ORIGINAL ARTICLE

Effect of Different Irradiation Times on Microhardness and Depth of Cure of a Nanocomposite Resin

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ABSTRACT

Objective: To determine the microhardness and depth of cure of nanocomposite using different irradiation times on both upper and lower surfaces of composite material.

Study Design: *In-vitro* experimental study design.

Place and Duration of Study: Dr. Ishrat-ul-Ebad Khan Institute of Oral Health Sciences, Dow University of Health Sciences and NED University of Engineering and Technology, Karachi, from March to May 2010.

Methodology: Total 120 cylinder shaped specimens; 60 specimens for depth of cure test and 60 specimens for micro hardness test were fabricated using A3 shade of nanocomposite (Filtek Z350 XT, 3M ESPE). For each irradiation time four groups were made (Group 1 = 20s), (Group 2 = 30s), (Group 3 = 40s) and (Group 4 = 60s). For each group fifteen specimens were used. The resin was placed and polymerized into a cylindrical plastic mold. Depth of cure was measured by using micrometer. Micro Vickers hardness was measured on both top and bottom surfaces. SPSS-16.0 was used for statistical analysis.

Results: There was statistically significant difference in the depth of cure between all groups showing the highest value in group 4 ($p < 0.001$). For hardness on top surface, there was a statistically significant difference in between groups 1 and 2 ($p=0.001$), groups 1 and 3 ($p < 0.001$), groups 1 and 4 ($p < 0.001$). There was no statistically significant difference between groups 2 and 3, groups 2 and 4 and in between groups 3 and 4. For hardness on bottom surface, there was statistically significant difference in between all groups showing the highest value in group 4 ($p < 0.001$).

Conclusion: Depth of cure and hardness was increased by increasing irradiation time. Hardness on the top surface was higher than bottom surface values.

Key words: *Nanocomposite. Depth of cure. Vickers hardness test. Irradiation time.*

INTRODUCTION

Composite resin has been widely used in dental restoration, due to low cost and conservative technique. To evaluate the elastic properties of composite resins, the methods which are mostly used are the Knoop and Vickers microhardness test.[1] Hardness tests are considered as an indirect method to evaluate the degree of polymerization of composite resins and which have already been reported to correlate with the degree of conversion of carbon double bonds.[2]

Depth of cure and microhardness are considered to be essential physical properties of composite resin materials that are relevant to the clinical technique of incremental packing and curing. Hardness is a property of a material that enables it to resist plastic deformation, usually by penetration. However, the term hardness may also refer to resistance to bending, scratching, abrasion or cutting.[3]

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Nanocomposite combine the advantages of hybrid and micro filled composite in the same restorative material showing favourable mechanical property, higher surface quality and increased wear resistance.[4] In addition to materials characteristics, light curing units significantly influence the degree of polymerization of light-activated composite resins. The most important features associated with the effectiveness of light curing seem to be the intensity of the light emitted the spectral output of the light source and the curing mode.[5]

Most LED equipments provide a power output of approximately 300 mW/cm^2 and have been reported to polymerize composite resins. LEDs are known to use less power, have a longer life and greater durability than conventional filament lamps. They have a narrow spectral range with a peak around 470 nm, which matches the optimum absorption wavelength for the activation of the camphorquinone photo initiator.[6] LED units generate minimal heat. The efficiency of conversion of electrical energy to useable curing energy is higher for blue LEDs than for conventional QTH lamps (14% vs. 1%, respectively).[7]

The irradiance of light emission depends on the power (Watts) of the curing unit as well as the time (seconds) and the surface area (cm^2) where the light is spread over. The energy density (irradiance x irradiation time)

influences the degree and depth of cure and the mechanical properties of light cured resin composite.[8] The degree of polymerization of these materials are basically proportional to the material thickness and irradiation time, which depend on some variables, such as type of material, composite shade, distance and quality of light source.[9]

The aim of this study was to determine the micro-hardness and depth of cure of nanocomposite material using different irradiation times and the difference in microhardness on both upper and lower surfaces of composite material.

METHODOLOGY

It was an *in-vitro* experimental study conducted at Dr. Ishrat-ul-Ebad Khan Institute of Oral Health Sciences, Dow University of Health Sciences and NED University of Engineering and Technology, Karachi, from March to May 2010. The specimens were fabricated for both depth of cure and Microhardness testing. After polymerization, 15 specimens were divided into each experimental group based on different irradiation times and then assessed for Microhardness and depth of cure test.

