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Original Article

A comparative study of the 5 mm-layer Vickers hardness model with bulk-fill resin-based composites

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Abstract

Aim: The aim of this study was to compare the Vickers hardness numbers (VHNs) of two bulk-fill resin-based composites (BFRBC) and a conventional hybrid resin-based composite (RBC) through the layers of a 5mm thickness model with two different light-curing time intervals.
Methodology: In the present study, a sonic-activated and dual-cure BFRBC, and a conventional hybrid RBC were used. Semi-cylindrical specimens 4 mm in radius and 5 mm in height were prepared using a two-piece stainless-steel mold (n=10). The BFRBCs allowed a single 5mm increment to be introduced into the molds, whereas hybrid RBC was incremented (2+2+1 mm). Two different time intervals were applied for the light-curing (irradiance of 1200 mW/cm^2) of each material (hybrid-sonic-activated bulk-fill, 20 s and 40 s; dual-cure bulk-fill, 7 s and 15 s). VHN measurements were carried out from top to bottom at every 1 mm of the specimen thickness. Data were analyzed using three-way and two-way ANOVA for the VHN and bottom/top ratios and Bonferroni correction for multiple comparisons (p=0.05).
Results: For each layer and time interval groups, there was a significant difference between the materials. The highest VHN was found within hybrid groups, whereas dual-cure bulk-fill groups showed the lowest results. Sonic-activated bulk-fill had the lowest bottom/top ratios, which were significantly different from those of the other materials. There was no significant difference between the different time intervals for bottom/top ratios within each material.
Conclusion: Increased irradiation intervals positively affected the VHN of hybrid and dual-cure bulk-fill. BFRBCs showed clinically acceptable bottom/top hardness ratios.

Keywords: bulk-fill, dental composite, hardness, Vickers

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Introduction

Developments in dental resin-based composites (RBCs) have resulted in the introduction of bulk-fill resin-based composites (BFRBCs) in the late 1990s. Commercially available BFRBCs offer single increment applications ranging from 4 to 10 mm according to manufacturers' instructions (1, 2). The increased depth of cure (DOC) of these materials is achieved by introducing alterations in the filler systems (lower particle content, more translucent fillers), modifications of initiator systems (novel photoinitiators, increased photoinitiator amounts), and monomer system revisions (3). As a result of the abundance of BFRBCs and their product-dependant mechanical and polymerizational properties and compositions, the classification of these materials is complicated (4). However, two main categories, 'low and high viscosity BFRBC' or 'flowable base and full-body BFRBC', are commonly mentioned in the literature (5).

Sonic-activated BFRBCs are materials that are hard to classify into these categories due to their ability to act in both low and high viscosity states via sonic activation with a specialized handpiece application (6). SonicFill 2 (SonicFill's successor and SonicFill 3's predecessor) (Kerr, USA) is claimed to successfully enable a 5 mm-depth of cure, carrying the advantages of both easily adapting to the cavity walls as a low-viscosity material and being able to be manipulated with hand instruments before light-curing as a high-viscosity material (7).

Dual-cured BFRBCs are another group of materials that are low-viscosity BFRBCs but also have other specific properties. These restorative materials incorporate chemical and photo-activation polymerization thus it is possible to chemically cure the total depth of the restoration after the light irradiation (3). Fill-Up (Coltene, Switzerland) is claimed to be ready to be polished three minutes after light irradiation at any material depth (10 mm), and a conventional RBC capping layer is optional, according to information provided by the manufacturer.

Hardness influences the quality of finishing and polishing, wear resistance, and in-service scratching of a material and is defined as the resistance to indentation. Therefore, hardness is relatively related to a restorative material's esthetic and mechanical properties, and its long-term success (8) and is often studied in the literature (9). Depth of cure (DOC) is a crucial quality that generally refers to the RBC thickness at which sufficient polymerization is achieved. The RBC hardness profiles used to measure DOC can be employed to ascertain degree of conversion as an alternative indirect method (10). There are a few studies investigating the VHN of sonic-activated and dual-cured BFRBCs utilizing a 2, 4, 5 mm thickness model. These models consist of top-bottom hardness measurements of the same specimens (11-13). Notwithstanding, this study stands out with its design, which provides VHN profiles at every 1mm layer with the same 5 mm sample in a fashion similar to work by Finan et al. (14), as opposed to using separate samples of different thicknesses.

The aim of this study was to investigate the VHN of two BFRBCs and a conventional hybrid RBC through the layers of a 5 mm thickness model with two different light-curing time intervals. The hypotheses of the study are as follows: (1) the difference in the RBC type will not affect the VHN at the same depth and for the same light-curing irradiation period, and (2) varying the light-curing irradiation periods and depth will not affect the VHN of the tested materials.

