymerized it was suggested that the hardness ratio should be greater than 80% as light passes through the composite, it is greatly reduced due to light scattering resulting in decrease in the effectiveness of cure at the bottom surface. [14]

In the present study, two thickness of nanofiled resin composite discs specimen were evaluated (2mm and 4mm) that were cured with two different LED light intensities; Standard (800 mW/cm^2) for 30 sec. and (1400 mW/cm^2) for 10 and 20 sec. as well. The hardness ratios% were then calculated for the 5 tested groups.

The results of the present study reveals that The highest mean value was recorded in **group 5:**4mm disc specimens cured with high intensity for 20 sec (91.16%), whereas the lowest mean was recorded in **group 4:** 4mm disc specimens cured with high intensity for 10 sec (73.34%). A significant difference was found between both groups (p=0.032). However, no significant difference was detected between **group 3:** High 10 (2mm) that recorded 85.56%, **group 5:** High 20 (4mm) recording 91.16% and **group 1 and 2:** standard (2mm and 4mm respectively) recording 86.96% and 81.08% respectively. From these results it could be noticed that the high intensity LED light curing system (1400 mW/cm^2 for 20 sec) was able to perform optimum polymerization and DC (91.16%) of 4mm bulk of nanofilled resin composite specimen. However, the same intensity but for only 10 sec failed to produce enough accepted polymerization and DC for 4mm bulk of nanofilled resin composite specimen but it could produce adequate polymerization and DC of 2mm bulk of nanofilled resin composite specimen. So the intensity of light as well as the exposure time might be greatly responsible for these results. However, interactions between light curing intensities and exposure time and between light curing unit and thickness of specimens significantly influenced microhardness results. According to the results of the current study, the depth or thickness of the resin composite followed by the duration of the exposure are the main factors influencing microhardness in composites and, therefore, in the degree of cure .

This was in agreement with other studies[7] who demonstrated that the hardness values were influenced by the interaction between curing light and exposure time and that specimens irradiated for 20 sec revealed higher microhardness values when the LED curing light was used. Moreover, irradiation for 40 s produced greater microhardness values at higher depths. On the other hand, some authors disagreeing to the hypothesis of the present study, demonstrated that depth of cure and microhardness were not affected by the curing light used (QTH or LED)[8]. Furthermore, it has been reported that LED technology polymerizes resin composites as well or better than some QTH lights [15,16]. The degree of conversion greatly affect the physical and mechanical properties of resin composite materials as the higher the degree of conversion, the better the biocompatibility of the resin materials, the better the mechanical properties, wear resistance, water sorption, subsequently the color stability of the resin composites [17, 18].

In this study, only one physical property was tested on one type of nanofilled resins composite polymerized with one type of LED light curing unit. So under the limitation of the present study, the following conclusions could be derived:

1. Depth of cure and microhardness values were greatly affected by the interaction between curing light intensities and irradiation time and material thickness as well.
2. Nanofilled resin composites irradiated with high LED light intensity (1400 mW/cm^2) for 20 sec. demonstrated a higher microhardness.
3. Composites might be cured in increments higher than 2 mm and up to 4mm when using LED high intensity light curing system with special caution to the irradiation time applied.

RECOMMENDATION

Further investigations are required to assess the effect of high intensity LED curing unit and depth of cure of 4mm resin composite restorations on shrinkage stress that may be induced during polymerization reaction and consequent marginal gap formation. More researches involving the use of other materials and multiple combinations of polymerization modes are necessary.

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