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Evaluation of Surface Roughness and Microhardness of New Generation Bulk-Fill Composites

Zehra Süsgün Yıldırım[1-a], Ezgi Sonkaya[1-b*], Zeliha Gonca Bek Kürklü[1-c]

[1] *Cukurova University, Faculty of Dentistry, Restorative Dentistry Department, 01330, Balcali, Adana, Türkiye.*
**Corresponding author*

Research Article

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ABSTRACT

Objectives: The main objective of the study was to evaluate and to compare the surface roughness and microhardness of three bulk-fill (ACTIVA Bioactive Restorative, SonicFill 2 Single Fill and SDR Flow Plus) and one conventional (G-aenial Posterior) composite resin at different depths.
Materials and Methods: Eighty disc-shaped composite resins (6 mm diameter, 2 mm height) were polymerized as recommended and then subjected to the appropriate finishing and polishing procedure. Transparent strips were placed between the samples before polymerization to evaluate the hardness at different depths (0-2mm and 2-4mm) of the bulk-fill composites. Microhardness was evaluated with a Vickers hardness tester and roughness was evaluated with an optical profilometer. One way ANOVA and Tukey multiple comparisons were performed for the statistical evaluation of microhardness and one way ANOVA was performed for roughness.
Results: No statistically significant difference was determined between the composite materials in respect of surface roughness ($p=0.336$). It was generally observed that as the layer thickness increased in all materials, the hardness values decreased ($p<0.0001$). SF was determined to have higher microhardness values in all the layers compared to the other samples ($p<0.001$).
Conclusions: While SF had the highest microhardness values in different layers, SDR was of equivalent value to a conventional composite. However, the hardness values of ACT in all layers were statistically significantly lower than conventional and other bulk-fillers.
Clinical Relevance: Within the limitation of this study, it may be recommended to use ACTIVA Restorative bulk-fill composite as dentin replacement in 2 mm layers and to cover the top surface with a composite.

Keywords: Bulk-fill composite, roughness, microhardness, profilometer, vickers.

Yeni Nesil Bulk-Fill Kompozitlerin Yüzey Pürüzlülüğü ve Mikro Sertliğinin Değerlendirilmesi

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ÖZ

Amaç: Çalışmanın temel amacı, üç bulk-fill (ACTIVA Bioactive Restorative, SonicFill 2 SingleFill ve SDR Flow Plus) ve bir konvansiyonel (G-aenial Posterior) kompozit reçinenin yüzey pürüzlülüğünü ve farklı derinliklerindeki mikrosertliğini değerlendirmek ve karşılaştırmaktır.
Materyal ve Metot: Seksen adet disk şeklindeki (6 mm çapında, 2 mm yüksekliğinde) kompozit reçine önerilen şekilde polimerize edildikten sonra uygun bitirme ve cilalama prosedürüne tabi tutuldu. Bulk-fill kompozitlerin farklı derinliklerindeki (0-2 mm ve 2-4 mm) sertliği değerlendirmek için polimerizasyondan önce numunelerin arasına şeffaf bantlar yerleştirildi. Mikrosertlik bir Vickers sertlik test cihazı ile ve pürüzlülük bir optik profilometre ile değerlendirildi. Mikrosertliğin istatistiksel değerlendirilmesinde one way ANOVA ve Tukey çoklu karşılaştırmaları, pürüzlülükte ise one way ANOVA uygulandı.
Bulgular: Çalışmada kullanılan kompozit malzemeler arasında yüzey pürüzlülüğü açısından istatistiksel olarak anlamlı fark saptanmadı ($p=0,336$). Genel olarak tüm kompozit reçinelerde tabaka kalınlığı arttıkça sertlik değerlerinin azaldığı gözlendi ($p<0,0001$). SF'nin diğer örneklere göre tüm katmanlarda daha yüksek mikrosertlik değerlerine sahip olduğu belirlendi ($p<0,001$).
Sonuç: SF farklı katmanlarda en yüksek mikrosertlik değerlerine sahipken, SDR sertlik açısından geleneksel bir kompozit ile eşdeğerdi. Bununla birlikte, ACT'nin tüm katmanlarındaki sertlik değerleri geleneksel ve diğer bulk-fillerden istatistiksel olarak anlamlı derecede düşüktü.
Klinik Çıkarım: Bu çalışmanın sınırları dahilinde, ACTIVA Restorative bulk-fill kompozitin dentin replasmanı olarak 2 mm'lik katmanlar halinde uygulanması ve üst yüzeyin bir kompozit ile kaplanması önerilebilir.