Materials and Methods

In the present study, two BFRBCs and a conventional nanohybrid RBC, shade A2, were used, as shown in Table 1. A total of 60 semicylindrical specimens were prepared using three RBCs and two different light-curing time intervals, which resulted in six test groups (n=10 specimens per group).

1. Specimen preparation

A two-piece stainless-steel mold was employed to achieve semi-cylinder (a vertically split cylinder)-shaped specimens with a 4 mm diameter and 5 mm height. The mold consisted of a piece with a flat surface (5 mm height, 1.5 cm depth,7 cm length) and a second piece in the same general shape with a 4 mm diameter semicylinder void. The mold was placed on a mylar strip with a glass slide underneath. Another mylar strip was also taped on the counterpart flat piece of the mold, and two pieces were stabilized during specimen preparation.

The conventional nanohybrid RBC was placed incrementally (2 mm, 2 mm, 1 mm) and light-cured after each increment. After the last increment, the RBC was covered with a mylar strip to minimize the effects of oxygen inhibition and a glass slide to allow compression for 10 s. The glass slide was then removed, and the RBC was light-cured as previous increments for two different curing time intervals, which are shown in Table 2. An LED (light-emitting diode) (Model BUILT-IN C, Guilin Woodpecker Medical Instrument Co., Guilin, Guangxi, China) light-curing device with an 8 mm diameter light tip and 1200 mW/cm^2 light intensity was utilized for the polymerization of the RBCs.

The sonic-activated and dual-cure BFRBCs allowed a single 5 mm increment placement into the molds. Next, the preparation steps were the same except for the curing time intervals of the dual-cure BFRBC and their removal time from the molds. Dual-cure bulk-fill composite specimens were removed from the molds three minutes after light curing, which is the curing time stated in the manufacturers' instructions. The bottom surface of the specimens was marked with a permanent marker, and the excess material was removed. A cylinder mold (3 cm diameter, 3 cm height) was used in order to embed the specimens (four specimens in per mold) in self-cure acrylic resin allowing the polishing and hardness measurement procedures to be simpler and faster. First, a piece of double-sided tape was placed on the bottom cover of the cylinder mold. Specimens were placed as their bottom surfaces were facing the same direction, and the flat surfaces faced the double-sided tape. The interior surfaces of the cylinder mold, which were then placed on the bottom cover, were isolated with Vaseline. The mold was filled with the self-curing acrylic resin, and after curing, the bottom cover was subsequently removed, and the acrylic cylinder was effortlessly pushed out of the mold due to Vaseline isolation.

The samples were ground with 1200 and 4000 grit SiC paper under water cooling using a polishing device (Forcipol 2V, Metkon Instruments Inc., Bursa, Turkey) to achieve an even and standardized surface for the tested materials (12, 13). After surface treatment, the specimens were stored in a humid and dark environment at 37^{0}C for 24 h in an EN 055 Nüve Incubator (Ücel Kimya Medikal, Sivas, Turkey) before hardness measurements were collected.

Table 1. The materials used in the study.

Material	Matrix	Filler type and size	Filler Loading	Manufacturer	Batch
Grandio Nanohybrid	Bis-GMA, TEDMA, UDMA	Ceramic glass fine particles (1µm), spherical silicium dioxide (20-60 nm)	87% wt 71.4% vol	Voco (Cuxhaven, Germany)	1545335
SonicFill 2 Sonic activated nanohybrid bulk-fill	Bis-GMA, TEGDMA, EBADMA (Ethoxylated bisphenol)	Silicon dioxide, barium glass	83.5% wt 83% vol	Kerr (Orange, CA, USA)	5790868
Fill-Up Dual-cure bulk-fill	TMPTMA (Trimethylol propane trimethacrylate), UDMA, Bis-GMA, TEGDMA, dibenzoyl peroxide, benzoyl peroxide	Dental glass, amorphous silicic acid, zinc oxide (2 µm)	65% wt 49% vol	Coltene-Whaledent (Altstatten, Switzerland)	G32310

Table 2. RBC increment and different light-curing time interval protocols used in the study.

Material	Placement	Light-curing time interval
Grandio	2 mm+2 mm+1 mm increments	I; 20 s and II; 40 s
SonicFill 2	5 mm single increment	I; 20 s and II; 40 s
Fill-Up	5 mm single increment	I; 7 s and II; 15 s

2. Vickers hardness measurement

The Vickers hardness of the specimens was measured using an HWDM-3 Highwood hardness tester (TTS Unlimited Inc., Osaka, Japan) with a load of 200 g applied for 30 s (14). On each sample starting from the top to the bottom, three measurements were recorded at every 1 mm through the specimen thickness. Thus, five mean VHN values were obtained for every specimen.

