Research Article

The effect of different curing distances on the microhardness of flowable bulk-fill composite materials

Ayah A. Alzaidy [1], **Manal H. Abd-alla** [1*]
[1] Department of conservative dentistry, College of Dentistry, Mustansiriyah University, Iraq
*Corresponding author: Manaer11@uomustansiriyah.edu.iq

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Abstract: Background: The microhardness of a composite resin is a vital parameter that is used to determine its clinical behavior. Measuring the microhardness of a composite resin has been used as an indirect method to assess its degree of conversion and extent of polymerization. The purpose of this in vitro study was to evaluate the effect of three curing distances (0, 2, and 4 mm) on the microhardness of the top and bottom surfaces of three types of flowable bulk-fill composite resins (smart dentin replacement, Opus bulk fill flow, and Tetric N). Material and method: Sixty-three specimens from the three types of composite resins (n=21) were fabricated using Teflon mold with a 4mm depth and a 5 mm internal diameter and cured for 20 seconds. For each material, three subgroups were fabricated according to the position of the light curing tip from the top surface; at 0, 2, and 4 mm distances. Microhardness was measured using the Vickers test with a 50-g load for 15 seconds for the top and bottom surfaces of all the samples. Results: The microhardness values were decreased in the following order; 0mm > 2mm > 4mm curing tip distance, for both sides and Tetric N had the highest microhardness values. Significant differences in microhardness were recorded between the top and bottom surfaces for all the specimens ($p<0.05$). Conclusion: Increasing the distances between the tip of the light cure and the surface of flowable bulk-fill resins can significantly decrease the microhardness of the bottom surfaces compared to the top surfaces.

Keywords: Microhardness; light cure tip distance; smart dentin replacement; Opus bulk fill flow; Tetric N flow

Introduction

Recently and with the industrial development in dental materials, wide varieties of dental resins have been introduced to the markets. These resins were represented to be used in all clinical situations with the best performance as well as the most convenient handling by the clinician. Bulk-fill materials were introduced as a substitute for the conventional resin to be cured with a single 4 mm incremental thickness instead of 2 mm layering [1]. Using these materials represented a convenient and less time-consuming technique in extensive posterior restorations or core build-up procedures. Bulk-fill composites with their high translucency and similar refractive index of the organic matrix and filler particles allowed enough light to penetrate for polymerizing the deeper layers. Additionally, the presence of more reactive photoinitiators improved the depth of cure resulting in a performance comparable to conventional composites, even when it was inserted in a single thicker increment [2]. Flowable bulk-fill resin composites, on the other hand, were introduced with a low viscosity resulting from lower filler content than conventional composites of about 37-53% by volume [3]. Bulk-fill materials with low-viscosity that have lower mechanical properties are suggested to be used in a single 4 mm thick increment as an internal layer to reduce polymerization stress [4].

Composite resins are different in their mechanical and physical properties according to their composition. The clinical success of any restoration depends on various factors that are related to the material used such as; degree of polymerization, polymerization shrinkage of the resin, linear coefficient of thermal expansion, wear resistance, modulus of elasticity, and depth of cure, etc.[1]. The microhardness of a material is one of the parameters that can both directly assess the resistance of the material to deformation [1] and indirectly measures its degree of conversion since it is considered the best indicator for the extent of polymerization [5]. One of the factors that affect the mechanical properties of any composite resin is the filler load within it. However, it has been demonstrated that light penetration through a composite restoration is greatly influenced by the filler size and content of the composite resin [6]. Some researchers have reported a reduction in radiant exposure of the curing light due to increasing the light curing distance between the composite surface and the tip of the light cure [7]. This reduction could be produced as a result of increasing the distance between the curing tip from the composite surface due to tooth morphology such as; cusp height, cusp steepness, and cavity depth, [8].

