

# Influence of bulk-fill composite types, shades and light-curing units on microhardness

 Serife Ozdemir<sup>1</sup>,  Evrim Eliguzeloglu Dalkilic<sup>2</sup>,  Fatemeh Hira Mazlouninia<sup>3</sup>,  Ahmet Hamdi Selcuker<sup>3</sup>

<sup>1</sup>Bezmialem Vakif University, Faculty of Dentistry, Department of Pediatric Dentistry, Istanbul, Turkey

<sup>2</sup>Bezmialem Vakif University, Faculty of Dentistry, Department of Restorative Dentistry, Istanbul, Turkey

<sup>3</sup>Bezmialem Vakif University, Faculty of Dentistry, Istanbul, Turkey

Copyright © 2020 by authors and Annals of Medical Research Publishing Inc.

## Abstract

**Aim:** The aim of this study was to investigate the microhardness of sonic-activated and high-viscosity bulk-fill resin-based composites (RBCs) in different shades polymerized by using two different light emitting diode (LED) light curing units (LCUs).

**Material and Methods:** Eight groups (n=4) of composite samples with two different shades (A2, A3) of sonic-activated bulk-fill RBCs (SonicFill™) and high-viscosity bulk-fill RBCs (Reveal HD Bulk) were prepared to evaluate the Vickers microhardness (VMH) values. To collect microhardness data for both the upper and the lower sample surfaces, the cylindrical bulk-fill RBC samples with a depth of 4 mm and diameter of 10 mm were cured with two different LED LCUs (Valo Ultradent, Ultradent Products Inc., South Jordan, UT; Demi Ultra, Kerr Dental, USA) with the light-guide tip positioned in contact with a glass slide on the upper surface of the samples. Three-way ANOVA and Student's t-test analyses were performed with the results obtained. Comparisons were performed using a significance level of  $p < 0.05$ .

**Results:** The type of bulk-fill RBC and the interaction between the type of LED LCU and shade had significant effects on the microhardness of the composites ( $p = 0.009$ ;  $p < 0.01$ ). Although the bottom surfaces of the two shades of SonicFill showed lower microhardness values than the top surfaces did ( $p < 0.05$ ), no significant difference was determined between the top and bottom surfaces of the different shades of Reveal HD bulk-fill ( $p > 0.05$ ). Although A2 SonicFill showed significantly higher microhardness values than A2 Reveal HD did when polymerized with the two LED LCUs ( $p < 0.05$ ), no significant difference was found between the A3 SonicFill and A3 Reveal HD when polymerized with DemiUltra ( $p > 0.05$ ).

**Conclusion:** Differences in microhardness among the materials are suggested to be dependent on the types and shades of bulk-fill RBCs and LED LCUs.

**Keywords:** Composite resins; curing lights; dental; polymerization

## INTRODUCTION

Since the 1960s, the main components of resin-based composites (RBCs) have been modified to achieve improved mechanical, biological and aesthetic properties (1,2). The abrasion resistance and mechanical properties of RBCs continue to be improved and offer an option for patients that restore the aesthetic appearance of teeth (3). Restoring deep cavities with RBCs requires time and effort, and postoperative sensitivity may still be observed (4). Therefore, there is increasing amount of interest and acceptability of the use of resin-based bulk-fill composites in clinical practice (3,5). A new type of RBCs with a larger size and a lower quantity of filler has been introduced to the market. These materials can be light cured in 4 or 5 mm increments instead of the conventional 2 mm increments (2,3,6). It is known that the bulk-filling technique reduces the chair time compared to the incremental technique

and reduces the risk of contamination and gap formation between the layers (2,5-7). Less chair time is important in pediatric clinical practice for better adhesion and less contamination (4). At present, different types of bulk-fill RBCs, such as low viscosity, high-viscosity, sonic-activated and dual-cured bulk-fill composite resins are commercially available.

