Original article

Resin composites top/bottom hardness and different light cure

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Abstract**Purpose:** The aim of this study was to evaluate microhardness and hardness ratio of two type hybrid resin composites using different irradiance light sources.**Materials and Methods:** Light curing units were an LED light-curing unit and an experimental quartz-tungsten-halogen (QTH) light-curing unit. The light-cured resin composites were Clearfil AP-X (shade A3) and Estelite Σ Quick (shade A3). Composite specimens of 2-mm thickness were polymerized in Teflon molds using an energy density of 24,000 mJ/cm². Light curing methods were LED 1,200 mW/cm² for 20 s and QTH 600 mW/cm² for 40 s. Just after light curing, the Knoop hardness was measured at the top and bottom surfaces of each specimen. The hardness ratio was calculated as follows: Knoop hardness of bottom surface/Knoop hardness of top surface.**Results:** Immediately after light curing, the Knoop hardness at the bottom surfaces of resin composites was significantly lower than that at the top surfaces with 1,200 mW/cm² 20 s for both resin composite ($p < 0.05$). There is no significant difference between the Knoop hardness at the top surfaces and the bottom surfaces with 600 mW/cm² 40 s for both resin composite ($p > 0.05$). The 600 mW/cm² 40 s showed significantly greater hardness ratio compared with that of the 1,200 mW/cm² 20 s for both resin composite ($p < 0.05$).**Conclusion:** The polymerization of resin composites at the bottom surface was inhibited compared with that of at the top surface using the regular irradiance of LED light.

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Key Words: energy density, hardness ratio, irradiance, Knoop hardness, resin composite**Introduction**

Resin composite polymerization results in volumetric shrinkage, and the stress created leads to a 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. 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 [5]. Therefore, the polymerization rate has a significant effect on strain development. It was reported that maximum flexural strength and modulus of light-cured resin composite was obtained by intermediate irradiance at the same energy density [6]. The use of an intense light source may cause more frequent marginal and wall gap formation [2,7,8]. Moreover, high irradiance of up to 2,000 mW/cm² lead to heat generation and that may harm the pulp [9,10,11].

Light-cured composites are usually polymerized from the resin surface near a light source, which causes polymerization-induced shrinkage of the resin toward the light. A slow-start light curing method (an initial low irradiance light, subsequently followed by high irradiance light) decreased curing stress, and improved marginal sealing and cavity wall adaptation [2,8,12,13,14,15,16]. Previous work had shown that when a composite was light cured with 270 mW/cm² 10 s + 5-s interval + 600 mW/cm² 50 s using an experimental quartz-tungsten halogen light-curing unit. The resin composite hardened earlier at the cavity base than at the surface [2,17], reduced volumetric shrinkage of resin composite restoration in the cavity [16]. This means that the homogenous polymerization of resin composites improved the resin composite adaptation to the cavity wall [2].

Thus, measurement of resin composite hardening is important for resin composite adaptation to the cavity wall. The direct method of quantifying the degree of conversion is infrared spectroscopy [18]. However, infrared spectroscopy technique is time-consuming. The microhardness of resin is an indicator of the degree of conversion [18,19], and a high correlation between the Knoop hardness (KHN) and infrared spectroscopy has been reported [18]. The hardness ratio was calculated as KHN of the bottom surface/KHN of the top surface [20]. The energy density is the important factor in degree of conversion of the light-cured resin composite. The energy density is calculated that irradiance multiplied by irradiation time. It was reported that conversion and material properties were the same result from similar energy density [21,22,23]. On the other hand, irradiance and irradiation time independently influenced degree of conversion and mechanical properties [6,24,25].

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 surface, bottom surface and the hardness ratio are not affected using different irradiance light source.

Materials and Methods

The materials, components, manufacturers, and batch numbers used in this study are listed in Table 1. The light curing units used were an LED light curing unit (Demi Ultra, Kerr, Orange, CA, USA) and an experimental quartz-tungsten-halogen (QTH) light curing unit (GC Corp., Tokyo, Japan) connected to a slide regulator. This QTH light curing unit has a control system of lamp voltage and the light radiant exitance was adjustable. The irradiance of the LED light curing unit was measured using a radiometer (L.E.D. radiometer, Demetron/Kerr, Orange, CA, USA) and an experimental QTH light curing unit was measured using a radiometer (Model 100 Optilux radiometer, Demetron/Kerr, Middleton, WI, USA). The light tip diameter of the LED light curing unit was 8 mm. However, the diameter of an experimental QTH light curing unit and 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 shade A3	silanated barium glass filler, silanated silica filler, silanated colloidal silica, Bis-GMA, TEGDMA, photoinitiator, catalyst, accelerator, pigments, camphorquinone, additional photo initiator, others; filler load 84.5 wt%	CHO132	Kuraray Noritake Dental
Estelite ΣQuick shade A3	super-nano spherical filler, silica-zirconia filler, Bis-GMA, TEGDMA, radical amplifier, camphorquinone; filler load 82 wt%	J2951	Tokuyama Dental

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

Two types of resin composites were polymerized using the two light curing methods; (1) LED 1,200 mW/cm² (light tip-resin distance: 0 mm) for 20 s; (2) QTH 600 mW/cm² (light tip-resin distance: 0 mm) for 40 s. Energy density of both light curing methods were 24,000 mJ/cm². Hybrid type of Clearfil AP-X (shade A3: Kuraray Noritake Dental Inc., Tokyo, Japan) and rapid cure type of Estelite ΣQuick (shade A3: Tokuyama Dental Corp., Tokyo, Japan) resin composites 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 methods described. Immediately after completion of the light curing, Knoop hardness (KHN) measurements were obtained from the top and bottom surfaces using a load of 100 g and a dwell time of 15 s (Hardness tester, model MVK-E, Akashi, Kanagawa, Japan). Microhardness was measured at the center of the twenty-four resin specimen surfaces. The hardness ratio [20] was then calculated. All experiments were performed at room temperature of 23 ± 2°C, with humidity of 50 ± 10%. Knoop hardness results (*n* = 6) and hardness ratio were compared and analyzed using Bonferroni/Dunn test.

