Depth of Cure of Dental Resin Composites: ISO 4049 Depth and Microhardness of Types of Materials and Shades

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Clinical Relevance

Achieving a high degree of cure throughout a 2 mm thickness of light-activated resin composite did not occur for many types and shades of resin composite. Clinicians should check the depth of cure by using the scraping method.

SUMMARY

The optimal degree of curing throughout the bulk of a visible light-activated dental resin composite is acknowledged to be important to the clinical success of a resin composite restoration.

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Unfortunately, the dentist has no means of monitoring the cure of the resin surfaces not directly exposed to the curing light. Techniques, such as the layered buildup of restorations in 2 mm increments with longer activation times than 20 seconds, have been suggested. This study investigated the depth of cure (DOC) of a commercial resin composite in three types: flowable, hybrid and packable and in three shades: B1, A3 and D3 after 20 second activation with a quartz halogen light (620 mW/cm²). Depth of cure was measured by scraping the uncured material and by using a Knoop Hardness profile, starting from the surface exposed to the light. Using a minimum Knoop Hardness ratio of 0.8 bottom/top only, the flowable in shade B1 achieved a 2 mm DOC. Using the less restrictive scraping test, only the B1 shade of flowable and hybrid significantly exceeded a 2 mm DOC. Knoop Hardness at the DOC obtained by scraping ranged from 55%-70% of the top surface hardness.

These data suggest that a 2 mm buildup layering technique may not result in adequate curing of the bottom layer for such a wide range of materials and that manufacturers need to pro-

vide quantitative information about DOC at specific activation times and light intensities for their entire range of resin materials and shades so that the dentist can devise a placement technique that will ensure adequate cure of the bulk of a restoration.

INTRODUCTION

The degree of cure of visible light activated dental resins was recognized as important to the clinical success of these materials soon after these materials were introduced. ¹⁻⁴ While the relative degree of cure of the external surface of a restora-

tion can usually be evaluated with simple techniques, the cure of the inner layers of resin is not similarly accessible to evaluation, and it was recognized early on that, unlike chemically activated resins, an adequate cure of the entire visible light activated restoration cannot be assumed, based on external surface properties. It has been shown that inadequate polymerization would result in a reduction in physical properties. Furthermore, components, such as residual monomer washed-out from the polymerized resin, may irritate soft tissue and predispose plaque accumulation, jeopardizing clinical success of the restoration.

The depth of cure of a visible-light activated resin has been the subject of considerable laboratory research.^{3,8-10} Even after more than 25 years of clinical use, there are still controversies about the depth of cure of a visiblelight activated resin.8 A number of different techniques have been employed to measure the properties of the polymerized resin composite most distant from the light source.11-12 These techniques include scraping away the unset material and measuring the remaining specimen. measuring top and bottom hardness and measuring top and bottom degree of conversion of double bonds in the polymer. 13-16 The scraping technique has been codified as the depth of cure measure in the ISO standard for dental resins 4049.12 To define depth of cure based on top and bottom hardness measurements, it is common to calculate the ratio of bottom/top hardness and give an arbitrary minimum value for this ratio in order to consider the bottom surface as adequately cured. Values of 0.80 and 0.85 have often been used. 13-14

Manufacturers and suppliers of dental resins rarely identify the basis for recommendations about depth of cure as they relate to light activation. An all too common recommendation is the use of some specific light exposure time to "cure" a 2 mm thickness. ¹⁵⁻¹⁷ It is well known that factors, such as resin type, filler levels, resin shade, intensity and spectrum of the activation light, influence the degree to which the bottom of a 2

Table 1: Resin Composites* Aeliteflo Aelite **Aelite BISCO** All Purpose **LS Posterior** Body **BISCO BISCO TYPE** Flowable Hybrid Packable **SHADES** B1, A3, D3 B1, A3, D3 B1, A3, D3 Ethoxylated Bisphenol A Dimethacrylate TEGDMA Χ Χ Glass filler Χ Χ Χ Χ Χ Amorphous silica

*Compositions taken from manufacturer's Material Safety Data Sheets BISCO (Schaumburg, IL, USA)

Table 2: Light Output Transmitted Through 1.3 mm Discs of Different Materials (mW/cm²)

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Resin Composites				
	Flowable	Hybrid	Packable	
B1	50	20	12	
A3	12	6	4	
D3	10	8	6	

mm thickness of material is cured,¹⁵ but little quantitative advice is typically available to guide the practitioner in adjusting placement technique.

