# *In vitro* Depth of Cure of Photo-activated Composites

R. TIRTHA, P.L. FAN,* J.B. DENNISON, and J.M. POWERS

*School of Dentistry, The University of Michigan, Ann Arbor, Michigan 48109, and *American Dental Association, Chicago, Illinois 60611*

*The* in vitro *depth of cure of six photo-activated composites was studied by measurement of hardness, transverse strength, and light transmission coefficients. Both Barcol hardness and transverse strength were sensitive techniques for characterizing depth of cure. As a group, the visible light-activated composites had higher values of depth of cure and higher transmission coefficients than did the ultraviolet light-activated composites.*

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## Introduction.

Photo-activated composite restorative materials offer the distinct clinical advantage of control over working time. Since photo-activated composites require no mixing at the time of insertion, there is also less potential for incorporation of air in the restoration. Concerns exist, however, about the depth of cure of photo-activated composites.

The *in vitro* depth of cure has been reported to be from 2 to 6 mm for ultraviolet light-activated composites,[1-3] and from 2 to 8 mm for visible light-activated composites.[3] Factors affecting depth of cure include chemical composition of the composite,[2] mold materials and dimensions,[3] and intensity of the light source.[4] Depth of cure is also related to the transmission coefficient of the composite and the wavelength of the activating light, since these parameters affect the amount of light available in successive layers of the composite for activation of polymerization.

The purpose of this research was to study the *in vitro* depth of cure of six commercial photo-activated composites by measurement of hardness, transverse strength, and transmission coefficients.

## Materials and methods.

The depth of cure of six commercial photo-activated composites was studied *in vitro* by measurement of the differences in hardness between the top and bottom surfaces and the transmission of light in samples of various thicknesses. Codes, shades, batch numbers, and manufacturers of the products tested are listed in Table 1.

Three samples (10 mm in diameter) for each condition were prepared against a plastic matrix sheet in brass molds with thicknesses of 0.95, 1.0, and 1.35 mm, and from 1.5 to 3.5 mm in increments of 0.5 mm. Samples were cured using the recommended ultraviolet light ($W^+$, $X^\dagger$) or visible light ($Y^\ddagger$, $Z^\S$) sources for recommended times (60 s) with the light centered on the sample.

Barcol hardness readings$^{\infty}$ were obtained for each sample three min after curing. Both top (nearest the light source) and bottom surfaces were tested in the center, and the difference in hardness ($\Delta H$) between the two surfaces was calculated. Mean values of $\Delta H$ and standard deviations were determined. Data were analyzed by analysis of variance,[5] and mean values of $\Delta H$ were compared by Tukey intervals[6] calculated at the 95% level of confidence.

Transverse strength was determined for composite F as a function of $\Delta H$ obtained under different conditions of sample thickness and application of light. Strength (S) was measured in transverse (three-point) bending with an apparatus as described elsewhere,[7] and was calculated by $S = 3\ PL/2\ b\ x^2$, where P is the load, l is the length (15 mm), b is the width, and x is the thickness. Samples (20 mm in length and 2.5 mm in width) were prepared in dies 1.5, 2.5, and 3.0 mm thick, and were polymerized by two methods to vary the depth of cure: (1) by sweeping the light source across the sample length for 60 s, or (2) by holding the light source in three adjacent spots for 60 s each. Values of $\Delta H$ were determined for these samples as already described. The data of S *vs.* $\Delta H$ were analyzed by analysis of regression.[8]

Light transmission curves were obtained for uncured samples of composites ranging in thicknesses from 0.95 to 3.5 mm utilizing a double beam ultraviolet-visible spectrophotometer$^{\P}$ with an integrating sphere$^{**}$ as described elsewhere.[9] Transmission spectra were obtained for visible light-activated composites between 400 and 700 nm and for ultraviolet light-activated composites between 285 and 400 nm. Transmission coefficients were determined by measurement of intensities at 470 nm and 365 nm, respectively, for the aforementioned composites as described below.

The intensity of a monochromatic beam of light passing through a translucent medium can be described by $I = I_o\ t_c^x$, where $I_o$ is the intensity of the incident beam, I is the intensity of light after passing through a material of thickness x, and $t_c$ is the transmission coefficient defined as the ratio of $I/I_o$ when x is a unit thickness.[10] When $\log I/I_o$ is plotted as a function of x, the slope of the resulting line is $\log t_c$. Values of $\log t_c$ were determined by analysis of regression.[8]

## Results.

The differences in Barcol hardness ($\Delta H$) between the top and bottom surfaces of the composites are plotted as a

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+Nuva Lite, #197 D, L.D. Caulk Co., Div. of Dentsply International, Inc., Milford, DE 19968

†Lee Light, #1209, Lee Pharmaceuticals, South El Monte, CA 91733

‡Fotofil Light, #1889, Johnson & Johnson Dental Products Co., East Windsor, NJ 08520

