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ORIGINAL ARTICLE

In vitro comparison of mechanical properties and degree of cure of bulk fill composites

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Abstract
Objectives The aim of our study was to measure and compare degree of conversion (DC) as well as micro- (indentation modulus, *E*; Vickers hardness, HV) and macromechanical properties (flexural strength, σ; flexural modulus, $E_{flexural}$) of two recently launched bulk fill resin-based composites (RBCs): Surefil® SDR™ flow (SF) and Venus® bulk fill (VB).
Materials and methods DC (n=6) was investigated by Fourier transform infrared spectroscopy (FTIR) in clinical relevant filling depths (0.1, 2, and 4 mm; 6 mm bulk, 6 mm incremental) and irradiation times (10, 20, 40 s). Micro- (n=6) and macromechanical (n=20) properties were measured by an automatic microhardness indenter and a three-point bending test device after storing the specimens in distilled water for 24 h at 37°C. Furthermore, on the 6-mm bulk samples, the depth of cure was determined. A field emission scanning electron microscope was used to assess filler size. Results were evaluated using one-way analysis of variance, Tukey's honest significance test post hoc test, a multivariate analysis (α=0.05) and an independent *t* test. Weibull analysis was used to assess σ.
Results VB showed, in all depth, significant higher DC (VB, 62.4–67.4 %; SF, 57.1–61.9 %), but significant lower macro- (VB, $E_{flexural}$=3.6 GPa; σ=122.7 MPa; SF, $E_{flexural}$=5.0 GPa; σ=131.8 MPa) and micromechanical properties (VB, E=7.3–8.8 GPa, HV=40.7–46.5 N/mm²; SF, E=10.6–12.2 GPa, HV=55.1–61.1 N/mm²). Both RBCs showed high reliability (VB, m=21.6; SF, m=26.7) and a depth of cure of at least 6 mm at all polymerization times. The factor "RBC" showed the strongest influence on the measured properties (η^2=0.35–0.80) followed by "measuring depth" (η^2=0.10–0.46) and "polymerization time" (η^2=0.03–0.12).
Conclusions Significant differences between both RBCs were found for DC, E, σ, and $E_{flexural}$ at all irradiation times and measuring depths.
Clinical relevance Curing the RBCs in 4-mm bulks for 20 s can be recommended.

Keywords Bulk fill · Composite · Macromechanical properties · Micromechanical properties · Degree of conversion

Introduction

Since the development of resin-based composites (RBCs), several improvements in their chemical composition as well as various filler reinforcements occurred, leading to a large category of materials [1]. Recently, a new category of flowable RBCs—so-called bulk fill RBCs—was introduced (Surefil® SDR™ flow, Dentsply, Caulk, USA and Venus® bulk fill, Heraeus Kulzer GmbH, Hanau, Germany) as bulk fill material and as liner in class I and II restorations. The particularity of the new material category is stated to be the option to place it in 4 mm thick bulks instead of the current incremental placement technique, without negatively affecting polymerization shrinkage, cavity adaptation or the degree of conversion (DC). Moreover, manufacturers stated that the polymerization shrinkage of those materials is even lower when compared to commonly used flowable and conventional RBCs [2]. Thus, problems related to polymerization shrinkage [3] like gap formation causing secondary caries due to bacteria colonization [4, 5], pulp irritation, post-operative sensibility when chewing [6], or cusp deflection when the "C" factor is high [7, 8] could be minimized. Manufacturers claimed that bulk fill

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materials can achieve a depth of cure of 6 mm [2], though no published investigations are available till now to confirm these statements. Nevertheless, the idea of placing a self-adapting material as bulk, saving time as well as improving material handling, is of great interest.

