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Effect of LED Light-curing on the Relative Hardness of Tooth-colored Restorative Materials

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Clinical Relevance

The use of high intensity LED curing units will result in the polymerization of tooth-colored restoratives using shorter times.

SUMMARY

Objective: To determine the relative hardness of GI-based restorative materials using both LED and QTH curing lights at different post–irradiation intervals. Methods: Four restorative materials (Z250, Beautifil, Dyract AP and Fuji II LC) with shade A3.5 were tested in conjunction with three LCUs (Astralis 3, Blue phase and Radii). The specimens were prepared using specially constructed molds, irradiating only the top surface to an equivalent energy density (J/CM). Vickers microhardness measurements were performed for both the top and bottom surfaces of all specimens following dry storage for 15 minutes in the dark. The measurements were repeated after 24 hours and 7 days. Mean VHNs and SD of the top and bottom surfaces of each specimen were calculated. Relative hardness values (RH) were also determined and mean RH was calculated for each group. Results: The top surface of the Z250, Beautifil and Dyract materials were significantly harder than the bottom surface ($p<0.05$) with all LCUs and at all time intervals. Conversely, a different trend was observed with Fuji II LC, where bottom surface VHNs were significantly higher than top surface values ($p<0.05$). Blue phase was the only LCU that recorded RH values above 80% for all tested material at all post-irradiation intervals. Conclusions: A high-intensity LED LCU used for 10 seconds resulted in RH values greater than 80%, with all four restoratives tested indicating a sufficient degree of monomer conversion with such a short curing cycle.

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INTRODUCTION

The development of resin-containing glass ionomer introduced restorations that actively help controlling recurrent caries, especially in high caries-risk patients.[1] These materials undergo hardening, partially through an acid-base reaction and partially through a polymerization reaction. RMGI varies considerably from materials that resemble GI cements to others that approximate light-cured resin composites.[2] They are categorized into: Resin-Modified Glass Ionomer (RMGI), Polyacid Modified Resin composite (PMRC) and the most recently introduced Giomers. The command set of these materials added to their merits over their predecessor, conventional glass ionomer restoratives. These materials also exhibit improved mechanical and physical properties and better handling characteristics.

Hand-in-hand with these developments, light curing units (LCU) have also progressed, with the emergence of high-intensity LCUs with light emitting diodes (LED). LCUs became very popular, since they have a number of advantages over conventional quartz tungsten halogen (QTH) LCUs. These advantages include being long-lasting, eliminating the need for filters and their ability to operate in a cordless fashion.[3] LED LCUs are typically smaller and lighter in weight, since cooling fans are not required. The high light intensity of these newly developed LCUs and the possibility of decreasing light exposure time while maintaining the desired degree of conversion is considered another advantage. Wiggins and others found that high-intensity LED LCUs resulted in the same degree of monomer conversion achieved with conventional QTH LCUs when only half the curing time was used.[4] This finding might not be applicable to GI-based/containing restorative materials, as their fluoride-containing glass particles may result in light attenuation and, consequently, adversely-affect monomer conversion. However, some reports suggested that, as time passes, decreased polymerization might be compensated for by post-irradiation delayed reaction, since light-activated restorative materials show progressive cross-linking of the resin phase after photo-activation.[5-6]

The degree of monomer conversion of resin composites can be measured using different testing techniques, either directly or indirectly. The direct methods, such as infra-red spectroscopy, laser Raman spectroscopy and nuclear magnetic resonance micro-imaging, are not frequently used, perhaps because they are time-consuming, complex and costly.[7] The indirect methods, which include scraping,[8] visual examination,[9] dye uptake[10] and surface hardness tests[11] are more widely reported in the literature. Although the scraping test gives no indication of the quality of cure, it has been accepted for use by standard organizations.[7] Surface hardness testing, on the other hand, has been used in many studies,[5-7] because it was shown to be a good indicator of the degree of conversion (DC).[11] Surface hardness can be measured either along the side of specimens, which will indicate gradual change in depth of cure, or on the upper and lower surfaces of disc-shaped specimens with a given thickness. The latter is used to measure the relative hardness, which is also considered a good indicator of DC.[12]

Based on the above, it is desirable to determine the RH of some GI- based restorative materials, especially those that more closely resemble resin composite in composition. This study determined the RH of a group of GI-based restoratives cured with two high-intensity LED LCUs compared to curing with one QTH LCU.

