Flexural strength and modulus of a step-cured resin composite

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In an attempt to reduce marginal contraction gaps, a step-curing mode of light polymerization of resin composite has been proposed. It was hypothesized that such an exposure mode, having an initial reduced curing rate, would result in composite having a lower modulus of elasticity than composite exposed in one step. The composite was initially exposed to power density levels of 50, 100, 150, or 200 mW/cm^2 for durations of 10, 20, or 40 s. The final exposure and the exposure of the control group were performed at 750 mW/cm^2 for 20 s. It was found that certain modes of two-step exposure resulted in a lower flexural modulus than did a one-step exposure of constant, high power density. Regression analysis showed, with statistical significance, that flexural modulus was relatively high following a short initial exposure at low power density or using a long initial exposure at high power density. Conversely, flexural modulus was relatively low following a long initial exposure at low power density or using a short initial exposure at high power density. The results were explained by probable differences in degree of conversion and cross-link density of the polymer network. □ *Cross-linking; degree of conversion; linear structure; stepped polymerization*

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A reduced rate of resin composite polymerization may give rise to reduced contraction in restorations owing to stress relief (1, 2). This reduced rate may be brought about by so-called 'step-curing', where relatively low power density is used during the first part of light exposure (3). Step-curing has been found to result in polymers with varying susceptibility to softening in ethanol (4). This indirect indication of a different polymer structure resulting from a reduced polymerization rate was speculated to be a manifestation of a polymer structure that is more linear and having fewer cross-links. Only a few studies have focused on the mechanical properties of step-cured resins. With respect to transverse bending properties, flexural modulus may be a more sensitive indicator of structural differences in the polymer matrix than is flexural strength (5). In general, flexural modulus decreases as degree of conversion decreases and as the linearity of the polymer structure increases (6).

Thus, this study measured the flexural strength and modulus of a step-cured resin composite where the first step of the exposure occurred at varying power density and for varying periods of time. The hypothesis tested was that an initial, reduced curing rate of a resin composite would result in the same flexural strength, but lower flexural modulus than using a rapid initial cure rate.

Materials and methods

Light exposure

The investigation used a proprietary resin composite (Z100, 3M ESPE, St. Paul, Minn., USA). According to the manufacturer, aside from the filler particles this material contains the commonly used methacrylate monomers BisGMA and TEGDMA and as photoinitiator the likewise commonly used camphorquinone–amine system. The curing lights used in the investigation were XL 3000 (3M ESPE) and Elipar Highlight (3M ESPE, Seefeld, Germany). The power densities of the curing lights were 450 and 750 mW/cm^2, respectively, as measured by means of a handheld dental curing radiometer (Model 100, Demetron Research Corporation, Danbury, Ct., USA). In the control group, specimens were exposed to 750 mW/cm^2 for 20 s. In groups exposed in two steps, the power density of the initial exposure was varied by reducing the input voltage to the XL 3000 curing unit by means of a rheostat. The voltage was regulated in such a manner that power density values of 50, 100, 150, or 200 mW/cm^2 were applied. The duration of initial low power density exposure was 10, 20, or 40 s. Immediately after the initial exposure, specimens were given a final exposure at 750 mW/cm^2 for 20 s.

Flexural strength and modulus

Uncured resin composite was placed in a box-shaped brass mold (length 5 mm, height 1.0 mm, and width 1.0 mm). The specimen was covered on both sides with a transparent matrix and then irradiated as described from one side of the specimen. The matrices were then removed, the specimens freed from the molds and stored in air at 37°C at ambient humidity for 2 months.

