Effect of Shade and Thickness on the Depth of Cure of Bulk-Fill Composites with Different Viscosities

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KEY WORDS
Bulk-fill composites; Microhardness; Thickness; Depth of cure; Shade;

ABSTRACT

Statement of the Problem: In an attempt to fasten and simplify the restoration process, a new class of composite resins, called the bulk fill composite resins have been introduced, which has been claimed to achieve a depth of cure (DOC) of 4 mm without affecting the properties of the material.

Purpose: The Purpose of this study was to investigate the effect of different shades, thicknesses and viscosities on the DOC of bulk-fill composites.

Materials and Method: Four bulk-fill composites [Filtek™ Bulkfill Flowable (FBF), Filtek™ Bulkfill posterior (FBP), Tetric N-Flow bulkfill(TNF), Tetric N-Ceram bulkfill(TNC)] and a conventional composite, Filtek™ Z250XT Universal (FZ) were evaluated. Samples (n=5) were made using two different shades (light and dark), thicknesses (2 and 4mm) and viscosities (flowable and sculptable). Microhardness was conducted on top and bottom surface using Vickers microhardness tester and DOC calculated as the bottom/top ratio. Statistical analysis was done using a mannwhitney test at p<0.05.

Results: DOC ranged between 52-95%. FBF composite exhibited the lowest overall hardness numbers. At 2-mm thickness, all the samples achieved an appropriate DOC. However, at 4-mm thickness, only the light shades for FBF and TNF samples achieved a DOC very close to 0.8. At 4-mm thickness, the light shades for FBF, TNF and FZ samples exhibited significantly higher DOC compared to dark shades. For 4-mm-thick samples, DOC of FB (dark and light shades) and DOC of TN (light shade) were different in the flowable type from the sculptable type.

Conclusion: Shade and viscosity influence DOC of bulk-fill composites at 4-mm depths. For bulk-fill composites, 20s light curing appears insufficient for 4mm bulk-fill placement.

Introduction

Currently, direct composite resins are the preferred materials for restoring small to medium cavities in posterior teeth on conditions in which the bonding and filling procedures can be properly performed [1]. Historically, in order to restore cavities using the incremental technique, the composite resin is cured at a maximum thickness of 2 mm. The main advantage of this technique is optimal cure throughout the material depth and decreased polymerization shrinkage [2]. On the other hand, the incremental technique is time-consuming, with higher risk of air bubbles being trapped between the layers and contamination of the operating field due to increased working time [3].

Recent developments in the technology of composite resin production have led to the introduction of bulk-fill composite resins, which can be cured in a thickness of 4–5 mm, resulting in a decrease in the duration of the restorative procedure [4]. Various studies have evaluated the physical properties of bulk-fill com-
posites resins, including creep [5], modulus of elasticity [6], cuspal deflection [7], microleakage [8] and wear resistance [9].

The measured mechanical properties place the bulk-fill composite resins, as a material category, between the nanohybrid and microhybrid composite resins and the flowable composite resins, suggesting a similar or even inferior clinical behavior of bulk-fill composite resins compared to nanohybrid and microhybrid composite resins [4]. Bulk-fill composite resins are also comparable with conventional composite resins in relation to water uptake and biocompatibility [10].

One of the most important factors in the failure of composite resin restorations is inadequate curing. Uncured composite resins might result in the failure of restoration because of increased chance of fracture, recurrence of caries or wear of the restoration. On the other hand, when the composite resin is not adequately cured, there is an increased risk of leakage of chemical materials from composite resin into the body tissues [11]. According to previous studies, depth of cure (DOC) of composite resins is affected by the type of composite resin photoinitiator, filler type, matrix, color and translucency, light spectrum of the light-curing unit and composite placement technique [12]. In addition, the thickness of the composite resin, irradiation time and the intensity of light affect the degree of conversion [13]. Typically, there are two methods for evaluating the adequate curing for a resin:

1) Degree of conversion using FTIR spectroscopy
2) Microhardness test

The majority of studies have indicated a good correlation between the degree of conversion and the microhardness test [14-16]. In the microhardness test, optimal DOC is defined as a depth with a hardness ratio of at least 0.8 of the hardness of composite resin surface [15-17]. While some researchers recommend the cure of bulk-fill composite resins at a thickness of 4 mm [18-19]; some others believe that the methods used in some studies have overestimated DOC of these composite resins and reiterate that polymerization of bulk-fill composite resins at 4-mm depths is inadequate [11,20].

