

Effect of pre-heating on depth of cure and surface hardness of light-polymerized resin composites

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ABSTRACT: Purpose: To evaluate the depth of cure and surface hardness of two resin composites when subjected to three preheating temperatures, three polymerization times and two types of curing lights. **Methods:** Two resin composites were used in this study (Esthet-X and TPH), three polymerization times (10, 20, 40 seconds), three preheating temperatures (70, 100, 140°F/21.1, 37.7 and 60°C), and two curing lights (halogen and LED). For depth of cure measurements, 180 specimens (4 mm in diameter and 2 mm in depth) were made for 36 combinations of variables. Four Knoop hardness measurements were obtained from both the top and bottom surfaces. For the surface hardness, another 180 (4 x 6 mm) cylindrical specimens were fabricated. Each specimen was sectioned in half and hardness measurements were made at 0.5 mm intervals. Statistical analyses were performed using the multifactor ANOVA at a level of significance of $\alpha = 0.05$. **Results:** For depth of cure, there was a statistical difference among all the main effects (time, temperature and curing light) for both composites ($P > 0.001$) when the % difference from the top was analyzed. Results indicate that there was an increase in hardness as the temperature of the composite was increased from 70 to 140°F for both composites for either the top or the bottom. The percent difference in hardness was greater when the LED curing light was used compared to the halogen curing light. Overall there was a greater change in hardness when the resin composite was polymerized at 140°F. Although the ISO standard was not met in many cases, there was a significant increase in hardness on both the top and bottom as temperature and curing time increased ($P < 0.001$). Results for the surface hardness showed that there was a significant statistical difference ($P < 0.001$) in hardness when the surface hardness at 0.5 and 3.5 mm were analyzed separately. There was a general increase in surface hardness for both the hybrid and microhybrid as time and temperature increased. For both hybrid and microhybrid groups, as the temperature increased, there was an increase in hardness and it was statistically different ($P < 0.001$). When the percent difference between 70 and 100°F or 70 and 140°F was evaluated, the greatest increase occurred between the 70 and 140°F and minimal increase between 100 and 140°F. Overall, the LED curing light provided a greater surface hardness for the hybrid at both depths than the halogen curing light. For the microhybrid, the halogen curing light provided the greatest surface hardness when the resin was polymerized for 40 seconds. (*Am J Dent* 2008;21:215-222).

CLINICAL SIGNIFICANCE: Heating resin-based composites to temperatures to 140°F (60°C) allows a reduction of irradiation times without compromising polymerization as indicated by hardness measurements. However, more clinical studies are needed to evaluate the effect of heated resin-based composites on the pulp. A unit to clinically pre-heat resin composites is commercially available which increases the hardness of the resin and may be beneficial to the dentist as well as the patients.

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Introduction

Most general dentists are placing tooth-colored restorations, and the number of composite restorations placed each year almost equals the number of amalgam restorations placed.¹⁻⁵ Many of these adult patients will want to have the same types of restorations placed in their children's mouths, either out of esthetic concerns, or because of worries from the amalgam "controversy". Dentists must be comfortable placing resin composite restorations if they are to be competitive in the fee-for-service market.

The properties of resin composites dictate the manner in which they are placed. A clean, dry field, proper etching, an appropriately designed preparation, and adequate curing time are critical for success in placing these restorations. Reducing the amount of time for curing would be beneficial to the practitioner as well as the patient, making procedures faster and perhaps more comfortable.

Calset^a is a device advertised as a way to decrease the amount of time required to polymerize resin composites. Recent design changes in the device have resulted in being able

to change the temperature from 130-140°F (55.4-60°C). The manufacturer claims that increasing the temperature of the resin prior to curing will result in a significant decrease in curing time (up to 80%) and will increase the degree of cure. Handling characteristics are said to be improved, allowing the composite to perform more like a flowable, while maintaining the properties of the original composite.⁶ In addition a recent publication has shown that pre-heating the resin composite leads to lower microleakage at the cervical margins.⁷

Shortening the irradiation period is not a new concept. Attempts to shorten the curing time have traditionally been focused on the type or intensity of curing lights, and also on altering the chemical properties of the resin. Published data is limited to a few in peer reviewed journals, while others were reports submitted to AdDent, Inc.⁶ Several of those studies were presented as abstracts at the International Association of Dental Research (IADR) and they evaluated the microleakage of a preheated resin, the effect of temperature on degree of conversion and polymerization rates, shrinkage, and surface hardness.⁸⁻¹³

One of the abstracts discussed the surface hardness of two types of resin composites, with polymerization performed at room temperature and 130°F (54.4°C).¹³ Five samples were made for each group, and the Knoop hardness (kg/mm²) was measured. The results of that study showed that overall surface hardness was increased, but not at statistically significant levels. However, there is evidence that if a composite is pre-heated, the monomer conversion rate is increased and therefore the duration of the irradiation period can be reduced.¹⁰ Daronch *et al*^{14,15} calculated the conversion rate of a pre-heated composite and found that by heating the resin composites to 140°F (60°C), the conversion rate increased between 31.6 to 67.3% and therefore less polymerization time was required. More investigation is necessary to determine if there is a real clinical benefit in heating composites.

