The Effect of Irradiation Time and Distance on the Microhardness of Three Commercial Nanohybrid Resin-Based Composites

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Received: September 13 2014
Accepted: October 27 2014

ABSTRACT

Purpose: The properties of resin-based composites as polymeric materials are related to the quality of polymerization. Microhardness measurement is an indirect method to predict this quality. Irradiation time and distance as factors related to light-curing process play important roles in this issue. The purpose of this study was to evaluate the effect of irradiation time and distance on the microhardness values of three different commercial nanohybrid resin-based composites.

Methods: A total of 180 disk-shaped specimens (60 specimens for each commercial resin-based composite) from three nanohybrid resin-based composites [Grandio (Voco), Simile (Pentron) and Tetric N- Ceram (Ivoclar Vivadent)] in A2 shade were prepared. The specimens were randomly subdivided in 6 subgroups (3 subgroups for evaluating irradiation time: 10 s, 20 s and 40 s, 3 subgroups for irradiation distance: 0 mm, 3 mm and 9 mm) which 10 specimens from each commercial resin-base composite were used for each subgroup. Vickers microhardness test was performed for the top and bottom surfaces of each sample using a microhardness tester under a 200 gr load and a dwell time of 15 s. Three random indentations were taken for each surface and a mean value was calculated. Data were analyzed by two and three way ANOVA and Tukey's post-hoc test at the 95% significance level.

Results: The microhardness values showed significant differences between subgroups for different irradiation times and distances (p value ≤ 0.001).).The only exception was Simile group which there was no significant difference for microhardness values between 0 and 3 mm distances. Grandio showed the highest microhardness values among others.

Conclusion: Increasing the irradiation time and decreasing the irradiation distance caused an increase in microhardness values. Also, the microhardness of the resin-based composites was affected by the chemical structure of the material.

Keywords:

Distance,
Irradiation,
Light Cure,
Microhardness,
Resin-Based Composite,
Time.

1. Introduction

Resin-based composites were introduced to dentistry in the early 1960s as restorative materials with better mechanical properties and clinical performances than acrylic resins and silicate-based materials [1]. Nowadays, because of the superior aesthetic properties, resin-based composites are the first choice in direct esthetic restorations and widely used in operative dentistry [2]. The first light curing systems for photo-activation of resin–based composites were devices emitting ultra violet light which were replaced by visible light systems such as QTH due to their adverse properties. Light curing systems must have the adequate efficiency to increase the conversion of monomer into polymer which is a critical factor to predict the quality of polymerization and as a result, the proper physical, mechani-

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cal and chemical properties of a resin-based composite restoration [3]. In addition to the factors related to the light curing process such as the size of the light-curing tip [4], irradiation time and distance, wavelength of the curing light and the intensity of light-curing units [5], the polymerization process is also influenced by the parameters related to the structure of resin-based composites. These parameters include the amount and type of monomer and coupling agent, the shade of resin-based composite [5], filler size and volume fraction, type and concentration of photo-initiator [6] and film thickness of resin-based composite [4].

Different direct and indirect laboratory tests have been used to evaluate the quality of polymerization process in resin-based composites. Hardness measurement is a widely used indirect method [7, 8]. The hardness is defined as the resistance of the material's surface against permanent deformation when a force is applied. In hardness test the area, width or depth of an indentation which is produced under a standard force by an indenter with a specific shape is measured by a microscope [1]. One of the recent advances in fabrication techniques of resin-based composites is the application of nanotechnology. In this method, the structures with the dimensions as small as 1 to 100 nm can be fabricated which have special physical and chemical properties due to their very small sizes. Some properties including more surface smoothness, better polishability and color characteristics have been attributed to the nanocomposites in comparison with microfilled and microhybrid composites whereas the flexural strength and microhardness are comparable with conventional posterior resin composites [9]. Two main groups of dental nanocomposites containing nanofiller particles are nanofills and nanohybrides which large particles of 0.4 to 5 micron with added nanometer sized particles are presented in the latter [1].