The aim of this study was to evaluate the effect of viscosity, shade and thickness on the DOC of bulk-fill composite resins. Bulkfill composite resins are divided into two groups based on viscosity: bulk-fill composite resins with low viscosity (flowable) and bulk-fill composite resins with high viscosity (sculptable). In this study, we evaluated Filtek Bulk-Fill and Tetric N-Ceram Bulk-Fill composite resins in two flowable and sculptable types and in two different shades. As a control group, we also studied a conventional nano-hybrid composite resin: Filtek Z250. The null hypothesis states that DOC of the evaluated composite resins is not affected by viscosity, shade and thickness.

Materials and Method

In this experimental study, four types of bulk-fill composite resins were evaluated: Filtek™ Bulk-Fill Flowable (3M ESPE, St Paul, MN, USA) (FBF), Filtek™ Bulk-Fill Posterior (3M ESPE, St Paul, MN, USA) (FBP), Tetric N-Ceram Bulk-Fill (Ivoclar Vivadent, Schaan, Liechtenstein) (TNC) and Tetric N-Flow Bulk-Fill (Ivoclar Vivadent, Schaan, Liechtenstein) (TNF); a conventional composite resin, Filtek™ Z250XT universal (3M ESPE, St Paul, MN, USA) (FZ) was also evaluated (Figure 1). Composition and brands of materials are shown in Table 1.

A1 shade was considered as a light shade (L) for FZ, FBP and FBF composite resins and IVA shade was considered as a light shade (L) for TNC and TNF composite resins. In addition, the A3 shade was considered as a dark shade for FBF, FBP and FZ composite resins and IVB shade was considered as a dark shade for TNC and TNF composite resins (D).

The samples were assigned to 20 groups and 5 samples were prepared for each group [21]. Steel molds, measuring 4 mm in diameter and 2 or 4 mm in thickness [22], were used to prepare the samples. After placing the mold on a glass slab and celluloid matrix strip, the composite resin was packed within it; then a glass slab and a celluloid matrix strip were placed on the upper su-

Figure 1: Resin composite samples
surface of the composite resin and the excess material was removed by exerting pressure on the glass slab.

The samples were light-cured for 20s using a Polywave LED light-curing unit (Bluephase N, Ivoclar Vivadent, Schaan, Liechtenstein) in "high" mode. Radiation intensity was measured with a radiometer (Ivoclar Vivadent, Schaan, Liechtenstein) before each curing procedure. The intensity of the radiation was 1200±40 mW/cm² during each curing procedure.

All the samples were incubated (Peco, Iran, Model: PI-455G) at 37°C in a dry environment within a light-proof container for 24 hours [19]. Then, the microhardness of the samples was measured by Vickers hardness machine (ZHVµ model, Zwick/Roel, United Kingdom). To measure the microhardness, first the samples were placed on the jig of the device and their surface was evaluated at ×40 magnification so that the location of the force on the surface was free of bubbles and other defects. Then a 300-gr load was applied to the sample for 10 seconds by a diamond pyramid-shaped indenter [23]. The loads were applied close to the center of the samples at a distance of 0.2 mm of each other. Then, with adjusting the electronic microscope index on the surface of sample, the diameter of the square indentation area was determined by tester. Finally, the surface and bottom Vickers microhardness of specimens’ calculations were made using computer processor of tester device using the formula:

$$VHN = \frac{(1.8544P)}{D^2}$$

In which, VHN represents Vickers hardness of material (Kg/mm²), P is the predetermined load applied on the sample (Kg) and D is the average diagonal distance (mm) of the square resulting from indentation of the pyramid tip of Vickers hardness tester [15]. To obtain the hardness number of each surface, 3 measurements were made on each surface and their mean was determined and recorded as the final hardness number for each surface. For each sample, two hardness numbers were obtained, which belonged to the top and the bottom of the samples. Then, by calculating bottom/top ratio, DOC of the samples was determined [11, 19] and the results were analyzed.