The bulk fill material Surefil® SDR™ (Smart Dentin Replacement, shrinkage decreased resin) flow contains a polymerization modulator, chemically embedded in the center of the polymerizable resin backbone of the SDR™ monomer, to lower polymerization shrinkage. The modulator has a high molecular weight. Due to the conformational flexibility around the centered modulator impart, the modulator is supposed to optimize flexibility and network structure of the SDR™ resin [9]. Investigations on RBCs with SDR™ technology showed significant lower shrinkage stress values [10] not only when compared to regular flowable RBCs, but also to nano- and hybrid RBCs or even to silorane-based composites [11]. De Biasi et al. investigated microhardness and raised concerns about its practical use due to its low Vickers hardness (HV) [12]. This was also confirmed by Ilie et al. [11] where Surefil® SDR™ flow showed the lowest surface hardness when compared to other commonly used RBCs (EsthetX Flow, Filtek Supreme Plus Flow, EsthetX Plus, Filtek Silorane, and Filtek Supreme Plus). However, when compared to the investigated flowable RBC of the same study, Surefil® SDR™ flow showed significant higher indentation modulus (E). In view of wear, surface roughness, gloss, color stability, and stain resistance, similar results to clinically successful RBCs were found [10]. Other experimental flowable RBCs with SDR™ technology—P&P Adaptable and P&P Universal (both Dentsply DeTrey)—also showed low shrinkage stress values [13]. Moreover, Surefil® SDR™ flow was used for luting fiber posts and resulted comparable regarding retentive strength like a dual resin cement commonly used [14].

This study evaluated and compared two bulk fill RBCs—Surefil® SDR™ flow and Venus® bulk fill—regarding their micro- and macromechanical properties and DC at different irradiation times and by simulating clinical relevant filling depth.

The tested null hypothesis were that: (a) there would be no significant difference between the two materials in view of macro- (flexural strength (σ), modulus of elasticity (E_{flexural})) and micromechanical properties Vickers hardness (HV) and indentation modulus (E) and degree of cure (DC) at any measured depth and irradiation time; (b) within one material, irradiation time and depth would not influence the measured properties.

Materials and methods

Two flowable bulk fill RBCs—Surefil® SDR™ flow (Dentsply, Caulk, USA, lot no.: 100407, 100507) and Venus® bulk fill (Heraeus Kulzer GmbH, Hanau, Germany, lot no.: 010026) were analyzed assessing DC and micromechanical properties (HV and E) as function of depth and polymerization time (10, 20, or 40 s) as well as the macromechanical properties (σ, E_{flexural}). According to manufacturers' information, Surefil® SDR™ flow consists of Ba–Al–F–B–Si–glass and St–Al–F–Si–glass as fillers (68 % per weight, 44 % per volume) and modified urethane dimethacrylate (UDMA), triethyleneglycol dimethacrylate (TEGDMA), and ethoxylated bisphenol-A-dimethacrylate (EBPDMA) as resin matrix. For Venus® bulk fill, Ba–Al–F–Si–glass and SiO_2 were given as fillers (65 % per weight, 38 % per volume) and UDMA and EBPDMA as resin matrix.

Degree of cure measurements

To evaluate DC, five different sample geometries were considered. Thin films (100 μm) as well as 2-, 4-, and 6-mm high molds (3 mm diameter) were filled in bulk. Additionally, three consecutive increments—each 2 mm high—were prepared in the mold of 6-mm height (6-mm incremental). Samples were cured by applying the curing unit (Elipar Freelight 2, 3 M ESPE, 1,226 mW/cm²) directly on the top of the particular mold, respectively, on the film surface covered by a transparent matrix strip. For each product, irradiation time (10, 20, and 40 s) and geometry (0.1, 2, 4 mm; 6-mm bulk; 6-mm increment) six samples were measured (n=6). Real-time measurements were made with a Fourier transform infrared (FTIR) spectrometer with an attenuated total reflectance (ATR) accessory (Nexus, Thermo Nicolet, Madison, USA). Therefore, the nonpolymerized RBC paste was put directly on the diamond ATR crystal in the mold as described above. FTIR spectra were recorded in real time for 5 min at the bottom of the samples irradiated according to the curing protocol presented above. Diameter of measured surface was 800 μm, wave number of the spectrum ranged between 4,000–650 cm^{-1} and the FTIR spectra were recorded with four scans at a resolution of 8 cm^{-1}.

