

Effect of Light-Curing Exposure Time, Shade, and Thickness on the Depth of Cure of Bulk Fill Composites

A Rodriguez • P Yaman • J Dennison • D Garcia

Clinical Relevance

Bulk fill composites can achieve higher depth of cure than conventional composite, but they do not always achieve a clinically adequate depth of cure. Time exposure and shade influence the depth of cure, especially at 4-mm thickness.

SUMMARY

Purpose: To investigate the effect of different light exposure times, shades, and thicknesses on the depth of cure (DOC) of bulk fill composites.

Methods and Materials: Two bulk fill composites, Tetric EvoCeram Bulk Fill (TBF) and Sonic Fill (SF), and a conventional composite, Filtek Supreme Ultra (FSU), were evaluated. Samples (n=10) were made using two different shades (light and dark), thicknesses (2 and 4 mm) and

exposure times (20 and 40 seconds). A Tukon 2100B-testing machine was used to obtain three Knoop hardness numbers (KHNs) measured at the top and bottom of each sample, and DOC was calculated as the bottom/top ratio. Statistical analysis was done using a Student *t*-test for comparisons between groups with a Bonferroni correction of $p < 0.004$.

Results: Top hardness values ranged from 79.79 to 85.07 for FSU, 69.49 to 91.65 for SF, and 51.01 to 57.82 for TBF. Bottom KHNs ranged from 23.54 to 73.25 for FSU, 45.74 to 77.12 for SF, and 36.95 to 52.51 for TBF. TBF had the lowest overall KHNs. Light-curing exposure time, shade, and material thickness influenced the DOC in most groups, especially at 4-mm depths. A higher bottom/top ratio was achieved when a 40-second cure was compared to a 20-second cure, when light shades were compared to dark shades, and when 2-mm increments were compared to 4-mm increments.

*Alexandra Rodriguez, DDS, MS, Department of Restorative Dentistry, University of Illinois at Chicago, Chicago, IL USA

Peter Yaman, DDS, MS, Department of Cariology, Restorative Sciences, and Endodontics, School of Dentistry, University of Michigan, Ann Arbor, MI, USA

Joseph Dennison, DDS, MS, Department of Cariology, Restorative Sciences, and Endodontics, School of Dentistry, University of Michigan, Ann Arbor, MI, USA

Daniela Garcia, DDS, MS, Department of Cariology, Restorative Sciences, and Endodontics, School of Dentistry, University of Michigan, Ann Arbor, MI, USA

*Corresponding author: 801 S. Paulina St., Room 321J, Chicago, IL 60612, USA, e-mail: alerod@uic.edu

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INTRODUCTION

Resin-based composites (RBCs) were introduced as restorative materials that would substitute for lost

tooth structures. Since the 1960s, the main components of RBCs have been modified to achieve improved mechanical, biological, and esthetic properties.¹ The filler particles enhance the mechanical properties of RBCs, such as modulus of elasticity, flexural strength, and hardness of the material; alter the thermal expansion behavior; and reduce their dimensional change.²⁻⁵ They also reduce the polymer content and consequently the polymerization shrinkage. There is also improvement in wear resistance, translucency, opalescence, radiopacity, water absorption, intrinsic surface roughness; and polishability as well as enhanced esthetics and handling properties.^{3,6} The filler sizes and shapes have been modified through time, and now most RBCs are hybrids, containing micro- and nanofiller particles and filler clusters.^{1,3,7,8} According to Lohbauer and others,⁹ RBCs should have a filler content of 60wt% to 87wt% to achieve acceptable characteristics.