Irradiation time is an important factor in light curing process which has been studied in many researches [4, 10, and 11]. In the study of Ceballos et al. [10] the effect of different curing times (20 s and 40 s) with different light sources (LED and QTH) on the Vickers microhardness of two different composite were evaluated. The results showed that increasing the irradiation time had no effect on the microhardness values up to 2.5 mm depth of composite but in higher than 2.5 mm there was an increase in Vickers microhardness. However, Alpöz et al. [11] ascertained that LED with the irradiation time of 40 s had higher Vickers microhardness than LED with the irradiation time of 20 s for the top surfaces.

Another factor affecting the light curing process is the distance between the light curing tip and the surface of resin-based composite. Since this parameter depends on the form and size of cavity [12], it cannot be controlled well. Zhu and Platt [13] evaluated the knoop microhardness values of resin-based composites with three different light sources and different irradiation distances of 0, 3, 6, 9, 12 and 15 mm. There was a reduction in microhardness values by increasing the distance. Aguiar et al. [14] concluded that the top surfaces were not influenced significantly by increasing the irradiation distance, but there was a significant reduction in Knoop microhardness values of bottom surfaces which were light-cured at a distance of 8 mm compared with 2 mm and 4 mm.

According to these controversies, the purpose of this study was to evaluate the effect of irradiation time and distance on microhardness values of three different commercial nanohybrid resin-based composites.

2. Materials and Methods

Three commercial nanohybrid composite [Grandio (Voco), Simile (Pentron (and Tetric N- Ceram (Ivoclar Vivadent)] in A2 shade were used in this study. Table 1 shows the compositions of resin-based composites.

2.1. Sample Preparation

A total of 180 disk-shaped specimens (10 mm diameter x 2 mm length) were fabricated in a teflon mold according to manufacturers' instructions where 60 specimens belonged to each commercial resin-based composite. Then, the specimens were photopolymerized with a QTH (Coltolux ® 75-Germany) light-curing unit. The light intensity of light-curing unit was measured with a radiometer (Optilux, Model 100, 10503, Kerr, USA), which was over 600 mW/cm2. For sample preparation the molds were placed on mylar strip on a glass slab and then were filled with resin-based composite and packed with a proper condenser. Subsequently, the resin-based composite was covered with another mylar strip and pressed with a glass slide to extrude excess material. The specimens were light-cured in close contact with their surfaces through the top mylar strip. The specimens were polished with a sequence of 600, 800 and 1200 grit silicon carbide paper under wet conditions and stored in distilled water in a dark incubator at 37 ˚C for 24 h to complete the polymerization process.

To evaluate the efficacy of irradiation time, a total of 90 specimens were evaluated. The 30 specimens for each commercial resin-based composite were randomly

Table 1. Compositions of tested resin-based composites.

Resin-based composite	Manufacture	Matrix	Filler type and size	Filler content (vol. %)	Manufacturer's recommended curing time
Grandio	Voco GmbH Cux-haven Germany	BisGMA, UDMA, TEGDMA, DMA	Glass–ceramic (1μm), SiO2(20-60nm)	71.4%	20 s
Simile	Pentron Clinical Technologies, Wallingford, USA	PCBisGMA, BisGMA,	Barium boro-silicate glass, nanoparticulate silica, zirconium silicate (5-20nm),Glass–ceramic SiO2 (0.04-0.7)	68%	10 s - 20 s
Tetric N-Ceram	Ivoclar/ Vivadent	UDMA, HDDMA	Barium aluminium silicate glass(0.4μm,0.7μm), ytterbium trifluoride(200nm), mixed oxide(160nm), Prepolymer	55–57%	20 s

subdivided in 3 subgroups which 10 specimens were used for each one (group 1 = curing time 10 s, group 2 = curing time 20 s and group 3 = curing time 40 s).