Results

Knoop hardness results of the top and bottom surfaces of resin specimens and the statistical comparisons are shown in Table 2. The results of the hardness ratio and the statistical comparisons are shown in Table 3.

Table 2 Knoop hardness at the top and bottom surfaces of resin composite

Light curing method		LED: 1,200 mW/cm ² 20 s	QTH: 600 mW/cm ² 40 s
Material		Mean (SD)	Mean (SD)
Clearfil AP-X	Top	62.7 (1.4) ^{a, A}	55.6 (1.3) ^A
	Bottom	56.1 (1.1) ^a	53.3 (2.1)
Estelite ΣQuick	Top	36.9 (0.6) ^{b, B}	33.4 (0.6) ^B
	Bottom	32.7 (0.5) ^b	32.6 (1.6)

Intergroup data designated with the same superscript lowercase letters for each top and bottom hardness are significantly different (*p* < 0.05).

Intergroup data designated with the same superscript uppercase letters for each light curing method are significantly different (*p* < 0.05).

Immediately after light curing, The KHN at the top surface of Clearfil AP-X and Estelite ΣQuick using LED 1,200 mW/cm² for 20 s was significantly higher than that of QTH 600 mW/cm² for 40 s (*p* < 0.05). The KHN at the bottom surfaces of resin composites were significantly lower than that of at the top surfaces for both Clearfil AP-X and Estelite ΣQuick resin composites using LED 1,200mW/cm² for 20 s (*p* < 0.05). There is no significant difference between the KHN at the top surfaces and the bottom surfaces for both Clearfil AP-X and Estelite ΣQuick using QTF 600 mW/cm² for

40 s ($p > 0.05$). The LED 1,200 mW/cm² for 20 s showed a significantly smaller hardness ratio than that of QTH 600 mW/cm² for 40 s for both Clearfil AP-X and Estelite Σ Quick ($p < 0.05$).

Table 3 Hardness ratio of resin composite

Light curing method	LED: 1,200 mW/cm ² 20 s	QTH: 600 mW/cm ² 40 s
Material	Mean (SD)	Mean (SD)
Clearfil AP-X	0.89 (0.01) ^{a, c}	0.96 (0.02) ^{a, d}
Estelite Σ Quick	0.89 (0.05) ^{b, d}	0.97 (0.03) ^{b, c}

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

Discussion

The KHN at the top surface of Clearfil AP-X and Estelite Σ Quick with LED 1,200 mW/cm² for 20 s was significantly higher than that of QTH 600 mW/cm² for 40 s. Light transmission through the light-cured resin composite is strongly affected by the opacity of the resin composite. The opacity of the resin composite is indicated by the refractive index mismatch [26] or contrast ratio [27]. The contrast ratio of Clearfil AP-X decreased during polymerization (increasing transparency) [17]. On the other hand, Estelite Σ Quick included radical amplifier to accelerate curing of resin composite. That why Knoop hardness at the top surface with LED 1,200 mW/cm² was higher than that of QTH 600 mW/cm² for both resin composites. 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. Clearfil AP-X include polygonal shape filler and Estelite Σ Quick include spherical shape filler. Therefore, it was suggested that the light reflectance of the resin polymer of Clearfil AP-X and Estelite Σ Quick was considerably different from that of the filler after curing.

The Knoop hardness at the bottom surfaces of resin composites were significantly lower than that of at the top surfaces for both Clearfil AP-X and Estelite Σ Quick resin composites with LED 1,200mW/cm² for 20 s. 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 [20,28,29].

The LED 1,200 mW/cm² for 20 s showed a significantly smaller hardness ratio than that of QTH 600 mW/cm² for 40 s for both Clearfil AP-X and Estelite Σ Quick. Both energy density were 2,400 mJ/cm². The LED 1,200 mW/cm² for 20 s showed a significantly smaller hardness ratio than that of the QTH 600 mW/cm² for 40 s for both Clearfil AP-X and Estelite Σ Quick resin composites. It was reported that when irradiance was increased the degree of conversion decreased linearly using the Fourier transform infrared spectroscopy [6]. When delivering a similar radiance exposure of 37,000 mJ/cm², a QTH 936 mW/cm² for 40 s light and an LED 825 mW/cm² units for 20 s showed a greater depth of cure than the Plasma arc curing 7,328 mW/cm² for 5 s light [27]. It was reported that the amount of activated starter radicals with a certain irradiance light is optimal to form cross-linked long-chain molecules [13]. A higher concentration of radicals stops crosslinking reaction earlier and leads to short-chain molecules [13]. That why high irradiance light of LED 1,200 mW/cm² inhibited polymerization of resin composite at the bottom surface even if 1,200 mW/cm² irradiance was regular irradiance of this LED light curing unit. There is no significant difference between the Knoop hardness at the top surfaces and the bottom surfaces for both Clearfil AP-X and and Estelite Σ Quick with 600 mW/cm² for 40 s. This result supported the optimal irradiance led to maximum hardness in the resin body [30]. Moreover, the use of bottom/top ratios for both hardness and conversion resulted in a linear relationship independent of filler size or filler loading [31].

It was suggested that the regular irradiance of QTH 600 mW/cm² for 40 s created more uniform polymerization of resin composite than the regular irradiance of LED 1,200 mW/cm² for 20 s.

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Conflict of Interest

There are no conflicts of interest to declare.

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