

Original Article

## The Effect of Bulk Depth and Irradiation Time on the Surface Hardness and Degree of Cure of Bulk-Fill Composites

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### ARTICLE INFO

#### Article History:

Received: 26 May 2016

Accepted: 28 August 2016

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#### Key words:

Bulk-fill Composites

Irradiation Time

Microhardness

Degree of Cure

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### Abstract

**Statement of Problem:** For many years, application of the composite restoration with a thickness less than 2 mm for achieving the minimum polymerization contraction and stress has been accepted as a principle. But through the recent development in dental material a group of resin based composites (RBCs) called Bulk Fill is introduced whose producers claim the possibility of achieving a good restoration in bulks with depths of 4 or even 5 mm.

**Objectives:** To evaluate the effect of irradiation times and bulk depths on the degree of cure (DC) of a bulk fill composite and compare it with the universal type.

**Materials and Methods:** This study was conducted on two groups of dental RBCs including Tetric N Ceram Bulk Fill and Tetric N Ceram Universal. The composite samples were prepared in Teflon moulds with a diameter of 5 mm and height of 2, 4 and 6 mm. Then, half of the samples in each depth were cured from the upper side of the mould for 20s by LED light curing unit. The irradiation time for other specimens was 40s. After 24 hours of storage in distilled water, the microhardness of the top and bottom of the samples was measured using a Future Tech (Japan- Model FM 700) Vickers hardness testing machine. Data were analyzed statistically using the one and multi way ANOVA and Tukey's test ( $p = 0.050$ ).

**Results:** The DC of Tetric N Ceram Bulk Fill in defined irradiation time and bulk depth was significantly more than the universal type ( $p < 0.001$ ). Also, the DC of both composites studied was significantly ( $p < 0.001$ ) reduced by increasing the bulk depths. Increasing the curing time from 20 to 40 seconds had a marginally significant effect ( $p \leq 0.040$ ) on the DC of both bulk fill and universal studied RBC samples.

**Conclusions:** The DC of the investigated bulk fill composite was better than the universal type in all the irradiation times and bulk depths. The studied universal and bulk fill RBCs had an appropriate DC at the 2 and 4 mm bulk depths respectively and using the recommended curing time of 40s can led to the slightly better value of DC in both composites.

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**Cite this article as:** Farahat F<sup>a</sup>, Daneshkazemi AR<sup>b</sup>, Hajiahmadi Z. The Effect of Bulk Depth and Irradiation Time on the Surface Hardness and Degree of Cure of Bulk-Fill Composite. J Dent Biomater, 2016;3(3):284-291.

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## Introduction

To achieve a high level of aesthetics, physical and strength properties close to natural tooth structure has been among the factors to be considered for spread of resin based composites (RBCs) in restorative dentistry in recent years. Since emergence of composites, a lot of changes have been made in their chemical structure and filler to eliminate or improve the defects [1]. In addition to the numerous advantages of composites used in restorative dentistry, the presence of characteristics like low polymerization shrinkage and depth have always caused restrictions in the field of application of these materials [2].

For many years, the use of composites with a thicknesses of less than 2 mm in order to achieve a restoration with a high degree of cure (DC) and minimal shrinkage polymerization and stress has been proposed as a principle [3,4]. However, using this technique in restoration of deep cavities is time consuming and also there is probability of air bubbles confinement or contamination between layers of the composite [5]. Thus, search for strategies for rapid conduction of deep dental cavities caused tendency to develop and marketing new RBC materials with capability of increasing the depth of polymerization in recent years.

A new type of RBCs named bulk fill has recently been introduced to the market which contains lower amount of filler with larger size. Presence of large sized filler (up to 2 $\mu$ m) in the structure of this composite in general terms reduces the level of connectivity between matrix and filler and can improve the transmission of curing blue light into the deeper point of composite restoration [6]. Moreover, according to the manufacturers, bulk fill composites have capability of replacement in deep cavities with thicknesses of 4 to 5 mm in one layer without the need for longer curing process or more severe irradiation. Thus, restoration time will be shorter and treatment will be faster [7].

Surface hardness is one of the important mechanical properties of dental composites. Hardness of resin composites can be defined as resistance against permanent indentation or penetration on the surface of restoration. This specification affects the capability of polishing and resistance to scratches of the material [8].