To evaluate the efficacy of irradiation distance, a total of 90 specimens were evaluated which 30 ones belonged to each commercial resin-based composite. The 30 specimens for each commercial resin-based composite were randomly subdivided in 3 subgroups which 10 specimens used for each one. (group1 = curing distance 0 mm, group 2 = curing distance 3 mm, group 3 = curing distance 9 mm). Two metal rings with the height of 3 and 9 mm were used to control the light-curing tip distance. In order to attain the 0 mm distance, the specimens were light-cured in close contact with their surfaces through the top mylar strip which was approximately 1 mm thick. It is worth mentioning that all the specimens were cured for 20 s.

2.2. Microhardness Test

Vickers microhardness test was performed for each sample at the top and bottom surfaces using a microhardness tester (Bareiss Prüfgerätebau GmbH, D-89610 Oberdischingen, Germany) under a 200 gr load and a dwell time of 15 s. Three indentations with the random distance of 1 mm were taken for each surface and a mean value was calculated. The microhardness was determined through the measuring the diameters of indentation which was produced by pyramidal square-base diamond indenter.

2.3. Statistical Analysis

Two and Three way ANOVA analysis with independent variables including commercial kind of resin-based composite (three variables), curing time (three variables), curing distance (three variables) depth of cure (two variables, top and bottom) Tukey's Post-hoc test with significance level of 95% were performed.

3. Results

3.1. Irradiation Time

Table 2 shows the Vickers microhardness for the top and bottom surfaces of three commercial resin-based composites with three different irradiation times. ANOVA analysis showed that the microhardness values were influenced by resin-based composite brand irradiation time ($p \leq 0.001$). The microhardness values on both top and bottom surfaces showed statistically significant difference between three subgroups. Therefore, increasing the irradiation time was an effective factor for improving the microhardness values ($p \leq 0.001$). Regardless of different variables, the mean values for top surfaces were more than bottom in all specimens. For top surfaces, the highest and lowest microhardness values were observed in Grandio groups with the irradiation time of 40 s (Microhardness: 119.32 VHN) and Tetric N- Ceram groups with the irradiation time of 10 s (Microhardness: 45.87 VHN), respectively. Grandio with 40 s irradiation time (Microhardness: 113.26 VHN) and Tetric N- ceram with 10 s irradiation time (Microhardness: 30.46 VHN) showed the highest and lowest microhardness values on bottom surfaces, respectively. The values of Vickers microhardness ratio (bottom/top) are presented in Table 2 where the highest value was 94% for Grandio with the irradiation time of 40 s and the lowest one was 66% for Tetric N-Ceram with the irradiation time of 10 s.

Table 2. The microhardness values and ratios % (bottom/top) for different irradiation times.

Composites \ Times		10 s Mean (SD)	10 s Ratio %	20 s Mean (SD)	20 s Ratio %	40 s Mean (SD)	40 s Ratio %
Grandio	Top	107.08 (4.76) VHN	83 VHN	115.92 (4.28) VHN	90 VHN	119.32 (6.16) VHN	94 VHN
	Bottom	89.09 (6.39) VHN		105.07 (6.25) VHN		113.26 (7.07) VHN	
Simile	Top	61.74 (3.87) VHN	70 VHN	67.75 (5.17) VHN	88 VHN	72.32 (4.33) VHN	90 VHN
	Bottom	43.48 (7.63) VHN		59.70 (4.53) VHN		65.13 (3.55) VHN	
Ttric N-Ceram	Top	45.87 (3.59) VHN	66 VHN	53.00 (2.27) VHN	82 VHN	56.46 (4.63) VHN	90 VHN
	Bottom	30.46 (4.86) VHN		43.71 (2.89) VHN		51.93 (4.15) VHN	