The most common monomers used in RBCs are the bisphenolglycidyl methacrylate created by Bowen in 1956¹⁰ and urethane dimethacrylate by Foster and Walker in 1974.¹¹ These are usually diluted with low-viscosity monomers, such as triethylene glycol dimethacrylate and ethylene glycol dimethacrylate.^{2,3,12,13} The reaction in which monomers get activated and join together to form polymers is called polymerization.^{12,14,15} For light-cured RBCs to polymerize, they require a photoinitiator that has an absorption spectrum that coincides with the output of the light-curing unit (LCU) used.^{3,4,16-20}

Depth of cure (DOC) of composites is generally studied to verify that the light-curing technique used for each specific material is appropriate to adequately cure the RBC so that it will have good mechanical properties and optimal conversion of unreacted monomers. In this way, microleakage, which can lead to secondary caries and toxicity of unreacted materials, is minimized. The DOC of composite resins may be affected by the composite photoinitiator, filler type, matrix, shade and translucency, intensity, and spectral output of the curing unit and possibly the technique used when placing the composite.¹⁸

The DOC of RBCs can be evaluated *in vitro* following standard laboratory tests.²¹ Microhardness tests have been shown to be reliable to determine the DOC of RBCs.^{22,23} Knoop hardness numbers (KHNs) and Vickers hardness tests have shown strong correlation between them as well as with tests used to determine degree of conversion,^{22,23,25,26} whereas the International Organization for Standardization's

scrapping method overestimates the DOC of composites.^{21-23,26,27}

The bottom-to-top ratio (B/T) of KHNs is a good estimate of the completeness of polymerization at a specific depth from the light source. An RBC is considered adequately cured for clinical use when the microhardness measurements of the bottom are 80% or higher than the measurements at the top of the same sample.^{24,26-29,36}

In 1994, Rueggerberg and others³⁰ determined that for 2-mm- and 3-mm-thick samples, duration, intensity of the light exposure, and their interaction contributed to the degree of conversion of RBCs. They recommended using 60 seconds of light cure with an intensity of at least 400 mW/cm² for each 2-mm layer of composite. More recently, in 2013, Ilie and others³¹ suggested that when using an LED curing light, a minimum of 20 seconds should be used for a 2-mm layer of composite. Other studies have shown that the DOC of RBCs is material specific^{25,26,29,32-37} and that, in general, increasing the light cure exposure time increased the DOC.^{30,31,36,38}

The shade of the composite can also affect the DOC. Authors have shown that lighter shades with greater translucency achieve higher DOC than darker shades.^{24,32,35}

Recently introduced RBCs can be cured in 4-mm increments (bulk fill) using a short exposure time, according to the manufacturers. Czasch and others³⁴ showed that bulk fill placement of these composites had a lower degree of conversion only at low polymerization times. The physical properties of several bulk fill composites have been studied for creep deformation,³⁹ elastic modulus, nanohardness,⁴⁰ cuspal deflection, microleakage,^{41,42} shrinkage, surface gloss, light sensitivity, and surface wear.⁴³

The purpose of this study was to evaluate the effect of different curing light exposure times, composite shades, and material thicknesses on the DOC of bulk fill composites. The null hypothesis was that for each composite material, there was no difference in DOC when light exposure time, composite shade, or sample thickness was varied.

METHODS AND MATERIALS

The materials used in this study are presented in Table 1. Cylindrical composite samples were made using a standardized split brass mold 10 mm in diameter and either 2 or 4 mm thick. The molds were cleaned with 2 × 2 gauze soaked in alcohol and then

Table 1: Filler Content, Filler Composition, Monomer Composition, Depth of Cure (DOC), and Time Exposure Recommended by the Manufacturers and Shades of RBCs Used in This Study

Filler Content (%Vol)	Filler Composition	Monomer Composition	DOC	Time Exposure	Shades
Filtek Supreme Ultra (3M ESPE, St Paul, MN, USA)					
63.3%	Silica, zirconia, and aggregated zirconia/silica cluster fillers	Bis-GMA, UDMA, TEGDMA, Bis-EMA, PEGDMA	2 mm	20 s	A1B, A3B
Sonic Fill (Kerr, MNF, Orange, CA, USA)					
69%	Barium, aluminum, boron, silicate	Ethoxylated bisphenol-A-dimethacrylate, bisphenolA-bis (2-hydroxy-3-methacryloxypropyl) ether	5 mm	20 s	A1, A3
Tetric EvoCeram Bulk Fill (Ivoclar/Vivadent Amherst, NY, USA)					
61%	Barium glass filler, ytterbium trifluoride, mixed oxide	Bis-GMA, Bis-EMA, UDMA	4 mm	10 s	IVA, AVB
Abbreviations: Bis-EMA, bisphenolglycidyl ethyl-methacrylate; Bis-GMA, bisphenolglycidyl methacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.					