Kolmogorov-Smirnov test was used to evaluate the normality of data. Since data were not normal, Mann-Whitney test was used to compare each variable individually. SPSS 24 was used for statistical analysis at a significance level of P<0.05.

**Results**

Table 2 presents the mean hardness of the top and the
Table 2: Bottom and Top Means and Standard Deviations (SD) of Vickers Hardness Numbers of Different Resin-Based composite

<table>
<thead>
<tr>
<th></th>
<th>4mm,dark</th>
<th>4mm,light</th>
<th>2mm,dark</th>
<th>2mm,light</th>
</tr>
</thead>
<tbody>
<tr>
<td>FZ</td>
<td>87.73(4.81)</td>
<td>87.86(2.03)</td>
<td>84.13(1.32)</td>
<td>90.59(2.51)</td>
</tr>
<tr>
<td></td>
<td>45.99(5.74)</td>
<td>58.86(5.38)</td>
<td>80.39(3.51)</td>
<td>81.99(5.22)</td>
</tr>
<tr>
<td>FBP</td>
<td>63.86(1.32)</td>
<td>59.99(2.03)</td>
<td>63.80(1.32)</td>
<td>62.59(2.51)</td>
</tr>
<tr>
<td></td>
<td>46.86(2.54)</td>
<td>42.26(2.08)</td>
<td>60.66(2.41)</td>
<td>59.66(2.53)</td>
</tr>
<tr>
<td>FBF</td>
<td>26.74(0.73)</td>
<td>29.13(1.14)</td>
<td>29.99(0.62)</td>
<td>29.33(1.43)</td>
</tr>
<tr>
<td></td>
<td>16.99(0.97)</td>
<td>23.19(1.30)</td>
<td>27.73(0.54)</td>
<td>28.06(0.89)</td>
</tr>
<tr>
<td>TNC</td>
<td>55.53(2.00)</td>
<td>55.79(1.07)</td>
<td>56.73(0.59)</td>
<td>51.16(1.86)</td>
</tr>
<tr>
<td></td>
<td>36.32(3.83)</td>
<td>38.06(1.70)</td>
<td>51.73(0.75)</td>
<td>50.06(0.49)</td>
</tr>
<tr>
<td>TNF</td>
<td>37.66(1.35)</td>
<td>34.39(1.69)</td>
<td>36.59(2.68)</td>
<td>34.73(1.94)</td>
</tr>
<tr>
<td></td>
<td>27.26(2.17)</td>
<td>27.46(1.42)</td>
<td>34.46(2.40)</td>
<td>32.92(1.90)</td>
</tr>
</tbody>
</table>

FZ: Filtek Z250 A1,A3; FBP: Filtek bulkfill posterior A1,A3; FBF: Filtek bulkfill flowable A1,A3; TNC: Tetric N-Ceram bulkfill IVA,IVB; TNF: Tetric N-Flow bulkfill IVA,IVB

bottom for each sample in terms of shade, viscosity and thickness. Among the composite resins evaluated, FBF composite resin exhibited the lowest hardness number at top and bottom; FZ composite resin exhibited the highest overall hardness values compared to the other materials. In addition, in all groups the top hardness was higher than the bottom.

According to statistical analyses, 2-mm-thick samples for all groups had significantly higher DOC compared 4-mm-thick samples (Figure 2). At 2-mm thickness, all the samples achieved an appropriate DOC (DOC>0.8). However, at 4-mm thickness, only FBF and TNF composite resins (light shades) achieved a DOC very close to 0.8.