For depth of cure, total 60 cylinder shaped specimens were fabricated using A3 shades of nanocomposite (Filtek Z350 XT, 3M ESPE). For each irradiation time four groups were made (Group 1 = 20s), (Group 2 = 30s), (Group 3 = 40s) and (Group 4 = 60s). Fifteen specimens were used for each group. The resin was placed into a cylindrical plastic mold with a diameter of 4 mm and a depth of 10.0 mm. The mold was packed with the resin, the specimens were then covered with acetate strips on both top and bottom surface to prevent the resin rich or oxygen inhibition layer which would influence polymerization.[10]

A glass slide was placed on top of the mold and gentle pressure was applied to extrude excess materials and to achieve a flat test surface. Before light activation the intensity of curing unit was measured with a radiometer (Demetron, Kerr Corporation, USA). The resin was then photo-polymerized using conventional LED light source, with the tip of the light guided 0.5 mm from the surface of the resin. The specimens with inaccurate dimensions and inadequately cured on top surface were excluded.

The depth of cure of the resin was determined using a standardized technique (ISO 4049:2000). Immediately after irradiation, uncured material was scraped away with a plastic spatula. The height of the cylinder of set resin was measured with an electronic micrometer (Kanon Nakamura MFG EMS 12, Japan) to an accuracy of 0.01 mm, and the result was divided by two. Depth of cure was assessed. Each sample was measured three times and the mean value of these three readings was recorded as the depth of cure.

For microhardness test total 60 cylinder shaped specimens (4 mm diameter x 4 mm height) were fabricated, grouped and photo-polymerized in the same manner as described above for the depth of cure test. Total number of measurements for the test was 120 (60 from top surface and 60 from bottom surface). Each specimen gave 6 measurements 3 from top surfaces and 3 from bottom surfaces. After the irradiation the specimens were stored in distilled water for 24 hours at 37°C in an incubator. Since approximately 75% of the light-curing process occurs in the first 10 minutes, and the curing reaction can continue for a period of up to 24 hours.[11] The specimens were removed from the storage mediums, blotted dry and positioned centrally beneath the indenter of a digital microhardness tester.

Three Vickers indentations (load: 0.1 kg; dwell time: 15 seconds) were performed on both upper and lower surfaces of each specimen, using a hardness tester (Wolpert 402MVD) according to the ISO 6507-3:1998 specification. The mean value of these three individual measurements was taken as the value of overall surface. The measurement of the original Vickers hardness, was measured optically by the diameter of the impression, generated by the Vickers diamond itself.[12]

For statistical analysis the mean effect of different irradiation times on microhardness and depth of cure were compared by using one way ANOVA (parametric analysis of variances) and for significant effect parametric post Hoc Tukey type was employed at 0.05 significance level. For comparison between top and bottom surface hardness paired sample t statistics was used on SPSS 16.0.

RESULTS

Depth of cure and the Vickers microhardness are summarized in Table I.

Table I: A summary of results; numbers in each group being 15.

Time	Depth of cure (mm)	Hardness on top (kg/μm²)	Hardness at bottom (kg/μm²)
Group 1			
At 20 seconds			
Mean ±	2.4473 ±	62.7533 ±	27.3667 ±
Std. deviation	0.02840	3.58506	3.83251
Group 2			
At 30 seconds			
Mean ±	2.5753 ±	69.2600 ±	32.3067 ±
Std. deviation	0.06739	6.14443	4.03245
Group 3			
At 40 seconds			
Mean ±	2.7700 ±	72.6067 ±	38.8400 ±
Std. deviation	0.04811	3.11894	3.35917
Group 4			
At 60 seconds			
Mean ±	2.9413 ±	73.4600 ±	45.0400 ±
Std. deviation	0.08070	3.34040	2.01983

There was statistically significant difference in between all groups ($p < 0.001$). Depth of cure of a nanocomposite was the highest in group 4 at 60 seconds of irradiation time, followed by the group 3, group 2 and group 1. The lowest depth of cure was found in group 1 at 20 seconds of irradiation time.

For micro Vickers hardness on top surface, there was statistically significant difference in between groups 1 and 2 ($p=0.001$), groups 1 and 3 ($p < 0.001$), groups 1 and 4 ($p < 0.001$). There was no statistically significant difference between groups 2 and 3, groups 2 and 4 and groups 3 and 4. Top hardness means of nanocomposite were the highest in group 4 at 60 seconds of irradiation time and lowest in group1 at 20 seconds of irradiation time. For micro Vickers hardness on bottom surface, there was statistically significant difference in between all groups ($p=0.000$). Hardness on bottom surface of a nanocomposite was at its highest in group 4 at 60 seconds of irradiation time, followed by the group 3, group 2 and group 1. This shows the lowest depth of cure in group 1 at 20 seconds of irradiation time.

