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ORIGINAL ARTICLE

Comparison of the microhardness of low-viscosity bulk-fill composite resins

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Abstract

The aim of this study is to compare the microhardness of five different low-viscosity bulk-fill composites. The bulk-fill composites used in the study; Estelite Bulk-fill flow (EBF), Filtek Bulk-fill (FBF), SureFil SDR flow (SDR), Tetric EvoFlow Bulk fill (TEFBF), X-tra Base (XBF). Cylindrical molds with a diameter of 5 mm and a height of 4 mm were used for the Vicker's Micro Hardness (VMH) test. Bulk-fill composite resins were placed in these molds at once. The polymerization of the composites was achieved for 20 seconds with the LED light curing. A total of 50 composite discs were prepared (n=10). Then the microhardness of the top and bottom surfaces was measured using MVK-H1 Microhardness Tester (Akashi Co, Tokyo, Japan. Depth of polymerization of each sample was recorded. Data were analyzed by using the *Kruskal Wallis* H and *Mann Whitney-U* tests. The bottom and top surface hardness values of the XBF composite samples (43.82±0.95 MPa, 47.87±0.59 MPa) were statistically significantly higher than the other bulk-fill composite samples (p<0.001). The bottom and top surface hardness values of the FBF composite (27.85±0.56 MPa, 22.05±1.40 MPa) were found to be statistically lower than other bulk-fill composite samples (p<0.001). Among the low-viscosity bulk-fill composites used in the study, except for FBF, the VMH values of the others were found to be above 0.80, and it was observed that they reached sufficient microhardness.

Keywords: Bulk fill composite resin, microhardness test, polymerization, viscosity

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Introduction

Composite resins with better structure can be obtained by improving the filler morphology in dental composite resins, progress in current dimethacrylate chemistry, and new monomer technologies [1]. In addition, in recent years, further simplification of the use of composite resins has been on the agenda, and clinicians want to perform high-quality clinical applications and shorten the procedure time [2].

The aim of the layering technique, which is the most common method used to place composite resins in the cavity, is to achieve the ideal polymerization conversion degree and sufficient hardness in composite resins [3]. However, this technique is clinically time consuming and has some disadvantages such as risk of contamination, loss of bond between composite resin layers, and formation of voids [4]. To solve these problems, new types of composite resins with the potential to polymerize in increasing thickness up to 4, 5 and 6 mm have been introduced to the dental market as "Bulk-fill" composite resins. In these composite resins, the focus is on transparent structure, alternative organic matrix, different initiators and various filler technologies [4,5]. Therefore, bulk-fill composite resins have potential benefits such as facilitating clinical applications and saving time. In addition, the bulk-fill placement technique prevents the formation of gaps and contamination between composite layers, and thus more compact restorations can be made [6]. By increasing the transparency of bulk-fill composite resins, they have higher light transmittance, which enables successful restorations [7]. Manufacturers have tried to increase the depth of polymerization in bulk-fill composites by various methods such as reducing the amount of filler, increasing the filler particle size, and using additional photoinitiators [8].

Today, bulk-fill composites are available in the market in two different forms: high and low viscosity. In high viscosity bulk-fill composites, the cavity can be completely restored with a single type of bulk-fill composite resin without the need for an additional covering layer. For low-viscosity bulk-fill composite resins, after being placed in the cavity, the restoration is completed by covering it with a 1.5-2 mm thick conventional composite [9]. Although the mentioned advantage of not needing the layering method is limited in this case, low viscosity bulk-fill composites do not require condensation. Thus, the application time is shortened and its compatibility with methacrylate-based composites makes its use widespread [7]. It has also been determined that the polymerization stresses of low-viscosity bulk-fill composites are lower than those of a traditional dimethacrylate composite resin [10]. Smart Dentin Replacement (SDR) composite is the first bulk-fill composite produced. However, since this bulk-fill composite is a material with low mechanical resistance to abrasion, it has been stated that the top layer should be finished with a conventional composite resin during the restoration. However, by adding high molecular weight monomers to the SDR bulk-fill composite, its content was renewed [8]. The polymerization depth determined by the ISO 4049 method for bulk-fill composites was found to be higher than expected. Instead, it is recommended to use Vickers microhardness measurements at the surface and at certain depths to determine the depth of polymerization [11,12]. Additionally, microhardness data for a given material provides information about its wear, polishability, and abrasive effect on antagonist teeth [13]. In addition, surface hardness measurement in composite resins can be used both to indirectly determine the degree of polymerization conversion and to measure the hardening depth of the composite resin [14]. Studies support the clinical use of bulk-fill composites, but further research on the mechanical properties of these composites is required [15,16]. The aim of this study is to compare the microhardness of low viscosity bulk-fill composites. The hypothesis of this study is that there is no difference in the microhardness of the low viscosity bulk-fill composites used.

