Original Article

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The effect of resin composite thickness and light exposure time on the curing depth of three high viscosity bulk-fill resin composites

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Objectives: To evaluate the influence of resin composite thickness and light exposure time on the depth of cure of three high viscosity bulk-fill resin composites by using the Knoop Microhardness number (KHN).
Material and Methods: Three bulk-fill materials (Tetric® N-Ceram Bulk Fill – TNBF; SonicFill™– SF; Filtek™ Posterior Restoration Bulk Fill – FBF) were prepared using a metal mold with four different thicknesses (2, 3, 4, and 5 mm) (*n*=15). Fifteen conventional nanocomposite (Filtek™Z350 XT; Z350) were prepared for 2 mm thickness. The 2, 3, 4 mm thickness groups were cured for 20 seconds and the 4, 5 mm thickness groups were cured for 40 seconds using a light emitting diode (LED) (Elipar™ S10, 3M ESPE) with standard curing mode with output wavelength range 430-480 nm, and 1,200 mw/cm^2 light intensity. All specimens were stored in the dark storage (37ºC, 24 hours) before submitting to KHN test. Data were analyzed using one-way ANOVA, Tamhane's T_2 post-hoc test, and independent samples *t*-test (α=0.05).
Results: With increasing specimen thickness, all bulk-fill materials showed significant decrease in KHN (p<0.05) except FBF-2mm that was not significant with FBF-3mm. At 4 mm, only FBF achieved the optimal mean percentages of bottom to top of KHN (80% KHR). With increasing light exposure time to 40 seconds, all bulk-fill materials increased in KHN and reached 80% KHR at 4 mm. However, at 5 mm, KHR of all materials was less than 80%.
Conclusion: With respect to KHR, all bulk-fill materials can safely be used for bulk filling up to 4 mm depth using 40 seconds of curing light.

Keywords: bulk-fill resin composite, depth of cure, light exposure time, microhardness

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Introduction

The use of light-cured resin composite material for restoring posterior teeth has continued to increase due to its ability to mimic the natural tooth color, to patient concern of mercury toxicity, and to avoid the discomfort of galvanic sensitivity in case of amalgam restoration. Moreover, tooth structure can be preserved by the use of a suitable adhesive. However, the limitation of light-cured resin composite material is the "depth of cure" (DOC). This refers to the maximum depth that resin composite can be adequately cured in the cavity. For conventional resin composites, an incremental technique is presently a golden standard for sufficient polymerization [1] in the placement of resin-based restorations. However, the incremental technique is time consuming and

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increases the risk of moisture contamination or voids trapped between layers. This is especially the case when restoring a deep cavity [2]. To overcome these disadvantages, the recently developed resin composites, called "Bulk-fill" resin composites, have become more popular. These materials have been reported to place up to 4-5 mm thickness in one increment without having to extend the light exposure time [3, 4]. The modifications in these bulk-fill resin compositions include new composite-filler technologies, modified pre-polymer shrinkage stress relievers, altered photo-polymerization modulator and a new initiator system [4]. These improvement aims to achieve the proper depth of cure in the deep cavity along with lower polymerization shrinkage [5].

Bulk-fill materials can be classified into two types, low-viscosity (fluid) and high-viscosity (packable) resin composites, based on the viscosity and application technique. However, the low-viscosity bulk-fill resin composite requires capping with conventional resin composite at the top of the cavity due to low mechanical properties of the material, low surface hardness, low modulus of elasticity [5], high water absorption and high polymerization shrinkage [6]. In contrast, the high viscosity bulk-fill resin composite can be placed without adding a capping layer. However, the restoration of posterior teeth with "bulk-fill" material in a deep cavity (as the manufacturer's instruction) is still a matter of concern because of the bulk material may not be cured properly, especially at the bottom of the cavity. Therefore, a study of the quality of the polymerization is both necessary and important. This can be done by evaluating the conversion of monomer to polymer, called "the degree of conversion (DC)" [7]. The DC depends on the amount of light absorbed, as well as scattered, within the material. These optical properties are influenced by the amount, size, and type of fillers, their color, the photo-initiator type and concentration, any refractive index mismatch, the nature of the light exposure source, and the exposure time [8]. Insufficient DC results in a decrease of the physical, mechanical [9] and biological properties [10] of resin composites. A definite DC for clinical used has not been established. However, it is known that there is an undesirable correlation of abrasive wear depth with DC value in the range of 55-65% [9]. Consequently a value of DC below 55% is not recommended for acceptable clinical performance [11].

Therefore, to achieve a good clinical outcome and for long-term durability of light-cured resin composites, the optimal depth and light exposure time for those bulk-fill materials in use should be investigated.