In paired sample t-test statistically significant differences were found between top and bottom surfaces of each composite with each irradiation time ($p=0.001$).

DISCUSSION

The use of LEDs for composite resin curing has increased because it produces a low increase in temperature during its use. They have a narrow spectral range which matches the optimum absorption wavelength for the activation of the camphorquinone.[3]

Depth of cure is considered as an essential physical property of composite resin and is widely used to evaluate the polymerization efficiency of light activated composite.[13] Schattenberg *et al.* reported that depth of cure of composite resins is mainly dependent on exposure time and the distance of the light guided tip of the light source from the composite resin.[14] McCabe *et al.* reported that an exposure time from 20 to 60 seconds can increase the depth of cure from 5% to 82%.[15] The results of this study were similar to those two studies showing significant increase in the depth of cure after increasing irradiation time.

Group 4 has the highest value of 3 mm depth of cure followed by group 3, group 2 and group 1. The lowest value of 2.44 mm was resulted from group 1, which was still higher than the value claimed by the manufacturers which is 2 mm in 20 seconds. This is contrary to a previous study conducted by Price *et al.* in 2003, who claimed that an exposure time of 40 seconds was considered to be a standard to cure a composite increment of 2.0 mm sufficiently over the years.[16]

In this study, nanocomposite material was used. At nano scale the strong interfacial interactions between the resin and fillers were demonstrated with high strength and high thermal stability which resulted in improved physical properties of the nanocomposite.[17]

According to ISO standard 4049:2009 the depth of cure of light cure resin should not be less than 1.5 mm. In this study, even the lowest value from the shortest curing time was 2.44 mm which was still higher than the value described in ISO standards.[18]

During light curing, the surface layer close to the light source is better polymerized than the layers far from it. Rueggeberg *et al.* in 1994 concluded that thick composite increments result in either partially or non-polymerized layers, which can compromise the quality of the restorations.[19] Lambrechts *et al.* reported that curing of composite in increments decreases polymerization shrinkage and provide complete polymerization which increases the hardness value.[20] Therefore, it can be concluded that in this study, if we would have placed composite resin incrementally than the increase in polymerization rate would have been resulted in increased hardness values.

Bouschlicher *et al.* in 2004 reported that in order to optimize clinical performance of restorations, the hardness of restorative material should at least be similar to that of the dentinal substrate not only superficially, but also in depth.[21] In this study, the top hardness values, resulted from group 3 (72.6 VHN) and from group 4 (73.46 VHN), were near to the value of dentine (80 VHN) in order to maintain the mechanical properties and marginal integrity of composite restoration.

On polymerization reaction kinetics, Murchison and Moore stated that application of the curing light for at least 40 seconds resulted in significantly higher hardness values than light-curing for 20 seconds.[22] In this study, the results were consistent with those of Murchison and Moore, the hardness value was increased by increasing curing time intervals. Top surface hardness value of composite in group 4 was resulted in the highest value of 73.46 HV.1 and in group 1 was resulted in the lowest value of 62.75 HV.1.

Calheiros *et al.* in 2006 reported that whenever a satisfactory degree of conversion is obtained, the superficial hardness is not further affected by the increase in time of exposure or composite shade.[23] This is in accordance with this study, there was no statistically significant difference in between group 2 and 3 and in between group 3 and 4 respectively.

Knezevicin *et al*. concluded that microhardness of the composite will reduce with increasing depth of resin.[25] As useable curing wavelengths are attenuated in the resin, less camphorquinone will be activated.[24] The results of this study were similar to that.

Katia in 2009 reported that with a 4 mm increment no light unit was able to promote satisfactory polymeri-

zation.[25] Similarly, this study results showed highly significant difference between the top and bottom surfaces. Top surface hardness values were consistently higher than those of the bottom surface values as a result of adequate polymerization.

This research will help dental professionals to understand the value of properties like hardness and depth of cure of composite. Both properties of a composite can be increased by increasing irradiation time and this in turn will improve the longevity of restorations.

In order to obtain more reliable results it would be advisable to place composite resin incrementally for specimen preparation in order to correlate with clinical situations. It has also been suggested to observe the affect of water or saliva storage on hardness of a nano-composite cured with different irradiation times.

CONCLUSION

Depth of cure and microhardness on both top and bottom surfaces were increased by increasing irradiation time. On top surface 30 seconds of irradiation time is enough to achieve sufficient hardness because there was no significant difference between 30 and 60 seconds of irradiation time. On bottom surface we can conclude that after 30 and 40 seconds of irradiation time hardness values remain half and after 60 seconds remain more than half when compared to the upper surface hardness values.

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