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Depth of cure and hardness of indirect composite materials polymerized with two metal halide laboratory curing units

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Abstract: The purpose of this study was to evaluate the depth of cure and Knoop hardness of indirect composite materials polymerized with different laboratory curing units. Five composite materials designed for fixed restoration veneer (Artglass, Ceramage, Epricord, Prossimo, and Solidex) were filled into a cylindrical mold and then light-exposed by using the respective proprietary laboratory curing unit or two metal halide curing units (Hyper LII and Twinkle X). Depth of cure was determined by a scraping technique, as described in ISO 4049. Composites also underwent Knoop hardness testing after immersion in water. The results (n = 5) were analyzed with the Kruskal-Wallis test and Dunn's multiple comparison test. For three materials (Prossimo, Artglass, and Epricord), depth of cure after polymerization with the Twinkle X unit was greater than that after polymerization with the respective proprietary units. For the Ceramage and Artglass materials, the Twinkle X unit resulted in the highest Knoop hardness number (KHN), whereas, for the Prossimo material, the Hyper LII unit resulted in the highest KHN. The metal halide units were effective in enhancing the post-polymerization properties of specific composite materials while reducing exposure time. (J Oral Sci 54, 121-125, 2012)

Keywords: laboratory curing unit; indirect composite; depth of cure; Knoop hardness.

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Introduction

Photopolymerized indirect composites are frequently used as veneers in cast restorations and metal-free restorations. Photoirradiation is an important process for obtaining certain physical properties, including low water sorption (1), low solubility to water (2), and wear resistance (3). The effects of light intensity and energy on the properties of direct composites have been investigated (4-7). Among mechanical tests, measurement of depth of cure and hardness of materials are simple methods for evaluating curing units. Studies have shown that irradiance substantially affected depth of cure (8,9) and hardness (4,6) of composite materials.

Indirect composite systems differ considerably from direct composite systems in monomer composition and curing apparatus. Direct composites are irradiated adjacent to a light source (frequently a quartz tungsten halogen lamp or light-emitting diode). Indirect composites are polymerized in a box-type laboratory curing unit. Because irradiation is performed extraorally, ultraviolet energy can also be used.

Several laboratory curing units equipped with metal halide lamps have been developed. Studies have shown that one such metal halide unit improved depth of cure (10) and hardness (2,11,12) of materials. In addition, use of a metal halide unit considerably reduced water solubility (1,11) and wear of indirect composites (3). However, there is only limited evidence on the polymerization performance of currently available metal halide curing units.

This study evaluated depth of cure and Knoop hardness of five indirect composites polymerized by using their respective proprietary curing units and two metal halide laboratory curing units.

Table 1 Indirect composite materials assessed

Trade name (Manufacturer)	Matrix	Filler (Filler loading, wt%)*	Lot number	Proprietary curing unit
Artglass (Heraeus Kulzer GmbH, Hanau, Germany)	Multifunctional methacrylate, Photoinitiator	Silicon dioxide, Silanized barium-aluminum-silicate glass (70)	010131	Dentacolor XS
Ceramage (Shofu Inc., Kyoto, Japan)	UDMA, Urethane diacrylate	Zirconium silicate (73)	100733	Solidilite
Epricord (Kuraray Medical Inc., Tokyo, Japan)	UTMA, TEGDMA, Photoinitiator	Organic filler, Glass, Silica (85.4)	00087B	α-Light II
Prossimo (GC Corp., Tokyo, Japan)	UDMA	Organic filler, Glass, Silica (65)	080404A	Labolight LV-II
Solidex (Shofu Inc.)	UDMA	Organic filler, Silica (78)	050860	Solidilite

UDMA: Urethane dimethacrylate; UTMA: Urethane tetramethacrylate; TEGDMA: Triethyleneglycol dimethacrylate
* Information supplied by manufacturers

Table 2 Laboratory curing units assessed

Trade name (Manufacturer)	Light source	Exposure time (s)	Wavelength (nm) Peak (nm)
Proprietary curing units			
Dentacolor XS (Heraeus Kulzer GmbH, Wehrheim, Germany)	Xenon lamp, 220 W × 1	180	374-591 484
Solidilite (Shofu Inc., Kyoto, Japan)	Halogen lamp, 150 W × 4	180	408-697 486
α-Light II (J. Morita Corp., Suita, Japan)	Halogen lamp, 320 W × 1 Fluorescent lamp, 27 W × 2	180	418-674 494
Labolight LV-II (GC Corp., Tokyo, Japan)	Fluorescent lamp, 27 W × 3	180	431-516 436
Metal halide curing units			
Hyper LII (Toho Dental Products, Saitama, Japan)	Metal halide lamp, 150 W × 2	90	329-549 366
Twinkle X (Toho Dental Products)	Metal halide lamp, 150 W × 2	90	314-456 366

Wavelength: wavelength range at which 80% of irradiance is included.
Peak: wavelength with the highest spectral irradiance.