This study examined how the depth of cure and surface hardness are affected by changing the temperature of light-polymerized conventional resin composites. In addition, the study assessed if an increase in temperature allowed a reduction in clinical curing time. It was hypothesized that preheating the composite would (1) increase the surface hardness and depth of cure and (2) decrease the time needed to polymerize a preheated composite.

Materials and Methods

Study design - This study was designed to include multiple variables in order to have a better understanding of how temperature and time may affect the surface hardness and depth of cure of different composites. Two different types of composites were used to determine if filler characteristics would affect the outcome variables of time and temperature. The composites chosen for this study were a microhybrid (Esthet-X^b) and a hybrid (TPH^b). A2 was chosen as the shade for both types of composites.

Two types of curing lights, namely a tungsten halogen (Spectrum 800^b) and an LED curing light (Smartlite iQ^b) were selected for this study. A photospectrometer (Varian Carry 5000^c) was used to verify the spectral irradiance of the lights of the curing units. The output intensity for the lights was checked before and during the study using a radiometer (Demetron Radiometer^d).

Three different polymerization times (10, 20 and 40 seconds) were used which are commonly cited in the literature as acceptable polymerization times. However, 20 seconds is the manufacturer recommended irradiation time and this time was used to see what effect polymerization time had on the outcome variables. The timers on the curing lights were verified for accuracy prior to use.

Three different temperatures were used (70, 100, and 140°F (21.1, 37.7 and 60°C). Typically resin composites are used at room temperature so 70°F was used as the control. Further analysis regarding the temperatures used is presented in the discussion.

The composite was placed in the Calset unit and after 20 minutes, the heated samples were immediately injected into the molds to prevent heat loss. The Calset device was used according to the manufacturer's instructions. Clean glass slabs were also pre-heated in an oven to the corresponding temperatures 1 hour prior to use. Each glass slab was used for one sample, and then immediately placed in the oven for re-heating.

This was done to ensure that the resin did not cool significantly while the sample was being prepared.

Depth of cure - For the surface hardness measurements and methods, the ISO standards for composite resins developed in conjunction with the ADA, were used.¹⁶ The standard requires that when a 2 mm-layer of resin composite is polymerized from the top, the bottom surface hardness should be 80% of the top surface hardness.

Five specimens were prepared for each combination of the parameters (two curing lights, two resin-based composites, three irradiation periods, three temperatures) resulting in 36 groups and a total of 180 specimens. The composite samples were packed into an aluminum mold measuring 4 mm in diameter and 2 mm in depth. Prior to packing the mold (that had been heated to the same temperature of the composite), a Mylar strip^e was placed on the glass slab, the mold was then placed and the composite packed. After placing the composite, a second Mylar strip was placed on top of the mold and a glass microscope slide was placed over the composite and then irradiated for the designated time. One minute after polymerization, the specimens were carefully removed from the mold and inspected for defects. Each specimen was stored in darkness at 98.6°F (37°C) for 24 hours before measurements were taken.

The Knoop hardness of the top and bottom of the composite specimen was measured with a Leco M-400^f hardness tester, and the readings for each surface were independently averaged and reported in Knoop Hardness Numbers (KHN). Four locations, 1 mm apart were measured for each specimen and surface and the data recorded. The difference in hardness between the top and bottom was calculated and recorded as the percent of the top surface.

Surface Knoop hardness - This portion of the study evaluated the polymerization hardness of the two resin composites which also had 36 combinations of factors for a total of 180 samples. The procedure to fabricate these specimens was similar to the depth of cure study except that the composite was packed directly into a standardized aluminum mold that measured 6 mm in depth and 4 mm in diameter. The irradiated resin composite was then removed from the mold and each specimen was stored in darkness at 98.6°F for 24 hours before hardness measurements were made.

Prior to the measurements, the specimens were first imbedded in a heated (194°F) modeling compound material^g with the bottom surface pressed into the compound; a leveling device was used to level the samples which were then rapidly placed in cold water to cool the compound. The specimens in this study were subjected to the heat of the compound for a very short time which should have had no effect on the depth of cure. Using the same previously used hardness tester, surface top hardness measurements were obtained from three random locations of each specimen and the Knoop values recorded (Fig. 1A).

A second set of measurements were made that required further preparation of the specimens. They were removed from the compound and re-imbedded horizontally to approximately half their diameter. The specimens were then sectioned lengthwise with a water-cooled diamond wheel (Low Speed Dia-

Table 1. Effect of temperature, time and light source on the top and bottom surfaces of a hybrid resin composite (percent difference).