RBC photopolymerization reactions initiate with visible blue light, based on the photoreactive systems that absorb photons at certain wavelengths of light from the light curing unit (LCU). It is known that inadequate polymerization may cause restorations to fail prematurely due to an increased incidence of secondary caries, tooth bonding failures, marginal defects, or restoration fractures. In addition, the biocompatibility of the restoration is adversely affected if the RBC is sufficiently polymerized (3). The latest advances in curing technology

**Received:** 21.11.2019 **Accepted:** 30.04.2020 **Available online:** 26.05.2020

**Corresponding Author:** Serife Ozdemir, Bezmialem Vakif University, Faculty of Dentistry, Department of Pediatric Dentistry, Istanbul, Turkey **E-mail:** serifeozalp@gmail.com

involve light emitting diodes and light curing units (LED LCUs) (8). In vitro studies have shown that bulk-fill RBCs placed at a thickness of 4 mm are sufficiently polymerized with moderate irradiances (1000 mW/cm<sup>2</sup>) and have a 20 second exposure time that corresponds to a radiant exposure of 20 J/cm<sup>2</sup>. There are many types of LCUs available for dentists. These LCUs often have different spectral radiant power magnitudes, light tip diameters, and radiant emittance (tip irradiance) levels, and these differences may adversely affect the ability of the units to improve the photoinitiation of the bulk-fill RBCs (6).

Manufacturers add different pigments to the structure of the composition to obtain a natural appearance of the tooth in terms of shade, fluorescence and opalescence. A wide range of bulk-fill RBCs is now available for clinicians' use. These RBCs are all expected to affect the light transmission of the restoration (9).

Surface hardness is one of the important mechanical properties of dental composites. The hardness of a resin composite affects the level of resistance to permanent indentation or penetration on the restoration surface. This specification affects the polishing property and level of resistance to material scratches. Mechanical properties, dimensional stability and solubility, color stability and biocompatibility are affected by the polymerization rate of composites (2).

For this reason, this study investigated the influence of different shades and types of bulk-fill RBCs on microhardness when they are polymerized with two different LED LCUs. Vickers hardness readings from the top and the bottom of the specimen were used to evaluate microhardness. The null hypothesis tested was that different LED LCUs would not affect the microhardness of different types of bulk-fill RBCs with different shades.

## MATERIAL and METHODS

In the present study, sonic-activated bulk-fill RBCs (SonicFill™, Kerr, USA) and high-viscosity bulk-fill RBCs (Reveal HD Bulk, Bisco, USA) with different shades (A2, A3) were used (Table 1). According to the power analysis performed, thirty-two cylindrical composite specimens (with a depth of 4 mm and diameter of 10 mm) were prepared with bulk-fill technique using teflon molds. The samples were polymerized with Valo (1000 mW/cm<sup>2</sup>, 395–480 nm, diameter of the tip 10mm; Ultradent Products Inc., South Jordan, UT) or Demi™ Ultra (1100 mW/cm<sup>2</sup>, 450–470 nm, diameter of the tip 8mm; Kerr Dental, USA) curing units using polyester matrix strips and thin microscope slides to obtain a flat surface (n:4). For photoactivation, the light guide tip was placed in contact with the glass slide on the top surface of the sample. Each specimen was light cured according to the manufacturers' instructions. After 10 sec irradiation, the specimens were removed from the mold and stored in 100% humidity at 37°C for 24 h to obtain the maximum amount of polymerization. For Vickers microhardness (VMH) measurements on the top and bottom surfaces,

an HMV microhardness tester (Shimadzu, Tokyo, Japan) was used with a load of 200 g for 10 sec. The VMH of each surface was recorded as the mean of the readings of four indentations obtained on the top (upper) and bottom (lower) surfaces of each specimen.

**Table 1. Brands, chemical compositions and manufacturers of the composite materials used in this study**

Brand	Chemical Composition	Manufacturer
SonicFill 2	Glass, oxide, chemicals	Kerr, CA, USA
	3-trimethoxysilylpropyl methacrylate, silicon dioxide, ethoxylated bisphenol-Adimethacrylate bisphenol-A-bis-(2-hydroxy-3-methacryloxypropyl) ether, triethyleneglycoldimethacrylate	
Reveal HD Bulk	Silica, Ba-glass	Bisco, Illinois, USA
	Ytterbium Fluoride	
	Urethane Dimethacrylate	
	BisGMA	
	3-(Trimethoxysilyl)propyl-2-Methyl-2-Propenoic Acid	
	Tert-butyl Perbenzoate	

The data were analyzed using IBM Statistical Package for Social Sciences (SPSS) 22 Software for Windows. The normality of the data were tested using the Kolmogorov–Smirnov and Shapiro–Wilk tests. Since the data (N) were normally distributed, the data obtained from the specimens were analyzed using three-way ANOVA and Student's t test. Comparisons were performed using a significance level of  $p < 0.05$ .