At 2-mm-thick samples, different shades had no effects on the DOC of various composite resins; however, when 4-mm-thick samples were evaluated, only the light shades for FBF and TNF samples achieved a DOC very close to 0.8 (Figure 2).

Comparison of different viscosities of composite resins at 2-mm thickness showed no significant difference in DOC of groups in terms of the type of viscosity (Figure 3). For 4-mm-thick samples, DOC of FB (dark and light shades) and DOC of TN (light shade) were different in the flowable type from the sculptable type.

Discussion

Bulk-fill composite resins, which can be cured at a thickness of 4-5 mm, were introduced to reduce the duration of restorative procedures [4]. Bulk-fill composite resins are cured in thicknesses greater than 2 mm due to some mechanisms:

1. Booster photoinitiators derived from benzoyl germanium with higher light-curing activity [24].
2. Polymerization modulators, such as urethane-based dimethacrylate monomers with high molecular weight, which reduce the stresses of polymerization [25].
3. Increased flowability for better adaptation [3].
4. Increased translucency through the use of mixed oxide fillers with refractive indexes equal to the resin matrix and use of glass fibers that increase the penetration of light into the composite resin [26].

Figure 2: Graph presenting the Depth-of-Cure B/T Ratios of Groups Comparing thickness(2 and 4mm) and shades (light and dark)
Some studies investigated the effect of different shades on the DOC and reported that darker shades exhibited lower microhardness than light shades. It has been reported that different composite resin compositions, filler size, weight, volume, and filler-to-matrix ratio have a significant effect on the composite resins’ DC and microhardness [27]. So, we concurrently evaluated the effect of viscosity, shade and thickness on the DOC of bulk-fill composite resins.

Consistent with the results of previous studies [19-20], hardness of the top was higher than that of the bottom. FZ composite resin exhibited the highest surface hardness, followed by FBP composite resin. Consistent with the results of previous studies [20,28], in each composite resin in this study, the surface hardness in the flowable type was significantly lower than that in the sculptable type, which could be due to the lower filler content of flowable composite resins. Studies have shown that the filler content of composite resins can affect their hardness and physico-mechanical properties [29]. The results of this study showed lower DOC in all the composite resins in 4-mm-thick samples compared to 2-mm-thick samples [Table 2], consistent with the results of previous studies [19-20].

A possible reason might be the lack of light penetration through the composite at increasing depths because a high percentage of the wavelengths are absorbed near the top surface of the composite and not available to excite co-initiators at greater depths [30].

Generally, manufacturers use methods such as in increasing translucency, increasing the amount of photoinitiators and use of additional photoinitiators to increase DOC of composite resins [31]. In this context, these composite resins have less light attenuation and more light transition rates compared to conventional composite resins [20].

Consistent with the results of previous studies [11], all the 2-mm-thick composite resin samples reached an adequate depth of cure (0.8) with a maximum curing time of 20 seconds. However, none of the 4-mm-thick samples in any group reached a DOC of 0.8, except for the light shades of flowable composite resins (FBF A1 and TF IVA), in which the DOC was very close to 0.8 (0.79). This finding is contrary to the claims made by the manufacturers about the DOC of composite resins at 4-mm thickness. It is not clear how manufacturers determine the DOC, since the scraping ISO 4049 method has overestimated the DOC [22] and also it is hard to standardize it [32], in this study, we used the Vickers hardness test to examine the DOC. Numerous studies have defined depth of cure based on hardness measurements performed on the top and bottom surface of a light-cured resin composite specimen and agree that a ratio of 0.80 may be used as a critical minimum acceptable threshold value [33-35].

