Original article

Polymerization behavior of resin composites using different irradiance lights

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Abstract

Purpose: This study aimed to evaluate the microhardness and hardness ratios of hybrid resin composites using different irradiance lights.

Materials and Methods: Light-curing units were light emitting diode (LED) light-curing unit (Demi Ultra). The light-cured resin composites were Clearfil AP-X (shade A3) and Clearfil Photo Bright (shade US). Composite specimens of 2-mm thickness were polymerized in Teflon molds using an energy density of 24,000 mJ/cm². Light irradiances were 1,200 mW/cm² 20 s and 600 mW/cm² 40 s. Just after light curing, the Knoop hardness (KHN) was measured at the top and bottom surfaces of each specimen using a hardness tester. The hardness ratio was calculated as follows: KHN of bottom surface/KHN. All experiments were performed at room temperature of 23 ± 2°C with humidity of 50 ± 10%.

Results: Immediately after light curing, the KHN at the bottom surfaces of resin composites was significantly lower than that at the top surfaces for every group ($p < 0.05$). The 1,200 mW/cm² 20 s showed significantly smaller hardness ratio compared with that of the 600 mW/cm² 40 s for both Clearfil AP-X and Clearfil Photo Bright resin composites ($p < 0.05$).

Conclusion: When the energy density was the same, the polymerization of resin composites at the bottom surface was more inhibited than that of at the top surface using high-irradiance light.

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Key Words: energy density, irradiance, Knoop hardness, polymerization, resin composite

Introduction

Resin composite polymerization results in volumetric shrinkage, and the stress created leads to the formation of a greater gap between the resin and cavity surfaces [1,2]. Such marginal gaps and consequent microleakage may cause marginal staining, postoperative sensitivity [3,4], and secondary caries.

On the other hand, when the bond strength exceeds the polymerization shrinkage stress, a crack is initiated in the tooth structure, usually the enamel [5-7], leading to direct communication with the oral cavity. It was reported that the white margin represented cracks in the enamel around resin composite restorations [7]. These cracks were located 30-50 µm from the composite-enamel interface, as observed using environmental scanning electron microscopy (SEM) under the 100% water-vapor-saturated condition [7].

Alternatively, increasing the velocity of light-cured resin composites decreased the composite adaptation to the cavity wall when a resin composite of a different composition was used [8]. Therefore, the polymerization rate has a significant effect on strain development. The internal hardness of the cured resins increased with argon ion laser output along with increasing intensity on wavelengths similar to those of light-curing units, but the maximum hardness was not always increased [9]. The use of an intense light source may lead to more frequent marginal and wall gap formation [2,10]. Moreover, maximum light irradiance of up to 2,000 mW/cm² may lead to heat generation, which might harm the pulp and gingiva [11-13].

Light-cured composites are usually polymerized from the resin surface near a light source, which causes polymerization-induced contraction of the resin toward the light. A slow-start light curing method to decrease curing stress was used to cure the composite with an initial low irradiance light, which was subsequently followed by high irradiance light. Excellent marginal sealing and cavity adaptation were achieved using this method [2,7,14-17]. Previous work had shown that when a composite was light cured with an initial irradiance of 270 mW/cm² for 10 s, a 5-s interval, and then an irradiance of 600 mW/cm² for 50 s. The resin composite hardened earlier at the cavity base than at the surface [2,18], and improved light-cured resin composite adaptation to the cavity wall [2,7,16,17]. This means that the homogenous polymerization of resin composites improved the resin composite adaptation to the cavity wall [2].

Thus, the measurement of resin composite hardening (in other words, degree of conversion at the cavity base) is important for resin composite adaptation to the cavity wall. The direct method of quantifying the degree of conversion is through infrared spectroscopy [19]. However, infrared spectroscopy is not a suitable technique for measuring the polymerization of resin composites immediately after the light cure, because the technique is time-consuming. The microhardness of resin is an indicator of the degree of conversion [19, 20], and a high correlation between the Knoop hardness (KHN) and infrared spectroscopy [19] has been reported. The hardness ratio was calculated as KHN of the bottom surface/KHN of the top surface [21].

The purpose of this study was to test the hypothesis that when the energy density is the same, polymerization of resin composites at the top and bottom surface and the hardness ratio are not affected using different light irradiance.

