Curing depths of a universal hybrid and a flowable resin composite cured with quartz tungsten halogen and light-emitting diode units

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This in vitro study evaluated curing depths of a universal hybrid resin composite with two viscosities (Tetric Ceram and Tetric Flow) after curing with 6 different quartz tungsten halogen and light-emitting diode curing units. Irradiance (light intensity) of the curing units varied between 200 and 700 mW/cm^2. The curing units were used for standard, soft-start, or pulse curing. Curing times were 20 and 40 s for standard curing, 3 + 10 s and 3 + 30 s for pulse curing, and 40 s for soft-start. Resin composite specimens, 5 in each group, with a diameter of 4 mm and a height of 6 mm, were made in brass molds and cured from one side at a distance of 6 mm. After 2 weeks, the specimens were ground longitudinally half through the specimen. Curing depth was then determined by measurement of Wallace hardness for each half millimeter starting at 0.5 mm from the top surface. For all curing units and for both resin composites an increased curing time led to statistically significantly higher depth of cure ($P < 0.0005$). Tetric Flow showed a statistically significantly higher depth of cure than Tetric Ceram ($P < 0.0005$). All curing units cured more than 2.0 mm of both composites from a distance of 6 mm at 20 s curing time. The value for 40 s was 3.0 mm. The composite closer to the surface than the depth of cure value was equally well cured with all curing units investigated. There was a significant linear correlation between the determined irradiance of the curing units and the depths of cure obtained (20 s: $r = 0.89$, $P < 0.025$; 40 s: $r = 0.91$, $P < 0.01$). □ *Composite; halogen; hardness; LED; light-curing*

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During the past decade, resin composites have replaced amalgam as the universal restorative material, although polymerization shrinkage is still considered a problem (1, 2). Shrinkage stresses at the bonding interface tend to destroy the bond between the resin composite and the tooth structure (3, 4). Several ideas have been introduced to reduce the shrinkage stresses, e.g. sandwich restorations, the use of flowable resin composites, and the use of soft-start and pulse-curing techniques (5–13). Flowable resin composite used as bottom increment has been recommended to improve the interfacial cervical adaptation and act as a stress-absorbing layer (14, 15). Labella et al. (16) argued that relatively high polymerization shrinkage of flowable resin composites may be offset by the relatively low modulus of elasticity of these materials and allow local distortion of the material rather than debonding. Several in vitro studies have investigated the effect of flowable resin composites on marginal seal with contradictory results, while little is known about their degree of conversion when used as bottom layer (13, 17–22).

A reduced initial irradiance, as in soft-start and pulse-cure, may result in slower development of stiffness of resin composites and prolong the compensating flow during polymerization (3, 10–12). In general, a more rapid polymerization and a higher degree of conversion lead to increased shrinkage stress (3). However, high conversion is important for good mechanical properties and biocompatibility (2, 23, 24). Irradiance diminishes as the curing tip is moved away from the resin composite, which may cause problems in the deepest part of the cavity (25).

The most widely used curing units for light-cured resin composites use quartz tungsten halogen light sources (QTH). These produce white light, which is then filtered to the range of blue light (400–500 nm). Only a small part of the spectral range emitted is effective for activating the photo-initiators. Heat generation is a major disadvantage of QTH lamps and increases with increasing radiation time (1, 26, 27). Other drawbacks of QTH devices are limited effective lifetime of the bulb and degradation of reflector and filter over time. Curing units using light-emitting diodes (LED) as a light source have been introduced in recent years. The LED features a narrow spectral output around 470 nm which coincides with the absorbance maximum of the initiator camphoroquinone. However, the first LED devices were characterized by low irradiance (26).

Asmussen (23) showed that, for a given resin composite, mechanical properties are a good indication of degree of conversion, and it has been shown that a higher degree of conversion is correlated with increased volumetric shrinkage (28).

The aim of this study was to evaluate the influence of the filler load of a hybrid resin composite material on curing depth by comparing the highly loaded restorative version with its less loaded flowable version. A further aim was to evaluate the effectiveness of six QTH and LED curing units used for different curing times. The first

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hypothesis tested was that the flowable resin composite shows greater curing depth than the universal resin composite, the second that an increased curing time results in increased curing depth, and the third that the new LED units would result in similar depths of cure as the QTH units when correlated to the irradiance determined by the radiometer.