This study investigated the depth of cure of three different shades (opacities) of a resin composite available in three different types that vary in viscosity and filler levels. Both the ISO scraping technique and a hardness profile (Knoop) from the top to the bottom of the specimen were used to evaluate depth of cure. The null hypothesis was that the shade and consistency of the resin composite would not interfere with the depth of polymerization.

METHODS AND MATERIALS

The resin composites employed are shown in Table 1.

Table 2 shows the amount of activation light transmitted through a 1.3 mm thickness of each of the types and shades. This was measured and decreased from flowable to hybrid to packable within a specific shade and from shade B1 to A3 or D3 within a material type.

Three specimens of each material type and shade, 4 mm in diameter and 6 mm deep, were condensed into Teflon molds. A 1 mm metal spacer was placed over the mold to hold the tip of the activating light back 1 mm from the surface of the resin. The specimens were activated for 20 seconds using a quartz halogen activation light (Visilux 2, Model 5520AA, Ser No 115449, 3M Dental Products, St Paul, MN, USA). Light output was checked with a hand-held radiometer (Power and Dose Meter, ACCU-CAL—30, Dymax Corp, Torrington, CT, USA) to ensure consistency between specimens

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(620mW/cm²). A 20 second light exposure using a common quartz halogen light¹8 was employed to simulate a typical clinical activation routine. Scraping away soft material after activation and measurement of the remaining specimen was followed by hardness measurements starting at the surface exposed to the light and proceeding through the length of the specimen.¹²

After activation, the specimens were immediately removed from the molds and the uncured material scraped away with a plastic spatula. The length of the remaining material was measured with a digital micrometer in three places and an average length was obtained. This value was divided by two to obtain the ISO 4049 depth of cure (DOC).12 Average values and standard deviations were calculated for the ISO DOC for each material and shade. After the DOC was measured, the specimens were embedded in epoxy resin and hemi-sectioned along their long axis. One sectioned half of each specimen was used for Knoop Hardness testing (KHN). A hardness profile was obtained by measuring starting at 0.5 mm from the top surface and measuring at 0.3-mm intervals to the bottom surface or until reliable Knoop Hardness could no longer be measured because the material was too soft. Knoop Hardness measurements were replicated three times at each position and an average determined. The hardness data for each of the three specimens was plotted against distance from the top surface, and a linear regression analysis was performed to estimate top hardness and rate of decline in hardness with depth from the top surface. The regression analysis data was also used in subsequent calculations to obtain Knoop Hardness values at specific distances from the top of the specimen. Confidence intervals from the regression analysis were employed to estimate error bars, when presenting KHN data.

RESULTS

Figure 1 compares the depth of cure as measured with the ISO 4049 scraping technique against the depth of cure estimated from the Knoop Hardness profiles using bottom/top hardness ratios of 0.85 and 0.80, respectively. Using the ISO criteria, 12 only the lightest shade (B1) of this resin system meets the 2.0 mm assumption for all three types of materials. Using the least restrictive KHN criteria of 0.8 of the top hardness, only shade B1 of the flowable yields a 2 mm depth of cure. In general, as expected from the light transmission data, the DOC is reduced with darker shades and more heavily filled types of material.

An alternate presentation of the data is shown in Figure 2. The DOC determined by the ISO technique was entered into the equations obtained from regres-

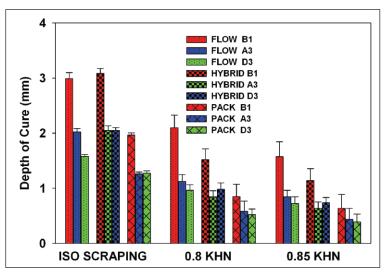


Figure 1. Depth of Cure of BISCO AELITE resin composites determined by ISO 4049 and from Knoop Hardness using either 80% or 85% of the hardness of the top surface.