Time (sec)	Curing light	Depth		Temperature (°C/°F)			P-value	% difference 70 & 100	% difference 70 & 140
				21.1/70	37.7/100	60/140			
10	Halogen	Top	Mean (SD)	36.45 (0.32)	50.47 (0.82)	59.45 (0.63)	< 0.001	38.4	63.1
		Bottom	Mean	19.56 (0.70)	32.51 (0.72)	45.64 (0.45)	< 0.001	66.2	133.3
	LED	Top	Mean	38.49 (0.57)	50.45 (0.58)	54.91 (0.82)	< 0.001	31.1	42.7
		Bottom	Mean	31.66 (0.40)	41.95 (0.71)	45.76 (0.36)	< 0.001	32.5	44.5
20	Halogen	Top	Mean	45.28 (0.23)	54.06 (0.50)	62.15 (0.49)	< 0.001	19.4	37.2
		Bottom	Mean	39.48 (0.38)	45.84 (0.69)	53.21 (0.55)	< 0.001	16.1	34.8
	LED	Top	Mean	41.35 (0.69)	54.64 (0.44)	59.47 (0.75)	< 0.001	32.1	43.8
		Bottom	Mean	35.15 (0.46)	49.72 (0.73)	52.83 (0.28)	< 0.001	41.5	50.3
40	Halogen	Top	Mean	46.29 (0.40)	56.73 (0.42)	62.48 (0.60)	< 0.001	22.5	35.0
		Bottom	Mean	41.62 (0.54)	54.50 (0.43)	56.52 (0.66)	< 0.001	31.0	35.8
	LED	Top	Mean	41.46 (0.43)	56.31 (0.43)	62.00 (0.45)	< 0.001	35.8	49.5
		Bottom	Mean	40.28 (0.50)	54.94 (0.41)	55.86 (0.39)	< 0.001	36.4	38.7

Table 2. Effect of time and temperature on the top and bottom surfaces of a microhybrid resin composite (percent difference).

Time (sec)	Curing light	Depth		Temperature (°C/°F)			P-value	% difference 70 & 100	% difference 70 & 140
				21.1/70	37.7/100	60/140			
10	Halogen	Top	Mean (SD)	38.04 (0.68)	44.32 (0.87)	50.02 (0.50)	< 0.001	16.5	31.5
		Bottom	Mean	14.86 (0.42)	16.45 (0.18)	21.89 (0.48)	< 0.001	10.7	47.3
	LED	Top	Mean	32.0 (0.40)	41.2 (0.60)	48.6 (0.70)	< 0.001	29.0	52.1
		Bottom	Mean	19.70 (0.70)	25.55 (0.38)	31.98 (0.25)	< 0.001	29.7	62.4
20	Halogen	Top	Mean	39.74 (0.95)	51.13 (0.33)	50.79 (0.59)	0.116*	28.7	27.8
		Bottom	Mean	22.45 (0.73)	27.16 (0.59)	35.01 (0.48)	< 0.001	21.0	56.0
	LED	Top	Mean	36.6 (0.50)	45.9 (0.60)	54.5 (0.20)	< 0.001	25.3	48.7
		Bottom	Mean	30.18 (0.68)	35.84 (0.61)	40.71 (0.20)	< 0.001	18.7	34.9
40	Halogen	Top	Mean	45.63 (0.45)	51.50 (1.52)	56.85 (0.52)	< 0.001	12.9	24.6
		Bottom	Mean	35.71 (0.68)	38.42 (0.70)	51.03 (0.30)	< 0.001	7.6	42.9
	LED	Top	Mean	37.7 (0.50)	51.1 (0.40)	58.6 (0.30)	< 0.001	35.5	55.5
		Bottom	Mean	36.28 (0.33)	42.03 (0.45)	48.43 (0.60)	< 0.001	15.8	33.5

* No statistical difference between 100 and 140°F.

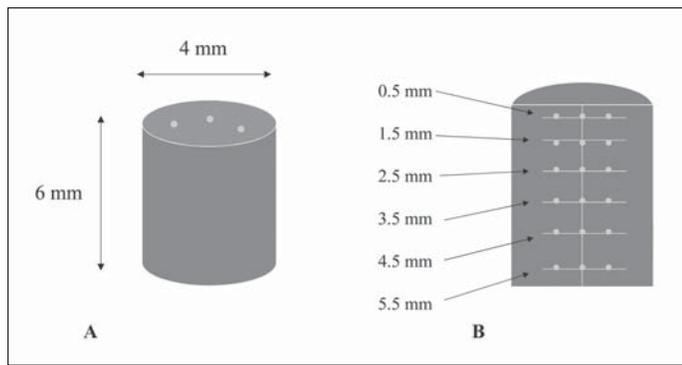


Fig. 1. A. Measurements at the top surface and B. cross sectional measurements.

mond Wheel Saw 650^h). The sectioned specimens were next polished using a HandiMet IIⁱ roll grinder in the following sequence of grits: 240, 320, 400 and 600. The final polish was obtained using an Ecomet IIⁱ grinder with 0.3 μm alumina powder. The polishing sequence was carried out using copious amounts of water to prevent unintended hardening by heating of the samples. Efforts were also made to shield the samples from ambient light during this preparation phase. The room lights were dimmed and a barrier shield was placed over the composite samples during preparation. Knoop hardness measurements were made at 0.5 mm, 1.5 mm, 2.5 mm, 3.5 mm, 4.5 mm, and 5.5 mm, or until unreliable measurements were obtained (Fig. 1B). At each interval, three measurements were made. The first was in the center of the long axis of the

specimen, and the other two were 1.0 mm on either side of the first, along the horizontal axis of the sample.