Many studies have been performed on the microhardness of the bulk-fill composite using different curing units [9], different curing times [10] and different curing distances [5]. The effect of light-curing distance on the curing of bulk-fill flowable composite is clinically pertinent because they are cured in 4 mm increments making the restoration bottom surface particularly vulnerable to light scattering further from the light source [11]. It was reported that the curing distance did not significantly affect the microhardness of high-viscosity bulk-fill composites [12]. Diab et al. in 2021[11] investigated the effect of light-curing distances on the curing (characterized by the hardness ratio) of two bulk-fill composites (Tetric N-Ceram bulk fill and Filtek bulk fill) in 4 mm thickness and cured for 20s at various distances: 0, 2, 4, 6, and 8mm. They concluded that both resins recorded a hardness ratio significantly lower at 8 mm. However, more evidence is still needed on the effect of different curing distances on the microhardness of new flowable bulk-fill resins (Opus bulk-fill flow and Tetric N flow bulk-fill). Low-viscosity bulk-fill materials are indicated to be used within deep and internal areas of cavities [4, 13]. Hence, reduction in light exposure for the bottom surfaces of the restoration could be a critical issue if used in deep cavities. Inevitable incomplete polymerization could compromise their mechanical properties. Therefore, this study was conducted to evaluate and compare the top and bottom surfaces' microhardness of three flowable bulk-fill resin composites cured at three light-curing distances; 0, 2, and 4 mm. The null hypotheses for this study were; firstly: There are no differences in the microhardness between the top and bottom surfaces of each specimen. Secondly: There are no differences in the microhardness of each resin composite at the three distances of cure. Lastly: There are no differences in the microhardness between the different flowable bulk-fill composites at each cure distance.

Materials and Methods

The flowable bulk-fill composite resins used in the study and their compositions are shown in Table 1.

Samples preparation and grouping

A total of 63 disk shape specimens were fabricated from the three materials (n=21) and divided into three groups: Group A using SDR, Group B using Opus bulk fill flow, and Group C using Tetric N flow. The specimens were fabricated using a Teflon mold with a 5mm diameter and 4mm depth. The mold was placed over a glass slide and filled with one increment of the material, then, it was covered by a Mylar strip to produce a flat surface. The light-curing was performed for 20 seconds, using a light-emitting diode (LED) system (Guilin Woodpecker Medical Instrument Co., Ltd. China). The light curing tip diameter was of 8 mm and light intensity of 1000-1200mW/cm^2. According to the light cure tip distance, three subgroups were fabricated from each group (n=7); Subgroups A1, B1, and C1 were cured with the light tip positioned at 0 mm from the top surface. For subgroups A2, B2, and C2, curing was performed at 2 mm away from the top surfaces using a ring spacer with a thickness of 2 mm, and for the third subgroups A3, B3, and C3, light-curing was performed at 4 mm away from the top surface of the specimen using a ring spacer with a thickness of 4 mm. Following curing, the samples were labeled with a red line on the top and a black line at the bottom. These specimens were organized in dry and dark containers at 37^o C for 24 hours. Each composite type was stored in a separate container and labeled with the group names, subgroup numbers, and sample numbers. The microhardness of the top and bottom surfaces for each specimen was tested using Digital Micro Vickers Hardness Tester TH714 (Beijing Time High Technology Ltd. China) using a 50-g load for 15 seconds [12]. Four indentations were taken for each surface (top or bottom) of each specimen, and the microhardness mean values were calculated for each side [15].

Table 1: The components of the flowable bulk-fill resin composite used in this study.

Products	Smart dentin replacement	Opus bulk-fill flow	Tetric N flow bulk-fill
Manufacturer	Dentsply Caulk, Milford, DE, USA	FGM, Joinville, SC, Brazil	Ivoclar Vivadent Inc Amherst, NY, USA
Recommended increment size	Up to 4 mm	Up to 4 mm	Up to 4 mm
Composite shade	Shade A1	Shade A1	Shade IVA
Method of activation	Visible light cure	Visible light cure	Visible light cure
Curing time	20 sec. by 550mW/cm²	20 sec.	≥ 500 mW/cm², 20 sec. ≥ 1000 mW/cm², 10 sec.
Resin Components	Modified UDMA resin, TEGDMA, polymerizable dimethacrylate resin; polymerizable trimethacrylate resin; camphorquinone (CQ) photoinitiator; ethyl-4(dimethylamino)benzoate photoaccelerator; butylated hydroxytoluene (BHT); fluorescent agent, and UV stabilizer.	UDMA monomers, Stabilizers, Camphorquinone Co-initiator (Quantities and specifications not provided by the manufacturer).	Bis-GMA, UDMA, TEGDMA.
Fillers type	Bareium-alumino-fluoro-borosilicate glass, silanated strontium alumino- fluoro-silicate glass, surface-treated fumed silicas, ytterbium fluoride, synthetic inorganic iron oxide pigments, and titanium dioxide.	Inorganic silicon dioxide (silica) loads, stabilizers, and pigments.	Barium glass, ytterbium trifluoride, and copolymers (71 wt%). Additives, initiators, stabilizers, and pigments are additional ingredients (<1.0 wt%).
Filler loading (wt/vol)	70.5 wt%., 47.4 vol%.	Not available.	68.2 wt%., 46.4 vol%.
Filler size	4.2 µm	Not available.	Ranges between 0.1 µm and 30 µm with a mean particle size of 5 µm.