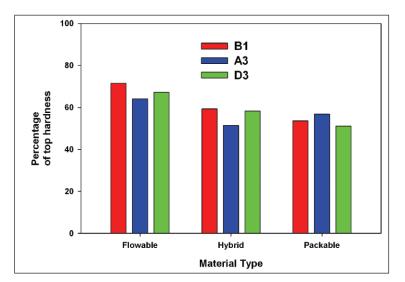


Figure 2. Percentage of top Knoop Hardness at depth of cure determined by ISO 4049.

sion analyses of the KHN profile data. The resulting KHN values were used to estimate the percentage of hardness at the ISO DOC compared to the top. With the exception of Flowable, the Knoop Hardness values of all materials and shades varied between 50% and 70% below the top surface at the ISO depth of cure.

DISCUSSION

The null hypothesis of this study, the shade and consistency not interfering with the depth of polymerization of a resin composite, was rejected due to the results (Figure 1). Although most clinicians would assume that shade D3 is darker than shade A3, both the light transmission data and depth of cure do not necessarily support this assumption. Using clinical

perception of relative darkness of shades may not result in adequate compensation if the clinician varies activation time to compensate for shade variation. If manufacturers use ISO 404912 to justify recommendations for curing times and depths of cure, the data in Figure 2 suggest a bottom hardness far below 80% of the top surface, with accompanying decreases in other mechanical properties. Since it has been shown that even a well polymerized resin composite can release some residual monomers and other reactive species, 19 it is reasonable to conclude that more substances would elute from poorly polymerized resin at the bottom of the restoration. These substances have the potential to irritate soft tissues and pulp, stimulate the growth of bacteria and promote allergic reactions. 7,20-21

The parameters used in this experiment are probably conservative compared to actual clinical situations. Based on measured light output, the curing light used would rank as a good quartz halogen activation light. The 1 mm distance between the light tip and resin surface is probably the minimum that is clinically feasible. Most recommendations for incremental buildup and curing of a dental resin suggest 2 mm thick layers. 18,22 This depth of cure is obtained for only one type and shade of material tested, using the least conservative hardness criteria. None of the sample groups met the 0.85 hardness ratio for a 2.0 mm thickness. The ISO criteria significantly inflated the depth of cure compared to hardness data for all materials tested.23 Even using this criteria, some of the types of material and shades did not have a DOC of 2.0 mm. The results of this investigation are in disagreement with some studies that have shown that 2mm increments are well polymerized²⁴⁻²⁵ but agree with other studies that show inefficiency of polymerization at a 2 mm depth.26 This study evaluated the same brand of materials, which is a limitation with respect to generalizing the conclusions. However, the objective was to show what happens to the depth of cure when composite formulation and shade are varied over a wide range from the standpoint of resin composites intended for different clinical applications. Clearly, studies should be done to evaluate the behavior of other brands of resin composite.

CONCLUSIONS

This study tested one brand of composite in flowable, hybrid and packable formulations in three shades (B1, A3 and D3) and indicates that the materials did not achieve a 2 mm depth of cure with 20 second light exposure when the bottom hardness of the specimens was measured. Despite using the ISO standard for depth of cure, not all materials met the DOC criteria at a 2 mm thickness. Since the clinician has no way of monitoring the degree of cure of the bottom of a resin composite

restoration, it is the opinion of the authors that the kind of testing reported in this study should be done by the manufacturers and suppliers of dental resin composites for all of their types of materials and shades, and the information be supplied to the dentist so that the placement technique can be modified to ensure reasonable properties for the full bulk of a resin composite restoration.