Statistical analysis - For the depth of cure, the percent difference between the top and bottom surfaces were calculated and a multifactor ANOVA was used to identify differences among three main effects: time, curing light and temperature for each type of composite. When differences were found, a Tukey post-hoc test at P> 0.05 level was used. To evaluate the effect of time for each temperature, the difference among the three temperatures was calculated and expressed as percent difference.

For the surface hardness, only two depth measurements were chosen for analysis from all of the data obtained from the internal surface hardness measurements. The 0.5 mm and 3.5 mm increments were chosen because reliable measurements were obtained at each of those depths for all samples that were prepared. A one-way ANOVA was used to identify separately any differences between the two main effects: temperature and time. If differences were found, a Student Newman's Keuls test was used to identify the differences among groups at a P> 0.05. For all tests, the hybrid and microhybrid resin composites were analyzed separately due to their inherent differences.

Results

Depth of cure - Results of the multifactor ANOVA are shown in Tables 1-4 for the hybrid and microhybrid composites respectively. There was a statistical difference among all the main effects (time, temperature and curing light) for both com-

Table 3. Effect of temperature and time on the top and bottom surface hardness on a hybrid resin composite.

Temp (°C/°F)	Time (sec)	Halogen top		Halogen bottom		% of top	LED top		LED bottom		% of top
		Mean	SD	Mean	SD		Mean	SD	Mean	SD	
21.1/70	10	36.45	0.32	19.56	0.70	54	38.49	0.57	31.66	0.40	82
	20	45.28	0.23	39.48	0.38	87	41.35	0.69	35.15	0.46	85
	40	46.29	0.40	41.62	0.54	90	41.46	0.43	40.28	0.50	97
37.7/100	10	50.47	0.82	32.51	0.72	64	50.45	0.58	41.95	0.71	83
	20	54.06	0.50	45.84	0.69	85	54.64	0.44	49.72	0.73	91
	40	56.73	0.42	54.50	0.43	96	56.31	0.43	54.94	0.41	98
60/140	10	59.45	0.63	45.64	0.45	77	54.91	0.82	45.76	0.36	83
	20	62.15	0.49	53.21	0.55	86	59.47	0.75	52.83	0.28	89
	40	62.48	0.60	56.52	0.66	90	62.00	0.45	55.86	0.39	90

*A 20-second polymerization time and 70°F (room temperature) were used as the controls. Groups connected by vertical lines were not statistically different.

Table 4. Effect of temperature and time on the top and bottom surface hardness on a microhybrid resin composite.

Temp (°C/°F)	Time (sec)	Halogen top		Halogen bottom		% of top	LED top		LED bottom		% of top
		Mean	SD	Mean	SD		Mean	SD	Mean	SD	
21.1/70	10	38.04	0.68	14.86	0.42	39	32.0	0.4	19.70	0.70	62
	20	39.74	0.95	22.45	0.73	56	36.6	0.5	30.18	0.68	82
	40	45.63	0.45	35.71	0.68	78	37.7	0.5	36.28	0.33	96
37.7/100	10	44.32	0.87	16.45	0.18	37	41.2	0.6	25.55	0.38	62
	20	51.13	0.33	27.16	0.59	53	45.9	0.6	35.84	0.61	78
	40	51.50	1.52	38.42	0.70	75	51.1	0.4	42.03	0.45	82
60/140	10	50.02	0.50	21.89	0.48	44	48.6	0.7	31.98	0.25	66
	20	50.79	0.59	35.01	0.48	69	54.5	0.2	40.71	0.20	75
	40	56.85	0.52	51.03	0.30	90	58.6	0.3	48.43	0.60	83

*A 20-second polymerization time and 70°F (room temperature) were used as the controls. Groups connected by vertical lines are not statistically different.

posites ($P > 0.001$) when the % difference from the top was analyzed. Time had the greatest influence (F -ratio= 12689.38) followed by temperature (F -ratio= 9347.01). Tables 1 and 2 indicate that there was an increase in hardness as the temperature of the composite was increased from 70-140°F for both composites for either the top or the bottom. The percent difference in hardness was greater when the LED curing light was used compared to the Halogen curing light. Overall, there was a greater change in hardness when the resin composite was polymerized at 140°F. Tables 3 and 4 show the percent difference between the top and bottom for the hybrid and microhybrid groups. The ISO 4049 resin based restorative materials standard requires that the bottom of the 2 mm thick sample has 80% of the hardness of the top. The LED light met the standard for all temperatures and polymerization times for the hybrid group. However, the halogen light did not meet the standard for the 10 seconds at either of the three temperatures ($P > 0.001$) with a % polymerization range between 54-77.