Anahtar Kelimeler: Bulk-fill kompozit, pürüzlülük, mikrosertlik, profilometre, vickers.

a susgunzehra@gmail.com https://orcid.org/0000-0002-1717-8214
b sonkayaezgi@yahoo.com https://orcid.org/0000-0001-9773-0955
c goncabek@mynet.com https://orcid.org/0000-0002-4726-7883

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Introduction

In recent years, almost only resin-based composite fillings have been used for restoration purposes in Restorative Dentistry. The clinical placement of these composites requires an incremental layering technique, but this is time-consuming and may lead to gaps between layers.[1] Technological developments have led to the production of bulk-fill resin-based composite materials which can fill cavities up to 4-6 mm in one application.[2]

The most important key to the success of bulk-fill resin-based composite materials is optimum polymerization, because polymerization to the full depth provides proper mechanical and physical properties. The polymerization of the restoration is directly related to the organic and inorganic composition of the material as well as the type and morphology of the filler contents.[3]

SDR Flow Plus is a new generation product of SDR, which has been used successfully since 2009. SDR technology has a patented structure of urethane dimethacrylate (UDMA), which results in less polymerization shrinkage and stretch, but it is recommended that the occlusal part is covered with conventional composite as SDR has low abrasion resistance.[4]

SonicFill is the only bulk-fill composite system with sonic activation, thereby allowing the possibility of placement in the cavity at low viscosity and modeling at a higher viscosity. SonicFill 2 SingleFill is a new product of the SonicFill family. SonicFill 2 has achieved significant improvements in polishability, overall aesthetics, wear resistance, and uptime using a new nanoscale zirconium oxide filler system. It can also be used without the need to coat the occlusal surface with a conventional composite.[5]

Activa, defined as a self-adhesive, dual-cure resin modified glass ionomer, is a mixture of modified polyacrylic acid, diurethane and other methacrylates, and contains 55.4% by weight bioactive glass and sodium fluoride. It is claimed by the manufacturer that Activa is durable, resistant to abrasion and breakage, and does not require an occlusal composite coating.[6]

The aim of this study was to evaluate the surface roughness and microhardness at different depths of Activa Bioactive Restorative, SonicFill 2 SingleFill and SDR Flow Plus, which are new generation bulk-fill composites, compared to G-aenial Posterior as a posterior conventional composite. The null hypothesis of the study was that there would be no difference between the bulk-fill composites compared to a conventional composite in respect of surface roughness and microhardness. It was also hypothesized that there would be no difference in the microhardness of different layer thicknesses of bulk-fill composites.

Materials and Methods

Sample preparation

In the present study, three bulk-fill (Activa Bioactive Restorative, SonicFill 2 SingleFill and SDR Flow Plus) and a conventional (G-aenial Posterior) composite resin materials were used. The properties of the composite resins used are given in Table 1. (Table 1) Composite samples were prepared by titanium molds and transparent polyester strips as described below:

- Disc I (6 mm Ø x 2 mm) top surface cured directly (Disc I: 0 mm – Disc I: 2 mm)
- Disc II (6 mm Ø x 2 mm) has a bottom surface, cured by LED from the top surface of disc I and the distance traveled by the light reaching the bottom surface is 4 mm in total (Disc II: 2mm – Disc II: 4mm).