Materials and methods

Brass molds with a diameter of 4 mm and a height of 6 mm were used to make resin composite specimens. The molds were filled with resin composite and covered with clear Mylar strips on each side. They were placed on a white metal surface and cured from the top. Six different curing units were used in this study, 4 QTH and 2 LED (Table 1). All were checked before the start with a radiometer (Optilux 100; Kerr/Demetron, Danbury, Ct., USA). The curing times, curing modes, and irradiance of the units are given in Table 1. The Elipar Trilight was used in the soft-start mode, which is only available in a 40 s program. The irradiance increases to full irradiance during the first 15 s and remains at this level for the rest of the curing period. The Bisco VIP was used in the pulse-cure mode and started with an initial cure of 3 s at 200 mW/cm^2, followed by a resting period of 3 min before a final cure of 10 or 30 s with an irradiance of 600 mW/cm^2. A distance of 6 mm between the curing light tip and the top surface of the specimen was used in all groups. The resin composites used were Tetric Ceram A3 (LOT E52766, Vivadent, Schaan, Liechtenstein) and Tetric Flow A3 (LOT E0037, Vivadent). Both Tetric materials are Bis-GMA/urethane dimetacrylate/TEGDMA based resin composites reinforced with barium glass, ytterbium trifluoride, barium-aluminum fluoride glass and high dispersed silica filler. Tetric Ceram contains 77.5 wt% and Tetric Flow 68 wt% filler. The mean filler size of the glass filler is 1 μm, of ytterbiumtrifluoride 0.24 μm, and of dispersed silica 0.04 μm. Five specimens were made in each group. After curing, the molds were stored in air for 24 h in the dark at room temperature before removing the specimens, which were then stored again in air at room temperature in the dark for 2 weeks until preparation for hardness testing. The specimens were embedded in acrylic cold mounting resin. After curing, each specimen was ground longitudinally half through the specimen. Hardness was then measured with a Wallace indentation hardness tester (H. W. Wallace, Croydon, UK) for each half millimeter, starting at 0.5 mm from the top surface. The Wallace hardness (H_w) measures the depth of penetration of a Vickers diamond under a pre-determined load of 100 g. The Wallace hardness number is thus a measure of softness: the higher the H_w, the softer the material (29).

Statistical analyses

For each specimen, a mean H_W value was calculated from the H_W values determined at the depths of 2.0 mm and less (0.5 mm, 1.0 mm, 1.5 mm, and 2.0 mm, respectively). The depth of cure for each specimen was then found by determining the greatest depth before a H_W value exceeding the minimal H_W value by 25% occurred. The choice of a 25% increase in H_W as determinant for the depth of cure was based on the following considerations. With a mean coefficient of variation of 12% of this study, a H_W value exceeding the minimal H_W by 25% is 2 SD distant from the mean. This implies that the probability of this value not being higher than the mean is less than around 2.5%.

Parametric as well as non-parametric statistical tests were used to analyze the results. Normally distributed data with homogeneous standard deviations were analyzed by Student's *t* test, one-way ANOVA, and in the case of statistical differences by Newman-Keuls' multiple range test (30). Remaining data were analyzed by Kruskal-Wallis H-test and by Wilcoxon's rank sum test (31). In the latter case, adjustments were made for the numerous pairwise comparisons by the Bonferroni method (32). A pre-set level of significance of 0.05 was used.

Results

Fig. 1 shows the results of a typical series of H_W measurement. In no case was a statistically significant increase in H_W found until a depth of more than 2.0 mm was reached. The depth of cure and maximal hardness attained (minimal H_W values) are given in Table 2. The minimal H_W results were distributed to allow for the use of parametric statistical methods. The values given in Table 2 were compared simultaneously for both materials, both curing times, and all curing units. It should be borne in mind that H_W really is a measure of softness, i.e. the higher the value of H_W, the softer the resin composite. The differences in minimal H_W were minor, and there were no