For the microhybrid resins (Table 4) the standard was met only when the halogen curing light was used for 40 seconds at a temperature of 140°F. For the LED group, the standard was met at 70°F with 20- and 40-second curing times. However, at 100 and 140°F, the 80% standard was met only after 40 seconds of polymerization.

Although the ISO standard was not met in many cases, there was a significant increase in hardness on both the top and bottom of both halogen and LED groups as temperature and curing time increased ($P < 0.001$).

When the effect of time was analyzed for the three curing temperatures (Tables 3, 4), there were no statistical differences

($P > 0.05$) for two of each of the hybrid and microhybrid resins at two of the polymerization times (20 and 40 seconds). All others indicated an increase in hardness when the polymerization time was increased.

Most manufacturers are recommending a polymerization of 20 seconds. Since most resins are currently used at room temperature (approximately 70°F), these two parameters were used as a standard for evaluating if different curing times (shorter 10 seconds or longer 40 seconds) were needed and if the temperature of the resin made any significant improvements at reducing the polymerization time. When the 10-second was compared to the 20-second polymerization time (room temperature) for the top or bottom by combining both curing lights and using the hybrid resin, (Table 1) there was a 14% decrease in hardness at 10 seconds and no measurable increase (0%) at 40 seconds for the top surface. However, there was a decrease of 32% between 10 and 20 seconds and 10% increase in hardness between 20 and 40 seconds for the bottom. When the same parameters were compared for 100 and 140°F, there was no statistical difference between the two with only a 5 to 7% decrease or increase in hardness for the top for both temperatures. For the bottom there was a 30% decrease in hardness when the resin was polymerized for 10 seconds at 100°F as compared to only 17% when polymerized at 140°F. For the microhybrid resin, very similar differences were found. These results indicate that at room temperature, the resins need to be polymerized at least for 20 seconds but as the temperature is increased, there is less need to increase the polymerization of the resin above 10 seconds.

Surface hardness - Results of the surface hardness are shown in Tables 5-6 and Figs. 2-5. A one-way ANOVA showed that there

Table 5. Effect of temperature and time on the surface hardness of a hybrid resin composite (percent difference).

Time (sec)	Depth	Curing light	Temperature (°C/°F)			P-value	% difference 70 & 100	% difference 70 & 140	
			21.1/70	37.7/100	60/140				
10	0.5 mm	Halogen	Mean (SD)	57.30 (0.26)	59.99 (0.19)	62.19 (0.21)	< 0.001	4.7	8.5
		LED	Mean	59.58 (0.30)	66.37 (0.12)	70.07 (0.21)	< 0.001	11.4	17.6
	3.5 mm	Halogen	Mean	29.61 (1.12)	39.89 (0.13)	44.73 (0.13)	< 0.001	34.7	51.1
		LED	Mean	41.83 (0.10)	52.76 (0.15)	57.67 (0.11)	< 0.001	26.1	37.9
20	0.5 mm	Halogen	Mean	61.19 (0.25)	63.41 (0.12)	66.30 (0.23)	< 0.001	3.6	8.4
		LED	Mean	62.29 (0.15)	69.01 (0.12)	73.29 (0.10)	< 0.001	10.8	17.7
	3.5 mm	Halogen	Mean	44.46 (0.17)	48.65 (0.14)	53.35 (0.14)	< 0.001	9.4	20.0
		LED	Mean	48.86 (0.13)	57.39 (0.12)	63.36 (0.11)	< 0.001	17.5	29.7
40	0.5 mm	Halogen	Mean	64.39 (0.14)	68.16 (0.10)	71.31 (0.17)	< 0.001	5.8	10.7
		LED	Mean	64.27 (0.13)	72.26 (0.12)	74.79 (0.21)	< 0.001	12.4	16.4
	3.5 mm	Halogen	Mean	48.39 (0.12)	49.91 (0.11)	61.39 (0.13)	< 0.001	3.1	26.9
		LED	Mean	56.37 (0.13)	65.17 (0.18)	69.11 (0.10)	< 0.001	15.6	22.6

Table 6. Effect of temperature and time on the surface hardness of a microhybrid resin composite (percent difference).