There are many methods to determine DOC and DC. One of the most common tests for assessing the DOC is a microhardness measurement. This is defined as the resistance to permanent indentation or penetration of the material. The DOC is considered to be acceptable when a resin composite achieves at least 80% of its maximum hardness [4].

Most of the reported DOC studies have focused on evaluating the microhardness of the bulk fill resin composites after exposure to light from a light-emitting diode (LED) for 20 seconds [12]. Therefore, the aim of this study was to evaluate the depth of cure of a conventional nanofill resin composite with a thickness of 2 mm and three high viscosity bulk-fill resin composites by using a Knoop microhardness (KHN) test. These were examined at 2, 3, 4, 5 mm thicknesses. Thicknesses of 2 to 4 mm were initiated using a LED for 20 seconds and thickness 4 and 5 mm were cured for 40 seconds. The hypotheses examined in this study were that:

1) At 2 mm thickness, all resin composites have no significant difference in KHN.

2) All bulk-fill resin composites will achieve a bottom-to-top hardness ratio of 0.80 at 4 mm using a LED with 20 seconds of exposure time.

Materials and methods

A conventional nanofilled resin composite (Filtek™ Z350 XT, shade A2, 3M ESPE, St. Paul, MN, USA) and three high-viscosity bulk-fill resin composites, which were Tetric® N-Ceram Bulk Fill - TNBF (shade A2, Ivoclar Vivadent, Schaan, Liechtenstein), SonicFill™ - SF (shade A2, Kerr, Orange, CA, USA), and Filtek™ Bulk Fill - FBF (shade A2, 3M ESPE, St Paul, MN, USA) were used in this study (Table 1).

Two hundred and twenty-five bulk-fill specimens were prepared using custom made metal molds with 2 mm diameter and four different thicknesses (2, 3, 4 and 5 mm) and fifteen Z350 specimens were prepared using 2x4 mm (thickness x diameter) as a control. A celluloid strip was placed on a glass plate and then the metal mold was placed on a celluloid strip. The bulk-fill resin composite was placed into the mold until it was slightly overfilled. A second celluloid strip was placed over the resin composite and another glass plate was gently pressed to extrude excess material. The specimens were then cured using a LED unit (Elipar™ S10, 3M ESPE, St. Paul, MN, USA) with the standard curing mode. The output wavelength ranges 430-480 nm, and 1,200 mw/cm^2 light intensity for 20 seconds for 2-4 mm thickness groups and 40 seconds for 4 and 5 mm thickness groups. This was achieved by positioning the light tip to be in contact with the glass slide on the top surface of the specimen. The light intensity of the curing light unit was calibrated using a LED radiometer (Bluphase® Meter II, Ivoclar Vivadent). All specimens were stored for 24 h in distilled water at 37ºC in the dark prior to KHN measurements.

Table 1 Manufactures' information about resin composite materials used in the study

Composite materials/ Type (Code)	Manufacturer	Shade/ LOT	Resin matrix	Filler	Filler content (wt%/ vol%)	Manufacturers' recommendation composite layer thickness	Manufacturers' recommendation light exposure time
Filtex™ Z350 XT Nanocomposite / Nanofilled (Z350)	3M ESPE, St. Paul, MN, USA	A2/ N576840	Bis-GMA, UDMA, TEDGMA, Bis-EMA	20 nm silica glass, 4 to 10 nm zirconia filler	79/63	2 mm	≥ 400 mW/ cm^2/20s
Tetric N-Ceram® Bulk fill / Nanohybride (TNBE)	Ivoclar Vivadent, Schaan, Liechtenstein	IVA/ T14296	Bis-GMA, Bis-EMA, UDMA	100 nm to 1 µm Ba Al silicate glass filler, prepolymer particles, 200 nm ytterbium fluoride filler, 160 nm mixed sphericle oxide filler	80/61	4 mm	≥ 1000 mW/ cm^2/10s
Filtek™Bulk Fill / Nanofilled (FBF)	3M ESPE, St. Paul, USA	A2/ N611596	AUDMA, AFM dimethacrylate, DDDMA, UDMA	20 nm silica filler, 4 to 11 nm zirconia filler, and 100 nm ytterbium fluoride filler	77/58	4 mm	≥ 1,000-2,000 mW/cm^2/20s
SonicFill™ Sonic-Activated bulk fill / Nanohybrid (SF)	Kerr, Orange, CA, USA	A2/ 5309506	Bis-GMA, TEGDMA, Bis-EMA	Silicon dioxide, barium glass	84/66	5 mm	≥ 550 mW/ cm^2/20s