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References

- Cook WD (1980) Factors affecting the depth of cure of UVpolymerized composites Journal of Dental Research 59(5) 800-808.
- Rock WP (1974) The use of ultra-violet radiation in dentistry British Dental Journal 136(11) 455-458.
- 3. Yearn JA (1985) Factors affecting cure of visible light activated composites *International Dental Journal* **35(3)** 218-225.
- Ruyter IE & Oysaed H (1982) Conversion in different depths of ultraviolet and visible light activated composite materials Acta Odontologica Scandinavica 40(3) 179-192.
- Shortall AC, Wilson HJ & Harrington E (1995) Depth of cure of radiation-activated composite restoratives—influence of shade and opacity *Journal of Oral Rehabilitation* 22(5) 337-342.
- Ferracane JL, Mitchem JC, Condon JR & Todd R (1997) Wear and marginal breakdown of composites with various degrees of cure *Journal of Dental Research* 76(8) 1508-1516.
- Sideridou ID & Achilias DS (2005) Elution study of unreacted Bis-GMA, TEGDMA, UDMA, and Bis-EMA from light-cured dental resins and resin composites using HPLC Journal of Biomedical Materials Research. Part B, Applied Biomaterials 74(1) 617-626.
- Nomoto R, Asada M, McCabe JF & Hirano S (2006) Light exposure required for optimum conversion of light activated resin systems *Dental Materials* 22(12) 1135-1142.
- 9. Kawaguchi M, Fukushima T & Miyazaki K (1994) The relationship between cure depth and transmission coefficient of visible-light-activated resin composites *Journal of Dental Research* **73(2)** 516-521.
- Shortall AC, Wilson HJ & Harrington E (1995) Depth of cure of radiation-activated composite restoratives—influence of shade and opacity *Journal of Oral Rehabilitation* 22(5) 337-342.
- 11. Yap AU, Soh MS & Siow KS (2002) Effectiveness of composite cure with pulse activation and soft-start polymerization *Operative Dentistry* **27(1)** 44-49.
- ISO Standard (2000) ISO 4049 Polymer based filling, restorative and luting materials *International Organization for Standardization* 3rd edition 1-27.
- 13. Bouschlicher MR, Rueggeberg FA & Wilson BM (2004) Correlation of bottom-to-top surface microhardness and conversion ratios for a variety of resin composite compositions Operative Dentistry 29(6) 698-704.
- Aravamudhan K, Rakowski D & Fan PL (2006) Variation of depth of cure and intensity with distance using LED curing lights *Dental Materials* 22(11) 988-994 Epub 2006.

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 Rueggeberg FA, Caughman WF, Curtis JW Jr & Davis HC (1993) Factors affecting cure at depths within light-activated resin composites American Journal of Dentistry 6(2) 91-95.

- 16. Jain P & Pershing A (2003) Depth of cure and microleakage with high-intensity and ramped resin-based composite curing lights Journal of the American Dental Association 134(9) 1215-1223
- Rueggeberg FA, Ergle JW & Mettenburg DJ (2000) Polymerization depths of contemporary light-curing units using microhardness *Journal of Esthetic Dentistry* 12(6) 340-349.
- 18. Caldas DB, de Almeida JB, Correr-Sobrinho L, Sinhoreti MA & Consani S (2003) Influence of curing tip distance on resin composite Knoop Hardness number, using three different light curing units *Operative Dentistry* 28(3) 315-320.
- 19. Tanaka K, Taira M, Shintani H, Wakasa K & Yamaki M (1991) Residual monomers (TEGDMA and Bis-GMA) of a set visible-light-cured dental composite resin when immersed in water *Journal of Oral Rehabilitation* 18(4) 353-362.
- Lee SY, Greener EH & Menis DL (1995) Detection of leached moieties from dental composites in fluids simulating food and saliva *Dental Materials* 11(6) 348-353.

- 21. Lee SY, Huang HM, Lin CY & Shih YH (1998) Leached components from dental composites in oral simulating fluids and the resultant composite strengths *Journal of Oral Rehabilitation* **25(8)** 575-588.
- 22. Aguiar FH, Lazzari CR, Lima DA, Ambrosano GM & Lovadino JR (2005) Effect of light curing tip distance and resin shade on microhardness of a hybrid resin composite Brazilian Oral Research 19(4) 302-306. Epub 2006 Feb 14.
- DeWald JP & Ferracane JL (1987) A comparison of four modes of evaluating depth of cure of light-activated composites *Journal of Dental Research* 66(3) 727-730.
- 24. Lindberg A, Peutzfeldt A & van Dijken JW (2005) Effect of power density of curing unit, exposure duration, and light guide distance on composite depth of cure Clinical Oral Investigation 9(2) 71-76 Epub 2005 Apr 7.
- Rueggeberg FA, Ergle JW & Mettenburg DJ (2000)
 Polymerization depths of contemporary light-curing units using microhardness Journal of Esthetic Dentistry 12(6) 340-349
- 26. Fan PL, Schumacher RM, Azzolin K, Geary R & Eichmiller FC (2002) Curing-light intensity and depth of cure of resinbased composites tested according to international standards *Journal of the American Dental Association* 133(4) 429-434.