## RESULTS

Three-way ANOVA results showed that there were significant effects of the bulk-fill RBC ( $p < 0.01$ ), bulk-fill RBC\*shade interaction ( $p < 0.01$ ), shade\*LCU interaction ( $p < 0.01$ ) and bulk-fill RBC\*shade\*LCU interaction on upper surfaces ( $p < 0.05$ ). No significant effect was observed for the shade ( $p > 0.05$ ), LCU ( $p > 0.05$ ) or bulk-fill RBC\*LCU interaction ( $p > 0.05$ ) (Table 2).

On lower surfaces, there were significant effects of the bulk-fill RBC ( $p < 0.01$ ), bulk-fill RBC\*shade interaction ( $p < 0.01$ ), and bulk-fill RBC\*shade\*LCU interaction ( $p = 0.009$ ;  $p < 0.01$ ). No significant effect was observed for the shade ( $p > 0.05$ ), LCU ( $p > 0.05$ ), bulk-fill RBC\*LCU interaction ( $p > 0.05$ ) or shade\*LCU interaction ( $p > 0.05$ ) (Table 2).

When the microhardness of the bottom and top surfaces of the composites were compared, the bottom surface of both shades of SonicFill 2 showed lower microhardness values than the top surfaces did ( $p < 0.05$ ). No significant difference was determined in the microhardness between the top and bottom surfaces of the Reveal HD Bulk

Table 2. Evaluation of the effects of bulk-fill RBCs, shade and LCU on VMH measurement on top and bottom surfaces

Independent Variables	Top		Bottom	
	F	p	F	p
Bulk-Fill RBC	155.876	<0.001**	50.228	<0.001**
Shade	1.116	0.301	0.108	0.746
LCU	0.107	0.747	0.218	0.645
(Bulk-Fill RBC)X (Shade)	17.106	<0.001**	20.037	<0.001**
(Bulk-Fill RBC)X(LCU)	0.103	0.751	0.278	0.603
(Shade) X (LCU)	12.592	0.002**	2.193	0.152
(Bulk-Fill RBC) X (Shade) X (LCU)	19.965	<0.001**	7.988	0.009**

Dependent Variable: Vickers Microhardnes Test (VHM), \*\*p<0.01 Three-Way ANOVA

Table 3. In-group and inter-group evaluation of VMH measurements on top and bottom surfaces between LCUs when A2 and A3 bulk-fill RBCs are used separately for composite materials

Bulk-Fill RBC	Shade	LCU	Top	Bottom	t <sup>1</sup>	p
			Mean+SD	Mean+SD		
SonicFill 2	A2	Valo	86.68±6.65	68.78±3.96	7.930	0.004**
		DemiUltra	89.98±2.55	71.20±2.76	10.280	0.002**
		t <sup>2</sup>	-0.926	-1.005		
		p	0.390	0.354		
	A3	Valo	82.13±3.16	62.25±5.78	10.558	0.002**
		DemiUltra	81.55±6.47	59.60±3.56	5.562	0.011*
t <sup>2</sup>		0.160	0.780			
	p	0.878	0.465			
Reveal HD Bulk	A2	Valo	61.75±5.69	51.30±7.85	3.082	0.054
		Demiultra	44.88±7.23	45.05±5.39	-0.035	0.974
		t <sup>2</sup>	3.670	1.312		
		p	0.011*	0.237		
	A3	Valo	55.80±8.84	51.00±5.91	0.687	0.541
		Demiultra	72.70±4.32	61.00±5.71	2.987	0.058
t <sup>2</sup>		-3.435	-2.433			
	p	0.014*	0.051			

<sup>1</sup>t: Paired Sample t-Test; <sup>2</sup>t: Student-t Test; \*p<0.05; \*\*p<0.01

(p>0.05) (Table 3).

When LED LCUs were compared, no significant difference was found between the bottom and top surfaces in the microhardness of both shades of the SonicFill 2 composites (p>0.05). However, on the top surfaces, the microhardness of both shades of the Reveal HD Bulk composite showed significant differences when polymerized with different LED LCUs (p<0.05) (Table 3).