Time (sec)	Depth	Curing light	Temperature (°C/°F)			P-value	% difference 70 & 100	% difference 70 & 140	
			21.1/70	37.7/100	60/140				
10	0.5 mm	Halogen	Mean (SD)	40.91 (0.28)	43.31 (0.18)	51.35 (0.16)	< 0.001	5.9	25.5
		LED	Mean	59.58 (0.30)	66.37 (0.12)	70.07 (0.10)	< 0.001	11.4	17.6
	3.5 mm	Halogen	Mean	3.61 (0.11)	3.75 (0.21)	4.66 (0.16)	< 0.001	3.9	29.2
		LED	Mean	41.83 (0.10)	52.76 (0.15)	57.67 (0.11)	< 0.001	26.1	37.9
20	0.5 mm	Halogen	Mean	52.21 (0.23)	54.47 (0.19)	60.00 (0.21)	< 0.001	4.3	14.9
		LED	Mean	62.29 (0.15)	69.01 (0.12)	73.29 (0.10)	< 0.001	10.8	17.7
	3.5 mm	Halogen	Mean	17.76 (0.33)	19.75 (0.22)	34.84 (0.17)	< 0.001	11.2	96.2
		LED	Mean	48.86 (0.13)	57.39 (0.12)	63.36 (0.11)	< 0.001	17.5	29.7
40	0.5 mm	Halogen	Mean	58.85 (0.31)	62.21 (0.15)	64.71 (0.19)	< 0.001	5.7	10.0
		LED	Mean	64.27 (0.13)	72.26 (0.12)	74.79 (0.21)	< 0.001	12.4	16.4
	3.5 mm	Halogen	Mean	31.09 (0.16)	37.43 (0.12)	40.37 (0.17)	< 0.001	20.4	29.9
		LED	Mean	56.37 (0.13)	65.17 (0.18)	69.11 (0.10)	< 0.001	15.6	22.6

was a significant statistical difference (P< 0.001) when the surface hardness at 0.5 and 3.5 mm was separately analyzed. There was a general increase in surface hardness for both the hybrid and microhybrid as time and temperature increased (Tables 5, 6). For both hybrid and microhybrid groups, as the temperature increased there was an increase in hardness and it was statistically significant (P< 0.001). When the percent difference between 70 and 100°F or 70 and 140°F was evaluated, the greatest increase occurred between 70 and 140°F with minimal increase between 100 and 140°F. Overall, the LED curing light provided a greater surface hardness for the hybrid at both depths than the halogen curing light (Table 5; Figs. 2-5). For the microhybrid, the halogen curing light provided the greatest surface hardness when the resin was polymerized for 40 seconds (Fig. 4).

When the hybrid was evaluated at 0.5 mm of depth, there was a ± 5% in polymerization hardness between the 10 and 20 and 20 and 40 seconds polymerization time and 100 to 140°F. However, at 3.5 mm of depth there was a 30% decrease in hardness between the 10 and 20 seconds and an increase of 12% between 20 and 40 seconds. When the temperature was increased to 100 and 140°F, there was a decrease of 1% compared to the 10 seconds and an increase of 10% when compared to 40 seconds polymerization time.

The microhybrid showed a ± 10% change in polymerization hardness between 10 and 20 and 20 and 40 seconds polymerization time and 100 to 140°F. At 3.5 mm of depth, there was a 46% decrease in hardness between the 10 and 20 seconds and an increase of 31% between 20 and 40 seconds. When the

temperature was increased to 100 and 140°F, there was a decrease of 42% compared to the 10 seconds and an increase of 22% when compared to 40 seconds of polymerization time.

Discussion

There are primarily two methods to determine the depth of polymerization of a resin composite: degree of monomer conversion and Knoop microhardness measurements. The hardness of a resin composite is commonly correlated to the mechanical strength, rigidity and resistance to occlusal degradation in the oral cavity. Previous studies have shown a correlation between degree of monomer conversion and Knoop hardness values.¹⁷⁻¹⁹ The authors chose a mechanical method of determining depth of cure using a Knoop hardness tester.

Traditionally, a 6 mm deep cylinder of composite is polymerized from the top and any uncured composite is scraped from the bottom of the sample. The cured portion is measured with calipers, the numbers are averaged and then divided by two to produce a mean depth of cure.¹⁶ There are however, some inherent problems with this method. The amount of force used to cut away the uncured composite can remove resin that is actually cured. Since this is done by hand, it is difficult to standardize the amount of force being used to scrape. There is also no way to know how hard the composite is at the bottom surface. Using an indenter was an alternative approach but was not available at the facility. Therefore, two different methods were used to determine the depth of cure. The first one was to determine the depth of polymerization of a 2 mm resin specimen. Most manufacturers recommend placing

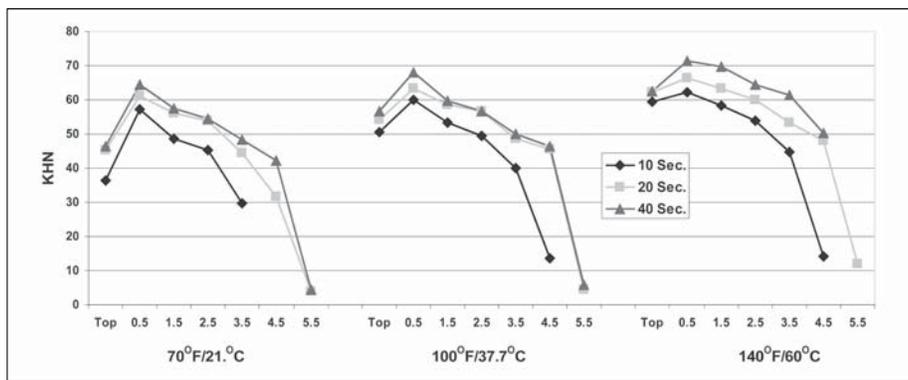


Fig. 2. Surface hardness of hybrid resin composite polymerized with the halogen curing light.