Table 1. Curing units included in the study

Curing unit	Type/mode	Power density (mW/cm^2)	Curing time (s)	Manufacturer
Demetron 2000	QTH/continuous	500	20 40	Demetron, Danbury, Ct., USA
Astralis 7	QTH/continuous	700	20 40	Vivadent, Schaan, Liechtenstein
Luxomax	LED/continuous	200	20 40	Akeda Dental, Lystrup, Denmark
Ultralume	LED/continuous	360	20 40	Ultradent Products, Inc., South Jordan, Ut., USA
Bisco VIP	QTH/pulse-cure	200 + 600	3 + 10 3 + 30	Bisco, Schaumburg, Il., USA
Elipar Trilight	QTH/soft-start	650	40	ESPE, Seefeld, Germany

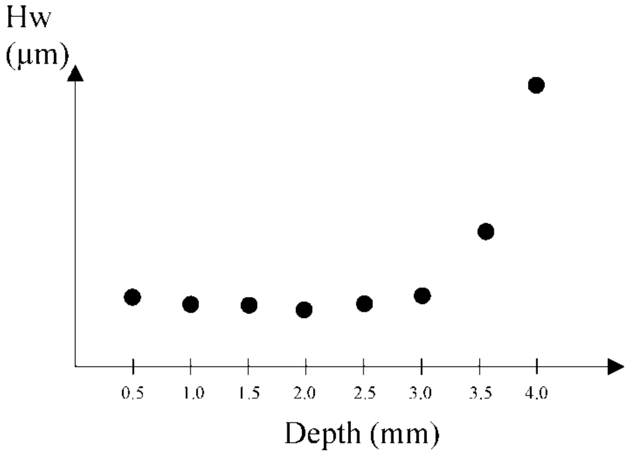

Fig. 1. Typical distribution of the Wallace hardness (H_W) measurements in relation to depth from the top surface. The exact values of Hw at a certain depth varied from one specimen to the next.

statistically significant differences between the curing times, between the two resin composites, nor between the curing units.

Because of lack of homogeneity of the standard deviations, the depth of cure data were analyzed by the non-parametric statistical methods described above. Table 2 gives the results of the statistical comparisons of the data obtained for each curing unit. Generally, prolonged curing yielded higher values of depth of cure, but statistically significant differences between the mean values were found only in 5 of the 10 cases. However, a statistical analysis of the *increases* in depth of cure obtained as a result of prolonged curing showed that prolonged curing significantly increased the depth of cure ($P < 0.0005$). Generally, higher depth of cure values were obtained with Tetric Flow than with Tetric Ceram, but statistically significant differences between the mean values were found only in 1 out of the 11 cases. However, a statistical analysis of the *differences* in depth of cure between the two materials showed that Tetric Flow yielded significantly higher depth of cure values than did Tetric Ceram ($P < 0.0005$). In order to compare the curing units, the depth of cure values of Tetric Ceram ($n_i = 5$) were pooled with those of Tetric Flow ($n_i = 5$) for each curing unit and curing time (i.e. $n_i = 10$). The mean values and standard deviations obtained fulfilled the prerequisites for use of parametric statistical methods; the mean values were compared by one-way ANOVA followed by Newman-Keuls' multiple range test. For the Bisco VIP curing unit, the curing times of 3 + 10 s and of 3 + 30 s were compared with those of the other curing units of 20 s and 40 s, respectively. At a curing time of 20 s, the curing units fell into 3 groups: Luxomax resulted in the significantly lowest depth of cure, Demetron 2000, Ultralume, and Bisco VIP, yielded higher and statistically similar depths of cure, and Astralis 7 yielded the significantly highest depth of cure. At a curing time of 40 s, Luxomax resulted in significantly lower depth of cure than all curing units except Demetron 2000, and Astralis 7 yielded a significantly higher depth of cure than did Luxomax and Demetron 2000. At both curing times, a significant linear correlation was found between the determined irradiances of the curing units

Table 2. Depth of cure and maximum hardness attained (minimal Hw) ($n = 5$)