Statistical analysis

The values of microhardness for all specimens were analyzed using IBM SPSS Statistics (SPSS for Windows, version 26; IBM Corp). The statistical significance for the mean values of microhardness among the different groups was calculated using one-way analysis of variance (ANOVA) and Tukey's test at the significance level of $\alpha = 0.05$.

Results

The means of the top and bottom microhardness of the studied materials are shown in Figure 1. Descriptive and inferential statistics of the microhardness values are shown in Table 2.

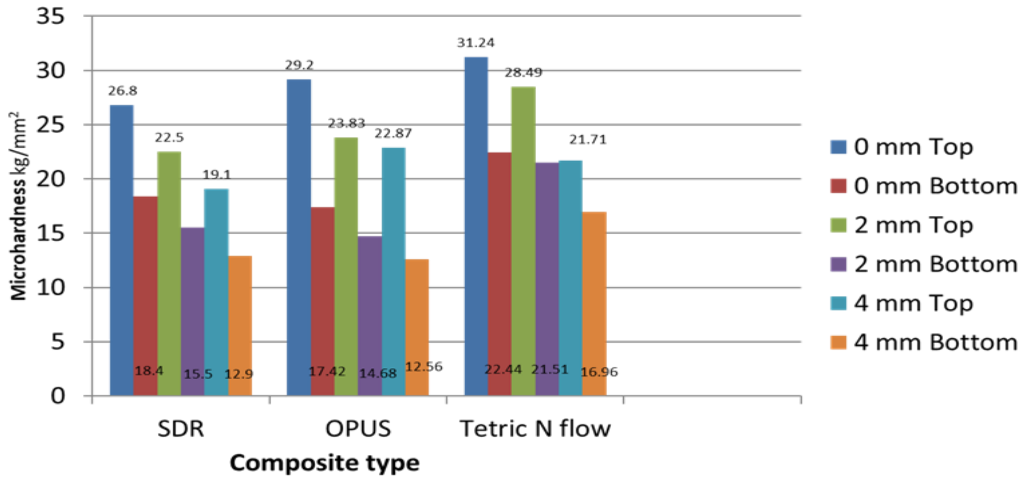

Figure 1: Bar chart shows the means of the top and bottom microhardness of SDR and Opus and Tetric N Ceram Bulk Fill groups at different light tip distances.

Table 2: The microhardness mean values, Min. Max, and SD for the top and bottom sides at the three curing distances for the three groups in kg/mm²

Groups	Light tip distance	Top/bottom	Mean *	SD	Max.	Min.
	0 mm	Top	26.84 [a] {*i*}	2.17	29.48	23.88
Group A		Bottom	18.45 [a] {*j*}(**s**)	2.76	21.3	14.07
SDR	2 mm	Top	22.50[b] (**u**)	2.17	26.92	20.33
		Bottom	15.51[b] (**v**)	1.57	18.3	13.54
	4 mm	Top	19.12[c] {*i*}	2.15	21.69	15.34
		Bottom	12.93[c] {*j*}(**x**)	1.65	15.72	10.65
	0 mm	Top	29.20[d] {*k*}	3.30	35.48	25.83
Group B		Bottom	17.42[d] {*l*}(**s,t**)	2.04	19.89	14.05
Opus flow	2 mm	Top	23.83[e]	1.90	26.77	21.73
		Bottom	14.68[e] {*m*}	1.37	17.14	12.76
	4 mm	Top	22.87[f] {*k*}	1.43	24.62	20.34
		Bottom	12.56 [f] {*l, m*}(**w**)	1.54	14.54	10.03
Group C	0 mm	Top	31.24 [g] {*o*}	5.47	39.09	25.65
Tetric N		Bottom	22.44 [g] {*n,p*} (**t**)	2.32	26.24	20.24
	2 mm	Top	28.49 [h] {*q*}(**u**)	2.92	33.25	25.67
		Bottom	21.51 [h] {*n,r*}(**v**)	1.22	23.09	19.62
	4 mm	Top	21.71 {*o,q*}	2.34	26.03	18.86
		Bottom	16.96 {*p,r*}(**w,x**)	2.46	19.27	12.13

* mean values with the same letter are sig. different at p< 0.05. [between top and bottom sides at same distance], { *between three distances*}, (**between the three groups**).