lubricated with mold-releasing agent (Al-Cote, Dentsply, Woodbridge, ON, Canada). The composites were inserted into the brass molds, and excess was removed with a metal spatula. The material was covered with a clear Mylar matrix, and a 1-mm-thick glass slide was pressed and stabilized with clips. The assembled samples were light cured with the Blue-phase LED curing light (Ivoclar/Vivadent Amherst, NY, USA) using a low-power setting (583 mW/cm²) as measured with a radiometer (Cure Rite Radiometer, Efos Inc, Mississauga, ON, Canada) during the study.

Ten samples for each of the selected shades (A1 and A3 for Filtek Supreme Ultra and SonicFill and IVA and IVB for Tetric EvoCeram) were light cured with each of the different time exposures (20 and 40 seconds). Once light cured, the glass slide was removed, and the samples were pushed out of the mold with finger pressure. A total of 240 samples were fabricated.

The samples were finished with a 600-grit silicon carbide paper (Mager Scientific, Dexter, MI, USA) on a rotary grinder-polisher (Micro Star 2000, Inc, Concord, ON, Canada) at a rotation speed of 102 rpm for one minute and then polished for an additional three minutes with moderate finger pressure with 800-grit silicon carbide paper (Mager Scientific). All samples were rinsed with water, dried, and stored in a manila envelope in a dark drawer at room temperature for 24 hours.

A Tukon 2100B-testing machine (Wilson Instrument, Norwood, MA, USA) was calibrated for micro-hardness testing. The Knoop diamond indenting tool was used to apply a 200-g load on the stabilized

samples with a dwell time of 15 seconds. Three measurements were recorded for both the top and the bottom surfaces of each sample. The indentations were measured using 20× magnification by positioning the cursors on both ends of the diagonal and measuring the length. The Tukon 2100B-testing machine gives the (KHNs), which are calculated using the formula

$$HK = L/I^2C_p,$$

where L represents the load applied (kg), I is the length of the long diagonal indentation (mm), and C_p is a constant to the projected area of the indentation (0.07028). The average of the three KHN readings on each surface were calculated and entered as a single data point to calculate the B/T ratio of each sample. Statistical analysis was done using a *t*-test for multiple comparisons between groups with a Bonferroni correction of *p* < 0.004.

RESULTS

Two light-curing exposure times, 20 and 40 seconds, and two shades were evaluated at 2- and 4-mm thicknesses to determine the effect on the DOC of one control and two bulk fill composites. Shade A1 for Filtek Supreme Ultra (FSU) and Sonic Fill (SF) and IVA for Tetric EvoCeram Bulk Fill (TBF) will be referred to as light, or "L," and shade A3 for FSU and SF and IVB for TBF as dark, or "D." The averaged top and bottom hardness for the three materials at the different times and shades are shown in Table 2. TBF had the lowest overall hardness numbers compared to the other materials, but this is related primarily to composition.