To determine the percentage of the remaining unreacted double bonds, the DC was measured by assessing the variation in peak height ratio of the absorbance intensities of methacrylate carbon double bond peak at 1,634 cm^{-1} and that of an internal standard (IS) peak at 1,608 cm^{-1} (aromatic carbon double bond) during polymerization, in relation to the uncured material. For the RBC Surefil® SDR™ flow, the reference peak was set at 1,600 cm^{-1} due to the absence of the aromatic carbon bond.

$$DC_{\text{height}}\% = \left[1 - \frac{\left(^{1,634\text{cm}^{-1}}/\text{IS}\right)\text{peak afte rcuring}}{\left(^{1,634\text{cm}^{-1}}/\text{IS}\right)\text{peak before curing}}\right] \times 100$$

Micromechanical properties

The variation in micromechanical properties (HV and *E*) was assessed on the 6-mm bulk samples prepared for the DC measurements. For this purpose, samples were stored in distilled water after curing for 24 h at 37°C, ground and polished under water in longitudinal direction from 3 mm diameter to 1.5 mm diameter with diamond abrasive paper (mean grain sizes: 20, 13, 6 μm) in a grinding system (EXAKT 400CS, Exakt, Norderstedt, Germany). Measurements were made with an automatic microhardness indenter (Fischerscope H100C, Fischer, Sindelfingen, Germany) starting from 0.1 mm under the surface, with 100 μm intervals between the measuring points. The test procedure was carried out force-controlled, where the test load increased and decreased with constant speed between 0.4 and 500 mN. Load and penetration depth of indenter (Vickers pyramid: diamond right pyramid with a square base and an angle of α=136° between the opposite faces at the vertex) were continuously measured during the load–unload hysteresis. Universal hardness is defined as the test force divided by the apparent area of indentation under the applied test force. From a multiplicity of measurements stored in a database supplied by the manufacturer, a conversion factor (0.0945) between universal hardness and HV was calculated by the manufacturer and entered into the software, so that the measurement results were indicated in the more familiar HV units. *E* was calculated from the slope of the tangent adapted at the beginning (at maximum force) of the nonlinear indentation depth curve upon unloading.

HV and *E* variations with depth and irradiation time were calculated for each product (Tables 3 and 4) based on data from six samples (360 measuring points). The depth of cure, usually acknowledged as the thickness of a RBC that is adequately cured [15] or rather as the depth where HV equals the surface value multiplied by an arbitrary ratio, usually 0.8 (HV-80 %) [16], was calculated. Therefore for each sample HV in the depth was compared to the related surface value and noted when it became less than 80 % (HV-80 %).

Flexural strength and flexural modulus

Determined in a three-point bending test according to ISO/DIN 4049:1998 was σ. The samples (n=20) were made by compressing the RBC material between two glass plates with intermediate polyacetate sheets, separated by a steel mold having an internal dimension of 2×2×16 mm. After curing (with three light exposures of 20 s per side, Elipar Freelight2, 3 M ESPE), the specimens were removed from the mold and any flash material was trimmed away with sandpaper (grit size P4000 (FEPA)). All specimens were then stored in distilled water at 37°C prior to testing for 24 h. Samples were loaded until failure in the universal testing machine (MCE 2000ST, quick test Prüfpartner GmbH, Langenfeld, Germany). The crosshead speed was 0.5 mm/min. The specimens were placed on a three-point bending test device, which is constructed according to the guidelines of NIST no. 4877 with 12 mm distance between the supports. During testing, the specimens were immersed in distilled water at room temperature.

Flexural strength was calculated from formula (1).

$$\sigma = \frac{3Fl}{2bh^2} \tag{1}$$

F is the maximum load (Newton), l is the distance between the supports (millimeter), b is the width of the specimen (millimeter), and h is the height of the specimen (millimeter).