Polymerization rate of composites also affects the mechanical properties, size stability and solubility, stability of colours and their biocompatibility [9]. Among various features, the ratio of bottom to top surface microhardness of RBCs in different irradiation times, called the degree of cure (DC), is widely used to evaluate the performance of dental RBCs in recent years [9]. Results of the references confirm the linear relationship between the surface and depth hardness of the composite restorations and their DC [7-10]. If the bottom to top surface microhardness of dental RBCs is more than 80 %, the DC of their restoration is acceptable [5,9].

In the studies conducted, different DCs have been reported for bulk fill and universal RBCs. For example, Czasch *et al.* showed that Venus Diamond and Surefil SDR composites have an acceptable DC if they are irradiated for 20 s to a depth of 4 mm [11]. The study by Tarle *et al.* showed that restorations with a depth of 4 mm with Quix Phil and X-TRA Fil composites have an acceptable DC if they are irradiated for at least 30 s, [6]. These researchers reported that by reducing the irradiation time to 10 s, especially for restorations with a depth of 4 mm, the microhardness of the bottom surface is greatly reduced, although in this case there is no significant change in the microhardness of top surface.

According to the above mentioned points, it can be concluded that the type of composites has a significant effect on the researches results. Most of the studies done by researchers in the field of dental materials have focused on different curing conditions or depth of restoration layers on the DC of the dental RBCs separately. For this reason in this study, the effect of curing time and material thickness on the DC of two groups of universal and bulk fill RBCs were considered. In addition, there was an attempt to investigate the validity of higher DC of bulk fill RBCs at thicker restoration layers and less curing time than the similar universal types as a part of the null hypothesis of the present work. Furthermore, investigation of acceptable DC of Tetric N Ceram bulk fill RBCs up to a depth of 4 mm, higher than universal type, based on their manufacturers' claim is another aim of this study.

## Materials and Methods

The materials evaluated in this in vitro study were

**Table 1:** Properties of the composites studied

Composite type	Tetric N-Ceram	Tetric N Ceram bulk fill
Manufacturer	Ivoclar Vivadent - Italy	Ivoclar Vivadent - Italy
LOT #	T24404	T29061
Matrix	Dimethacrylate	Dimethacrylate
Filler	Barium Glass, Ytterbium Trifluoride, Mixed Oxide and Copolymer	Barium Glass, Ytterbium, Trifluoride, Prepolymer and Mixed Oxide
Filler (volume %)	55-57	53-55
Filler (weight %)	80-81	75-77

two nanohybrid resin based composites (RBCs) including universal (Tetric N Ceram) and bulk fill (Tetric N Ceram Bulk Fill) with shade A3 (Table 1). A total of 96 samples were prepared and then equally divided into 12 groups ( $n = 8$ ) including the two types of investigated resin based composites (bulk fill and universal), the three various sample height (2,4 and 6 mm) and finally the two applied curing times (20 and 40 seconds) in the present work. In other words, each of 12 groups of samples consisting of 8 specimens were made from one of the two used composite types with a height of 2 or 4 or 6 mm and curing time of 20 or 40 seconds.

#### *Sample preparation*

Preparation of the specimens was performed in cylindrical Teflon moulds with a diameter of 5 mm and height of 2, 4 and 6 mm. The moulds were first mounted on the top of a glass slab with the dimensions of  $1 \times 76 \times 76$  mm and a Mylar strip (PD, Switzerland) and then the mould was filled in bulk with one of the two investigated universal and bulk fill composites. The upper surface of the mould was covered with a second Mylar strip and glass slab. A load of 500 g was applied on the top of glass slab for 30 seconds to ensure consistent packing of the samples and elimination of the extra composite materials.

Afterwards, the load and top slab were removed and the samples were light cured only from the upper surface using LED Demi light curing unit

(Demi- Kerr- USA) with an output intensity of 1000 mW/cm<sup>2</sup> for 20 or 40 seconds. The tip (circular with a diameter of 10 mm) of light curing unit was kept centered and in direct contact with the top Mylar strip. It should be noted that the power density of the light curing unit was checked after curing every 5 specimens using a radiometer (Demetron, Kerr-USA). After removing the top Mylar strip, the samples were pushed out of the mould and unpolymerized extra composites material of the top surface edge was deleted with a bistoury blade. Then the surface of all composite samples was polished in the presence of water using red, orange, yellow and white Sof Lex (3M ESPE- USA) polishing discs for 5 seconds, respectively. The top surfaces of the samples were identified with an indelible mark. Finally, the samples were rinsed with water and kept in distilled water for 24 hours at 37 ° C in the light proof incubator (Memmert IPP55plus, Germany) chamber.