Knoop Microhardness number (KHN) was measured on the top (0 mm) and bottom surfaces of each specimen using a microhardness tester (FM-ARS-9000, Future-Test Corp., Kanagawa, Japan) with a load of 100 g for 15 seconds at room temperature (25±1C). Five random indentations were performed on each surface of every specimen. Data was recorded as the Knoop microhardness number (KHN), which was computed by measuring the dimensions of the indentations using the formula:

$KHN = 14230 (F/d^2)$

where F is the indentation load (g), and d is the diagonal of the indentation (μm). This was then converted into the Knoop microhardness ratio (KHR) according to the following equation:

% KHR = (bottom KHN mean value/top KHN mean value) x 100

Statistical Analysis

The KHN at the bottom surfaces of different thicknesses of each material were compared using one-way ANOVA and Tamhane's T_2 post-hoc test. The comparison between the KHN at 4 mm and 5 mm thicknesses when using 20 seconds and 40 seconds irradiation times were compared using independent samples *t*-test.

Statistical analyses were performed using IBM SPSS Statistics for windows (version 16). The variance parameter α=0.05 was considered to be statistically significant in all tests.

Results

The mean and standard deviation of Knoop microhardness numbers (KHN±SD) and Knoop microhardness ratio (KHR) at the bottom of all materials tested with different thickness using 20 seconds and 40 seconds of light curing time are shown in Table 2 and 3, respectively. Statistical analysis indicated that mean microhardness was affected by resin thickness (F=30.578, $P < 0.001$) and light curing time in 4, 5 mm resin thickness (F=6.258, $P < 0.001$). At 2 mm thickness, the conventional resin composite (Z350) had the highest KHN (52.801.76) followed by SF (52.462.28) and FBF (51.392.29); however, there was no significant difference among those three groups ($P < 0.05$). Additionally, TNBF had the lowest KHN (35.781.09) and showed a significantly different value from the other groups ($p<0.05$). By increasing the specimen thickness, all groups showed significant decrease in KHN at 2, 3 and 4 mm ($p<0.05$) except FBF-2 mm and FBF-3 mm that showed no statically significant difference (Table 2).

With respect to 80% of Knoop microhardness ratio (80%KHR), only FBF achieved 3 and 4 mm thickness, while TNBF attained at 3 mm. All groups were not polymerized at the 5 mm depth when using LED curing unit for 40 seconds (Table 3).

Table 2 Mean Knoop Microhardness number (KHN) (±SD) and Knoop Microhardness ratio (KHR) at the bottom of all materials tested with 20 seconds of light exposure time.

Groups (n=15)	2mm		3mm		4mm	
	Mean(SD)	KHR	Mean(SD)	KHR	Mean(SD)	KHR
Z350	52.80 (1.76)	86.88	-	-	-	-
SF	52.46 (2.28)	92.98	44.12 (1.44)	79.59	30.65 (2.43)	55.05
TNBF	35.78 (1.09)	94.98	31.57 (1.01)	88.55	29.53 (1.05)	79.43
FBF	51.39 (2.29)	94.53	49.39 (1.25)	91.13	43.26 (1.44)	80.08

Table 3 Mean Knoop microhardness (KHN) (±SD) and Knoop Microhardness ratio (KHR) at the bottom of bulk-fill materials tested at 4 mm and 5 mm with 40 seconds of light exposure time.

Composite materials	Light exposure time (seconds)	4 mm		5 mm	
		Mean (SD)	KHR%	Mean (SD)	KHR%
SF	40	44.52 (1.27)	82.84	24.45 (2.58)	44.82
TNBF	40	35.48 (2.05)	91.27	23.53 (0.79)	66.31
FBF	40	51.12 (1.28)	93.71	38.42 (2.09)	72.78

With regard to the light curing time, all 4 mm thickness bulk-fill resin composites with 40 seconds of light curing produced significantly higher KHN than did 20 seconds. For this 40 seconds light curing time, 4 mm thickness bulk-fill resin composite achieved 80% KHR while 5 mm thickness specimens had KHR lower than 80% (Table 3).

FBF had the highest KHN at 4 and 5 mm thicknesses, while TNBF had the lowest KHN. In all bulk-fill resin composites, KHN of the 4 mm thickness specimen was significantly higher than that for samples with 5 mm thickness. Noticeably, FBF with 3 and 4 mm thickness were statistically different. However, 5 mm thickness FBF with 40 seconds light curing had significantly lower microhardness and lower KHR than 2 mm thickness FBF with 20 seconds light curing. Moreover, considering the position of measurement, all specimens demonstrated a significant difference in microhardness between top and bottom areas of measurement ($p<0.05$), which KHN on the top surface was higher than that on the bottom surface (Table 4).