METHODS AND MATERIALS

Brand names, specifications, compositions and manufacturers of the four tested restorative materials are listed in Table 1. Shade A3.5 was used for all materials in order to standardize light absorption/attenuation. Table 2 shows the brand names of three LCUs, their specifications and manufacturers.

Twelve circular, black acrylic molds (30 mm in diameter and 2 mm thick) were specially constructed, each with five holes (3.5 mm in diameter). Four groups of specimens were prepared, one from each material. Three molds were assigned to the four groups. Each group was then subdivided into three subgroups, with five specimens made with one mold. The specimens from each mold were cured with one of the three LUCs. Each restorative material was placed in one of the mold's holes over a Mylar strip (Universal strips of acetate foil, Italy) and a glass slab, which was painted black. Every effort was made to prevent the inclusion of air voids while inserting the material in the molds. Another Mylar strip and glass slab were placed over the inserted material and pressure was applied for 30 seconds. For the Fuji II LC material, the capsules were activated and mixed according to the manufacturer's instructions before inserting into the molds. The glass slide was then removed, and the mold was covered with a specially designed metal cover that allows the tip of the LCU to coincide with the center hole.

Curing through the Mylar strip was followed according to the time given in Table 2. For the QTH LCU, the metal cover used to aim the light guide tip acted as a spacer so that the light intensity received by each specimen was maintained at 350 mW/cm^2. This resulted in comparable total energy for all LCUs (Table 2). The reduced light intensity of the QTH unit was verified with a radiometer (Model 100, Optilux Radiometer, Kerr Corporation, Orange, CA, USA). The specimens were stored dry in the dark for 15 minutes. Microhardness measurements were then performed

Table 1: *Materials Brand Names, Shade, Specification, Composition and Manufacturer*

Material Brand Name	Specification	Composition	Manufacturer
Filek Z-250	Universal hybrid composite (visible light cure)	Resins: Bis GMA, TEGDMA. Filling: Zirconia Silica (0.5-0.7 μm) 66% volume	3M ESPE Dental Products, St Paul, MN, USA
Beautifil	Giomer (S-PRG restorative material) Syringe type (visible light cure)	S-PRG filler, glass fillers, BisGMA, catalyst	Shofu Inc, Kyoto, Japan
Dyract AP	Polyacid modified resin composite (Compule) (visible light cure)	Resin: UDMA, TCB resin Fillers: SrF_2, (Sr)FALSi glass.	Dentsply, De Trey, Konstanz, Germany
GC Fuji II LC	Resin modified glass ionomer. Capsules (dual set, visible light cure)	Liquid: copolymer of maleic acid and polyacrylic acid, HEMA, water. Powder: fluoro-alumino-silicate glass.	GC Corporation, Tokyo, Japan

Bis GMA= Bisphenol A-glycidyl methylmethacrylate.
TEGDMA= triethylene glycol dimethacrylate.
UDMA=urithane dimethacrylate.
TCB resin=tetracarbonic acid-hydroxyethylmethacrylate ester.

Table 2: *The Light Curing Units Names, Manufacturers and Specifications*

Light Curing Unit	Manufacturer	Lamp	Light Intensity (mW/cm^2)	Irradiation Time (seconds)	Energy Density (J/cm)
Astralis 3	Ivoclar-Vivadent Schaan, Liechtenstein	Quartz-tungsten-halogen	Reduced to 350	40	14J/cm
Radii	SDI Inc	Light-Emitting Diode	1400	10	14J/cm
Blue phase	Ivoclar Vivadent Schaan, Liechtenstein	Light-Emitting Diode	1100	10	11J/cm

using a microhardness tester with a Vickers indenter (HMV-2000, Shimadzu Corporation, Kyoto, Japan) and a 100g load was applied for 15 seconds. Three indentations were performed to the top irradiated surface, and three corresponding indentations were made in the bottom surface. The second, third, fourth and fifth holes were subsequently packed, cured and subjected to microhardness testing in the same way as the first. Care was taken so that the light beam was prevented from scattering towards the previously prepared specimen. This was achieved using the formerly mentioned metal cover. Microhardness measurements were repeated for all specimens after 24 hours and 7 days. The specimens' preparation, storage and testing were carried out at room temperature. Mean VHNs of the top and bottom surfaces of each specimen were calculated. The overall means for each test subgroup were then calculated. Relative hardness (RH), which is the ratio of hardness of the bottom surface relative to the top, was also calculated for each specimen at each time interval. The mean RH was also calculated for each subgroup.