Polymerization was performed with an LED light source (FreeLight 2, Elipar, 3M ESPE) for the times mentioned as in Table 1. (Table 1) After demoulding, a point was placed on the side edge of the bottom surface with an acetate pen to identify the top and bottom surfaces. The samples were stored dry in the dark for 24 hours before analysis. All the samples were prepared by a single researcher in the same environment at the same time of day.

The composite samples were finished and polished dry and unidirectionally using Sof-Lex (3M ESPE) Al_2O_3 polishing discs for 20 s at 15,000 RPM. Each disc has been used once and were changed according to their grain thickness: Coarse 100 µm, Medium 29 µm, Fine 14 µm, and Super Fine 8 µm, respectively. After each disc, the samples were washed with distilled water and dried with air for 5 s.[7]

Microhardness Measurement

Microhardness measurements were made for different depths of bulk-fill composites. Conventional composites were obtained as 2 mm, and bulk-fill composites were obtained as 4 mm before separating them with transparent strips. The discs produced at 4 mm were divided into 2 mm. Thus, 4 measurements were obtained from a bulk-fill composite sample, 0 - 2mm top and bottom from Disc I, and top and bottom from 2 - 4mm Disc II. A total of 140 measurements were obtained from 70 disc-shaped composite resin samples.

Surface Vickers hardness (VH) was determined using a Vickers hardness tester (Micro Hardness Tester, Wilson, Buehler, USA) and a 100 g load (0.981 N) applied for 15 seconds, slope: 10 gf/s. Three indentations were recorded for each sample at different points of the irradiated top and non-irradiated bottom surfaces. VH values were expressed as N/mm2 (MPa).

Roughness Measurements

Roughness measurements were made only for the top surface of the composite resins. A total of 40 disc-shaped composite resin samples were obtained at 2 mm for conventional and bulk-fill composites. The surface roughness of each specimen was measured using an optical profilometer (Filmetrics, Profilm3D, USA) with white light interferometry. Two- and three-dimensional profiles were obtained from the surfaces with a measurement 250x250 µm field of view and 0.44 µm spatial sampling at x4 magnification. The roughness values (Ra) of each specimen were recorded as µm. Three profiles were obtained from each specimen, and the arithmetic average of them was calculated. The profilometer was calibrated against a

reference block before each measurement. Surface images were obtained with AFM (Atomic Force Microscopes) for each composite sample.

Topographic imaging with Atomic Force Microscopy

Three-dimensional (3D) topographic images of 10 x 10 μm^2 sample surfaces were acquired with an atomic force microscope (AFM; Park Nx10, Suwon, Republic of Korea). The silicon probe tip was used with a scanning rate of 1 Hz and a resolution of 256 x 256 pixels.

Statistical analysis

The study datas were analyzed statistically using SPSS Statistics 21 software (IBM Corpn., Armonk, NY, USA). One-Way Variance analysis (ANOVA) and Tukey multi-comparisons were applied to evaluate microhardness, and One Way ANOVA for the analysis of roughness.

Table 1: Properties of the dental composites used.