Materials and Methods

The materials, components, manufacturers, and batch numbers used in this study are listed in Table 1. The shades of resin composites used in this study, A3, correspond to Vita classical shade (Vitapan Classical, Vita Zahnfabrik, Bad Zäckingen, Germany). The light curing unit used was an LED light-curing unit (Demi Ultra, Kerr, Orange, CA, USA). The irradiance of the LED light curing unit was measured using an L.E.D. radiometer (Demetron, Kerr, Orange, CA, USA). The light tip diameter of the LED light-curing unit was 8 mm. However, the diameter of a curing radiometer was 7 mm. Then, the light tip diameter was changed from 8 mm to 7 mm using black masking tape.

Table 1 Study materials

Material	Components ^a	Batch No.	Manufacturer
Clearfil AP-X (AP) Shade: A3	silanated barium glass filler, silanated silica filler, silanated colloidal silica, Bis-GMA, TEGDMA, photoinitiator, catalyst, accelerator, pigments, others, Filler load: 84.5 wt%	9B0091	Kuraray Noritake Dental
Clearfil Photo Bright (PB) Shade: US	prepolymerized organic filler, silanated silica filler, silanated colloidal silica, silanated silica glass filler, Bis-GMA, TEGDMA, urethane tetramethacrylate, hydrophilic aliphatic dimethacrylate, photoinitiator, catalysts, accelerators, pigments, others, Filler load: 82.0 wt%	690008	Kuraray Noritake Dental

^aAbbreviations: Bis-GMA, bisphenol A-glycidyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate

The resin composites were polymerized using the two light-curing techniques; (1) LED 1,200 mW/cm² (light tip-resin distance: 0 mm) for 20 s; (2) LED 600 mW/cm² (light tip-resin distance: 6 mm) for 40 s. Hybrid-type resin composites of Clearfil AP-X (shade A3: Kuraray Noritake Dental, Tokyo, Japan) and Clearfil Photo Bright (shade US: Kuraray Noritake Dental) were placed in a Teflon mold (wide, 3 mm; long, 7 mm; and deep, 2 mm) with polyethylene strips at the bottom surface, and the composite was covered with polyethylene strips and slide glass to prevent the formation of an oxygen inhibited layer. Then, the resin composite was polymerized using the two curing techniques described. Immediately after completion of the light curing, Knoop hardness (KHN) measurements were obtained from the top and bottom surfaces of each of the two resin specimens using a load of 100 g and a dwell time of 15 s. (Hardness tester, model MVK-E, Akashi, Kawasaki, Japan). Microhardness was measured at the center of the resin specimen surface. In the case of measurement at the bottom surface of the resin, specimens were taken out of the mold as rapidly as possible following completion of the light curing and turned over. Thereafter, the Knoop hardness was measured. The Knoop hardness measurements were performed at the top and bottom surfaces of each specimen ($n = 6$). Knoop hardness results were compared and analyzed using the Kruskal-Wallis test and Bonferroni/Dunn test. The hardness ratio [21] was then calculated. All experiments were performed at room temperature of $23 \pm 2^\circ\text{C}$, with humidity of $50 \pm 10\%$.

Results

Microhardness and hardness ratio

Knoop hardness results of the top and bottom surfaces of resin specimens together with the Bonferroni/Dunn test comparisons are shown in Table 2, and the Kruskal-Wallis test result was ($p < 0.01$). The results of the hardness ratio and the statistical comparisons are shown in Table 3.

Table 2 Knoop hardness number (KHN) at the top and bottom surfaces of resin composite

Light curing method		LED: 1,200 mW/cm ² 20 s		LED: 600 mW/cm ² 40 s	
Material		Mean (SD)	Median (IQR)	Mean (SD)	Median (IQR)
Clearfil AP-X	Top	65.2 (1.2) ^{a, A}	64.6 (63.8-66.4)	59.5 (1.2) ^{b, A}	59.5 (59.1-60.2)
	Bottom	56.7 (1.2) ^a	56.9 (56.7-57.0)	55.7 (1.7) ^b	56.1 (55.2-56.6)
Clearfil Photo Bright	Top	38.0 (0.8) ^a	38.3 (37.4-38.5)	39.7 (1.7) ^b	39.0 (37.9-40.2)
	Bottom	33.8 (1.5) ^{a, B}	33.7 (32.3-34.4)	36.7 (1.1) ^{b, B}	36.5 (36.2-37.5)

Intergroup data designated with the same lowercase letters for each top and/or bottom hardness are significantly different ($p < 0.05$). Intergroup data designated with the same uppercase letters for each light curing method are significantly different ($p < 0.05$).