Multi-way analysis of variance (MANOVA) was used to statistically analyze the data at the $p<.05$ level of significance. The data was further analyzed using the Kruskal-Wallis and Mann-Whitney U tests. The data was first entered into a computer using Microsoft Excel version 7 (Microsoft Corporation, Redmond, WA, USA) and analysis was conducted with the SPSS statistical package (Statistical Package for the Social Science; SPSS Inc, Chicago, IL, USA).

RESULTS

Table 3 shows the means and standard deviations of VHNs of top and bottom surfaces of all materials as a function of LCU and post-irradiation time interval. Table 4 shows the means and standard deviations of RH values of the four materials with the three LCUs. Z250 and Fuji II LC were the only materials with an RH above 80% regardless of the LCU and time interval used. Blue phase was the only LCU that recorded RH values above 80% among all tested materials at all time intervals (Figure 1).

MANOVA revealed significant differences among the mean VHNs of the materials ($p<.001$). The Kruskal-Wallis test indicated significant differences in mean hardness values achieved with the three LCUs at the three time intervals ($p<.05$) except for Fuji II LC top surface at 15 minutes and Fuji II LC bottom surface at 24 hours ($p>.05$). Statistically-significant differences ($p<.01$) were also revealed when the Kruskal-Wallis test was used to compare the different materials cured with each LCU and at all post-irradiation times. However, no significant difference was found among the VHNs recorded for the bottom surface of the Dyract AP specimens cured with the QTH LCU ($p>.05$).

Table 3: *Means ± ST D of VHNs for Each Material at Each Time Interval with the Three Light Curing Units*

	15 Minutes		24 Hours		7 Days	
	Top	**Bottom**	**Top**	**Bottom**	**Top**	**Bottom**
Z250/Astralis 3	90.77 ± 0.77	75.61 ± 0.58	94.0 ± 0.36	79.25 ± 0.40	94.05 ± 0.42	82.43 ± 0.49
Z250 Radii	74.62 ± 0.54	63.55 ± 0.26	84.16 ± 0.64	70.75 ± 0.55	86.86 ± 0.87	76.84 ± 0.65
Z250 Blue phase	76.2 ± 2.85	73.36 ± 3.67	91.00 ± 3.11	82.72 ± 2.71	95.89 ± 1.47	91.26 ± 3.53
Beautifil/Astralis 3	56.82 ± 2.30	28.57 ± 4.32	70.98 ± 4.81	37.52 ± 3.04	72.28 ± 2.46	52.43 ± 5.86
Beautifil/Radii	54.62 ± 6.61	36.72 ± 5.46	69.32 ± 10.26	42.38 ± 9.17	79.79 ± 3.08	66.28 ± 3.61
Beautifil/Bluephase	72.86 ± 0.88	61.07 ± 0.44	80.3 ± 0.57	68.74 ± 0.59	85.18 ± 0.35	74.33 ± 0.94
Dyract AP/Astralis 3	56.47 ± 1.72	34.95 ± 1.17	68.09 ± 5.68	35.87 ± 1.03	69.36 ± 4.56	36.18 ± 1.15
Dyract AP/Radii	47.06 ± 4.29	25.8 ± 6.19	50.68 ± 2.76	26.51 ± 1.20	52.64 ± 2.15	36.57 ± 1.05
Dyract AP/Bluephase	51.34 ± 0.29	47.85 ± 1.37	58.94 ± 0.64	50.65 ± 0.63	58.56 ± 0.09	53.26 ± 0.70
Fuji II/Astralis 3	54.34 ± 4.34	56.67 ± 3.53	85.26 ± 6.34	77.52 ± 3.75	119.83 ± 18.0	99.85 ± 3.30
Fuji II/Radii	52.91 ± 5.34	63.71 ± 3.61	80.53 ± 2.25	81.94 ± 1.22	110.04 ± 1.83	99.87 ± 1.90
Fuji II/Bluephase	49.88 ± 2.07	57.28 ± 1.37	64.75 ± 1.69	82.60 ± 0.63	80.45 ± 0.52	82.27 ± 0.43

Table 4: *Relative Hardness Means ± SDs of the Tested Materials with the Different Curing Units at the Three Post-irradiation Times*