Product name	Type	Shade and cure time	Composition	Filler size and content	Manufacturer
G-aenial Posterior	Micro-hybrid composite	A2, 2mm for 20s	UDMA, Silicon dioxide, Fluoro alumino-silicate glass, Composite filler, Pigment, Photo initiator.	>100 nm; Fluoroaluminosilicate, Inorganic filler <100 nm; Fumed silica, Pre-polymerized fillers 16-17 µm; Strontium and lanthanoid fluoride wt/vol %: 77/65	GC, Japan.
ACTIVA BioActive-Restorative	Flowable bulk-fill	A2, 4mm for 20s	Blend of diurethane and other methacrylates with modified polyacrylic acid (44.6%), 1,4-Butanediol dimethacrylate, UDMA, Bis-GMA.	Reactive ionomer glass fillers (Amorphous silica 6.7 %) (Sodium fluoride 0.75 %) 55.4 wt % of bioactive glass and sodium fluoride.	Pulpdent, USA.
SonicFill 2 SingleFill	Sonic-activated flowable and sculptable bulk-fill	A2, 5mm for 20s	Bis-GMA, Bis-EMA, TEGDMA	Ba-B-Al-Si-glass, SiO2 wt/vol %= 83.5/66	Kavo Kerr, USA.
SDR Plus	Flowable bulk-fill base	A2, 4mm for 40s	Modified UDMA, BisEMA, TEGDMA.	Ba-Al-F-B-Si-glass, Sr-AlF-Si-glass 0.02–10.0µm (mean 4.2) wt/vol %= 68/45	Dentsply Sirona, USA.

*UDMA: Urethane dimethacrylate / Bis-GMA: Bisphenol A-glycidyl methacrylate / Bis-EMA: bisphenol A diglycidyl methacrylate ethoxylated / TEGDMA: triethylene glycol dimethacrylate

Results

No statistically significant difference was determined between the materials in respect of top surface roughness (p=0.336). (Table 2) AFM images of the samples are given in Figure 1. (Figure 1)

The hardness values of different composite resins were compared at the same thickness: (Figure 2) The top and bottom surfaces of the samples at 0-2 mm thickness were evaluated, SF>GC>SDR>ACT was determined. SF was statistically different from the other materials (p<0.001), and the difference between GC and ACT was statistically significant (p<0.001).

In the evaluation of the top surface of the samples at 2-4 mm thickness, SF>ACT>SDR was observed, and SF was determined to be statistically significantly different from the other materials (p<0.001).

In the evaluation of the bottom surface of the samples at 2-4 mm thickness, SF>SDR>ACT was observed, and SF was determined to be statistically significantly different from the other materials (p<0.001)

The hardness values of the same material at different layer thicknesses: (Figure 2)

In all the materials, a decrease was seen in the hardness values as the layer thickness increased. A statistically significant increase was seen in hardness between the bottom surface of SF 0-2mm thickness and the top surface of SF 2-4mm (p<0.0001).

When the difference in hardness was evaluated between the layer thicknesses of SF, there was determined to be a statistically significant difference between SF 0-2mm thickness top surface and 2-4mm thickness bottom surface (p<0.0001). There was determined to be a statistically significant difference between SF 0-2 mm thickness bottom surface and 2-4mm thickness top and bottom surfaces (p<0.0001, p=0.019, respectively). The difference between the top and bottom surfaces of SF 2-4 mm thickness was statistically significant (p<0.0001).

When the difference in hardness was evaluated between the layer thicknesses of ACT, there was determined to be a statistically significant difference between the top and bottom surface ACT 0-2 mm thickness and the bottom surface 2-4 mm thickness (p=0.006, p<0.0001, respectively). The difference between the top and bottom surface of ACT 2-4 mm thickness was statistically significant (p<0.0001).

When the difference in hardness was evaluated between the layer thicknesses of SDR, there was determined to be a statistically significant difference between the top surface of SDR 0-2mm thickness and the top and bottom surfaces of 2-4mm thickness (p=0.002, p<0.0001, respectively). The difference between the bottom surface of SDR 0-2 mm thickness and the bottom surface of 2-4 mm thickness was statistically significant (p=0.013).