3.2. Irradiation Distance

Vickers microhardness for three resin–based composites and different irradiation distances are summarized in Table 3. There was statistically significant difference between the groups based on the type of resin-based composite and irradiation distance ($p \leq 0.001$). Microhardness values were statistically influenced by different irradiation tip distances and ,therefore, reducing the irradiation distance played an important role in improving microhardness on both top and bottom surfaces ($p \leq 0.001$). The only exception was Simile group where there was no significant difference for microhardness values between 0 and 3 mm distances. According to Table 4, the highest and lowest mean microhardness values for top surfaces were 115.92 VHN and 45.36 VHN for Grandio with 0 mm irradiation distance and Tetric N-Ceram with 9 mm irradiation distance, respectively. Grandio with 0 mm irradiation tip distance (Microhardness: 105.07 VHN) and Tetric N-Ceram with 9 mm irradiation tip distance (Microhardness: 31.41 VHN) showed the highest and lowest bottom microhardness values, respectively. The values of Vickers microhardness ratio (bottom/top) for three resin-based composites are shown in Table 3. Vickers microhardness ratio for light-cured Grandio at a distance of 0 mm (90%) was the highest one and light-cured Tetric N-Ceram at a distance of 9 mm showed the lowest one (69%). Regardless of the type of the resin-based composite or irradiation distance, the top microhardness values were higher than bottom ones in all specimens.

4. Discussion

There are different laboratory tests for evaluating the polymerization of the resin -based composites [7, 8]. Some of these tests including differential thermal analysis (DTA), infrared spectroscopy (FTIR) and Ra-

Table 3. The microhardness values and ratios % (bottom/top) for different irradiation distances.

Composites \ Distances		0mm Mean (SD)	0mm Ratio %	3mm Mean (SD)	3mm Ratio %	9mm Mean (SD)	9mm Ratio %
Grandio	Top	115.92 (4.76) VHN	90 VHN	105.70 (4.45) VHN	87 VHN	97.46 (5.69) VHN	88 VHN
	Bottom	105.07 (6.25) VHN		92.75 (6.81) VHN		86.67 (7.87) VHN	
Simile	Top	67.25 (5.17) VHN	88 VHN	64.55 (6.10) VHN	84 VHN	60.21 (5.93) VHN	70 VHN
	Bottom	59.70 (4.53) VHN		54.69 (7.02) VHN		42.74 (6.87) VHN	
Ttric N-Ceram	Top	53.00 (2.27) VHN	82 VHN	49.4 (2.31) VHN	74 VHN	45.36 (4.67) VHN	69 VHN
	Bottom	43.71 (2.89) VHN		36.83 (3.33) VHN		31.41 (3.59) VHN	

man spectroscopy determine the degree of conversion as direct tests. The others including microhardness measurement, optical microscopy and scrapping test evaluate the efficacy of polymerization indirectly through the depth of cure measuring [15]. As shown in literatures, microhardness is a good predictor of degree of conversion and also a good indicator to determine some mechanical properties of the materials such as strength and stiffness [16, 17]. However, some studies such as Taher [18] found no accurate correlation between the hardness and degree of conversion. Furthermore, Bouschlicher et al. [17] refused to support any relation between these two parameters with composite formulation.

The purpose of this study was the evaluation of important curing parameters like irradiation time and distance on microhardness values of three different commercial nanohybrid composites. The results of current study showed that light-cured Grandio at a distance of 0 mm for 40 s yielded the highest value. Microhardness test evaluates the efficacy of polymerization indirectly. Appropriate polymerization in different depths of a resin-based composite restoration depends on the bottom to top microhardness ratio which should be higher than 80%. For the ratios of 80% or more, the degree of polymerization or bottom to top conversion (DC) is approximately 90% or more which means 90% of ultimate conversion has been occurred at the top surface of resin based composite [16]. According to the study of Yap et al. [19], in the proportion of 100% the polymerization will be considered as a completely effective process. However, the ratios about 80% are also adequate. In current study, the values of microhardness ratio were higher than 80% for all groups with 20 s and 40 s irradiation times, but with the irradiation time of 10 s only Grandio passed the 80%. In evaluating the effect of distance, the microhardness ratios for all groups in the distance of 0 mm were higher than 80%, but the microhardness ratio for Tetric N- Ceram with the irradiation distance of 3 mm and for both Tetric N- Ceram and Simile with the distance of 9 mm were less than 80%. Different chemical compositions of matrix and also the size and distribution of fillers in resin-based composites can change this proportion. Similar to other studies, there were higher microhardness and degree of conversion values for top surfaces in comparison to bottom surfaces [4, 20 and 21]. The explanation is that the reduced microhardness value for bottom surfaces is directly related to the attenuation in light intensity due to the light scattering while passing through the composite mass.