	15 Minutes	24 Hours	7 Days
Z250			
Astralis 3	0.83 ± 0.01	0.84 ± 0.01	0.88 ± 0 .01
Radii	0.85 ± 0.01	0.84 ± 0.01	0.88 ± 0.01
Blue phase	0.96 ± 0.01	0.91 ± 0.01	0.95 ± 0.03
Beautifil			
Astralis 3	0.51 ± .08	0.53 ± 0.06	0.72 ± 0.07
Radii	0.69 ± 0.13	0.62 ± 0.13	0.83 ± 0.04
Blue phase	0.84 ± 0.01	0.86 ± 0.01	0.87 ± 0.01
Dyract AP			
Astralis 3	0.62 ± 0.04	0.53 ± 0.04	0.52 ± 0.02
Radii	0.54 ± 0.10	0.52 ± 0.02	0.69 ± 0.02
Blue phase	0.93 ± 0.03	0.86 ± 0.01	0.91 ± 0.01
Fuji II LC			
Astralis 3	1.11 ± 0.16	0.92 ± 0.07	0.85 ± 0.11
Radii	1.22 ± 0.17	1.02 ± 0.03	0.91 ± 0.01
Blue phase	1.15 ± 0.03	1.28 ± 0.03	1.02 ± 0.01

The top surfaces of Z250, Beautifil and Dyract AP were found to be significantly harder than the bottom surfaces ($p<.05$) for all LCUs and at all time intervals. On the other hand, a different trend was observed for Fuji II LC, where the bottom surface VHNs were significantly higher than the top ($p<.05$). Fuji II LC showed an exception to this trend in specimens cured with the QTH LCU at 15 minutes and 7 days and Radii at 15 minutes and 24 hours, where there was no significant difference between the top and bottom surfaces hardness values ($p>.05$)

DISCUSSION

Some reports indicated that a light intensity of at least 300 mW/cm^2 is required to adequately cure 2 mm-thick specimens of resin composite.[13-14] Other investigators questioned the clinical relevance of using a high-intensity curing light if longer exposure time, such as 40 seconds, was used.[15-16] However, it would be beneficial if the curing time of restorative materials can be reduced without adverse consequences.

Hardening was evaluated for three post-irradiation times, starting with 15 minutes. This short time interval was chosen in this study in order to better simulate the clinical situation, since restorations usually function shortly after their placement. Interestingly, in many published research studies, testing is conducted after 24 hours, which allows the specimens to reach their maximum hardening.[18]

In the current study, the microhardness technique was successfully used to produce accurate and readable indentations that were easy to measure for the GI-based materials after only 15 minutes from initiation of the setting. This is contrary to a previous investigation by Moon and others, which indicated that, at such an early stage, hardness measurements could not be used to accurately differentiate between curing techniques.[20]

The resin composite Z250 had RH values that were above 80% under all test conditions, even at the 15-minute time interval. This finding suggests that high intensity LED LCUs can reduce the time necessary for curing this microhybrid resin composite, which uses camphroqinoune as a photo-initiator. However, the same findings were not found for the PAMRC and Giomer materials that were tested. This can be attributed to the type and size of their fillers. PARMRC and

Giomer contain relatively large particles, which adversely affect their ability for light transmission. It seems that, in addition to the inherent properties of each material, such as its composition, particle size and shade, these materials require special, unique curing energy (intensity x time) to reach the ultimate curing level of RH that is equal to or higher than 80%. On the other hand, Fuji II LC (RMGI) had RH values of more than 100% in several measurements in this study. Perhaps this is due to the light curing of this material, which prohibits the acid/base reaction to proceed at the same rate at the top surfaces as compared to the subsurface deeper bulk of the specimens.[11-23] Despite the bottom surface receiving a lesser amount of energy, it had higher VHN values. This was more evident at the earlier testing time—15 minutes—than at later times. The positive clinical implication of this phenomenon is that the deepest part of restorations made with these materials will reach optimum RH at an early stage immediately after their insertion. At an early stage of RMGI development, most of their manufacturers stressed the importance of incremental placement of these materials.[24] However, findings related to this work do not support this concept, as their chemical reaction could serve as a significant advantage, allowing curing throughout the bulk of the restoration and, as a result, permit bulk application. This relates well to the *in-vitro* and *in-vivo* success of the so-called sandwich technique.[25-27]