Table 2: Surface roughness (Ra) of the materials.

	n	Mean	SD
GC	10	.1131	.14182
ACT	10	.1046	.05837
SF	10	.1007	.06138
SDR	10	.0827	.03273
Total	40	.1003	.08168

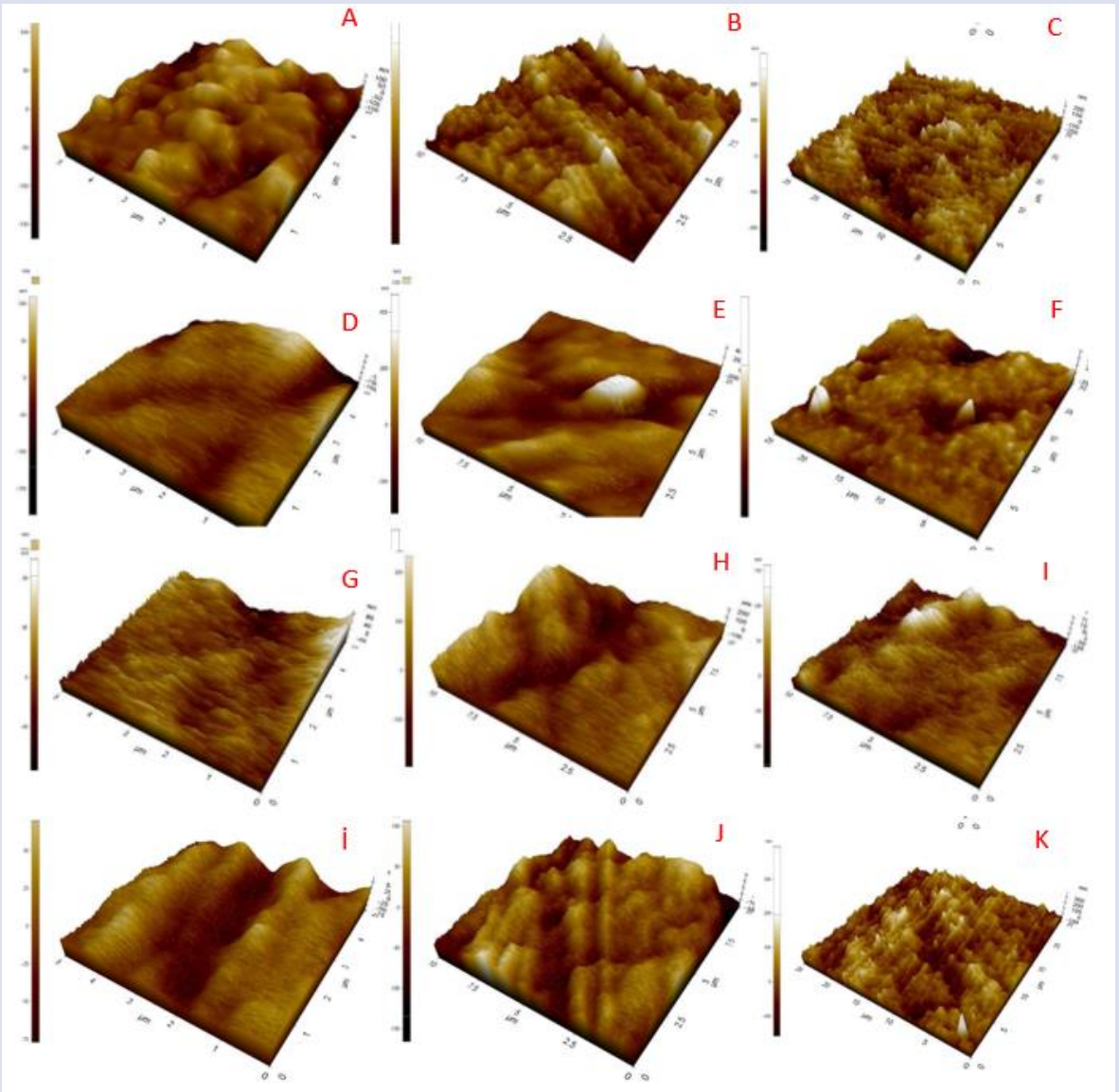

Figure 1. *3D AFM images of (A) GC Top surface 5µm (B) GC Top surface 10µm (C) GC Top surface 25µm (D) ACT Top surface 5µm (E) ACT Top surface 10µm (F) ACT Top surface 25µm (G) SF Top surface 5µm (H) SF Top surface 10µm (I) SF Top surface 25µm (İ) SDR Top surface 5µm (J) SDR Top surface 10µm (K) SDR Top surface 25µm.*