Table 3 Hardness ratio of resin composite

Light curing method	LED: 1,200 mW/cm ² 20 s	LED: 600 mW/cm ² 40 s
Material	Mean (SD)	Mean (SD)
Clearfil AP-X	0.88 (0.01) ^a	0.94 (0.01) ^a
Clearfil Photo Bright	0.88 (0.01) ^b	0.94 (0.02) ^b

Intergroup data designated with the same lowercase letters for each irradiation are significantly different ($p < 0.05$).

Immediately after light curing, the KHN at the bottom surfaces of resin composites were significantly lower than that of at the top surfaces for every group ($p < 0.05$). Those hardness ratios were <1 . The KHN at the top surface of Clearfil AP-X using 1,200 mW/cm² for 20 s was significantly higher than that of 600 mW/cm² for 40 s ($p < 0.05$). The KHN at the bottom surface of Clearfil Photo Bright using 1,200 mW/cm² was significantly lower than that of 600 mW/cm² for 40 s ($p < 0.05$). The 1,200 mW/cm² for 20 s showed a significantly smaller hardness ratio than that of 600 mW/cm² for 40 s for both Clearfil AP-X and Clearfil Photo Bright resin composites ($p < 0.05$).

Discussion

After the light curing, hardening at the bottom surface of the resin composite specimens was significantly lower than that at the top surface for every group. Light-cured composites are usually polymerized from the resin surface near a light source. Therefore, the micro-hardness at the top surface resin composite was significantly higher than that of at the bottom surface [21]. The Knoop hardness at the top surface of the Clearfil AP-X using 1,200 mW/cm² for 20 s was significantly higher than that obtained using 600 mW/cm² for 40 s. This result suggested that higher irradiance light provides higher degree of conversion [22]. The Knoop hardness at the bottom surface of the Clearfil Photo Bright using 1,200 mW/cm² for 20 s was significantly lower than that of 600 mW/cm² for 40 s. Both energy densities were 2,400 mJ/cm². It was reported that optimal irradiance led to maximum hardness in the resin body [9]. Moreover, light transmission through the light-cured resin composite is strongly affected by the opacity of the resin composite. This opacity is different before and after the resin composite is cured. The opacity of the resin composite is indicated by the contrast ratio [22]. The contrast ratio is the ratio of reflectance backed by black and white standards, with the identical sample backed by a white background and added to the diffuse reflection from the sample. The increased diffuse reflection is referred to as the luminous reflectance of the sample backed by the white background. The greater the transparency of the sample disk, the greater the increase in diffuse reflection at the white background; contrast ratio takes a smaller value. A completely opaque material hides both the white and black backgrounds, resulting in a contrast ratio of 1. The contrast ratio for a translucent material ranges from 0-1 [22]. Almost all resin composite materials decrease the contrast ratio (increase transparency) during polymerization. It was thought this type of resin composite showed a slight acceleration of curing of the composite at the bottom surfaces when using a low-irradiance light. The contrast ratio of Clearfil AP-X decreased during polymerization (increasing transparency), while that of Clearfil Photo Bright increased during polymerization (increasing opacity) [18]. When a material with light reflectance of the filler is close to that of the resin composite monomer (polymer), the transparency of the resin composite is increased. Therefore, it was suggested that the light reflectance of the resin polymer of Clearfil AP-X and Clearfil Photo Bright was considerably different from that of the filler after curing.

The 1,200 mW/cm² for 20 s showed a significantly smaller hardness ratio (0.88) than that of the 600 mW/cm² for 40 s (0.94) for both Clearfil AP-X and Clearfil Photo Bright resin composites. This result supported the fact that the hardness of resin composite samples was highly correlated with the bottom-to-top degree of conversion ratios using the FTIR method, and was independent of the filler and filler loading [23]. It was reported when delivering a similar radiance exposure of 37 J/cm², a QTH of 936 mW/cm² for 40 s and an LED of 825 mW/cm² for 20 s units achieved a greater depth of cure than the PAC 7,328 mW/cm² for 5 s light [24]. It was concluded that a lower irradiance of 600 mW/cm² for 40 s produced a more uniform hardening of both resin composites. It was reduced light energy density post-gel contraction while maintaining the degree of conversion in the resin composite [25].

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Conflicts of Interest

Junji Tagami received honorarium from Kuraray Noritake Dental.

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