	Depth of cure (mm)					Minimal Hw (μm)	
	Mean ± *s*	Median	Lowest	Highest	Statistics	Mean ± *s*	Statistics
Demetron 2000, 20 s, Tetric Ceram	2.7 ± 0.27	2.5	2.5	3.0	a	11.9 ± 0.9	ab
Demetron 2000, 20 s, Tetric Flow	2.8 ± 0.27	3.0	2.5	3.0	a	13.4 ± 1.1	ab
Demetron 2000, 40 s, Tetric Ceram	3.4 ± 0.42	3.5	3.0	4.0	ab	12.2 ± 2.2	ab
Demetron 2000, 40 s, Tetric Flow	3.8 ± 0.27	4.0	3.5	4.0	b	13.5 ± 1.5	ab
Luxomax, 20 s, Tetric Ceram	2.1 ± 0.22	2.0	2.0	2.5	a	11.4 ± 0.8	ab
Luxomax, 20 s, Tetric Flow	2.4 ± 0.22	2.5	2.0	2.5	a	13.1 ± 1.0	ab
Luxomax, 40 s, Tetric Ceram	3.0 ± 0.00	3.0	3.0	3.0	b	13.8 ± 3.0	ab
Luxomax, 40 s, Tetric Flow	3.2 ± 0.27	3.0	3.0	3.5	b	15.0 ± 1.4	b
Bisco VIP, 3 + 10 s, Tetric Ceram	3.0 ± 0.00	3.0	3.0	3.0	a	13.4 ± 1.7	ab
Bisco VIP, 3 + 10 s, Tetric Flow	3.1 ± 0.55	3.0	2.5	4.0	ab	13.7 ± 0.9	ab
Bisco VIP, 3 + 30 s, Tetric Ceram	3.9 ± 0.42	4.0	3.5	4.5	bc	12.3 ± 1.3	ab
Bisco VIP, 3 + 30 s, Tetric Flow	4.5 ± 0.00	4.5	4.5	4.5	c	12.4 ± 0.7	ab
Astralis 7, 20 s, Tetric Ceram	3.5 ± 0.50	3.5	3.0	4.0	a	13.2 ± 2.8	ab
Astralis 7, 20 s, Tetric Flow	3.9 ± 0.42	4.0	3.5	4.5	a	13.3 ± 1.7	ab
Astralis 7, 40 s, Tetric Ceram	4.3 ± 0.27	4.5	4.0	4.5	a	14.0 ± 2.6	ab
Astralis 7, 40 s, Tetric Flow	4.7 ± 0.45	5.0	4.0	5.0	a	14.0 ± 1.2	ab
Ultralume, 20 s, Tetric Ceram	2.8 ± 0.27	3.0	2.5	3.0	a	13.4 ± 1.3	ab
Ultralume, 20 s, Tetric Flow	3.1 ± 0.22	3.0	3.0	3.5	ab	13.2 ± 0.9	ab
Ultralume, 40 s, Tetric Ceram	3.5 ± 0.35	3.5	3.0	4.0	ab	12.9 ± 1.7	ab
Ultralume, 40 s, Tetric Flow	4.2 ± 0.27	4.0	4.0	4.5	b	15.0 ± 1.6	b
Elipar Trilight, 40 s, Tetric Ceram	3.8 ± 0.27	4.0	3.5	4.0	a	11.0 ± 0.8	a
Elipar Trilight, 40 s, Tetric Flow	4.5 ± 0.00	4.5	4.5	4.5	b	12.9 ± 0.5	ab

s = seconds; *s* standard deviation.
For each curing unit separately, depth of cure values characterized by same letter are not statistically different at $\alpha \leq 0.05$. Minimal Hw values characterized by the same letter are not statistically different at $\alpha \leq 0.05$.

(Table 1) and the pooled depth of cure values obtained (20 s: $r = 0.89$, $P < 0.025$; 40 s: $r = 0.91$, $P < 0.01$).

Discussion

A high degree of conversion is necessary to obtain good physical properties and low cytotoxicity of resin composites (24). It is therefore important for the clinician to know whether the thickness of the resin composite layer applied will be optimally cured. A high degree of conversion is partly related to the total irradiance reaching the material, which depends on the properties of the curing unit and the distance between the curing tip and the resin composite. The degree of conversion can be measured with techniques such as FTIR or Raman spectroscopy (33, 34). However, these techniques are time-consuming and complex. Asmussen (23) measured Wallace hardness and tensile strength and found that for a given resin composite they were well correlated to degree of conversion, and Rueggeberg et al. showed that degree of conversion is correlated to volumetric shrinkage (28).