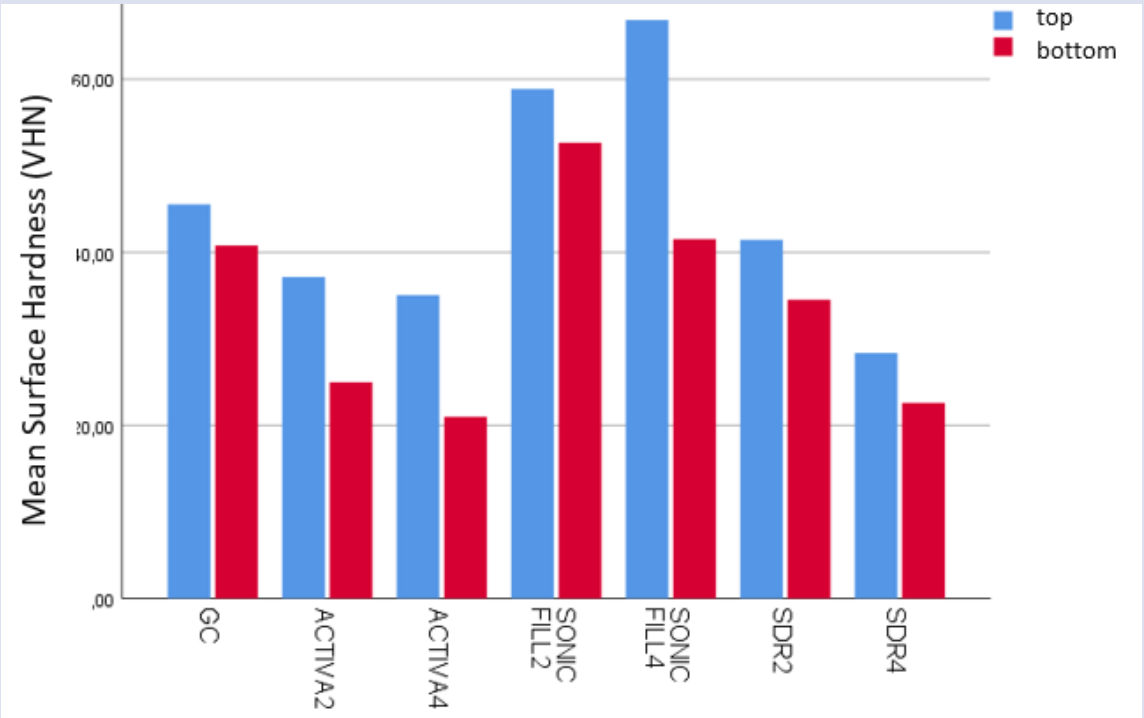

Figure 2. *Top and bottom surface hardness of the materials. VHN – Vickers hardness number.*

Discussion

The use of bulk-fill composite resins, which can be applied as a large mass with low technique sensitivity, has become more widespread among dental practitioners, especially for large cavities, because of advantages such as the reduced clinical working time. However, the surface hardness and roughness of bulk-fill composites applied to posterior teeth must be within acceptable clinical limits.[8] According to the results of this study, which aimed to investigate this situation, the surface roughness values of bulk-fill and conventional resins were similar, and SF was seen to have higher microhardness values at different layer thicknesses than the other bulk-fill and conventional composites. Therefore, the null hypothesis of the study was partially rejected.