§Translux Light, #2398, Reach Electronics, Inc., Lexington, NE 68850

∞Barcol Indentor, Barber-Colman Co., Motor Division, Rockford, IL 61101

¶ACTA C III UV-Visible Spectrophotometer, Beckman Instruments, Inc., Irvine, CA 92664

**ASPH-U Integrating Sphere, Beckman Instruments, Inc., Irvine, CA 92664

TABLE 1
CODES, SHADES, BATCH NUMBERS, AND MANUFACTURERS OF PHOTO-ACTIVATED COMPOSITES STUDIED

| Code Composite (Light Source) | Product (Light Source) | Shade | Batch Number | Manufacturer |
|---|---|---|---|---|
| Ultraviolet light-cured | | | | |
| A (W) | Nuva Fil (Nuva Lite) | Light | 061579 | L.D. Caulk Co. Div. of Dentsply Int'l., Inc. Milford, DE 19968 |
| B (W) | Nuva Fil (Nuva Lite) | Yellow | 1025793 | L.D. Caulk Co. |
| C (X) | Leefill (Lee Light) | Universal | 9344 | Lee Pharmaceuticals South El Monte, CA 91733 |
| Visible light-cured | | | | |
| D (Z) | Durafill (Translux Light) | Gray Opaque | 0KT79/12 DEZ80 | Phasealloy, Inc. El Cajon, CA 92021 |
| E (Z) | Durafill (Translux Light) | Gray | 0KT79/12 DEZ80 | Phasealloy, Inc. |
| F (Y) | Fotofil (Fotofil Light) | Universal | 9A-801 | Johnson & Johnson Dental Products Co. East Windsor, NJ 08520 |

function of sample thickness (x) in Fig. 1. The data were analyzed by analysis of variance, and a Tukey interval of 1.9 Barcol units was computed to show differences among means at different thicknesses. If $\Delta H$ of 5 Barcol units is considered to be a critical change in hardness, then corresponding values of critical depth of cure can be read from Fig. 1. These values are listed in Table 2 and varied from 1.4 mm for composite B to 2.7 mm for composite F.

The transverse strength (S) of composite F was determined for 14 samples polymerized under different conditions to vary $\Delta H$. A plot of log S *vs.* $\Delta H$ is shown in Fig. 2. The correlation coefficient (r) of the line determined from analysis of regression was 0.960. The critical value of r above which the hypothesis of independence of S and $\Delta H$ could be rejected was 0.532 at the 95% level of confidence. For $\Delta H$ of 5 Barcol units, composite F decreased in strength from 82 to 71 $MN/m^2$, a change of 13%.

Plots of log $I/I_o$ *vs.* sample thickness (x) for the composites are shown in Fig. 3. Slopes of these curves determined from linear regression are listed in Table 2 with the corresponding correlation coefficients. The transmission coefficients ($t_c$) listed in Table 2 were calculated from these slopes as described. Values of $t_c$ ranged from 0.03 $mm^{-1}$ for composite B to 0.49 $mm^{-1}$ for composite F.

The critical depth of cure determined from Fig. 1 by assuming a critical $\Delta H$ of 5 Barcol units is plotted as a function of the transmission coefficient ($t_c$) of the composites in Fig. 4. The correlation coefficient determined from analy-

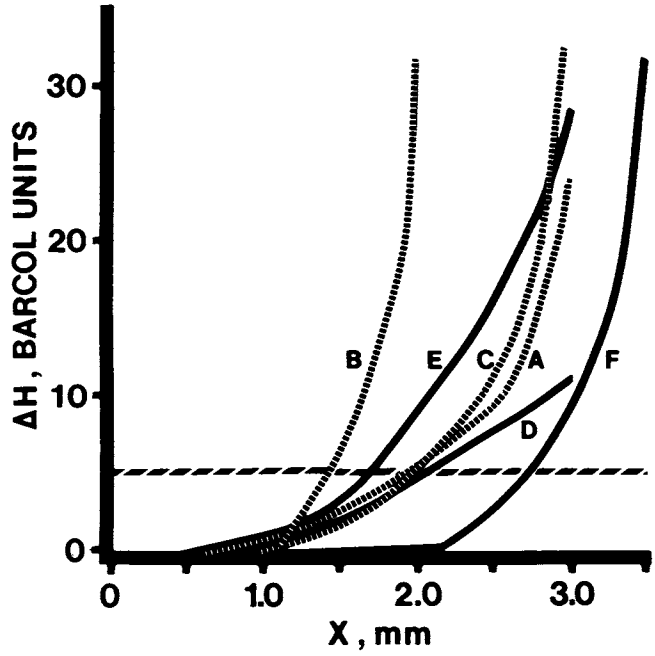


Fig. 1 – Graphs of differences in hardness ($\Delta H$) between top and bottom surfaces of composite *vs.* thickness (x) of composite for six composites. The critical difference in hardness (5 Barcol units) is indicated by the broken line.