Materials and Methods

Preparation of Samples

In this *in vitro* study, five different low viscosity bulk-fill composites were used: Estelite Bulk-fill flow (EBF, Tokuyama Dental Corp, Ibaraki,

Japan), Filtek Bulk-fill flow (FBF, 3M Espe, St.Paul, USA), SureFil SDR flow (SDR; Dentsply DeTrey, USA), Tetric Evo Flow Bulk-fill (TEFBF, Ivoclar Vivadent Schaan, Liechtenstein), X-tra Base (XTB, Voco, GmbH, Cuxhaven, Germany) were used. The materials used in the study and their contents are given in Table 1. Cylindrical molds with a diameter of 5 mm and a height of 4 mm made of polytetrafluoroethylene were used to prepare the samples [17]. Transparent tapes were placed on the upper and lower surfaces of these molds. Sample size was calculated as 50 with a power of 0.80 (effect size=0.53 and α=0.05). A total of 50 samples, 10 in each group, were prepared for the measurements to be made on the VMH device (n = 10). Before the polymerization of the samples, the power of the light device was checked using a radiometer (Bluephase Meter, Ivoclar Vivadent). The samples prepared for the VMH test were polymerized for 20 seconds using an Elipar Freelight (3M-ESPE Seefeld, Germany) LED light source (480 nm wavelength and 1200 mW/cm2). During the polymerization process, the tip of the light device was used perpendicularly and in contact with the samples. They were kept in incubator with 100% humidity at 37 °C for 24 hours.

Vicker's Microhardness Test

The samples were polished with SiC sandpaper (#1200) for 5 seconds to remove the outer resin layer and to obtain a standardised and stable surface. Then the VMH measurement of the samples was started.

For the VMH test, a 200 g load was applied to the samples for 10 seconds using a Vickers microhardness tester (MVK-H1, Akashi Co, Tokyo, Japan) [18]. Six measurements were recorded on both sides of each sample and averaged for the statistical analysis. (Figure 1 and 2) Hardness rate of each sample;

VMHmean=VMHbottom surface/VMHtop surface was determined by the formula.

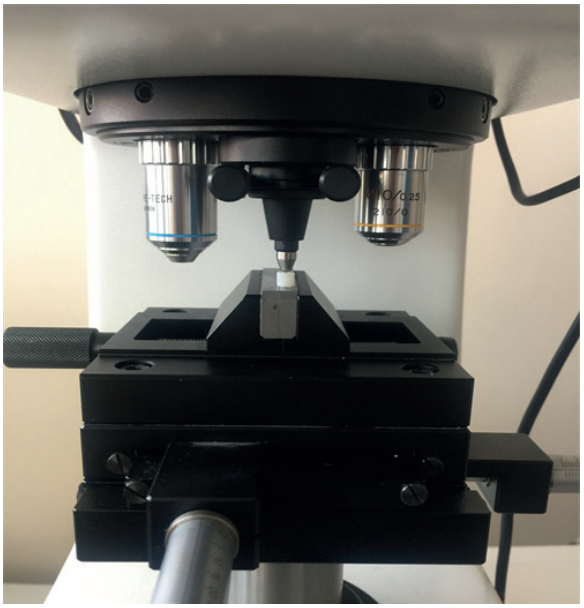

Figure 1. Vickers Microhardness Tester.

Table 1. Composite resins used in the study, manufacturer companies, matrix and filler types, filler amount.