The Vickers microhardness (VH) test device was selected for use in this study as it requires a minimum area on the sample surface for the test and the method is simple and rapid results are provided.[9-11] The size of the Vickers hardness indentation is larger than the size of the filler particles in the material complex, so in the calculation the VH includes not only the filler component but also the surrounding softer resin matrix. In this context, VH indirectly considers cross-linking of the entire matrix network.[12, 13]

Profilometers and AFM have become the most preferred methods for the measurement of surface roughness of dental materials as they do not cause deformation of the sample surface and the results are highly accurate.[14] In the literature related to the measurement of the surface roughness of bulk-fill composites, there has been reported to be no difference between SDR and Sonic Fill in non-polished samples of different bulk-fill composites, and they have shown the lowest mean surface roughness compared to other bulk fillers.[15] In a review evaluating surface roughness, 32 of 43 studies compared microhybrid/nanohybrid composites with other composite types such as suprananofill, nanofill, microfill, bulk-fill, and flow. In 14 of the 32 studies, the smoothest or at least one smooth surface was obtained in the microhybrid/nanohybrid samples of all the tested materials.[16] In this study, no difference was found between GC posterior, a microhybrid composite, and all the other materials in respect of surface roughness following a polishing procedure.

In studies that have applied bulk-fill and conventional composites to cavities 4mm in depth, higher bonding strength and microhardness results have been obtained with bulk-fill composites.[17, 18] The reasons for this include the better light permeability due to the higher translucency of bulk-fill composites, and the variations in filler particles in the organic and inorganic matrixes such as increased molecular weight of the monomers, the addition of new stress-relieving monomers, the addition of pre-polymer particles and fiberglass rod segments, and the presence of plasticizing monomers, alternative and more reactive photo initiators, and polymerization modulators. [17,19,20] It has been suggested that due to all these modifications, they have less polymerization shrinkage and a higher degree of conversion.[21] According to the results of the current study, Sonic Fill had a statistically significantly higher level of microhardness than conventional G-aenial posterior composite. Sonic technology is recommended to facilitate the bulk

placement of the resin up to 5mm and the better spread to the cavity by increasing the flow of the resin.[22, 23]

In a study that compared the effect on microhardness of sonic and incremental composite placement, the bond strength and microhardness values obtained from SonicFill resin were higher than from the other bulk-fill resins and showed similar results to the conventional resins placed with the incremental technique. Sonic Fill flow is optimized and can penetrate the cavity better because of the converters reacting to sonic energy and the barium glass and silicone dioxide in the inorganic particles. [22, 23] Sonic energies have been shown to reduce the viscosity of the material by up to 87% with the technology. This means that by using SF in clinical practice, the time to construct a restoration is shortened by up to 30%.[24] The amount of filler particles of this material is said to be 83.5% by the manufacturer. This high rate could be responsible for minimizing the stress caused by polymerization shrinkage of the material. [25] All these above-mentioned modifications explain the higher hardness value obtained in the SonicFill measurements in all the layers compared to the other composites in the current study.

Although clinical procedures are simplified and working time is shortened by materials which allow single increment application, the problem of low microhardness may be encountered. [24] There are studies in literature showing that lower microhardness values have been obtained with bulk-fill resins compared to conventional resins applied with an incremental technique. [20, 26] Similarly in another study, the microhardness of bulk-fill filling materials with high viscosity was found to be lower than the microhardness of conventional composites. The surface microhardness of the experimental groups of low viscosity materials was found to be even lower. [27] In the current study, when the hardness values of the top surface were evaluated, the conventional G-aenial posterior composite was seen to have higher microhardness values than SDR and ACT. That this result is expected from conventional composite can be attributed to the increasing polymerization associated with increased light dispersion of the greater number of particle/resin matrix interfaces because of the high filler content because of 2mm application as recommended by the manufacturer. [28]

SDR is a fluid material with modified monomers and a relatively low amount of filler content (68% by weight). According to the manufacturer's information, as the low inorganic particle content decreases the organic matrix-filler particle interface contact, the depth of polymerization is increased by absorbing lighter during polymerization of the material. Moreover, low viscosity bulk-fill SDR includes a photoactive group embedded in urethane-based methacrylate monomers, which can enter a reaction with camphorquinone. The manufacturer claims that such an interaction helps to modulate the hardening reaction, and because of the higher flexibility of urethane groups, polymerization is reduced, and a more homogenous polymer structure is formed. [29, 30] The results of the current study are consistent with this view that these modifications of SDR have surface roughness and hardness which may not show a statistically significant difference from those of conventional composites.