TABLE 2
CRITICAL DEPTH OF CURE AND TRANSMISSION COEFFICIENT FOR PHOTO-ACTIVATED COMPOSITES

| Code | Critical Depth of Cure,* mm | Slope of log $I/I_o$ *vs.* x, $mm^{-1}$ | Correlation Coefficient† | $t_c$, $mm^{-1}$ |
|---|---|---|---|---|
| F | 2.7 | −0.313 | 0.998 (0.811) | 0.49 |
| E | 1.7 | −0.552 | 0.997 (0.878) | 0.28 |
| D | 2.1 | −0.589 | 0.998 (0.878) | 0.26 |
| C | 2.0 | −0.690 | 0.986 (0.754) | 0.20 |
| A | 2.0 | −1.158 | 0.927 (0.878) | 0.07 |
| B | 1.4 | −1.520 | 0.956 (0.950) | 0.03 |

*Thickness at which $\Delta H$ of 5 Barcol units occurred.
†The critical value of r for the 95% level of confidence is given in parentheses.

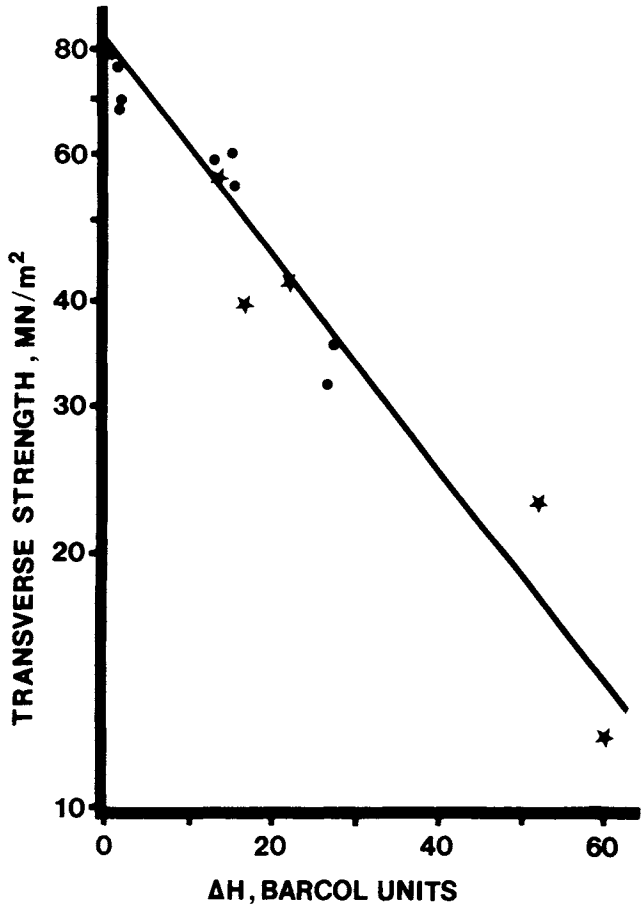


Fig. 2 – Graph of log transverse strength (S) *vs.* difference in hardness (ΔH) on a semi-log scale for 14 samples of composite F polymerized under different conditions to vary ΔH. • represents data for light source held in three spots for 60 s each, whereas x represents data for light source sweeping for 60 s.

sis of regression was 0.818. The critical value of r was 0.811 at the 95% level of confidence. As the transmission coefficient increased, the critical depth of cure also increased for the composites as a group, although there was a considerable amount of error (33%) associated with the correlation.

## Discussion.

The measurement of Barcol hardness of the top and bottom surfaces of a composite to evaluate depth of cure *in vitro* is a sensitive technique in which differences in hardness greater than 1.9 Barcol units are statistically significant. Values of critical depth of cure reported herein are in agreement with values reported by others[1-3] using a scraping technique for samples made in a metal mold. Larger values of depth of cure would be expected for samples made in poly (tetrafluorethylene) molds.[3] Correlations between these *in vitro* methods and *in vivo* results, however, have not been made.

The three-point bending test also appears to be a sensitive method to evaluate depth of cure of photo-activated composites. The inadequately polymerized bottom surface of a sample allows the knife edges of the sample holder to penetrate the sample. The load at failure is directly proportional to the actual thickness squared of the cured sample, whereas x (which includes both cured and uncured thicknesses) is used in computing the transverse strength (S).