#### *Measurement of microhardness and the degree of cure*

After removing the samples from distilled water, the microhardness of top and bottom surfaces of them was measured by Future Tech (Japan-Model FM 700) Vickers hardness (VHN) tester. To perform the test, six selective indentations (3 on both the top and bottom surfaces) were made with 50 g load and dwell time of 15 seconds using a pyramid shaped diamond indenter head tip. The

location of indentation points on the surface and depth of composite samples were selected so that they had at least 2 mm distance from the sample edges and 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 depth Vickers microhardness of specimens' calculations were made using computer processor of tester device based on Eq. 1 and final displayed results on the Vickers tester screen were recorded for further statistical consideration. The three measurement microhardness values on the top and bottom were averaged to obtain a single value for surface and depth Vickers microhardness of each specimen.

$$\text{VHN} = (1.8544P) / D^2 \quad (1)$$

In which, VHN represents Vickers hardness of material ( $\text{Kg}/\text{mm}^2$ ), 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. After determining the amount of top and bottom microhardness, the degree of cure (DC) of each sample was calculated according to Eq. 2.

$$\text{Degree of Cure(\%)} = \frac{\text{bottom Microhardness}}{\text{top Microhardness}} \times 100 \quad (2)$$

#### Statistical analysis

Statistical analysis was performed with IBM package for the social sciences (SPSS Inc., IBM Corporation- New York, USA) Statistics Version 18 for Windows. Three-way analysis of variance (ANOVA) was applied to investigate the effect of various studied restorative RBCs materials, thickness and irradiation times on mean surface and depth microhardness and the DC of samples. The post-hoc Tukey's HSD (Honestly Significant Difference) test was used for pair-wise comparison between the means when ANOVA test is significant.

Also, independent *t*-test had been applied to compare between two types of investigated bulk fill and universal composites and their depth and surface microhardness. In addition, one-way ANOVA have been used to evaluate the influence of sample height and irradiation time on the mean

surface and depth microhardness and DC followed by Tukey's test was used for pair wise comparison between the means when ANOVA test is significant. The significance level of statistical analysis was set  $p \leq 0.05$ .

#### Results

The mean values of surface and depth microhardness (VHN) of the tested points and the calculated degree of cure (DC) for are displayed in Table 2.

ANOVA showed the significant effect ( $p=0.001$ ) of the samples' thickness on depth microhardness of universal Tetric N Ceram RBCs; however, based on *Tukey's* test output, this effect was not significant ( $p = 0.389$ ) in the thicknesses of 4 ( $31.67 \pm 3.74$  VHN) and 6 mm ( $30.24 \pm 3.28$  VHN). On the other hand, it's obvious from the ANOVA results that the influence of irradiation time was noticeable ( $p = 0.002$ ) on the depth microhardness of the universal Tetric N Ceram RBC samples.

Based on the ANOVA results, in the investigated Tetric N Ceram bulk fill composite change of samples thickness between 2 ( $92.77 \pm 13.2$  VHN), 4 ( $77.75 \pm 7.27$  VHN) and 6 mm ( $68.33 \pm 7.83$  VHN) had a significant ( $p = 0.001$ ) effect on their depth microhardness. The results of *Tukey's* test also confirms that change of sample thickness between 2, 4 and 6 mm have significant effect on the depth microhardness of investigated bulk fill RBCs at ( $p \leq 0.003$ ). Also, ANOVA results indicate that different irradiation times did not have a significant effect ( $p = 0.121$ ) on depth microhardness of bulk fill samples although the effect of this factor on depth microhardness of bulk fill was higher than the surface microhardness ( $p = 0.147$ ). In addition, it was observed from the ANOVA results that increasing the thickness of the samples and irradiation time had no significant ( $p \geq 0.05$ ) effect on the surface microhardness of both studied universal and bulk fill RBCs.