When the microhardness of the top layer was evaluated in the current study, ACT was determined to have the lowest microhardness. Low viscosity SDR, which currently has an indication for use as a base liner, was seen to have a higher hardness value than ACT, even on the lowest surface. Despite being a bioactive bulk-fill composite, ACT is not a recommended material for any additional occlusal coverage, and the low filler ratio content (56% by weight) can cause low hardness values. In a study which examined the Knoop Hardness values of bulk-fill composites with 5 different properties, the bioactive flowable composite ACT was seen to have the lowest hardness values. In the same study, no statistical difference was determined between the hardness values in different layers of ACT placed in bulk. It can be seen that the material not being affected by the increased thickness has undergone a chemical polymerization reaction caused by the glass ionomer components. [31] In another study which compared bulk-fill resins, conventional glass ionomer, and ACT before and after thermocycles, ACT showed mechanical properties (diametral tensile strength, flexural strength) comparable to those of bulk-fill resin composites, and lower hardness values. [32] The hardness of a material with a high blend of diurethane and other methacrylate with modified polyacrylic acid (44.6%) content and a low inorganic particle amount may be reduced compared to conventional resin composites. From these results, which are consistent with the findings of the current study, ACT, which is a low viscosity material, can be recommended as a liner for clinical dentin replacement.

To be able to accept that a composite resin has been sufficiently cured, generally the arbitrary minimum value of the bottom-to-top surface hardness ratio should be in the range of 0.8-0.85. At 0-2mm thickness, other than ACT, the bottom-to-top surface hardness ratio of the other composites was above this value and at 0-4mm thickness, it was below this value for all the bulk-fill composites.[33] However, in both conditions, ACT had the lowest cure depth values.

In a study which compared the microhardness and degree of conversion of bulk-fill composites, SDR, which was within the bulk-fill composites, was seen to have uniform conversion values throughout the 4 mm depth of the restorations. [24] The microhardness of high and low viscosity bulk-fill and conventional composites tested at 4 mm showed no different from the top surface values in another study. [27] In the results of the current study, there was seen to be a significant decrease in microhardness between the top and bottom layers in all the bulk-fill composites other than SF. Composite type and differences in cure depth, and even the general degree of conversion may be due to the viscosity of composites in uncured form. Composite viscosity is affected by the monomer component and filler particle content, and the

reaction kinetics and ultimately the degree of conversion is an important parameter as this parameter is affected by the activity of reactive species.[3]

In vitro situations and no aging process can be evaluated as a limitation of this study. In addition, that the method of obtaining different layer thicknesses was the application of a metal mold allowing the placement of a second 2 mm layer by separating with a clear band after the placement of the first 2 mm layer could also constitute a limitation of the study. Even if it is attempted to perform the application in the shortest possible time, the interface common to the 0-2mm and 2-4mm layers could be affected by the light. That the 2-4mm top surface was harder than the 0-2mm bottom surface in the SF group could have been a result of this.

Conclusions

The results of this study evaluating the surface properties of bulk-fill composites compared with a conventional composite demonstrated no difference between the materials in respect of roughness. When the hardness values were examined, SonicFill had the highest values in all different layers. SDR showed similar results to a conventional composite and ACT had the lowest microhardness values. Moreover, ACT did not have a sufficient depth of cure at both 0-2mm and 2-4mm thicknesses.

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