Table 2: Bottom and Top Means and Standard Deviations (SD) of Knoop Hardness Numbers of Different Resin-Based Composites								
	2 mm L		2 mm D		4 mm L		4 mm D	
	20 s Mean (SD)	40 s Mean (SD)	20 s Mean (SD)	40 s Mean (SD)	20 s Mean (SD)	40 s Mean (SD)	20 s Mean (SD)	40 s Mean (SD)
Filtek Supreme Ultra								
Top	82.00 (0.72)	79.79 (0.67)	83.23 (2.18)	81.04 (4.62)	85.07 (4.27)	83.09 (1.71)	84.23 (2.65)	84.03 (1.49)
Bottom	73.25 (0.73)	72.56 (0.92)	58.55 (4.72)	70.94 (4.45)	39.99 (5.68)	55.58 (4.36)	23.54 (2.94)	46.00 (4.39)
SonicFill								
Top	82.77 (3.26)	91.65 (2.85)	69.49 (3.59)	75.94 (1.48)	72.57 (2.86)	81.17 (1.28)	74.28 (1.74)	74.53 (2.02)
Bottom	74.49 (2.18)	77.12 (2.61)	62.32 (1.67)	70.81 (2.05)	59.18 (4.70)	71.77 (2.84)	45.74 (3.83)	60.18 (2.19)
Tetric EvoCeram Bulk Fill								
Top	55.95 (0.88)	57.79 (0.57)	57.82 (0.73)	53.72 (0.69)	51.01 (1.76)	54.85 (1.97)	56.14 (1.29)	52.66 (1.90)
Bottom	47.99 (1.41)	52.51 (0.38)	50.40 (1.04)	48.94 (1.05)	36.95 (2.34)	45.64 (2.63)	36.95 (2.93)	39.16 (1.73)

Exposure time comparisons are shown in Table 3. For 2-mm-thick samples, the exposure time did not have a significant influence on the DOC except for light shade SF and dark shade FSU. At 4-mm thickness, all materials had a statistically significant higher DOC when cured for 40 seconds compared to 20 seconds.

The different shades of composites (Table 4) did not have a significant influence in 2-mm-thick samples except for FSU at 20-second and SF at 40-second exposure times. When 4-mm-thick samples were evaluated, the light shade for all materials had a statistically significant higher DOC compared to the dark shade.

In general, 2-mm-thick samples achieve a higher DOC than 4-mm-thick samples (Table 5). At 2-mm thickness, only the dark shade FSU samples did not achieve an adequate DOC when cured for 20 seconds (.70). For light shades at 4 mm, only SF (.82) had an average DOC higher than 80% cure after 20 seconds of exposure, but both SF (.88) and TBF (.83) had 80% cure after 40 seconds. For a darker shade at 4 mm,

only SF (.81) had 80% DOC after 40 seconds of exposure.

DISCUSSION

This study measured top and bottom KHNs to calculate the DOC for two bulk fill composites and a conventional composite by evaluating different light-curing exposure times, shades, and thicknesses.

All top KHNs were higher than their corresponding bottom hardness for all samples (Table 2). As mentioned in previous studies, the hardness of the RBC materials decreases when the thickness of the material increases.^{22,26,35,37} In the present study, top and bottom hardness values were material specific (Table 2), coinciding with results of other studies.^{25,26,29,32-37} FSU had the smallest range of top KHN (5.27) followed by TBF (6.80). SF had a wider range of top KHN (22.15) and also had the highest top surface value of all groups (91.65). In general, TBF had the lowest top KHN (51.01). The wide range of top KHNs of SF might be related to its surface characteristics, which looked grainy and dark under the microscope, making it difficult to read the Knoop

Table 3: <i>Depth-of-Cure B/T Ratios of Groups Comparing Exposure Times; Mean (Standard Deviation) of Filtek Supreme Ultra (FSU), SonicFill (SF), and Tetric EvoCeram Bulk Fill (TBF)</i>												
	2mm L		p value	2mm D		p value	4mm L		p value	4mm D		p value
	20s	40s		20s	40s		20s	40s		20s	40s	
FSU	0.89 (0.01)	0.91 (0.01)	1	0.7 (0.07)	0.88 (0.08)	<0.001	0.47 (0.08)	0.67 (0.06)	<0.001	0.28 (0.04)	0.55 (0.06)	<0.001
SF	0.90 (0.03)	0.84 (0.04)	0.003	0.90 (0.04)	0.93 (0.03)	0.128	0.82 (0.06)	0.88 (0.04)	0.003	0.62 (0.05)	0.81 (0.04)	<0.001
TBF	0.86 (0.02)	0.91 (0.01)	0.045	0.87 (0.02)	0.91 (0.02)	0.611	0.73 (0.05)	0.83 (0.05)	<0.001	0.66 (0.05)	0.74 (0.04)	<0.001