The highest microhardness values were at 0 mm followed by 2 mm, and 4 mm light tip distances, respectively, at both sides for all the groups. Tetric N flow had the highest microhardness values followed by Opus flow, then SDR.

The microhardness of the top versus the bottom side at the three curing distances showed significant differences between the top and bottom values of SDR and Opus flow ($p<0.05$). However, for Tetric N, there were significant differences between the top and bottom sides at 0 and 2mm curing distances, with no significant difference at 4 mm curing distance.

Comparing the microhardness for the three light tip distances showed significant differences between 0 and 4 mm curing distances for the top and bottom sides. This was true for the three composite types. However, significant differences were recorded for Opus flow between 2 and 4 mm for the bottom sides. For Tetric N, significant differences were seen between 2 mm and both 0 mm and 4 mm.

While the microhardness of the three materials at the same curing distance, for the top sides showed no significant difference between the three groups except between SDR and Tetric N flow a significant difference was recorded ($p=0.008$) at a 2 mm curing distance. However, for the bottom sides and at 0 and 4 mm distances, the differences were significant between Tetric N and both SDR and Opus flow ($p<0.05$). However, at a 2mm curing distance, the difference was significant between SDR and Tetric N only.

Discussion

In the current study, microhardness values for the top surfaces were significantly greater than those of the bottom surfaces for all three distances. Thus, the first null hypothesis was rejected. Significant decrease was recorded between the top to bottom microhardness at all the three curing distances except for Tetric N at 4 mm. This result can indicate that a greater amount of light energy reached the photoinitiator at the top surface, thus starting the polymerization reaction and higher values of microhardness were reached. It has been reported that resin composites have the property of dispersing the light energy of the curing light. Thus, when the light passes through the bulk of the composite, light intensity is reduced due to the light being scattered by filler particles and the resin matrix [16]. It has been reported that a 2 mm thickness of a composite resin is sufficient to reduce the light intensity to 6% of its initial value [5]. This results are supported by other studies reporting that composite resins showed a gradual decrease in microhardness as the depth of cure increases, and this drop is more accentuated for depths beyond 2 mm [17]. It has been reported that when the thickness of a low viscosity resin composite increases, the hardness values will be decreased [18].

For the same material, the results of this study showed that the microhardness values were in the following order; 0mm > 2mm > 4mm curing tip distance, for both sides with significant differences between 0mm and 4mm ($p<0.05$). This was true for all three types of the used flowable composites. Thus, the second null hypothesis was also rejected. This result can be attributed to the distance between the light tip and the resin composite as increasing the distance affects the light intensity that reaches the material. It has been reported that 1mm of air reduces the light intensity by approximately 10%, thus reducing the polymerization depth and the degree of conversion that affects the microhardness [8]. Similar results were reported for the microhardness of Filtek Z250 and Filtek Supreme [19] and SDR and Tetric EvoCeram Bulk Fill [5]. Comba et al. in 2020 [4] showed that SDR produced a significant decrease in microhardness at a depth lower than that recommended by the manufacturer and a significant decrease was evident at 2.15 mm depth.

For the third null hypothesis of this study, Tetric N flow showed significantly higher microhardness values than the other two composites at the bottom side. While for the top side, the difference was significant between SDR and Tetric N flow at a 2mm distance. Thus, the third null hypothesis was also rejected. This result could be attributed to the differences in the composition of these three materials. Many material attributes would affect the scattering and absorption of light including; filler content, filler size and shape, the resin matrix, photoinitiators, pigments, and even the type of surface treatment of the fillers that enables them to attach to the resin matrix [19]. Although SDR and Tetric N flow have a close volume content of fillers of 47% (with 4.2 μm in size) and 46% (0.1 to 30 μm in size) respectively, the size of the fillers within Tetric N had a wider range. Such a result is in accordance with the results of other studies (18, 20). It has been reported that factors other than filler volume can affect the microhardness such as the size and shape of the fillers (20). The lower percentage of fillers with larger filler particles can reduce light scattering at filler-matrix interfaces and ensure more light penetration into deeper layers (18). Although other researchers support that a resin with a higher filler size and volume may produce a higher surface hardness (21), it has been reported that different types of monomers can affect the microhardness of resins (22). The polymerization of resins depends on different factors, such as their chemical structure and concentration of monomers and photoinitiators (18). It was reported by Kelic et al. in 2016 (20) that a resin containing Bis-GMA, which is the most vicious and least flexible monomer, tends to be higher in microhardness. Therefore, low values of microhardness are not unexpected for the three experimented composites in this study as they contained UDMA as the main monomer, which has the least viscosity. At the same time, Tetric N flow combines several other monomers such as Bis-GMA and TEGDMA, which can increase the stiffness of the polymer matrix. These differences in the matrix components of Tetric N may produce a higher microhardness in comparison to the other two materials. For Opus flow, enough data concerning filler content and size was not available by the manufacturer; therefore, no logical explanation can be reached.