However, some studies have reported a depth of cure of >0.8 for 4-mm-thick bulk-fill composite resin samples, which is acceptable [28]. Other studies, consistent with the present study, have reported DOC of <0.8 in 4-mm-thick samples of the bulk-fill composite resins [19-20,22]. The differences in the results of studies might be explained by differences in sample prepara-

![Figure 3: Graph presenting the Depth-of-Cure B/T Ratios of dark and light shades Comparing viscosity (high and low)](image-url)
tion conditions, storage of the samples and the method used to determine the DOC. Composite resin type, mold type and diameter, the use of a lubricant, the intensity of the light-curing unit, the storage conditions of the samples, the method of testing and the amount of load used were varied in different studies [11]. The effect of mold size has been examined for opaque cylindrical molds and it was found depth of cure decrease as mold size diameter decreased [36]. Black molds produced shorter depths of cure than a stainless steel mold when a light shade of composite is cured [37].

When a mold is used, a pronounced effect by the walls resulted in decreased hardness as the mold wall is approached and the severity of this effect was dependent on the color of the mold. It is believed that this is due to absorption/reflection characteristics of light by the walls, with the white molds showing the least effect [38].

The mold used in our study was made out of metal. This blocked transmission of all light outside of the central 4-mm of the light guide tip. However, a metal mold is specified in the ISO standard 4049 and has also been recommended by different authors so as not to overestimate depth of cure [36, 38-39]. This metal mold created testing conditions closer to clinical conditions where a metallic matrix is placed around the boxes in Class 2 preparations [11].

The results of this study indicated no significant differences in the curing depths between the dark and light shades except between two flowable bulk-fill composite resins with 4-mm thickness. However, Rodriguez et al. [19] concluded that when bulk-fill Tetric Evoceram and Sonic Fill composite resin samples with 4-mm thickness were light-cured for 20 seconds, there was a significant difference in curing depth between dark and light shades and Consistent with the results of other studies, DOC in light shades was greater than that in dark shades [40]. According to previous studies, size, radioactivity, translucency and pigments in the filler particles affect light transmission of the material [41]. Pigments in dark shades limit the light transmission and reduce the degree of polymerization [30]. It seems that due to lower filler content and higher translucency of flowable bulk-fill composite resins compared to sculptable type, presence of more pigments in the dark shade resulted in a decrease in curing depth when the thickness of composite has been increased resins to 4 mm [42].

The results of this study indicated no significant difference in the DOC between flowable and sculptable composite resins at 2-mm thickness; however, at 4-mm thickness, in flowable type the DOC was significantly different from that in sculptable type in all the groups, except the one in IVB dark shade of TNC bulk-fill composite resin. Consistent with the results of this study, some researchers have reported greater DOC in flowable bulk-fill composite resins compared to sculptable composite resins [3]. This variation in the DOC of bulk-fill composite resins with different viscosities might be related to the difference in their filler content. By increasing filler-to-matrix ratio, the degree of conversion decreases since high filler content prevents the development of polymer chains [43]. In addition, as the amount of filler increases, the amount of light scattering increases and the translucency for the blue color decreases [26].

Bulk-fill flowerless exhibited large filler size with dominant polygonally shaped features compared with conventional flowable resin composites, as seen with a scanning electron microscope. The filler load was slightly increased, but the filler-matrix interface was assumed to be decreased, due to the bigger size of the filler particle. Hence, it allows more curing light to transmit through the composite and improve the DOC [44].

In this study only four types of bulk-fill composite resins were studied; therefore, it is suggested that other bulk-fill composite resins should be studied and the effects of other variables, including the intensity of radiation, type of light-curing unit and its distance from the surface of composite resin, on their DOC be examined in future studies.

**Conclusion**

- Shade and viscosity influence DOC of bulk-fill composites at 4-mm depths.
- None of the composite resins investigated in this study reached a curing depth of >0.8 mm at 4-mm thickness. (Samples of flowable composite resins in light shade exhibited DOC very close to 0.8).
- Under the limitations of this study, for bulk-fill com
Acknowledgment
This research has been supported by Zanjan University of Medical Sciences and Health Services grant for number A-12-1040-3 research proposal.

Conflict of Interest
The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article

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