Before the test, the height (a, mm) and width (b, mm) of each specimen were measured with a micrometer screw, after which they were subjected to 3-point loading with

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Table 1. Flexural strength of resin composite exposed using two-step exposure (MPa; means ± *s*; N = 10). The final exposure was performed using 750 mW/cm^2 for 20 s

Power density of initial exposure (mW/cm^2)	Duration of initial exposure (s)			
	0	10	20	40
50		190 ± 28	182 ± 30	190 ± 46
100		185 ± 46	199 ± 37	186 ± 36
150		193 ± 51	188 ± 47	198 ± 37
200		206 ± 52	215 ± 33	167 ± 33
Control	180 ± 29			

Analysis of variance showed that the mean values are not statistically different at $P = 0.05$.
s = standard deviation.

l = 3 mm between the cylindrical supports. The radii of supports and plunger were 0.5 mm. The crosshead speed of the testing machine (Instron Universal Testing Machine, Model 5566, High Wycombe, UK) was 1 mm/min. Flexural strength (S, MPa) was calculated as

$$S = 3F \cdot l/(2 \cdot a^2 \cdot b) \quad (1)$$

where F in N is the force at fracture (7). Flexural modulus (E, GPa) was calculated as

$$E = \beta \cdot l^3/(4 \cdot a^3 \cdot b) \quad (2)$$

where β is the slope in N/mm of the straight-line relationship between force and deflection of resulting flexural curve (7). Ten specimens were made for each group.

Statistics

Data were treated statistically by analysis of variance and regression analysis (8, 9). The pre-set level of statistical significance was $\alpha = 0.05$.

Results

Flexural strength values are presented in Table 1 and flexural modulus values in Table 2. There were no significant differences among the flexural strength groups. In contrast, statistical analysis of modulus of elasticity results indicated significant differences among the curing modes. The influence of energy density (power density multiplied by exposure duration) on flexural strength and flexural modulus was not significant. Excluding the control group, and considering values obtained using two-step exposure only, two-factor factorial ANOVA of modulus data showed an influence of initial exposure duration ($P < 0.001$), no influence of initial exposure power density, and a significant influence of the interaction of these two factors ($P < 0.01$). Since analysis of variance does not take the order of the variables into account, multiple component regression analysis was performed using the relationship

$$Z = a + b_1 \cdot X + b_2 \cdot Y + b_3 \cdot XY \quad (3)$$

in which Z is the modulus, X the power density, Y the exposure duration, XY the interaction, a the axis interception, and b_1, b_2, and b_3 are the regression coefficients. The analysis yielded the following relationship

$$Z = 7.0 - 0.0082 \cdot X - 0.024 \cdot Y + 0.00025 \cdot XY \quad (4)$$

in which all three regression coefficients statistically differed from zero. The relationship is depicted in Fig. 1 and has a correlation coefficient of R = 0.84.

Table 2. Flexural modulus of resin composite exposed using two-step exposure (MPa; means ± *s*; *n* = 10). The final exposure was performed using 750 mW/cm^2 for 20 s

Power density of initial exposure (mW/cm^2)	Duration of initial exposure (s)			
	0	10	20	40
50		^{a}6.0 ± 0.48	^{ab}6.3 ± 0.41	^{a}6.0 ± 0.34
100		^{a}6.0 ± 0.55	^{a}6.1 ± 0.50	^{ab}6.2 ± 0.44
150		^{a}5.8 ± 0.35	^{a}5.8 ± 0.25	^{b}6.5 ± 0.31
200		^{a}5.8 ± 0.35	^{a}5.9 ± 0.31	^{ab}6.2 ± 0.37
Control	^{b}6.5 ± 0.38			

Analysis of variance showed that the mean values are statistically different at $P = 0.0005$. Mean values with the same superscript letter do not differ from each other at $P = 0.05$.
s = standard deviation.