Table 4: Depth-of-Cure B/T Ratios of Groups Comparing Shades; Mean (Standard Deviation) of Filtek Supreme Ultra (FSU), SonicFill (SF), and Tetric EvoCeram Bulk Fill (TBF)

	2mm 20s		p value	2mm 40s		p value	4mm 20s		p value	4mm 40s		p value
	L	D		L	D		L	D		L	D	
FSU	0.89 (0.01)	0.7 (0.07)	<0.001	0.91 (0.01)	0.88 (0.08)	0.261	0.47 (0.08)	0.28 (0.04)	<0.001	0.67 (0.06)	0.55 (0.06)	<0.001
SF	0.90 (0.03)	0.90 (0.04)	0.61	0.84 (0.04)	0.93 (0.03)	<0.001	0.82 (0.06)	0.62 (0.05)	<0.001	0.88 (0.04)	0.81 (0.04)	<0.001
TBF	0.86 (0.02)	0.87 (0.02)	0.13	0.91 (0.01)	0.91 (0.02)	1	0.73 (0.05)	0.66 (0.05)	0.001	0.83 (0.05)	0.74 (0.04)	0.003

hardness indentations. The surface appearance might be related to the ultrasonic vibration by the hand piece used to apply the composite and the higher filler content. The different hardness between materials might be related to their filler content by volume SF (69%), FSU (63.3%), and TBF (61%) as well their components (Table 1). None of the materials had KHNs comparable to the hardness of enamel as verified by Cardoso and others⁴⁴ (253 ± 26 to 478 ± 39 KHN).

It is possible that the sizes, radiopacity, translucency, and pigments of these filler particles influenced the light transmission through the material, affecting the DOC.³¹ The combination of monomers used in the three materials was also different, which, according to some studies, can influence the material properties.^{3,13,16-19}

The photoinitiators used are not specified by the manufacturers, and the DOC and other properties of a material depend on the photoinitiator's emission spectrum coinciding with the light output of the LCU.^{3,4,16-20} A slight mismatch in the wavelength of the light source and the sensitivity of the photoinitiator could limit the ability to successfully

maintain free radicals, which are responsible for the polymerization process.^{14,15}

Exposure Time

Some authors have found that when increasing the thickness of the samples, the exposure time should also increase to achieve a higher DOC.^{22,30,33,36-38} In general, the materials studied showed that the exposure time had a greater effect on the DOC of 4-mm samples compared to 2-mm samples (Table 3).

FSU was used as the control in this study. The manufacturers recommend light curing "body" (B) shades in 2-mm increments for 20 seconds. In the present study, 2-mm increments of A1B shade reached an adequate DOC when light cured for 20 seconds (0.89). This outcome was comparable to the results of the study done by Ilie and others,¹ who suggested that a minimum of 20 seconds of light-curing exposure time should be applied to a 2-mm increment of material light cured with an LED LCU. According to the present study, 2-mm samples of A3B shade required a higher exposure time (40 seconds) to obtain a B/T ratio higher than 0.80 (0.88).

Table 5: Depth-of-Cure B/T Ratios of Groups Comparing Thickness; Mean (Standard Deviation) of Filtek Supreme Ultra (FSU), SonicFill (SF), and Tetric EvoCeram Bulk Fill (TBF)

	L 20s		p value	D 20s		p value	L 40s		p value	D 40s		p value
	2mm	4mm		2mm	4mm		2mm	4mm		2mm	4mm	
FSU	0.89 (0.01)	0.47 (0.08)	<0.001	0.7 (0.07)	0.28 (0.04)	<0.001	0.91 (0.01)	0.67 (0.06)	<0.001	0.88 (0.08)	0.55 (0.06)	<0.001
SF	0.90 (0.03)	0.82 (0.06)	0.001	0.90 (0.04)	0.62 (0.05)	<0.001	0.84 (0.04)	0.88 (0.04)	0.012	0.93 (0.03)	0.81 (0.04)	<0.001
TBF	0.86 (0.02)	0.73 (0.05)	<0.001	0.87 (0.02)	0.66 (0.05)	<0.001	0.91 (0.01)	0.83 (0.05)	<0.001	0.91 (0.02)	0.74 (0.04)	<0.001