The VHN at 5 mm divided by the VHN at 1 mm was calculated as the bottom/top hardness ratio for the depth of polymerization evaluations.

Statistical analysis

Statistical analysis of data was performed using the SPSS 21.0 (IBM SPSS Inc., Armonk, NY, USA). Three-way ANOVA was performed to evaluate the hardness results regarding different RBCs, depth, and time intervals (p=0.05). The bottom/top ratio data regarding different RBCs and time intervals were analyzed with two-way ANOVA (p=0.05). Bonferroni correction was employed for multiple comparisons for both hardness and the bottom/top ratio assessments.

Results

According to the ANOVA results of the study, RBC material, depth, and light-curing duration significantly affected the hardness values (p<0.001) (Table 3). The Grandio groups demonstrated the highest hardness results, followed by SonicFill 2 and Fill-Up (Table 4).

Grandio and Fill-Up performed statistically better in time interval II at each depth layer (p<0.05), whereas no significant difference was found between time intervals at each depth layer for SonicFill 2 (p>0.05) (Table 4). In Fill-Up and Grandio groups for both time intervals, no significant difference was found between varying depths (p>0.05) (Table 4).

Table 3. Three-way ANOVA analysis of Vickers hardness results

	Type III Sum of Squares	df	Mean Square	F	p
Material	262498.720	2	131249.360	16652.968	<0.001
Depth	1137.757	4	284.439	36.090	<0.001
Time	1049.978	1	1049.978	133.222	<0.001
Material * Depth	1918.484	8	239.811	30.427	<0.001
Material * Time	558.093	2	279.047	35.406	<0.001
Depth * Time	20.231	4	5.058	0.642	0.633
Material * Depth * Time	124.152	8	15.519	1.969	0.051

Table 4. Mean±SD Vickers hardness values of tested groups

Depth	Time	Material		
		Fill-Up	SonicFill 2	Grandio
1 mm	I	45.67±1.12 (a, x, A)	72.43±2.6 (b, x, A)	109.18±3.86 (c, x, A)
	II	49.02±0.89 (a, x, B)	72.35±1.3 (b, x, A)	116.21±3.1 (c, x, B)
2 mm	I	44.59±1.57 (a, x, A)	70.77±2.14 (b, xy, A)	108.8±3.16 (c, x, A)
	II	48.98±1.09 (a, x, B)	69.71±1.66 (b, xy, A)	113.52±4.69 (c, x, B)
3 mm	I	45.06±1.19 (a, x, A)	67.34±3.17 (b, yz, A)	108.65±5.17 (c, x, A)
	II	48.83±1.07 (a, x, B)	67.65±1.8 (b, y, A)	115.99±3.53 (c, x, B)
4 mm	I	44.91±1.21 (a, x, A)	64.41±2.83 (b, z, A)	105.94±4.46 (c, x, A)
	II	49.4±1.61 (a, x, B)	63.5±2.04 (b, z, A)	114.68±3.61 (c, x, B)
5 mm	I	44.91±1.21 (a, x, A)	56.79±3.01 (b, t, A)	109.89±4.01 (c, x, A)
	II	49.38±1.17 (a, x, B)	58.97±1.92 (b, t, A)	113.52±4.74 (c, x, B)

The lowercase 'abc' letters refer to the rows for RBC type comparisons. The lowercase 'xyzt' letters refer to the columns for varying depth VHN comparisons within the same time interval. The uppercase 'ABC' letters refer to the columns for I-II.time interval VHN comparisons within the same depth. Values with the same letters are not significantly different.

In time interval I for SonicFill 2, there was a significant difference in the VHN between depth layer comparisons ($p<0.05$), except in the 1 and 2 mm, 2 and 3 mm, and 3- and 4-mm comparisons ($p>0.05$) (Table 4). In time interval II for SonicFill 2, there were significantly different VHN when comparing the 1 and 2 mm and 2- and 3-mm pairs ($p>0.05$) (Table 4).

The ANOVA results of bottom/top hardness ratios revealed that whereas the time intervals did not affect the bottom/top hardness ratios significantly ($p>0.05$), the material type effect was significant ($p<0.001$) (Table 5). For both time intervals, there was no significant difference between the Fill-Up and Grandio bottom/top hardness ratios ($p>0.05$), although the SonicFill 2 ratios were significantly lower ($p<0.05$) (Table 6). Furthermore, a significant difference was not observed between time intervals I and II for any of the RBCs ($p>0.05$) (Table 6).