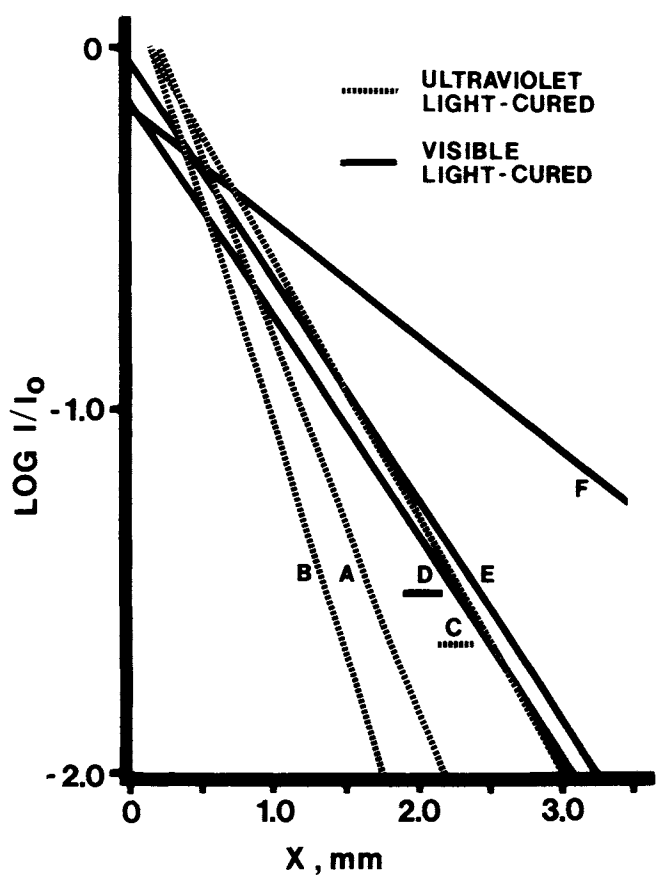


Fig. 3. – Graph of log $I/I_0$ *vs.* thickness (x) for six composites. The slope of each line is log $t_c$. The values of log $I/I_0$ of 0, – 1, and – 2 represent values of $I/I_0$ of 100%, 10%, and 1%, respectively.

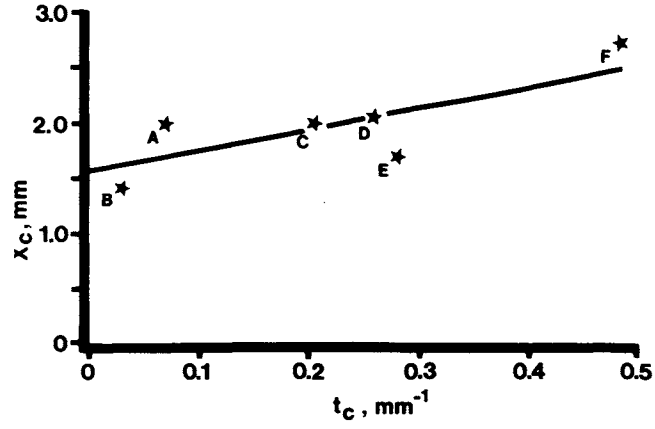


Fig. 4 – Graph of critical depth of cure ($x_c$) *vs.* transmission coefficient ($t_c$) for the photo-activated composites.

Composites with more shallow depths of cure would be expected to show larger decreases in transverse strength than would those with deeper depths of cure.

The visible light-activated composites (D, E, and F) as a group had higher values of critical depth of cure and higher transmission coefficients ($t_c$) than did the ultraviolet light-activated composites. The relatively poor correlation (r = 0.818) between critical depth of cure and $t_c$ suggests that other factors are important in the polymerization process.

Two factors are the type and concentration of the activator-initiator system and the intensity of the light source used to polymerize the composite. It is expected that if these two factors were constant, then a higher depth of cure would result from a higher value of $t_c$. The transmission coefficient itself is influenced by the wavelength of light and by the refractive indices of the resin and fillers, as well as by the size, shape, and amount of filler particles. Thus, darker and more opaque shades of composites might be expected to have lower values of $t_c$. To offset a lower value of $t_c$, the manufacturer may adjust the activator-initiator system to accommodate less light or recommend curing in thinner layers or for longer times.

Clinically, different shades of a manufacturer's photo-activated composite may require different curing techniques. Application of the composite in layers and adjustment of the exposure time or intensity of the light source may be necessary to polymerize some shades of composite completely, compared to the universal shade for which curing conditions are usually well-defined. Since the ultraviolet light-cured composites have low values of the transmission coefficient, curing of these composites should be given extra attention.

## Conclusions.

The depth of cure of six photo-activated composites was studied *in vitro* by measurement of hardness, transverse strength, and transmission coefficients. Barcol hardness was a sensitive technique for evaluation of depth of cure. A difference in hardness between the top and bottom surfaces of one composite of 5 Barcol units was equivalent to a 13% decrease in transverse strength. As a group, the visible light-activated composites had higher values of depth of cure and higher transmission coefficients than did the ultraviolet light-activated composites.

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