According to the three-way ANOVA results, surface microhardness of the investigated universal and bulk fill composites was not significantly affected by the type of composite material ( $p = 0.388$ ) and any change of RBC samples thickness ( $p = 0.592$ ) while the effect of different curing time on the surface hardness of bulk fill sample compared with universal type was slightly

**Table 2:** Results of surface and depth microhardness and degree of cure of the studied composites.

Resin composite	MT (mm)	IT (sec)	N	MTH (Kg/mm <sup>2</sup> )	SD	MBH (Kg/mm <sup>2</sup> )	SD	DC (%)	SD
Universal (Tetric N Ceram)	2	20	8	44.51	2.54	33.98	1.91	76.52	5.36
		40	8	45.72	2.69	35.38	1.43	77.68	6.51
		Total	16	45.12	2.60	34.68	1.79	77.10	5.80
	4	20	8	44.42	2.40	29.97	3.48	67.91	10.44
		40	8	45.31	2.21	33.36	3.35	73.82	8.70
		Total	16	44.87	2.28	31.67	3.74	70.87	9.78
	6	20	8	44.71	2.44	28.16	2.19	63.07	4.81
		40	8	46.63	3.10	32.31	2.89	69.65	8.58
		Total	16	45.67	2.87	30.24	3.28	66.36	7.53
bulk fill (Tetric N Ceram Bulk Fill)	2	20	8	44.88	3.10	40.74	3.74	90.95	8.06
		40	8	46.03	6.81	42.63	3.09	94.58	17.35
		Total	16	45.46	5.15	41.69	3.46	92.77	13.20
	4	20	8	44.97	2.98	34.64	2.19	77.42	8.16
		40	8	46.25	1.40	36.04	2.33	78.08	6.80
		Total	16	45.61	2.35	35.35	2.30	77.76	7.27
	6	20	8	45.20	3.10	29.68	2.43	65.93	7.03
		40	8	47.38	2.75	33.37	3.07	70.71	8.28
		Total	16	46.29	3.05	31.53	3.29	68.33	7.83

MT= Material thickness, IT= Irradiation time, N= Number of samples, MTH= Mean top surface microhardness, SD= Standard deviation, MBH= Mean bottom surface microhardness and DC= Degree of cure

meaningful ( $p = 0.032$ ). Further, the results of three-way ANOVA and *Tukey's* test were showed that variation of all investigated factors including composite type, thickness of samples and curing times had a significant ( $p \leq 0.001$ ) effect on the depth microhardness of studied resin composites.

Statistical analysis of the results related to the DC of composite samples using the three-way ANOVA method showed the significant superiority ( $p = 0.001$ ) of DC value of bulk fill samples over the universal type in similar depth and irradiation time. Also, in both composite materials, by increase of samples height, the DC value of the samples decreased significantly ( $p = 0.001$ ) and

the value of this parameter for curing time of 40 seconds was slightly ( $p = 0.040$ ) longer than 20 s. In addition, according to the three-way ANOVA results, the effect of irradiation time on the DC of two composites studied in the same thickness did not show a considerable significance ( $p = 0.676$ ). Further, it's clear from the three-way ANOVA results that the influence of variation of sample thickness on the DC of investigated composites with the same curing time is also considerable ( $p = 0.010$ ).

## Discussion

Bulk fill RBCs are materials that, according to the

manufacturers' claim, can be cured in thicknesses of 4 or even 5 mm at one stage [9]. Validity of this claim was performed by investigating the effect of irradiation times and different thicknesses on the performance of Tetric N Ceram bulk fill RBCs based on the DC in this study. Furthermore, the results were compared with similar Tetric N Ceram universal RBC. On the other hand, comparison of the performance of the two composites under the study in similar depths and irradiation times showed the significant superiority of DC in bulk fill type over universal types in different thicknesses of 2, 4 and 6 mm.

The ratio microhardness of bottom to top surface as a common criterion for evaluating the DC of the dental RBC restoration [10,12,13] is used in this study. According to the possibility of increasing the DC of composite samples in a time interval after the curing process [14-18] as well as increasing their degree of polymerization by increasing the temperature of storage room [19], in this study all tests were performed 24 hours after curing the samples and keeping them in light proof incubator space at mouth temperature (37 °C).

The present results show that universal samples at 2 mm depth and bulk fill samples at 2 and 4 mm depths had an acceptable DC based on 80% microhardness drop-off hypothesis from top to bottom surface of specimens. This is in agreement with comparable studies [5,7,8,11] that showed different bulk fill materials had higher depth of cure than the common universal composites.