No significant difference was found between the microhardness of the composite shades when both of the bulk-fill composites were polymerized with Valo LCU (p>0.05). On the top surfaces, no significant difference was determined between the microhardness of the A2 and A3 shades of the SonicFill 2 composite when polymerized with DemiUltra (p>0.05). On the bottom surfaces, the A3-SonicFill composite showed lower microhardness values

than the A2-SonicFill composite did when polymerized with DemiUltra ( $p < 0.05$ ). On both surfaces, the A2 Reveal HD Bulk composite showed lower microhardness values than the A3 Reveal HD Bulk did when polymerized with DemiUltra ( $p < 0.05$ ) (Table 4).

**Table 4. Evaluation of VMH measurements at top and bottom surfaces between composite shades when using SonicFill and Reveal HD Bulk composite materials separately on light devices**

LCU	Bulk-Fil RBC	Shade	Top	Bottom
			Mean±SD	Mean±SD
Valo	SonicFill 2	A2	86.68±6.65	68.78±3.96
		A3	82.13±3.16	62.25±5.78
		t	1.236	1.862
		p	0.263	0.112
	Reveal HD Bulk	A2	61.75±5.69	51.30±7.85
		A3	55.80±8.84	51.00±5.91
		t	1.132	0.061
		p	0.301	0.953
DemiUltra	SonicFill 2	A2	89.98±2.55	71.20±2.76
		A3	81.55±6.47	59.60±3.56
		t	2.422	5.152
		p	0.052	0.002**
	Reveal HD Bulk	A2	44.88±7.23	45.05±5.39
		A3	72.70±4.32	61.00±5.71
		t	-6.611	-4.059
		p	0.001**	0.007**

**Student-t Test; \* $p < 0.05$ ; \*\* $p < 0.01$**

When the composite materials were compared, the A2 SonicFill composite showed significantly higher microhardness levels than the A2 Reveal HD Bulk composite did when polymerized with both of the LED LCUs ( $p < 0.05$ ) (Table 4), and no significant difference was found between the microhardness values of the A3 SonicFill and A3 Reveal HD Bulk composites when polymerized with DemiUltra ( $p > 0.05$ ) (Table 5).

**Table 5. Evaluation of VMH measurements on top and bottom surfaces between bulk-fill RBCs using Valo and DemiUltra LCUs separately in shades**

Shade	LCU	Bulk-Fil RBC	Top	Bottom
			Mean±SD	Mean±SD
A2	Valo	SonicFill 2	86.68±6.65	68.78±3.96
		Reveal HD Bulk	61.75±5.69	51.30±7.85
		t	5.697	3.975

		p	0.001**	0.007**
	DemiUltra	SonicFill 2	89.98±2.55	71.20±2.76
		Reveal HD Bulk	44.88±7.23	45.05±5.39
	t	11.768	8.633	
	p	<0.001**	<0.001**	
A3	Valo	SonicFill 2	82.13±3.16	62.25±5.78
		Reveal HD Bulk	55.80±8.84	51.00±5.91
	t	5.607	2.721	
	p	0.001**	0.035*	
	DemiUltra	SonicFill 2	81.55±6.47	59.6±3.56
		Reveal HD Bulk	72.70±4.32	61.00±5.71
	t	2.276	-0.416	
	p	0.063	0.692	

**Student-t Test; \* $p < 0.05$ ; \*\* $p < 0.01$**

## DISCUSSION

In the present study, the microhardness values of sonic-activated and high viscosity bulk-fill RBCs that had different shades and polymerized with different LED LCUs were determined. The results showed that the microhardness values of the different types of bulk-fill RBCs with different shades varied depending on the LED LCU used. Thus, the null hypothesis was accepted.