The universal testing machine stored the force during bending and the deflection of the beam in a file. The bending modulus was calculated from formula (2).

$$E_{\text{flexural}} = \frac{\text{Fl}^3}{4\text{bh}^3 y} \tag{2}$$

y is the deflection at load point [mm].

Field emission scanning electron microscope

For each product, one specimen (1×1×0.5 cm) was manufactured with an irradiation time of 60 s and treated for 1 h in a chemical dry cleaning process with oxygen plasma in vacuum (45–50 W). Afterwards, surfaces were investigated (magnification, ×10,000; signal, secondary electrons SE2; working distance, 4 mm; electron high tension, 10 kV) with a field emission scanning microscope (FE-SEM; Zeiss Supra® 55 VP, Zeiss NTS GmbH, Oberkochen, Germany) and the most representative picture was chosen for assessing fillers' size.

Statistical analysis

The results for DC, HV, and *E* within each material, each measuring depth and each curing time, respectively, were compared using one-way analysis of variance (ANOVA) and Tukey's HSD post hoc test (α=0.05; SPSS 18.0, Chicago, IL, USA). An ANOVA multivariate analysis and partial eta square statistic was used to investigate the influence of the parameters "RBC", "measuring depth" and "polymerization time" on *E*, HV, and DC. For the properties E_{flexural} and σ, the influence of "RBC" was assessed. Additionally, a Weibull analysis was used to assess σ.

A common empirical expression for the cumulative probability of failure P at applied stress is the Weibull model:

$$P_f(\sigma_c) = 1 - \exp\left[-\left(\frac{\sigma_c}{\sigma_0}\right)^m\right]$$

where, σ_c is the measured strength, m is the Weibull modulus, and σ_0 is the characteristic strength, defined as the uniform stress at which the probability of failure is 0.63. The double logarithm of this expression gives $\ln\ln\frac{1}{1-P} = m\ln\sigma_c - m\ln\sigma_0$.

By plotting lnln(1/(1-P)) versus ln(σ), a straight line results, with the upward gradient m, whereas the intersection with the x-axes gives the logarithm of the characteristic strength.

Results

The influence of the parameters "RBCs", "measuring depth", and "polymerization time" as well as their interaction products was analyzed in an ANOVA multivariate test (Table 1). DC and the mechanical properties—HV, E, σ, and $E_{flexural}$—were selected as depended variables. The significance values of these three main effects were less than 0.05, indicating that they contribute all to the model. The "RBCs" was the parameter exerting the strongest influence on all measured properties (higher eta square values). The influence of the "measuring depth" was stronger on the micromechanical properties (HV and E) than on DC, whereas the influence of polymerization time, though significant, was very low.

A one-way ANOVA was used to identify detailed differences in the measured properties within each material as function of polymerization times (horizontal lines in Tables 2, 3, and 4) and geometries (vertical lines in Tables 2, 3, and 4).

A significant increase (p<0.05) in DC (Table 2) with increasing polymerization time was found for Surefil® SDR™ flow only at 4 mm (between 10 and 40 s) and 6 mm depth bulk placement (between 20 and 40 s) whereas for Venus® bulk fill this statement is only valid at 4 mm (between 10 and 20 s, respectively, 40 s) and 6-mm depth (bulk, between 10 and 20 s; incremental, between 10 and 40 s).

The DC at 6-mm depth bulk versus incremental placement was significantly lower only at low polymerization times (10 and 20 s for Surefil® SDR™ and 10 s for Venus® bulk fill). Comparing both RBCs, it can be seen that Venus® bulk fill had a statistically significant higher DC (about 5 %) for all irradiation times and measuring depths.

Concerning the variation of E (Table 3), results showed for both RBCs significant (p<0.05) lower values for 0.1 mm when compared to 2 mm depth as well as statistically equal values for 2 and 4 mm depth at all polymerization times. Similar trend is also valid for HV (Table 4). As for the incremental thickness, the HV-80 % was not reached in the 6-mm samples at any polymerization time in both measured RBCs.