Many factors such as the size and composition of the filler, material translucency and intensity of LED, curing time, monomer composition and concentration of photo initiator can have considerable effects on the DC of dental RBCs [17]. Increase of filler content and also application of irregularly shaped filler in composites composition can lead to increased contact area of the resin and filler and reduce the amount of light transmittance through the RBCs [20]. Light transmission also reduces in composites with large sized filler (0.05-2 $\mu$ m) [18]. On the other hand, one of the best ways to enhance the DC of RBCs might be to increase the materials translucency by matching the refractive indices of fillers and matrix. Differences in the refractive indices of filler and the composite matrix may increase light scattering, which consequently

reduces the DC of the composite restorations [17].

The present study shows that the DC of Tetric N Ceram bulk fill RBC is more than universal Tetric N Ceram type. Considering the properties of studied RBCs (Table 1), it is clear that despite the similarity in shape and size of fillers, the higher translucency due to using different filler chemical composition [8] and lower filler content of bulk fill material (75-77 wt%, 53-55 vol%) compared with universal type (80-81 wt% , 55-57 vol%) can be some factors affecting present results. Several researches [3,7,21] such as Moszner *et al.* [20] and Bucuta *et al.* [8] have been done to evaluate the effects of amount and size of filler and the materials translucency on the DC of bulk fill and universal RBCs that their findings support the results of this work.

The chemical composition of the filler and matrix can have significant effects on the DC of RBCs. In the filler combination of Tetric N Ceram bulk fill composite prepolymerized fillers (PPF) containing barium glass and silica minerals is used. Based on other studies [7,8,11,21], due to the application of PPF, RBCs like Tetric N Ceram bulk fill are able to achieve a high filler load while maintaining a low specific surface between inorganic fillers and organic matrix, as a part of PPF is actually organic. Today, new types of photoinitiator such as Ivocerin and Benzoyl Germaniumare are used in the composition of PPF of bulk fill RBCs instead of the common type such as Camphorquinone (CQ) [22-24]. The higher capability of these materials in creation of free radicals per molecule unit can improve the light sensitivity of RBCs [25-28]. These changes have a positive effect on the light absorbance ability and DC of RBCs.

The manufacturers' instruction of placing the Tetric N Ceram bulk fill and Tetric N Ceram universal RBCs up to 4 mm and 2 mm bulks respectively and light curing for 10 s without a loss in acceptable DC seems to be of great interest for clinicians. Results of present study recommend an increase of the curing time from 20 to 40 s in clinical condition due to its marginally significant effects on the DC of both investigated RBCs. Also, present results indicate that in both investigated bulk fill and universal RBCs, the value of DC significantly decrease with any increasing of the

sample thickness. Effects of material thickness and curing time on the performance of RBCs have been investigated in many research works [5,8,11,13,29-31]. Some others studied the effect of material thickness on the DC of various bulk fill RBCs for curing time of 20 s [5,8,30]. Their results show that in some types of bulk fill RBCs such as SDR the DC remains acceptable with increasing the material thickness only up to 4 mm in agreement with present results while some other bulk fill RBCs such as X-Tra, Venus and Tetric Evoceram can have reasonable depth of cure up to 6 or 8 mm in contrast to this study.

It seems that different results of many studies conducted on the microhardness of bulk fill and universal RBCs can be related to various factors such as material composition, time interval passing from samples preparation and performing tests, type of moulds, type and temperature of storage room and test design [32]. In general, evaluating the microhardness of the RBCs at 1 mm depth in addition to present investigated thickness of 2, 4 and 6 mm for different curing time of 10 and 30 s can provide a more comprehensive study.

## Conclusions

Within the limitations of present in vitro study, it can be stated that the DC of Tetric N Ceram bulk fill RBCs is higher than similar universal type for all values of curing time and material thickness. Also, the producer recommended irradiation time of 20 s and appropriate curing depth up to 4 and 2 mm is sufficient for investigated bulk fill and universal RBCs, respectively. According to the marginally significant effect of the curing time on the DC of both Tetric N Ceram composite samples, increasing the irradiation time from 20 to 40 s in clinical condition is suggested.

**Conflict of Interest:** None declared.

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