In this study, the three-way ANOVA results showed that the shade and LCU parameters did not affect the microhardness of bulk-fill RBCs and that the type of bulk-fill RBC parameter, bulk-fill RBC\*shade interaction parameter, and bulk-fill RBC\*shade\*LCU interaction parameter had a significant effect on the microhardness of the top and bottom surfaces of the composites. This result is in disagreement with the results of previous studies that investigated the effect of composite shades on the microhardness values of composites. The difference in the results between our study and previous studies may be due to the fact that only brighter shades, such A2 and A3, were preferred for use in the present study. Faria-e-Silva et al. (9) and Kramer et al. (10) reported that different factors such as resin composite type, shade and translucency, increased thickness, distance from the tip of the light curing unit, postirradiation time and size and distribution of filler particles can limit the curing depth. Sabatini (11) also reported that the surface hardness of composites depends on the type of LCU used for polymerization, whereas previous studies (10,13) have shown that the type of composite affects the performance of LCUs. When the microhardness values of the top and bottom surfaces of the bulk-fill RBCs were compared, the bottom surfaces of SonicFill showed lower microhardness values than did the top surfaces. In contrast, similar

microhardness results were found between the top and bottom surfaces of the Reveal HD Bulk groups. Previous studies that examined the degree of polymerization of composite resins showed that in addition to light-curing conditions, chemical-related factors also play an important role in polymerization. In general, the filler size from some commercially available bulk-fill RBCs increased to 20 µm, which reduced the percentage of total particle volume. As a result, there was a decrease in the filler-matrix interface and light scatter, which allowed light to penetrate deeper areas (12). Light transmission directly affects the properties of a composite and ultimately restoration performance. Many factors related to the material, including the increment thickness and the various optical properties, such as the refractive index mismatch between the organic matrix and the inorganic filler, the size and distribution of the filler particles and the added pigments, may affect light transmission through a composite (13). The different results between the top and bottom microhardness values of these two different types of bulk-fill RBCs used in the present study may be due to the different chemical compositions, different filler particles and percentages of total particle volume.

The light-curing composites were polymerized by radical photopolymerization. After photons are absorbed by photoinitiators, free radicals are formed in the presence of activators. Then, the free radicals trigger the polymerization reaction, and the monomers turn into polymers. To achieve an adequate degree of polymerization of a light-activated resin, the light absorption spectrum of the photoinitiator should fully or partly overlap with the radiation spectrum of the LCU used for photopolymerization. Camphorquinone (CQ) is the most widely used photoinitiator and has a peak sensitivity of approximately 470 nm in the blue range of the visible light spectrum (7). Recently, different photoinitiators have been added to bulk-fill RBCs. The manufacturer reported that CQ is found in SonicFill as a photoinitiator. However, the photoinitiator type of Reveal was not obtained from the manufacturer. In both shades of SonicFill, the specimens showed similar microhardness values when they were polymerized with the two different LCUs. However, on the top surfaces, Reveal HD Bulk showed different microhardness results when polymerized with Valo with a wide band spectrum of 395 to 480 nm and DemiUltra with a narrow band spectrum of 450 to 470 nm. The variation in the microhardness results of the Reveal HD Bulk composite, which was polymerized with different LED light curing devices, may be because of the difference in wavelengths of the LCUs and the initiator used in this composite resin.

The composite shade associated with the translucency of the material had a strong effect on the amount of irradiation that reached the bottom of the composite and polymerization (9). Monomer compositions and inorganic contents affect the shade and translucency of composite materials (14). In darker shades, such as C2, the light energy reaching the inferior layers of the composite is lower than

that of brighter shades, such as A2 and A3, mainly because a large percentage of radiated light is absorbed by the pigments of the composite (14). Accordingly, in the present study, on the bottom surfaces, A3 shades of SonicFill 2 showed lower microhardness values than did A2 shades of SonicFill 2 when polymerized with DemiUltra. However, similar microhardness values were found between the A2 and A3 shades of SonicFill 3 and between the A2 and A3 shades of Reveal Bulk-fill when polymerized with Valo. Although only brighter shades were used in the current study, the effect of shade on the microhardness values of bulk-fill RBCs also changed when different LED LCUs were used.

According to these results, the Valo LED LCU with a wide band spectrum polymerized both of the bulk-fill composites with different shades adequately, and the resulting composites had similar microhardness values. However, DemiUltra with a narrow band spectrum yielded different microhardness values between the A2 and A3 shades of both bulk-fill composites.