Material name	Manufacturer	Organic matrix type	Filler type	Filler ratio (% weight/ % volume)
Estelite Bulk-fill Flow (EBF)	Tokuyama Dental Corp, Ibaraki, Japan	Bis-GMA, Bis- MPEPP, TEGDMA,	Suprananospherical filler Silica, Zirconia, Ytterbiumtrifluoride.	% 70/56
Filtek Bulk-fill Flow (FBF)	3M Espe, St.Paul, USA	UDMA, BISGMA, Bis-EMA, Procrylat resin	0.01 to 5 µm—based on silica, zirconia and ytterbiumtrifluoride	% 64,5/42,5
SureFil SDR flow (SDR)	Dentsply DeTrey, USA	Modifiye UDMA, TEGDMA,EBPADMA	Ba-B-F-Al silikat cam SiO2, amorföz Sr-Al silikat cam	% 68/44
Tetric EvoFlow Bulk-fill (TEFBF)	Ivoclar Vivadent Schaan, Liechtenstein	Dimethacrylates Copolymers	Barium glass Ytterbium trifluoride	% 68/46
X-tra Base Bulk-fill (XBF)	Voco GmbH Cuxhaven, Germany	MMA, Bis-EMA Aliphatic di-methacrylate (UDMA	Barium glass ceramic, fumed silica	% 75/58

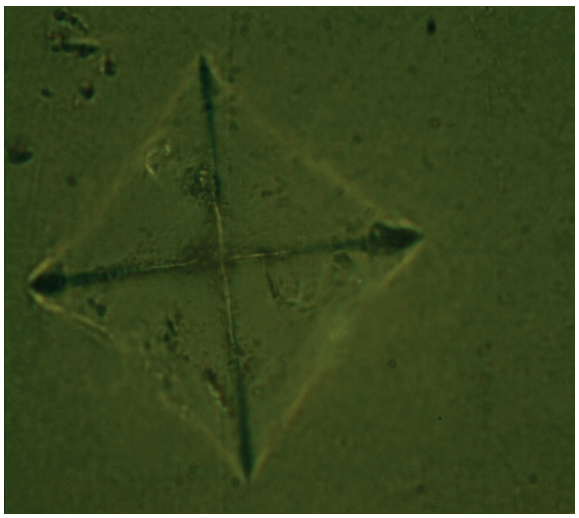

Figure 2. The image formed in the vickers hardness test.

Ethics Committee Approval

This study protocol was carried out in accordance with the relevant guidelines of the Principles of the Declaration of Helsinki and was approved by the Clinical Research Ethics Committee of Afyonkarahisar Health Sciences University report numbered 2023/2.

Statistical Analysis

Statistical evaluations of the obtained data were made using SPSS23.0 (IBM SPSS Statistics, Armonk, USA). The upper and lower microhardness values obtained from the samples were analysed by Kruskal wallis H-test according to the normality of the data and multiple comparisons were made by Mann Whitney-U test. Significance level was set at $p < 0.05$.

Results

The bottom and top surface hardnesses and average hardness rates of the composite samples are presented in Table 2 and Table 3. The bottom and top surface hardness values of the XBF composite samples (43.82±0.95 MPa, 47.87±0.59 MPa) were statistically significantly higher than the other bulk-fill composite samples (p<0.001). The bottom and top surface hardness values of the FBF composite (27.85±0.56 MPa, 22.05±1.40 MPa) were found to be statistically lower than other bulk-fill composite samples (p<0.001). There was a statistically significant difference between the top and bottom surface hardness values in all bulk-fill composite groups. XBF (0.91±0.01HR) composite had no difference between SDR (0.89±0.03 HR) (p=.059), while it had statistically higher VMH rates compared to the other composites (p<0.05).

Discussion

In this study, VMH values of five different low viscosity bulk-fill composites were tested. Among the tested materials, FBF composite was found to have the lowest VMH value. According to these results, the null hypothesis was rejected. In the VMH test of a composite resin, the ratio

Table 2. Upper and lower surface hardness values of composite samples and mean and standard deviations of hardness ratios.