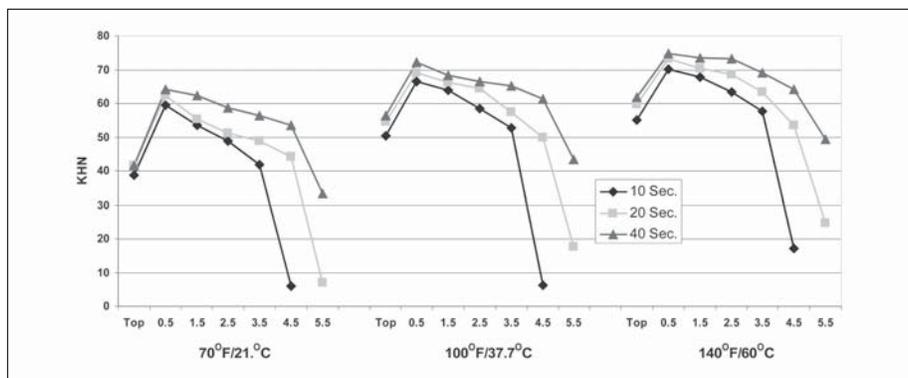


Fig. 3. Surface hardness of hybrid resin composite polymerized with the LED curing light.

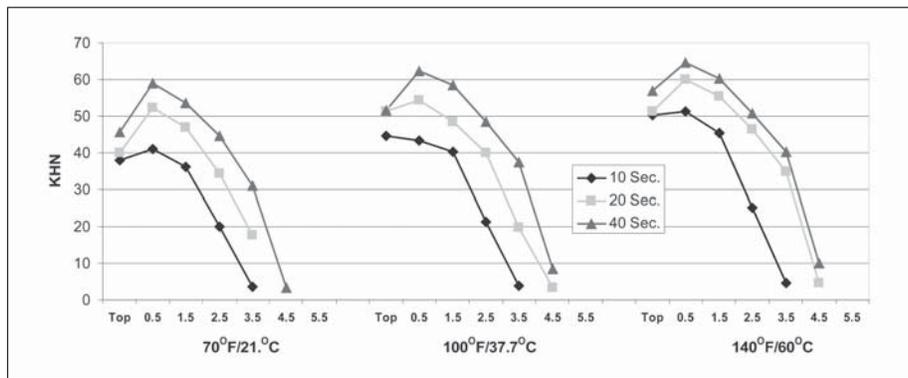


Fig. 4. Surface hardness of microhybrid resin composite polymerized with the halogen curing light.

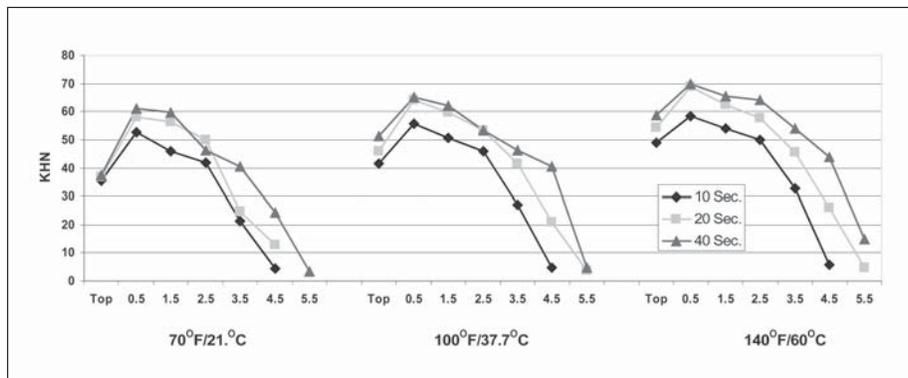


Fig. 5. Surface hardness of microhybrid resin composite polymerized with the LED curing light.

the composite in 2 mm increments so samples were made and the polymerization depth calculated as 80% of the top hardness.

As was explained in the methodology section of this manuscript, the second method involved measuring how deep the resin was polymerized. This provided a detailed assessment

of the actual hardness as the depth increased.

Using the traditional method to determine the depth of cure leads one to believe that the curing time has a greater influence on the depth rather than the temperature. However, the real effect of temperature is seen when looking at the hardness

values. The samples may not have cured noticeably deeper, but the increase in hardness at the deeper levels is seen when looking at the full data table. The figures presented in the results section provide an adequate view of this data.

For this study, three temperatures were evaluated. Seventy degrees (70°F/21.1°C) was chosen because it represents the temperature of a hypothetical typical dental office temperature. One hundred degrees (100°F/37.7°C) was chosen because it was between the 70-140°F produced by the Calset device, and because it is close to the normal intraoral temperature of the mouth, and 140°F/60°C was chosen because it is the maximum temperature produced by the Calset unit.