The microhardness value of a composite resin when exceeds 50 VHN was proposed by some authors to be considered ideal (23). However, these values have been recorded for the microhardness of conventional composite resins, not for flowable bulk-fill composite. According to the results of this study, all three flowable bulk-fill composite resins used showed lower microhardness at the bottom surfaces, and the values were decreased by increasing the light tip distances from the top surface. Hence, a 4 mm thickness cured for 20 s with a light intensity of 1000-1200 mW/cm2 was insufficient to obtain similar top-to-bottom parameters, especially when it is cured with a higher than 0 mm light tip distance. The hardness depends on the extent of the reaction and the degree of crosslinking produced during the monomer curing process (24). Increasing photo-activation time may activate the initiator and generate free radicals that start the polymerization, presenting a synergistic effect on the polymerization rate. In this way, it is possible to extend the photo-activation time to achieve a higher conversion (25). Therefore, low microhardness values for flowable bulk-fill resin support its use as dentin replacement that requires a veneering layer of a micro-hybrid composite resin with high microhardness to withstand the posterior occlusal load (26, 27).

Conclusion

Within the limitations of this study, it can be concluded that:

The microhardness values of the bottom surfaces can be significantly decreased compared with the top surfaces by increasing the distances between the tip of the curing light from the resin surface from 0, 2, to 4 mm, and this was true for the three tested flowable bulk-fill composite materials. For each material, the microhardness values were significantly higher at 0 mm than at 4 mm light tip distance for both the top and bottom surfaces. However, Tetric N flow had the highest microhardness values, with significant differences from SDR and Opus flow only at the bottom sides.

Conflict of interest: None.

Author contributions

AAA and MHA; study conception and design. AAA; data collection. AAA and MHA; Methodology, statistical analysis and interpretation of results. AAA; original draft manuscript preparation. AAA and MHA; Writing - review & editing. Supervision; MHA. Both authors reviewed the results and approved the final version of the manuscript to be published.

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تأثير مسافات المعالجة الضوئية المختلفة على الصلادة الدقيقة للمواد المركبة ذات التعبئة السائبة القابلة للتدفق

طالبة الماجستير اية عاصم الزيدي , استاذ مساعد دكتور منال حسين عبدالله

المستخلص:

الخلفية:الصلادة الدقيقة للراتنج المركب هي معلمة حيوية تستخدم لتحديد جودة المركب. تم استخدام قياس الصلادة الدقيقة للراتنج المركب كطريقة غير مباشرة لتقييم درجة التقارب حيث ثبت أنه أفضل مؤشر لمدى البلمرة . الهدف من الدراسة: تقييم تأثير ثلاث مسافات معالجة ضوئية (0 ، 2 ، و 4 مم) لثلاثة راتنجات مركّبة قابلة للتعبئة بالحشو السائل (**Tetric N, Opus bulkfill Flow , SDR**) على الصلادة الدقيقة لاسطحها العلوية والسفلية. المواد والطرق: تم تصنيع 63 عينة من الأنواع الثلاثة من الراتنجات المركبة (العدد = 21) بقطر داخلي يبلغ 5 مم وعمق 4 مم وتم التصليب لمدة 20 ثانية. لكل مادة ، تم تصنيع ثلاث مجموعات فرعية وفقًا لموضع طرف الضوء من السطح العلوي ؛ على مسافات 0 و 2 و 4 مم. تم قياس الصلابة الدقيقة باستخدام اختبار فيكرز بحمل 50 جم لمدة 15 ثانية للأسطح العلوية والسفلية لجميع العينات. النتائج: تم تسجيل فروق ذات دلالة إحصائية في الصلادة الدقيقة بين الأسطح العلوية والسفلية للعينات عند المسافات الثلاثة للراتنجات الثلاثة المختبرة. **Tetric N**كان له أعلى قيم للصلادة الميكروية. الخلاصة: زيادة المسافات بين طرف المعالجة بالضوء وسطح راتنجات التعبئة السائبة القابلة للتدفق يمكن أن تؤثر بشكل كبير على الصلابة الدقيقة لاسطحها السفلية مقارنة بالاسطح العلوية.