Materials and Methods

Materials

The materials used are shown in Table 1. Five indirect composite materials were assessed: Artglass (Heraeus Kulzer GmbH, Hanau, Germany), Ceramage (Shofu Inc., Kyoto, Japan), Epricord (Kuraray Medical Inc., Tokyo, Japan), Prossimo (GC Corp., Tokyo, Japan), and Solidex (Shofu Inc.). All materials were designed for veneering metallic restorations, and the Artglass and Ceramage materials are also used in metal-free restorations. Dentin color (shade A2) paste was selected for each of the materials. The laboratory curing units used in this study are summarized in Table 2. The Dentacolor XS unit (Heraeus Kulzer GmbH, Wehrheim, Germany) is equipped with a xenon stroboscopic light source, the Solidilite (Shofu Inc.) and α-Light II (J. Morita Corp., Suita, Japan) units use different halogen light sources, the α-Light II and Labolight LV-II (GC Corp.) units use fluorescent tubes, and the Hyper LII and Twinkle X units (Toho Dental Products, Saitama, Japan) use metal halide lamps with different types of reflector.

Depth of cure

The depth of cure of a material was determined by a scraping technique, as described in ISO 4049 (13). A cylindrical stainless steel split mold with a height of 6 mm and diameter of 4 mm was filled with composite material, and the specimen surface was covered with a piece of polyester film. Specimens were irradiated in one of three units (proprietary unit, Hyper LII, or Twinkle X),

Table 3 Depth of cure (mm) of composite materials exposed with three curing units

Material	Proprietary unit			Hyper LII			Twinkle X			K-W
	Median	Mean	SD	Median	Mean	SD	Median	Mean	SD	
Prossimo	3.6 b	3.6	0.1	4.3 a,b	4.3	0.2	4.5 a	4.6	0.2	S
Artglass	3.2 b	3.2	0.1	3.9 a,b	3.9	0.1	4.2 a	4.2	0.2	S
Epricord	3.6 b	3.6	0.1	3.8 a,b	3.8	0.1	3.9 a	3.9	0.2	S
Ceramage	3.7	3.7	0.1	3.6	3.7	0.1	3.8	3.9	0.2	NS
Solidex	3.7	3.7	0.1	3.6	3.6	0.2	3.8	3.8	0.3	NS

K-W: Kruskal-Wallis test; S: significant difference observed among three units; NS: no significant difference (Kruskal-Wallis test, $P > 0.05$). Identical letters indicate that the values are not significantly different (Dunn's test, $P > 0.05$).

Table 4 Statistical categories for depth of cure for the five materials

Curing unit	Prossimo	Artglass	Epricord	Ceramage	Solidex
Hyper LII	A	A	A		
		B	B	B	B
Twinkle X	C	C	C		
		D	D	D	D

Identical letters indicate no significant difference between composites (Dunn's test, $P > 0.05$).

at the center of the unit floor. Immediately after polymerization, the composite material was removed from the mold and wiped with alcohol-treated gauze after the uncured material was scraped away with a plastic spatula. Five specimens were prepared from each of the five composite materials. The remaining length of specimens was measured with a micrometer (MDC-SB, Mitutoyo Co. Inc., Kawasaki, Japan) and defined as depth of cure.

Knoop hardness

A Teflon mold with a thickness of 2 mm and diameter of 10 mm was filled with indirect composite. The material was covered with a piece of polyester film and irradiated for the time period shown in Table 2. After removal from the mold, specimens were stored in distilled water at 37°C for 24 h. The top surfaces of specimens were grounded with 2,000-grit silicon carbide paper and polished with felt using 3-, 1-, and 0.25-µm diamond suspension. The Knoop hardness number (KHN) was determined using a universal indenter (MVK-C, Akashi Ltd., Yokohama, Japan) under a 0.49-N load for 30 s dwell time. For each specimen, the mean KHN was calculated from five indentations for each of five specimens.

Statistical analysis

Equality of variances in each group was analyzed by using the Levene test. Multiple comparisons were performed by using the Kruskal-Wallis test for depth of cure and KHN, followed by Dunn's nonparametric multiple comparison test. A P value of less than 0.05 was defined as statistically significant in all tests. GraphPad Prism 5 software (GraphPad Software Inc., CA, USA) was used for the analysis.