	Top surface vickers microhardness value (MPa)	Bottom surface vickers microhardness value (MPa)	*p*
Estelite Bulk-fill Flow (EBF)	39,93±1,47^{ad}	35,6±1,15^{a}	0,001
Filtek Bulk-fill Flow (FBF)	27,85±0,56^{b}	22,05±1,40^{b}	0,001
SureFil SDR Flow (SDR)	39±1,51^{a}	37,72±0,65^{a}	0,001
Tetric EvoFlow Bulk-fill (TEFBF)	40,85±0,55^{d}	33,25±0,72^{c}	0,001
X-tra Base Bulk-fill (XBF)	47,87±0,59^{c}	43,82±0,95^{d}	0,001

Values with the same letters in the same column were not statistically different. (p=0.05)

of the lower surface hardness value to the upper surface hardness value gives the hardness rate of the composite resin. Polymerization of the composite resin is considered sufficient when this hardness rate is at least 80%. When this ratio is 1, it means that the polymerization of the composite resin is complete [19,20]. The inorganic content of composite resins is responsible for mechanical and physical properties [21]. It is stated that there is a proportion between the inorganic content of composite resins and their hardness values. For this reason, it has been shown that composite resins have different hardness rates due to the difference in inorganic and matrix content [22]. In this study, the microhardness values of the FBF composite with a low filler ratio (64.5% by weight) were found to be lower than the other composites included in the study. The filler content of the XTB (75% by weight) composite was higher and the microhardness values were found to be higher than other bulk-fill composites. In a study, it was found that the decrease in the filler amount of composite resins caused the surface hardness of the composite to decrease [23]. The low bottom and top surface hardness values of the FBF composite can also be attributed to the presence of zirconium particles in the filler structure. It has been stated that zirconium negatively affects polymerization by reducing light transmittance due to its high refractive index [24]. In a study by Besegato et al. [25], they found that FBF composite exhibited mechanical behavior that could compromise the quality and longevity of the restoration. In XTB and SDR bulk-fill composites, the manufacturer reduced the amount of filler and increased the filler size. As a result, by reducing the surface area between the filler content and the matrix, light scattering was reduced and the composite was enabled to reach sufficient hardness after polymerization [7]. SDR is the first bulk-fill composite produced. The modified UDMA resin in its structure contains a photoactive group. This content allows deeper penetration of light during the polymerization of the SDR composite and reaches a higher microhardness rate [26]. In our study, the VMH value of the SDR bulk-fill composite was found to be 89% and reached sufficient microhardness. Aggarwal et al. [27] found the VMH rate in SDR bulk-fill composite samples to be over 80%, in parallel with the current study. In a study investigating the polymerization depths of traditional flowable composites and "bulk-fill" composites, the microhardness ratio in SDR samples was found to be over 80% [28]. TEFBF contains a translucent filler. This allows light to pass through the material during its polymerisation. [27] In addition, this composite contains Ivocerin (a photoinitiator based on dibenzoyl germanium). According to the manufacturer, it has higher photocuring activity than camphorquinone due to its higher absorption in the region between 400 nm and 450 nm. It can be used without the addition of an amine as a co-initiator. It forms at least two radicals capable of initiating radical polymerisation; therefore, it is more effective than the camphorquinone/amine system. [29-31] In our study, the TEFBF microhardness ratio (0.81) was found to be above 80% and it can be said that this bulk-fill composite has sufficient microhardness. In a study by Sousa-Lima et

al.[32] the microhardness ratios of TEFBF and a conventional composite were analysed. The results of the study were found to be compatible with the results of our study. Differences in results in studies examining the microhardness of bulk-fill composite resins may be due to the use of different light devices or the molds used being made of different materials [11]. In the research conducted, instead of using metal molds for samples to be prepared from composite resin, polymethylmethacrylate molds were used, which have low light absorption and allow light to easily reach the lower surface without decreasing its intensity [33,34].

Conclusion

Considering the limitations of the current study, it can be said that EBF, SDR, TEFBF and XTB bulk-fill composite resins with VMH values above 0.80 have reached sufficient microhardness, thus these bulk-fill composite resins show sufficient polymerization depth for clinical use. For FBF composite resin, the VMH value was found to be below 0.80. In addition, the bottom and top surface hardness values of FBF composite resin were found to be significantly lower than other bulk-fill composite resins.

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Conflict of interest

There is no conflict of interest between the authors of this study.

Data availability statement

Data can be requested from the authors.

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