Comparing both RBCs, Surefil® SDR™ flow showed statistically significant higher values for E (about 3 GPa) and HV (about 15 N/mm²) at all irradiation times and measured depths.

The investigated macromechanical properties σ and $E_{flexural}$ revealed for Surefil® SDR™ flow a significantly higher σ (131.8±5.8 MPa) and $E_{flexural}$ (5.0±0.4 GPa) when compared to Venus® bulk fill (σ=122.7±6.9 MPa; $E_{flexural}$= 3.6±0.4 GPa). For both materials, a very high Weibull modulus was reached (21.6 and 26.7) attesting a high reliability of both RBCs (Fig. 1). Comparing FE-SEM pictures (Fig. 2), fillers in Surefil® SDR™ flow are consistently smaller than fillers of Venus® bulk fill.

Table 1 Influence of material, measuring depth and polymerization time on the micromechanical properties, indentation modulus (E) and Vickers hardness (HV), as well as degree of conversion (DC) and macromechanical properties—flexural strength (σ) and flexural modulus ($E_{flexural}$)

Factor	E	HV	DC	σ	$E_{flexural}$
RBCs	0.84	0.80	0.63	0.35	0.80
Measuring depth	0.46	0.24	0.10		
Polymerization time	0.03	0.04	0.12		

The influence of all parameters was statistical significant (α=0.05). Table contains the partial eta square values. The higher the partial eta squares, the higher the influence of the selected factor on the measured properties

Discussion

Two recently launched bulk fill flowable RBCs—Surefil® SDR™ flow and Venus® bulk fill—considered to be used as cavity liners and bulk fill materials in class I and II restorations were investigated. For this purpose, specimens were measured by a FTIR spectrometer, a microhardness indenter, a three-point bending test device and a FE-SEM. It must however be considered that the measurements were done with a modern high-intensity LED curing unit which was applied at mold upper surface. Placing clinical restorations often means higher distances [17–19] between less effective curing units [20] and RBC surface. Therefore, the clinical values of the measured properties could be lower.

The substantial reduction in polymerization shrinkage and particularly the ability to place the RBCs as 4-mm bulks claimed by both manufacturers has led to further interest about the composition of the measured products. For both

Table 2 Degree of cure (%) 5 min after curing of Surefil® SDR™ flow and Venus® bulk fill measured at 0.1, 2, 4, 6 mm (bulk and incremental) depth of the samples are detailed in mean values and standard deviations (in parentheses)

Measuring depth Polymerization time	0.1 mm	2 mm	4 mm	6 mm-bulk	6 mm-incremental
Surefil® SDR™ flow					
10 s	58.9 $A_{1,2,3}$ (2.9)	60.1 $a_{2,3}$ (1.8)	58.3 $A_{1,2}$ (1.7)	57.1 a_1 (3.0)	61.0 A_3 (3.1)
20 s	61.1 A_2 (1.5)	59.5 a $_{1,2}$ (2.2)	59.7 $AB_{1,2}$ (1.7)	58.2 a_1 (1.7)	60.7 A_2 (2.1)
40 s	60.4 $A_{1,2}$ (3.1)	59.6 a_1 (1.9)	61.2 $B_{1,2}$ (2.1)	60.1 $b_{1,2}$ (2.0)	61.9 A_2 (2.4)
Venus® bulk fill					
10 s	65.0 $A_{1,2}$ (1.9)	65.0 a $_{1,2}$ (1.5)	62.9 $A_{2,1}$ (2.3)	62.4 $a_{2,1}$ (2.5)	65.6 $a_{1,2}$ (1.5)
20 s	64.9 A_1 (1.7)	65.0 a_1 (1.5)	66.1 $B_{1,2}$ (2.8)	65.6 $b_{1,2}$ (2.0)	66.7 $ab_{1,2}$ (1.6)
40 s	64.6 A_1 (1.6)	65.7 $a_{1,2}$ (1.8)	667.92 $B_{3,1,2}$ (1.6)	66.1 $b_{1,2}$ (2.8)	67.4 $b_{2,3}$ (1.5)