In the clinic, a minimal distance between the light tip and the resin composite can only be obtained for the last increment of resin composite. The distance between the light tip and the bottom of the resin composite increment applied increases with increasing cavity depth. To imitate the clinical situation when the first layer of a Class II restoration is cured, all specimens were cured from a distance of 6 mm.

It has been recommended that flowable resin composite be used as bottom layer in Class II resin composite restorations in order to obtain better marginal seal. This has been verified in some studies (17, 18), while in others no effect was found (19, 21). However, the improvement in marginal adaptation shown in some studies could possibly be explained by a decreased degree of conversion and hence by decreased shrinkage. The aim of the present study was to compare a universal resin composite with its flowable version from a depth of cure point of view. Statistical analysis of the difference in depth of cure for both materials showed that Tetric Flow yielded statistically significantly higher depth of cure than did Tetric Ceram ($P < 0.0005$). A reason for this is probably that higher filler content in the universal version of the resin composite results in increased scattering of the light (35). It can therefore be assumed that the results are applicable for all combinations of universal/flow resin composites. The first hypothesis was accepted.

Harrington et al. found (36) that the irradiance at the resin composite surface decreased to approximately 1/3 when the light tip was held at a distance of 7 mm from the resin composite surface rather than in close contact with it. Rueggeberg et al. (28) have recommended that increments not thicker than 2 mm should be used when the resin composite is cured for 60 s with a QTH unit. Prati et al. (37) discussed on the basis of their results what happens in a clinical situation when a 2-mm increment is inserted in an 8-mm deep cavity. They stated that considering the reduction of power density through air and through composite only 4% of initial power density reaches the deeper layer of resin composite in contact with the dentin wall. This is in contrast to the present results. All the curing units used in this study were able to cure more than the recommended 2 mm despite a light tip distance of 6 mm and a curing time of 20 s (for Bisco VIP 3 + 10 s). For a curing time of 20 s the depth of cure was found to vary between 2.1 and 3.9 mm and for a curing time of 40 s between 3.0 and 4.7 mm. The increase in curing time led to a statistically significant higher depth of cure ($P < 0.0005$) for all the curing units and the second hypothesis was therefore accepted.

Curing units using LED were introduced in the early 21st century. These use junctions of doped semiconductors (p-n junctions) to generate light and require no filter to produce blue light. They have a narrower light emission spectrum compared to QTH units, between 440 and 490 nm, which falls within the camphoroquinone absorption spectrum (38). When first introduced, the irradiance of LED units was limited compared to QTH units. The first units used small LEDs arranged in an array where the number of LEDs was limited by the surface area. In newer LEDs the conventional diodes have been replaced by large-surfaced emitting LED chips, and irradiances up to 900 mW/cm^2 are now possible (39). Advantages of LED are that they are small, they can be made cordless and portable and they are quiet since they contain no fan. A disadvantage, however, is that most models are unable to cure composite containing photoinitiators with absorbance maxima lower than that of camphoroquinone. Both LED curing units in this study showed higher irradiances than the first LED units, but irradiances that were lower, as measured by the hand-held radiometer, compared to the QTH units. The LED Ultralume and the QTH Demetron 2000 units differed by 140 mW/cm^2, but showed depths of cure of similar magnitude. This is in agreement with the findings of Hofmann et al. (40) and may be explained by the fact that the higher irradiance registered for the QTH unit includes spectral ranges that do not activate camphoroquinone (41). Still, there was a statistically significant linear correlation at both curing times, between the determined irradiances of the curing units (Table 1) and the pooled depth of cure values obtained (20 s: $r = 0.89$, $P < 0.025$; 40 s: $r = 0.91$, $P < 0.01$). Therefore the third hypothesis was accepted.

In this present study there was no statistically significant difference in minimal H_W between the different curing units or curing times, which indicates that all curing units and curing times were able to cure the resin composites closer to the curing units to the same degree. It can be concluded that all curing units were able to cure more than 2.0 mm of the resin composite studied from a distance of 6 mm at a curing time of 20 s (for Bisco VIP 3 + 10 s). An increase in curing time to 40 s (for Bisco VIP 3 + 30 s) resulted in a statistically significantly higher depth of cure. Based on the results of the present study, it seems that the curing time of the studied material per 2 mm increment may be reduced from the 60 s previously recommended to 20 s, and that with a curing time of 40 s the increment thickness can be increased to 3.0 mm.

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