Hansen and Asmussen showed that the cavity depth is most often 4-5 mm in lower premolars, 5-6 mm in upper premolars and lower molars, and 5-7 mm in upper molars. They also mentioned that 15% of the cavities in upper molars are 28 mm deep [22]. In addition to these findings, the most common recommendation is to consider 1mm distance between the resin composite and light curing tip [23]. In the study of Caldas et al. [24], the effect of different irradiation distances (0, 6 and 12 mm) were evaluated. They concluded that the surface hardness will be decreased by increasing the irradiation distance which is similar to the findings of current study. In the present study the only exception was Simile group which there was no significant difference between 0 and 3 mm distances. In the study of Aguiar et al. [14], no significant difference was shown between 2 and 4 mm distances, but the difference with 8 mm was significantly high. It has been shown that 1 mm reduction in distance leads to 10% reduction in light intensity. [25]

The time variable was also evaluated in this study. The hardness of resin composites is almost constant for the curing times above 40 s [26]. The manufacturer's recommended curing times per 2mm layer are shown in Table 1. The results of this study showed a significant improvement in microhardness by increasing the irradiation time from 10 s to 40 s. It is noticeable that the findings are consistent with a study by Lima et al. [21] who evaluated the effect of curing time (20 s and 40 s) and curing device on the knoop microhardness values of a nanofilled resin composite. They reported that increasing the curing time from 20 s to 40 s increased the microhardness but had no effect on the degree of conversion. However, they stated that because there was no attenuation in light intensity at the top surface, increasing the irradiation time was effective on bottom surfaces more than top ones. One of the impacting factors on composite DC and indirectly on hardness is the shade of composite. In the study of Anfe et al. [27], the microhardness values were influenced by translucency of resin-based composites [5]. To ensure the accuracy of exam, all the used in this study were selected of A2 shade. According to the obtained data, the microhardness values for Grandio were higher compared to the other resin-based composites. Although all were nanohybrid composites, it can be interpreted with regard to the different compositions of material.

The findings of our study are consistent with Cekic-Nagas et al. [12] and Mota et al. [28] which ascribed the higher microhardness of Grandio to its higher filler loads instead of filler size. The compositions of the examined resin-based composites are shown in Table 1. The microhardness values of resin-based composites are directly related to the filler contents [29]. The results of the current study showed this following ranking which is similar to their filler contents by volume (Grandio > Simile > Tetric N- Ceram). In addition to the filler contents, the higher microhardness of Grandio is related to the existence of fillers with large particles [30]. Regarding to the study of Hahnel et al [31], by increasing the filler content, the mechanical properties will be improved due to a stronger interfacial bond between the resin matrix and filler particles. All of the resin-based composites have a minimum of 60 vol% filler particles. In the commercial formulae of Grandio, there is 71.4% vol% fillers (SiO_2 nanoparticles) added to larger particles of 1.5 µm barium allumino boro silicate. Poggio et al. [32] ascribed the higher microhardness values of Grandio to the higher filler content and large particles. The microhardness is also influenced by the composition of resin-matrix. Unlike Simile, Grandio contains TEGDMA, which is a monomer with smaller structure in dimensions in comparison with the Bis-GMA and UDMA and according to the study of Sideridou et al. [33], it has higher DC than the other two. Moreover, it has been shown that the higher microhardness of Grandio is due to TEGDMA in resin monomer formulation which decreases the viscosity and with increasing the further reaction of monomer [17]. According to the study of Moraes et al. [34] and ours, the different composition of composites explains the significant statistical differences between the microhardness values.

5. Conclusions

In conclusions, the microhardness values and consequently, the mechanical properties of the resin-based composites will be improved by increasing the irradiation time and decreasing the irradiation distance. In addition to the factors related to the light-curing process, the microhardness of a nano hybrid resin-based composites was affected by the chemical structure of the material.

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