Uppercase letters (in vertical line) and subscript numbers (in horizontal line) indicate statistically homogenous subgroups (Tukey's HSD test, α= 0.05 was used for every column and line). Same letters (within one geometry, in vertical line) and lowercase letters (within one polymerization time, in horizontal line) indicate statistical similar groups (Tukey's HSD test, α=0.05)

RBCs, the manufacturers renounced to bisphenol-A-dimethacrylate (Bis-GMA) and only formed the organic matrix out of other dimethacrylates [21, 22]. As a result, the RBCs are supposed to be less viscous because UDMA, TEGDMA, and ethoxylated EBPDMA form more flexible polymers than Bis-GMA [23–26]. Moreover, Bis-GMA is said to be more hydrophilic [27] and consequently runs a higher risk of water uptake and degradation than the more hydrophobic EBPDMA [28]—used in both RBCs—thus reducing the risk of discoloration [29].

In our study, DC was mainly influenced by the type of RBC (η^2=0.63). Combined with our results, the claimed significant lower DC of Surefil® SDR™ flow in comparison to Venus® bulk fill [2] as well as its stated high DC when compared to other common RBCs (EsthetX Flow, Filtek Supreme Flow, Tetric Evo Flow, Filtek Silorane) [30] can be confirmed within the limitations of our experimental setup. Unless it has to be pointed out that through different matrix compositions of the two RBCs, DC cannot be rated because each monomer and additional group implicates different properties and different molecular architecture, thus a higher DC does not necessarily mean higher mechanical properties as also confirmed by the measured mechanical properties. Furthermore by increasing the concentration of monomers [31] or diluents [32] the DC can be artificially kept high without improving mechanical properties. This was obviously not done in the analyzed materials, since the measured mechanical properties performed well as already investigated and confirmed for Surefil® SDR™ flow when comparing it to different types of modern RBCs [11]. Scougall-Vilchis et al. claimed that microhardness largely depends on the filler particles (size, weight, volume) as well as on the chemical composition of the RBC when—like in our study—the test device produces larger indents than the size of the fillers [33]. Therefore, it can be stated that the measured HV values present the average microhardness of both, fillers and matrix. Comparing micromechanical properties of Venus® bulk fill—concerning values on surface

Table 3 Indentation modulus *E* [GPa]—of Surefil® SDR™ flow and Venus® bulk fill measured at 0.1, 2, 4, and 6 mm depth of samples cured for 10, 20, or 40 s as 6 mm high bulk and stored for 24 h in distilled water at 37°C, is detailed in mean values and standard deviations (in parentheses)

Measuring depth Polymerization time	0.1 mm	2 mm	4 mm	6 mm bulk
Surefil® SDR™ flow				
10 s	10.7 AB_1 (0.6)	12.0 a_2 (0.5)	12.2 A_2 (0.7)	11.5 a_2 (0.6)
20 s	10.6 A_1 (0.4)	11.9 a_2 (0.4)	12.2 A_2 (0.5)	11.1 a_1 (0.7)
40 s	101.8 B A_1 (0.4)	11.9 $a_{2,3}$ (0.4)	12.2 A_3 (0.6)	11.6 a_2 (0.8)
Venus® bulk fill				
10 s	7.3 A_1 (1.1)	8.4 a_2 (0.6)	8.6 A_2 (0.9)	7.3 a_1 (0.4)
20 s	7.6 A_1 (0.3)	8.6 a,b_2 (0.4)	8.8 A_2 (0.4)	7.7 b_1 (0.5)
40 s	7.7 A_1 (0.9)	8.8 b_2 (0.3)	8.8 A_2 (0.4)	8.5 c_2 (0.3)

Uppercase letters (in vertical line) and subscript numbers (in horizontal line) indicate statistically homogenous subgroups (Tukey's HSD test, α= 0.05 was used for every column and line). Same letters (within one geometry, in vertical line) and lowercase (within one polymerization time, in horizontal line) indicate statistical similar groups (Tukey's HSD test, α=0.05)