Discussion

Recently, a new type of bulk-fill resin composite, high-viscosity bulk-fill, has been introduced into the market. It has been claimed that the main advantage of this material is the ability to be placed in bulk of thickness 4-5 mm without capping. Further, the recommended light exposure time and the light curing unit can be the same as those used with conventional resin composites [13]. However, adequate polymerization, especially in the deeper parts of filling, is an important factor for clinical success. Increasing the depth of cure of bulk-fill resin composite may require an increase in translucency. These requirements can be achieved by modifications in the filler content and/or organic matrix with advanced technology [14].

Table 4 Mean Knoop microhardness at the top and bottom areas of three bulk fill resin composites when light cured for 20 and 40 seconds.

Groups	TNBF		FBF		SF	
	Top	Bottom	Top	Bottom	Top	Bottom
2mm 20s	37.62	35.78	54.47	51.39	56.94	52.46
3mm 20s	36.02	31.57	54.19	49.39	55.43	44.12
4mm 20s	37.18	29.53	54.06	43.26	55.66	30.65
4mm 40s	38.87	35.48	54.55	51.12	53.74	44.52
5mm 40s	35.40	23.53	52.79	38.42	54.76	24.45

Theoretically, translucency varies inversely as the amount of filler particles [5]. Therefore, a simple method for improving material translucency is to reduce the amount of filler. Some types of bulk-fill resin composites contain a lower filler amount and an enlarged filler size. However, increased filler size has been shown to decrease light transmission. This is due to lower light scattering at the filler interface when the particles are smaller than the wavelength of the incident blue light. When cross-linking starts, the density and the refractive index of the polymer matrix increase, approaching the refractive index of the filler. Thereby, scattering is reduced and then light transmission increases during curing. Thus, the best way to increase the depth of cure might be to increase the material translucency by matching the refractive index of filler and matrix [13].

In this study, microhardness was measured on the top and bottom surface of resin composites. Particular attention, with statistical analysis, was paid to the bottom surface as in this area is difficult to obtain optimal polymerization. Moreover, these values were transformed to Knoop hardness ratio (KHR) for more distinctive analysis. In addition, conventional resin composite (Z350) was tested at 2 mm thickness because it could represent the optimal polymerization and hardness values.

Considering the hardnesses of the various composites measured at 2 mm thickness, conventional resin composite (Z350) revealed similar hardness value when comparing with three bulk-fill materials. Among bulk-fill groups, SF had the highest KHN when measured at 2 mm thickness with 20 seconds light exposure time. This may be attributed to the higher percentage of filler loading of SF [15].

For the SF bulk fill resin composite, although it obtained high hardness, it showed the lowest depth of cure. This is represented by a low %KHR when using both 20 and 40 seconds curing time at the different thicknesses. Comparing the amount of fillers to the other groups, this material has the highest percentage filler loading. This may reduce its translucency and lead to lower the light-curing depth. One study [16] reported the similar results as SF bulk fill resin composite could not effectively cure at 4 mm depth with 20 seconds irradiation using a polywave LED with output intensity of 1, 170 mw/cm^2. However, other studies report that the depth of cure of SF is much greater, reaching 4-5 mm depth with 20 seconds of light exposure time [4, 13, 17]. The dissimilar results may be due to the difference in the specimen preparation, measurement process, and the difference of bulk fill material that used to compared with SF.

FBF provided the highest %KHR at all thicknesses when using both 20 and 40 seconds of irradiation. Additionally, FBF achieved more than 80% of hardness at 4 mm thickness when using the LED for only 20 seconds. This result may due to the lowest percentage of filler loading of FBF; therefore, more light can be transmitted to the bottom of the cavity than the other composites.

For the TNBF group, the %KHR reached 80% at 3 mm thickness using the LED for only 20 seconds. This bulk-fill contains a new photo-initiator system, Ivocerin™ (a dibenzoyl germanium compound), which absorbs visible light over a wider range of wavelengths between 370 to 460 nm. In addition, the filler in TNBF is more spherical, which enhanced the translucency and so the depth of cure[4]. The observed depth of cure in this study is similar to a previous report [17]. In this 80% of the maximum top value was not reached at 4 mm depth when using 20 seconds curing time.

According to the results from this study, the stated hypothesis that all the selected materials are equivalent can be rejected as there are the significant differences in the hardness of materials at 2 mm thickness. In addition, the depth of cure of the bulk fill group could not reach 4 mm when using the LED for 20 seconds. However, FBF did reach 80% of hardness at 4 mm.