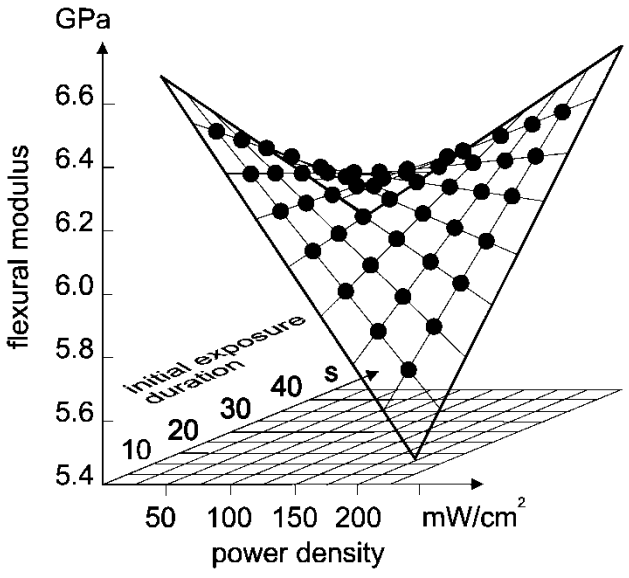

Fig. 1. Relationship between flexural modulus of a step-cured resin composite depicting the influence of exposure duration and power density during the initial exposure. The depicted relationship is based on multiple regression analysis of values presented in Table 2 and shown in equation (4).

Discussion

A resin composite was polymerized according to one of several step-curing modes, and flexural strength and modulus were determined. The research hypothesis expressed in the Introduction was validated: the flexural strength was not influenced by variation in initial exposure power density or duration, whereas certain modes of two-step exposure resulted in lower flexural modulus than did a one-step exposure of constant, high power density.

In view of the significant differences found in flexural moduli, flexural strength proved to be not a very sensitive indicator of structural differences. Differences in flexural modulus may be explained primarily by differences in chemical composition, degree of conversion, and degree of cross-linking (6). Obviously, for the same monomer composition, as is the case in the present investigation, only the latter two factors play a role.

With respect to degree of conversion, according to the so-called total energy concept (10–17), up to a limiting value, the degree of conversion is determined by the total amount of light received by the resin composite, i.e. the energy density: Thus, one would expect a significant correlation between modulus and energy density. However, no such correlation was found. Although certain deviations from the total energy concept are possible, as may be inferred from several recent studies (4, 18–21), it would seem that an explanation of the relationship exhibited in the Figure cannot be based exclusively on degree of conversion and that other factors must be involved.

With respect to the degree of cross-linking, and assuming a constant degree of conversion, differences may arise because of differences in the rate of cure as earlier discussed (22). At low initial cure rate, only a minor portion of camphorquinone content is activated, and relatively few growth centers are formed. Consequently, the propagation of polymerization will predominantly add one molecule of monomer after the other in a growing polymer chain manner. The result is that a predominantly linear polymer is formed before the final exposure is supplied. On the other hand, at high initial cure rates, many camphorquinone molecules are activated, forming many growth centers that increase the tendency to form a branched polymer.

On the basis of the above considerations, the saddle-shaped relationship presented in the Figure may be explained by differences in both degree of conversion and degree of cross-linking. It can be seen that, at low initial exposure power density and short initial exposure duration, flexural modulus is high. In this case, the energy density delivered in the initial exposure is so low that the main part of polymerization takes place during the high rate of the final exposure, giving the same properties as those of a resin composite in the control group. At low initial exposure power density, modulus decreases with increasing exposure duration because an increasingly higher proportion of polymerization occurs at a reduced rate, leading to an increasingly linear polymer. Using short initial exposures, modulus decreases with increasing power density, again because an increasingly higher proportion of polymerization occurs at a reduced rate. Finally, at high initial exposure power density, modulus increased with exposure duration. This behavior is best explained as the result of degree of conversion increasing with increasing energy density.

The resin composite used in the present study was a proprietary material based on commonly used monomers and initiators (23, 24). Although the material possibly contains minor amounts of undisclosed, polymerization enhancing compounds, it may still be considered as being representative for most resin composites on the market. This consideration implies that also the polymerization kinetics and results obtained in the present study may be of a general nature to many contemporary resin composites.

As mentioned in the Introduction, it is the aim of a step-curing exposure mode to reduce the stress development in restorations of resin composite. Such exposure modes may preserve marginal integrity to a higher degree, but the present study has shown that mechanical properties of the resin composite may be adversely affected. Only clinical studies can resolve whether the exposure mode should optimize marginal integrity or mechanical properties of photo-cured composite restorations.

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