Table 4 Vickers hardness HV (N/mm^2) –and depth of cure (HV-80 %) [N/mm^2] of Surefil® SDR™ flow and Venus® bulk fill measured inat 0.1, 2, 4, and 6 mm depth of samples cured for 10, 20, or 40 s as 6 mm high bulk and stored for 24 h at 37°C in distilled water, is detailed in mean values and standard deviations (in parentheses)

Measuring depth Polymerization time	0.1 mm	2 mm	4 mm	6 mm bulk	HV-80 %
Surefil® SDR™ flow					
10 s	55.1 A_1 (2.3)	57.8 a_2 (3.4)	58.2 A_2 (4.1)	55.4 a_1 (3.8)	44.1
20 s	59.1 A_1 (3.3)	61.1 b_2 (2.1)	59.8 $A_{1,2}$ (2.9)	59.0 $b_{1,2}$ (3.4)	47.3
40 s	59.1 A_1 (1.3)	60.2 $ab_{1,2}$ (1.8.)	59.5 $A_{1,2}$ (2.9)	58.9 b_1 (2.6)	47.3
Venus® bulk fill					
10 s	40.7 A_1 (4.0)	46.1 a_2 (3.1)	46.5 A_2 (4.7)	39.1 a_1 (2.9)	32.6
20 s	41.4 A_1 (1.5)	45.8 a_3 (1.4)	46.4 A_3 (0.9)	43.0 b_2 (1.4)	33.1
40 s	42.7 A_1 (4.9)	46.4 a_2 (2.1)	46.4 A_2 (1.0)	46.0 c_2 (1.9)	34.2

Uppercase letters (in vertical line) and subscript numbers (in horizontal line) indicate statistically homogenous subgroups (Tukey's HSD test, α= 0.05 was used for every column and line). Same letters (within one geometry, in vertical line) and lowercase (within one polymerization time, in horizontal line) indicate statistical similar groups (Tukey's HSD test, α=0.05)

and in 2-mm depth after curing for 20 s—with literature data [11], HV and *E* result like a commonly used microhybrid flowable RBC (EsthetX Flow) and a nanohybrid flowable RBC (Filtek Supreme Flow; for *E*). When comparing the neat dimethacrylates, Sideridou et al. showed that DC increases in the order Bis-GMA<Bis-EMA (EBPDMA)< UDMA<TEGDMA [23]. However, there must be an upper limit in increasing concentration of dimethacrylates with lower molecular weight because polymerization shrinkage would either increase [34]. The low polymerization shrinkage for Surefil® SDR™ flow shall result from the addition of the "polymerization modulator", a chemical moiety in the resin backbone increasing flexibility and thus relaxing the polymerized network without harming DC (when compared to another common flowable RBC (EsthetX Flow, Dentsply)) [30]. Moreover, the extreme lowered polymerization shrinkage stress claimed by the manufacturer has been confirmed in other studies, showing for Surefil® SDR™ flow significant lower polymerization stress (1.1±0.1 MPa) even when compared to the low- shrinkage silorane-based composite Filtek Silorane [11]. Unfortunately, there are no published studies concerning the polymerization shrinkage of Venus® bulk fill. But with low contraction stress, the cavity adaptation increases and it allows the dentist to place the composite in a favorable way. Nevertheless, investigations on polymerization shrinkage in various bulks could be useful as an increased "C" factor, caused by lower unattached RBC surface, raises cusp deflection [8].