In summary, this study shows that an exposure time of 20 seconds using LED curing unit is not enough to reach KHR > 80 % in bulk-filling of 4 mm thickness. To achieve an optimal hardness at 4 mm depth, extending the LED light curing time to 40 seconds is strongly recommended for all three bulk-fill materials. This is much longer than manufacturer's recommendation that suggested 10 seconds for TNBF, and 20 seconds for SF and FBF cured with LED unit with intensity > 1,000 mW/cm^2. Although this laboratory test did not exactly simulate the clinical situation (a dentinal tubule may be a better light transmitter than the metal mold), it does show the necessity of extending the light exposure time when using bulk-fill in a deep cavity.

Conclusion

The satisfactory depth of cure of three photo-polymerized high viscosity bulk-fill resin composites decreases with thickness. Increasing the exposure time to 40 seconds increases the depth of cure. Placement of 5 mm thickness with 40 seconds of light exposure is not recommended for any of the materials under investigation.

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Conflict of interest

Wasa Puechpaiboon declares that she has no conflict of interest. Suppason Thitthaweerat declares that he has no conflict of interest. Piyapanna Pumpaluk declares that she has no conflict of interest.

Ethical approval

This article does not contain any studies with human participants or animals performed by any of the authors.

References

1. Ferracane JL. Resin composite-state of the art. *Dent Mater* 2011; 27: 29-38.
2. Kapoor N, Bahuguna N, Anand S. Influence of composite insertion technique on gap formation. *J Conserv Dent* 2016; 19: 77-81.
3. Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. *Clin Oral Investig* 2013; 17: 227-235.
4. Alrahlah A, Silikas N, Watts DC. Post-cure depth of cure of bulk fill dental resin-composites. *Dent Mater* 2014; 30: 149-154.
5. Ilie N, Bucuta S, Draenert M. Bulk-fill resin-based composites: an in vitro assessment of their mechanical performance. *Oper Dent* 2013; 38: 618-625.
6. Jang JH, Park SH, Hwang IN. Polymerization Shrinkage and Depth of Cure of Bulk-Fill Resin Composites and Highly Filled Flowable Resin. *Oper Dent* 2015; 40: 172-180.
7. Anusavice KJ, Phillips RW, Shen C, Rawls HR. Phillips' science of dental materials. 12 ed. St. Louis, Missouri: Elsevier Sounders; 2013.
8. Leprince JG, Leveque P, Nysten B, Gallez B, Devaux J, Leloup G. New insight into the "depth of cure" of dimethacrylate-based dental composites. *Dent Mater* 2012; 28: 512-520.
9. Ferracane J, Mitchem J, Condon J, Todd R. Wear and marginal breakdown of composites with various degrees of cure. *J Dent Res* 1997; 76: 1508-1516.
10. Caughman WF, Caughman GB, Shiflett RA, Rueggeberg F, Schuster GS. Correlation of cytotoxicity, filler loading and curing time of dental composites. *Biomaterials* 1991; 12: 737-740.
11. Silikas N, Eliades G, Watts DC. Light intensity effects on resin-composite degree of conversion and shrinkage strain. *Dent Mater* 2000; 16: 292-296.
12. Reis AF, Vestphal M, Amaral RCD, Rodrigues JA, Roulet JF, Roscoe MG. Efficiency of polymerization of bulk-fill composite resins: a systematic review. *Braz Oral Res* 2017; 28;31:e59:37-48.

13. Bucuta S, Ilie N. Light transmittance and micro-mechanical properties of bulk fill vs. conventional resin based composites. *Clin Oral Investig* 2014; 18: 1991-2000.
14. Li X, Pongprueksa P, Van Meerbeek B, De Munck J. Curing profile of bulk-fill resin-based composites. *J Dent* 2015; 43: 664-672.
15. Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. *J HYPERLINK "https://www.ncbi.nlm.nih.gov/pubmed/12131887" Prosthet HYPERLINK "https://www.ncbi.nlm.nih.gov/pubmed/12131887" Dent* 2002 ; 87: 642-9.
16. Tarle Z, Attin T, Marovic D, Andermatt L, Ristic M, Taubock TT. Influence of irradiation time on subsurface degree of conversion and microhardness of high-viscosity bulk-fill resin composites. *Clin Oral Investig* 2015; 19: 831-840.
17. Kim EH, Jung KH, Son SA, Hur B, Kwon YH, Park JK. Effect of resin thickness on the microhardness and optical properties of bulk-fill resin composites. *Restor Dent Endod* 2015; 40: 128-35.