In the present study, two different bulk-fill composites; high viscosity and sonic-activated composites, were preferred. When these two different bulk-fill composites were compared, the A2 SonicFill 2 composites showed higher microhardness values than the A2-Reveal composites did when polymerized with the two LED LCUs. This result may be due to the high filler content of SonicFill (82%). However, no information regarding the filler content of the Reveal composite was available for comparison. The mechanical properties are known to be directly related to the filler content of the composite. Consequently, the SonicFill composite could be better polymerized by being activated with sonic waves, resulting in higher microhardness values.

## CONCLUSION

This study evaluated microhardness of sonic activated and high viscosity bulk-fill RBCs in two shades polymerized with different LED LCUs.

In addition to that only A2 and A3 shades were used, darker shades such as C2 could change the microhardness results. According to the results of this study, when we compare LED LCUs and shades, sonic activated bulk-fill RBCs and high viscosity bulk-fill RBCs give different results. When comparing the upper surfaces of the materials, there was no difference between the LCUs in both shades for the SonicFill, whereas in the Reveal HD Bulk group there were statistically significant differences between the LCUs in both shades. Within the limitations of this study, the microhardness of sonic-activated and high viscosity bulk-fill RBCs may vary depending on type of material, LED LCUs and shades.

*Competing interests: The authors declare that they have no competing interest.*

*Financial Disclosure: There are no financial supports.*

*Ethics committee approval: Ethics committee permission was not required for this review.*

## REFERENCES

1. Rodriguez A, Yaman P, Dennison J, et al. Effect of Light-Curing Exposure Time, Shade, and Thickness on the Depth of Cure of Bulk Fill Composites. *Oper Dent* 2017;42:505-13.
2. Farahat F, Daneshkazemi AR, Hajiahmadi Z. The Effect of Bulk Depth and Irradiation Time on the Surface Hardness and Degree of Cure of Bulk-Fill Composites. *J Dent Biomater* 2016;3:284-91.
3. Maghaireh GA, Price RB, Abdo N, et al. Effect of Thickness on Light Transmission and Vickers Hardness of Five Bulk-fill Resin-based Composites Using Polywave and Single-peak Light-emitting Diode Curing Lights. *Oper Dent* 2019;44:96-107.
4. Oter B, Deniz K, Cehreli SB. Preliminary data on clinical performance of bulk-fill restorations in primary molars. *Niger J Clin Pract* 2018;21:1484-91.
5. Ilie N, Luca BI. Efficacy of Modern Light Curing Units in Polymerizing Peripheral Zones in Simulated Large Bulk-fill Resin-composite Fillings. *Oper Dent* 2019; 44:416-25.
6. Shimokawa CAK, Turbino ML, Giannini M, et al. Effect of light curing units on the polymerization of bulk fill resin- based composites. *Dent Mater* 2018;34:1211-21.
7. Gan JK, Yap AU, Cheong JW, et al. Bulk-Fill Composites: Effectiveness of Cure With Poli- and Monowave Curing Lights and Modes. *Oper Dent* 2018;43:136-43.
8. Kaya MS, Bakkal M, Durmus A, et al. Structural and mechanical properties of a giomer-based bulk fill restorative in different curing conditions. *J Appl Oral Sci* 2018;26:20160662.
9. Faria-E-Silva AL, Fanger C, Nguyen L, et al. Impact of Material Shade and Distance from Light Curing Unit Tip on the Depth of Polymerization of Composites. *Braz Dent J* 2017;28:632-7.
10. Krämer N, Lohbauer U, García-Godoy F, et al. Light curing of resin-based composites in the LED era. *Am J Dent* 2008;21:135-42.
11. Sabatini C. Comparative study of surface microhardness of metacrylate-based composites resins polymerized with light-emitting diodes and halogen, *Eur J Dent* 2013;7:327-35.
12. Lima RBW, Troconis CCM, Moreno MBP, et al. Depth of cure of bulk fill resin composites: A systematic review. *J Esthet Restore Dent* 2018;30:492-501.
13. Leonard DL, Charlton DG, Roberts HW, et al. Polymerization efficiency of LED curing lights. *J Esthet Restore Dent* 2002;14:286-95.
14. Theodoridis M, Dionysopoulos D, Koliniotou-Koumpia E, et al. Effect of Preheating and Shade on Surface Microhardness of Siloran-Based Composites. *J Inversting Clin Dent* 2017;8.