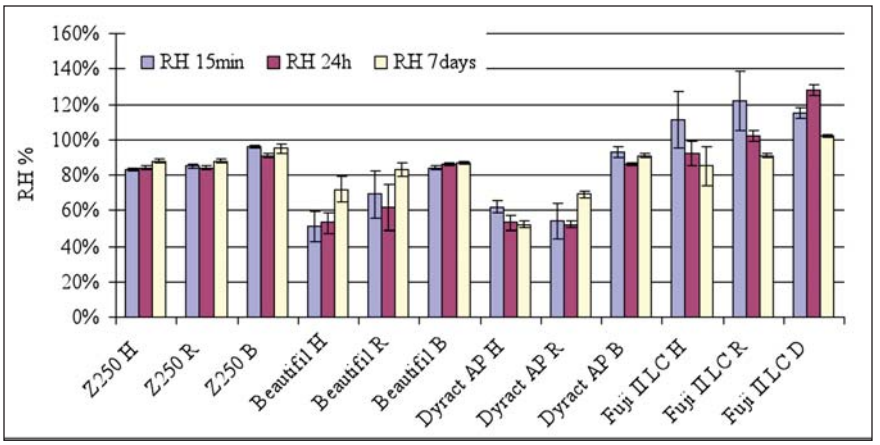

Figure 1: *Mean RH values of the investigated materials as a function of LCUs and post-irradiation time. The error bars represent the standard deviations. H: Astralis 3, R: Radii, B: Bluephase LCUs.*

Ferracane (1985) demonstrated good correlation between increasing hardness and increasing DC; however, he also concluded that an absolute hardness number could not be used to predict DC when different composites were compared.[28] Hence, RH ratios ranging from 89%-90% have been used as criteria for adequate conversion at a specific sample thickness.[29-30] These ratios are considered to reflect the relative extent of conversion of a deeper surface to that of the top exposed surface. This assumption has also been validated by Bouschlicher and others.[31]

In the current study, the recorded RH values dropped at certain time intervals, then increased at later ones. This drop was due to the uneven progression of polymerization in both the top and bottom surfaces over time; that is, the increase in the top surface hardness over such time surpassed that of the bottom surface. Therefore, reliance on RH alone as a sole indicator of curing effectiveness may be unreliable for these materials. The findings of this study support the work of Nakfoor and others, who stated that RH alone may be misleading, and actual hardness values must be examined along with RH.[32] Hence, it is wise to consider both the hardness values and RH and not depend on only one for the evaluation of curing effectiveness.

In the current study, post–irradiation time was found to have an effect on the hardness values of all tested materials with minor exceptions, that is, comparing means at 15 minute and 24-hour time intervals, which confirmed previous findings that resin composite does not reach the maximum degree of polymerization immediately after curing.[19,33] Following a specific period of time, the degree of polymerization of the resin composite leveled off to the maximum value in top and bottom surfaces regardless of differences in VHN immediately after curing among the different curing units. It seems that, after a certain degree of viscosity is reached, the diffusion rate of the free radicals slows down, therefore, it becomes more difficult for residual monomer to be involved in further reaction.[35] It should also be noted that, despite the significant differences recorded, post-irradiation polymerization cannot compensate for the lower degree of cure of resin composite attained at earlier setting stages.

The significant differences in the recorded VHN values and degree of cure among all the tested materials found in the current study were in agreement with the findings of Asmussen[12] and Chung.[35] Such differences are possibly attributed to variability in both matrix and fillers.

The similarities that were found in both the tested resin-composite (Z250) and PAMRC (Dyract AP), in that the top surface hardness was significantly higher than the bottom surface, was also in agreement with previously reported findings.[36-37] The Giomer material (Beautifil) revealed similar results in this respect. This could be due to a number of reasons. The shape, size, opacity, distribution and content of inorganic fillers can significantly affect light transmission through the

thickness of an increment of composite material. The more the light is distorted as it passes through the thickness of the increment, the lower the RH ratio.[38]

In general, the recorded hardness values of the tested materials in the current study were relatively higher than that recorded in other studies.[39-40] This may be due to differences in the methodology followed, such as storage conditions. In the current study, the specimens were stored dry, rather than wet.[5] Specimen storage under wet conditions might result in plasticizing the specimen surfaces through hydrolysis, which may affect the level of surface hardness.

Variability in the applied load by the Vickers indenter affects the obtained hardness readings. Therefore, in order to compare surface hardness values among studies, the applied load must be controlled.[41] While a Vicker's indenter was used in the current study, other studies used Knoop. However, Puskus and others stated that there is a high correlation between the hardness values obtained with both the Vicker and Knoop indenters.[42]

CONCLUSIONS

Under the conditions of the current study, it can be concluded that high-intensity LED LCUs applied for only 10 seconds resulted in relative hardness values that were either equivalent to or higher than those obtained with a control QTH LCU with 350 mW/cm^2 intensity applied for 40 seconds in the majority of test conditions.

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