Twenty-four hours was deemed reasonable, as the post-polymerization stability of resin composites has been established by several studies.^{20,21} Previous research has shown that when a polymerized resin is subjected to heat for a sustained period of time, there can be an increase in degree of cure.¹²

The proposed hypothesis for the hardness and depth of cure was validated. A preheated composite increased the hardness and depth of cure for both types of curing lights and composites (hybrid and microhybrid). It has been clearly shown by Daronch *et al*^{14,15} that there is a strong correlation between temperature and monomer conversion. There are many factors affecting a pre-heated composite prior to polymerization. One such factor is that an increased temperature decreases composite viscosity and enhances radical mobility, resulting in higher conversion rate and a harder composite.²³⁻²⁵ The second hypothesis regarding using a shorter exposure to polymerize the composite was also validated. By reducing the exposure by 25 to 50% and increasing the temperature, the authors were able to attain comparable conversion rates to a room temperature resin composite. The theory being that even though fewer radicals are formed due to the shorter exposure time, there is a greater mobility due to increased temperature, and therefore a greater conversion rate.^{14,15}

When the top hardness measurements were compared to the measurements at 0.5 mm; the 0.5 mm was harder than the top measurement. Under closer evaluation using a high power stereo measuring microscope (x100), it was noted that the sectioned samples from the depth of cure study had a layer of resin at the surface that was approximately 50-100 μ m in thickness. This "resin-rich" layer appeared to be lacking the filler component of the composite, and therefore had a notably softer surface with the filler missing. However, the oxygen inhibited layer was not chemically analyzed.

It is not known if a pre-heated resin composite has any effect on the pulpal tissue when placed into a prepared tooth. There are studies that suggest that pulpal damage may occur with an increase in pulpal temperature of 41.9°F (5.5°C). Friedman⁶ found that there was an increase of only 34.8°F (1.6°C) when a 130°F (54.5°C) composite is injected into a tooth with 1 mm of dentin remaining. There were few references in the literature regarding the temperature of the material prior to placement. Rueggeberg *et al*²⁶ found an increase averaging 46.2°F (7.9°C) when a resin was heated to 140°F (60°C). Other *in vitro* studies^{27, 28} showed a higher risk of causing pulpal damage by increasing the polymerization time of high intensity curing lights.

An interesting finding was that lower conversion rates were

found at room temperature than at the other two temperatures. This finding highlights the importance of using resin materials that have not been stored in the refrigerator. Another interesting finding was that overall; there was a modest enhancement in hardness by polymerizing the composite at 140°F as compared to 100°F. However, in a clinical situation there might be a delay between removing and dispensing the composite from the Calset unit, placing the composite in the cavity, contouring and polymerization. Assuming a time delay of about 1 minute, the preheated composite will cool down substantially, so pre-heating the composite to 140°F might still be beneficial in terms of conversion rate even if the composite is polymerized for 10 seconds instead of 20 seconds at room temperature when using an LED curing light.

For the microhybrid resin at 3.5 mm, adequate hardness was only obtained at 40 seconds for the halogen light and 20 seconds for the LED curing light (Table 6). This variation in hardness is worth discussing. Two different filler size of resin composites were used. While temperature, time and conversion correlated well with the use of the hybrid resin composite, it did not correlate well with the microhybrid. This fact demonstrates that the absolute conversion rate for one type of composite cannot be extrapolated to other brands and shades due to variations in filler composition and photoinitiators. Careful evaluation needs to be made for different types of resin composite.

If a 20-second polymerization time and a room temperature resin is used as the standard for polymerization in the average private office and the ISO 4049 is used as the standard to determine adequate hardness, the following recommendations can be made. If placing the resin in 2 mm increments using a halogen or LED curing light, then polymerizing the resin for 10 seconds at 100 or 140°F is all that is necessary.

If the hybrid resin is polymerized in increments larger than 2 mm then pre-heating the resin and using a 10- to 20-second polymerization time is adequate with either an LED or halogen curing light. If a microhybrid is used and polymerized with a halogen light, adequate polymerization of the deeper layers can only be obtained using a minimum of 20 seconds. If an LED curing light is used, then 10 seconds is adequate for this type of resin composite.

In conclusion, preheating resin composites with a commercially available (Calset) composite warmer increases the monomer conversion rate and increases the depth of cure and hardness of the tested composites. LEDs are more efficient at polymerizing the tested composites and produced statistically significantly better results than the halogen curing light. Shorter polymerization times with a pre-heated resin can produce similar hardness values as a room temperature resin with longer curing times. These results are valid for the resins evaluated in the laboratory and the effects in a clinical situation cannot be concluded from this study.

- a. AdDent, Inc., Danbury, CT, USA.
- b. Caulk/Dentsply, Milford, DE, USA.
- c. Varian Inc., Palo Alto, CA, USA.
- d. Kerr/Sybron, Orange, CA, USA.
- e. Henry Schein, Melville, NY, USA.
- f. LECO, St. Joseph, MI, USA.
- g. Miltex, Inc., York, PA, USA.
- h. South Bay Technology, San Clemente, CA, USA.
- i. Buehler, Lake Bluff, IL, USA.

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