Table 5. Two-way ANOVA analysis of bottom/top hardness ratios.

	Type III Sum of Squares	df	Mean Square	F	p
Material	0.575	2	0.288	212.587	<0.001
Time	0.001	1	0.001	0.491	0.486
*Material * Time*	0.012	2	0.006	4.267	0.018

Table 6. Mean±SD bottom/top hardness ratio values of tested groups

Time	Material		
	Fill-Up	SonicFill 2	Grandio
I	0.99±0.02 (a, A)	0.78±0.04 (b, A)	1.01±0.04 (a, A)
II	1.01±0.02 (a, A)	0.82±0.03 (b, A)	0.98±0.06 (a, A)

The lowercase 'abc' letters refer to the rows for RBC type comparisons within the same time interval. The uppercase 'ABC' letters refer to the columns for I-II.time interval bottom/top hardness ratio comparisons within the same RBC type. Values with the same letters are not significantly different.

Discussion

There are very few studies regarding the VHN of BFRBCs regarding polymerization time and material thickness. The present study was designed to investigate the effects of two different light-curing time intervals and material thickness on VHN for a dual-cure, a sonic activated BFRBC, and a nanohybrid RBC. The first hypothesis was rejected because the restorative material type exhibited significant differences in VHN within the same thickness and polymerization period. The second hypothesis, however, was partially rejected because the depth and light-curing period effects on the VHN were only significant for some of the test groups.

A conventional nanohybrid RBC, Grandio, which is used in other hardness studies in the literature (18, 19), was selected as a control group in this study. Poggio et al. compared the VHN of three microhybrids, two nanohybrids (including Grandio), and a nanofil RBC using 20 s, 40 s, and soft-start 40 s polymerization protocols (18). Similar to the present study, the highest values were observed in the Grandio groups, and Grandio had bottom/top ratios higher than 0.80, which indicates that they are clinically acceptable. Jafarzadeh-Kashi et al. evaluated the VHN of Grandio and two other nanohybrid RBCs at three different irradiation times (10, 20, and 40 s) (20). Similar to our study, previous results found that Grandio, having the highest filler percentage (vol), yielded the highest VHN among the tested RBCs. Increasing the irradiation periods improved the surface hardness of Grandio in both studies, supporting the light-curing time as a parameter affecting the VHN of RBCs. There is also a study comparing Grandio hardness values to two BFRBCs each had similar filler ratios (86% and 85.5% (wt)) to each other and SonicFill 2 (83.5% wt), but higher than Fill-Up's (65% wt) (19). They also reported that Grandio exhibited higher VHN than the two microhybrid BFRBCs tested in the study.

As a member of the bulk-fill family, SonicFill 2 is drawing attention due to its unique sonic-activated insertion technique; thus, studies regarding its various properties are increasing in the literature. Monterubbiasnessi et al. compared two sonic-activated (SonicFill, SonicFill 2), a low-viscosity (SDR), a high-viscosity (Filtek BF), a dual-cure (Fill-Up) BFRBCs to evaluate the degree of conversion and VHN (13). It has been concluded that sonic-activated (especially SonicFill 2) BFRBCs had the highest VHN, and Fill-Up exhibited moderate results between 40-50 VHN, which is relatively similar to the present study (44.59±1.57-49.4±1.61 including both time intervals). In another study conducted by Moharam et al., SonicFill 2 had higher VHN in both 4 mm bulk and incremental insertions (80-90 VHN) than it did in our findings, which were between 56-72 VHN (21). This difference may be attributed to the study designs of each paper, especially the aforementioned VHN measuring technique, semi-cylindrical sample shape, and 5 mm sample thickness. On the other hand, a high-viscosity BFRBC (X-tra Fil) has similar filler particle ratios with Grandio. Both have Bis-GMA, TEGDMA, and UDMA monomers and are by the same manufacturer exhibited significantly higher VHN than SonicFill 2, which is similar to the results of our study.

Kim et al. reported that increasing the thickness (2, 3, and 4 mm) decreased the VHN of the tested materials: four BFRBC (including SonicFill), a flowable conventional RBC, and a condensable nanohybrid RBC (11). Among these materials, SonicFill had the highest hardness values, contrary to our findings, even though there are differences between its successor, as previously mentioned. The results were explained by the fact that SonicFill is comprised of higher inorganic filler particle ratios. Babishi et al. investigated the effects of various beverages on VHN of four bulk-fills and a conventional RBC (Filtek Z 350) (22). In this study, SonicFill 2 was revealed to have the highest VHN among the other BFRBCs, and in agreement with our study, it underperformed significantly ($p<0.05$) than Filtek Z 350 which is also a nanohybrid but has lower filler particle ratios than Grandio.