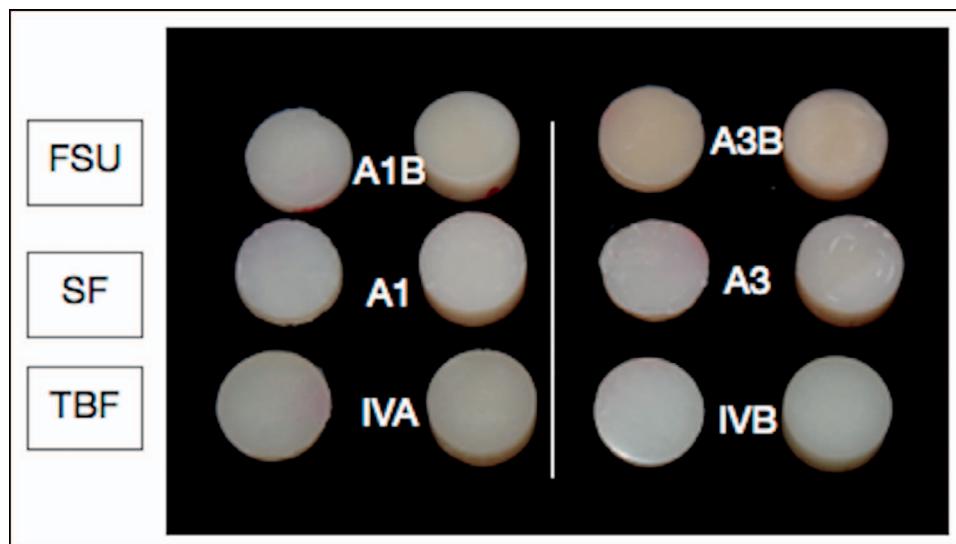


Figure 1. Samples of light and dark shades of the composites illustrating variation in shade as related to thickness of the samples; 2 mm (left) and 4mm (right).

SF samples of 2-mm-thickness shade A1 had a higher DOC when cured for 20 seconds (0.90) compared to 40 seconds (0.84). The statistical difference between these groups was significant ($p=0.003$, which is very close to the Bonferroni correction value of $p<0.004$). If the sample size would have been larger, there is a possibility of not finding any difference between light curing for 20 or 40 seconds for 2-mm-thick light shade samples. In 4-mm-thick samples, increasing the light exposure time increased the DOC of samples evaluated (0.82 to 0.88), as has been stated in other studies.^{22,30,33,36-38}

The effect of time exposure on DOC of TBF was similar to FSU. Two-millimeter samples of either IVA or IVB shade showed an adequate DOC when light cured for 20 seconds. When thickness was increased to 4 mm, samples exposed to a 40-second light cure showed significantly higher DOC than samples cured for 20 seconds, but only the light shade had a value above 0.80 (0.83).

Shade

The effect of shade has shown that lighter shades reach a higher DOC than darker shades (Table 4). Koupis and others³⁵ compared shades A2 and A4 light cured for 40 seconds, finding higher DOC on A2 samples. Moore and others²⁴ compared shades B1, A3, and D3 and also found higher DOC for the lightest shade (B1). In the present study, FSU had a higher DOC when A1B shade was compared to A3B, except in the 2-mm samples light cured for 40 seconds, where there was no statistically significant difference. When the shade of the A3B FSU composite is compared to other dark shade samples,

it can be noticed that FSU has a higher chroma, possibly containing more pigments or less translucency, which leads to a lower DOC at higher depths compared to other materials (Figure 1).