Results

Medians, means, and statistical results for depth of cure and KHN of the composites are presented in Tables 3 through 6. Because the Levene test showed lack of homogeneity for the standard deviations, all test results were analyzed using the Kruskal-Wallis test, followed by Dunn's nonparametric multiple comparison test.

The Prossimo, Artglass, and Epricord materials polymerized with the Twinkle X unit had greater depth of cure values than the same materials polymerized with their proprietary units (a and b). For the Ceramage and Solidex materials, no significant difference in depth of cure was observed among the three units. Depth of cure after polymerization with the Twinkle X and Hyper LII units was not statistically different among the five composites (Table 3). Depth of cure values after polymerization with the two metal halide units were divided into two categories (Table 4). For both units, the Prossimo material had greater depth of cure than the Ceramage and Solidex materials.

The results of Knoop hardness testing are summarized in Table 5. The Ceramage and Artglass materials had higher KHNs after polymerization with the Twinkle X unit than after polymerization with their proprietary units (a and b). For the Epricord and Solidex materials, no significant difference in KHN was observed among the three units. The Prossimo material had a higher KHN after polymerization with the Hyper LII than after polymerization with its proprietary unit (a and b). There was no statistical difference in KHNs between the Twinkle X and Hyper LII units for any of the five materials (Table 5). The KHNs obtained after polymerization with the Hyper LII unit were divided into two categories, whereas KHNs obtained after polymerization with the Twinkle

Table 5 Knoop hardness numbers for composite materials exposed with three curing units

Material	Proprietary unit			Hyper LI			Twinkle X			K-W
	Median	Mean	SD	Median	Mean	SD	Median	Mean	SD	
Ceramage	58.1 b	57.5	5.7	70.2 a,b	67.3	7.5	68.7 a	70.0	3.3	S
Artglass	51.7 b	53.0	4.5	56.8 a,b	55.4	7.6	61.9 a	61.8	1.6	S
Epricord	48.7	46.9	4.7	52.4	52.0	2.2	56.4	55.2	7.1	NS
Solidex	41.5	43.5	5.7	50.0	50.4	5.6	50.6	49.6	3.9	NS
Prossimo	27.8 b	29.0	2.6	42.7 a	44.8	15.4	34.3 a,b	35.7	6.0	S

K-W: Kruskal-Wallis test; S: significant difference observed among three units; NS: no significant difference (Kruskal-Wallis test, $P > 0.05$). Identical letters indicate that the values are not significantly different (Dunn's test, $P > 0.05$).

Table 6 Statistical categories for KHN for five materials

Curing unit	Ceramage	Artglass	Epricord	Solidex	Prossimo
Hyper LII	E	E	E	E	
		F	F	F	F
Twinkle X	G	G	G		
		H	H	H	
			I	I	I

Identical letters indicate no significant difference between composites (Dunn's test, $P > 0.05$).

X unit were divided into three categories (Table 6). For both units, the Ceramage material had a higher KHN than the Prossimo material.

Discussion

This study compared depth of cure and KHN for five indirect composite materials after using their proprietary curing units and two metal halide curing units. Previous studies showed that a metal halide curing unit (Hyper LII) considerably enhanced the post-polymerization properties of indirect composite materials (1-3,10-12,14,15). As described above in the Introduction, the aim of the present study was to evaluate depth of cure and KHN of five composite materials polymerized by means of three curing units. The present research also evaluated differences between two metal halide units with different assemblies.

The results for depth of cure showed no significant difference between proprietary units and the Hyper LII unit for all five materials. In addition, there was no significant difference in depth of cure between the Hyper LII and Twinkle X metal halide units for all five materials. However, depth of cure significantly differed between proprietary units and the Twinkle X unit for three composite materials, probably because, although both metal halide units use the same light source, the Twinkle X unit emits higher light energy than the Hyper LII unit due to differences in the structure of the reflector and the assembly of the curing units.

The results of hardness testing showed that the KHNs of two materials (Ceramage and Artglass) were highest with the Twinkle X unit, whereas the KHN of the Prossimo material was highest with the Hyper LII unit. These results suggest that both metal halide units can potentially improve polymerization performance of indirect composite materials while reducing exposure time. Satisfactory polymerization of composite materials in a shorter period of time would be beneficial for dentists and dental technicians. However, so-called quick curing has been found to induce excessive polymerization shrinkage within the thick layer of composite materials and may negatively affect bonding between layered substrates (16,17). Clinicians and technicians should therefore be aware that laboratory curing units and curing conditions should be carefully selected with regard to the clinical cases and situations in which materials are going to be applied.

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