There is limited information regarding Fill-Up in the literature because it is a relatively novel restorative material. Aggarwal et al. tested four bulk-fills, a flowable conventional RBC, and a condensable microhybrid RBC and concluded that Fill-Up showed relatively similar VHN (bottom (4 mm): 34.56, top: 44.2) to our findings (12). As opposed to the present study, the bottom/top ratio obtained for Fill-Up by Aggarwal et al. (0.78) was less than 0.80. Dry and ethanol VHN of eight BFRBC, a flowable RBC, and Grandio were investigated in Leprince et al.'s comparative study (17). In agreement with the present study, Grandio showed higher VHNs (dry and ethanol) than any other material (including SonicFill and Fill-Up), and the dry VHN results were similar to our data (time interval II) for Grandio, Fill-Up, and SonicFill. The differences with respect to our study were the sample thickness (2 mm) and the light-curing time for Fill-Up (40 s), and the bottom/top ratio was not included in Leprince et al.'s study. In an analysis of residual monomer study (23), Fill-Up was observed to exhibit a higher residual monomer release than a nanohybrid RBC. It was also argued that less Bis-GMA and UDMA elution from Fill-Up compared with the other tested RBCs might be attributed to its dual-curing property. This argument may support the nonsignificant VHN difference through the 5mm thickness and the high bottom/top ratios for Fill-Up in our study.

Some studies have shown that by increasing the thickness of the sample, insufficient light energy is delivered to the bottom of the sample (24-26). However, in both time intervals, the Fill-Up and Grandio groups showed no significant difference between varying depths, whereas the SonicFill 2 groups demonstrated significant differences in a few depth layer comparisons. These results can be partially explained by the lower amount of light transmission compared to Fill-Up and Grandio. During the curing process, the light that passes through the RBC is absorbed and scattered based on the particulate size of fillers and refractive indices of the resin matrix and fillers (27, 28). There were higher microhardness values for top surfaces in comparison to the bottom surfaces. The explanation is that the reduced microhardness value for the bottom surfaces is directly related to the attenuation in light intensity due to the light scattering while passing through the composite mass.

The time variable was also evaluated in the current study. The manufacturer's recommended curing times are shown in Table 1. Increasing the curing time is expected to increase the microhardness (29). The results of this study showed a significant improvement for Grandio and Fill-Up by increasing the irradiation time at each depth layer, whereas no significant difference was found between time intervals at each depth layer for SonicFill 2. Such findings could be multifactorial. One of these factors might be the difference in the chemical composition of the matrix, which has been reported to affect the surface microhardness of resin composites. Moreover, other parameters might also be responsible for the difference in surface microhardness values among the different tested materials including, filler particles size, morphology and distribution (30), particle shape and density, monomer type and ratio, the degree of polymer cross-linking, and the degree of conversion; which all vary greatly between the various products present in the market (21).

Several studies have defined depth of cure based on hardness measurements performed on the top and bottom surfaces of a light-cured resin composite specimen (11, 31-33). Generally, in the studies, different incremental thicknesses of 2, 4, and 6 mm were examined on different samples (34, 35). In this study, hardness values were evaluated in 1 mm layers, similar to Garoushi et al.'s study (15), but they evaluated different samples for varying depths (1, 2, 3, and 4 mm). Furthermore, the hardness values obtained were used to calculate a bottom/top hardness ratio and judged to be adequate when this ratio was over 80% or more. Based on this observation, all tested RBCs could be used up to depths of 5 mm.

One of the factors indirectly impacting the hardness is the shade of the RBC. In the study of Anfe et al. the microhardness values were influenced by the translucency of resin-based composites; thus, ensuring the accuracy of the test, all the RBCs in this study were selected to have shade A2 (36).

The limitations of the present study are that only two BFRBC varieties were compared with a conventional hybrid RBC, and only LED light was used as it is more commonly available. No thermocycling was carried out, the lack of which excludes the effects of mechanical and thermal stresses that are otherwise inevitable in the oral environment.

Conclusions

Further in vitro and in vivo investigations should be carried out regarding the properties of Fill-Up and SonicFill 2 in light of the current study and its limitations. The following conclusions were drawn according to this study:

- A nanohybrid RBC yielded the highest VHN values, followed by SonicFill 2 and Fill-Up.
- All tested RBCs could be used up to a depth of 5 mm based on the clinically acceptable bottom/top hardness ratios, which may indicate adequate polymerization levels for these materials.

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