The comparisons of shade for SF showed that there was no influence of shade on 2-mm-thick samples light cured for 20 seconds, whereas 2-mm-thick samples light cured for 40 seconds showed a significantly higher DOC for A3 shade (0.93) compared to A1 (0.84). A similar result was found by Lazarchik and others,³² who established that at certain thicknesses, 2-mm-thick samples of shade A4 had a tendency to be harder than the A1 shade. There is a possibility that the darker shade had a higher concentration of photoinitiators that would be activated at decreased depths (2 mm), or it is more translucent to compensate for the pigmentation.³² As seen in Figure 1, the A3 shade of SF was different from the A3B shade of FSU. Light shade samples of 4-mm depths light cured for either 20 or 40 seconds had a higher DOC than the dark shade groups; this has also been shown in previous studies.^{24,32,35}

At 2-mm depths, the shades of TBF did not influence the DOC. For 4-mm-deep groups, IVA shade reached significantly higher DOC than IVB.

Thickness

All the materials had significantly lower DOC for 4-mm samples compared to 2-mm samples, except for SF shade A1 cured for 40 seconds, which had no difference in DOC between thicknesses (Table 5). Other studies have also established that DOC decreases when material thickness increases.^{22,26,35,37}

FSU did not reach an adequate DOC for 2-mm shade A3 samples cured for 20 seconds (0.47) or for any 4-mm-thick sample. One of the reasons for this can be attributed to the composition of the FSU composite, which has microfilled clusters that can increase the light dispersion, as mentioned by DeWald and others.²³ Koupis and others³⁵ also stated that the filler composition of RBCs influences their translucency, hardness values, and DOC.

According to the manufacturer, SF should reach a 5-mm DOC of more than 86% when light cured for 20 seconds with a light output of 550 mW/cm². The manufacturer does not mention the mechanism and/or components that this material uses to achieve a higher DOC compared to conventional composites. All SF 2-mm samples had a higher DOC than the 4-mm samples, except for the light shade cured for 40 seconds, where the DOC of 2- and 4-mm thickness was statistically similar. Most likely, the translucency of the light shade composite and its photoinitiator and monomer compositions permit the light to activate the polymerization process throughout a 4-mm increment of material when light curing for a 40-seconds time period.

For SF, all the groups evaluated had a mean B/T ratio higher than 0.80, except 4-mm samples of A3 shade cured for 20 seconds (0.62). From the groups that achieved a mean DOC of 0.80, the 2-mm A1 shade cured for 40 seconds, 4-mm A1 shade cured for 20 seconds, and 4-mm A3 shade cured for 40 seconds had around 25% of the samples below a B/T ratio of 0.80. This shows that this material does not always achieve an adequate DOC even if it is light cured for 40 seconds. With the results of this study, it is difficult to predict a DOC of 5 mm when using SF as recommended by the manufacturer.

TBF is advertised to achieve a DOC of 4 mm when light cured with an LED LCU ≥ 1000 mW/cm² for 10 seconds. In the present study, a light output much lower than the recommended one was used (583 mW/cm²) for a longer period of time (20 or 40 seconds). The manufacturers claim that this material has an additional photoinitiator (polymerization “booster”) that ensures a long working time. The exact properties of this photoinitiator that may influence the properties of the composite are unknown. All 2-mm-thick samples of TBF reached an adequate DOC above a B/T ratio of 0.80. From the 4-mm-thick samples, only the IVA shade cured for 40 seconds achieved an acceptable DOC (0.83), although 25% of these samples were below the 0.80 B/T ratio.

CONCLUSIONS

- At greater thickness (4 mm), a longer exposure time increases the DOC for all materials tested.
- Shade had a greater influence at increased depths, showing higher DOC for light shades compared to dark shades.
- Bulk fill composites achieved the highest DOC. At 4-mm thickness, all samples of only SF A1 shade samples achieved a DOC higher than 0.80. SF A3 shade and TBF IVA shade samples achieved an average DOC above 0.80, but about 25% of the samples did not reach that threshold.

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Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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