Statistics revealed for HV a strong influence (η^2=0.80) and for σ a moderate influence (η^2=0.35) of the factor "RBC"; moreover, *E* (η^2=0.84) and $E_{flexural}$ (η^2=0.80) were nearly equally strongly depended on the material. Therefore, the first tested hypothesis must be rejected. In the macro- and micromechanical tests, Surefil® SDR™ flow proved to be significantly superior to Venus® bulk fill. Reasons for this behavior might be found in both, inorganic and organic compounds. Surefil® SDR™ flow differs from Venus® bulk fill in the matrix composition as it contains additional TEGDMA and a polymerization modulator [30]. With the addition of the more flexible side groups containing TEGDMA, viscosity can be decreased [35] and with the formation of more homogenous copolymer networks,

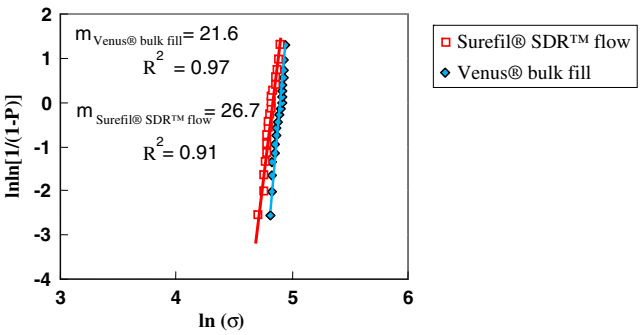

Fig. 1 Evaluation of the Weibull parameter (*m*) for Surefil® SDR™ flow and Venus® bulk fill using the variables *P* (probability of failure) and σ (flexural strength). R^2 is the coefficient of determination

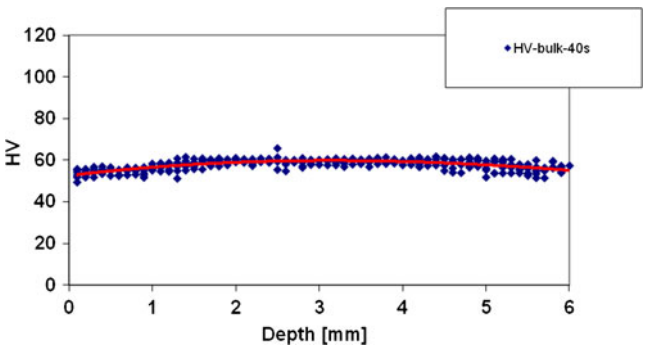

Fig. 2 Evaluation of the Vickers hardness (HV) with increasing specimen depth for Surefil® SDR™ flow when cured as bulk for 40 s (360 measuring points)

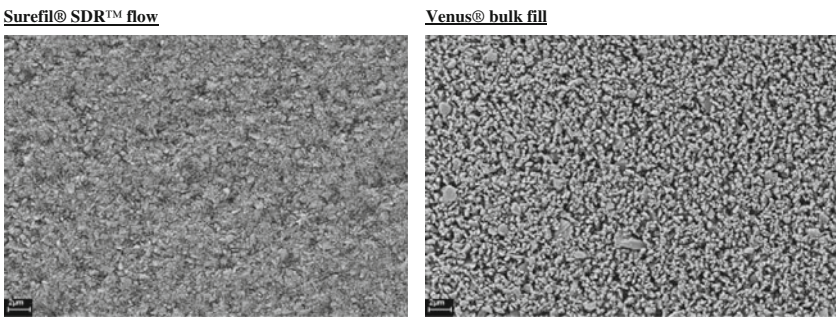

Fig. 3 FE-SEM pictures of the measured materials

polymerization shrinkage decreases either [36]. When comparing experimental composites with different types and contents of fillers, Lee et al. found out that viscosity of RBC increases when filler volume increases [37]. Decreased viscosity is desirable for Surefil® SDR™ flow to reach similar levels of flowability, as its filler content (68 % per weight, 44 % per volume) strongly differs from the filler content of Venus® bulk fill (65 % per weight, 38 % per volume). With increasing filler volume, the flexural strength and modulus as well as hardness improve [38, 39]. Comparing the results for the micromechanical properties to a study investigating five nanohybrid RBCs (Miris2, N'Durance, Premise, Simile, Venus Diamond) with the same experimental setup, Venus® bulk fill and Surefil® SDR™ flow show lower values than all of the measured materials [40]. The recommendation for an irradiation time of 20 s and a 4-mm bulk placement for Surefil® SDR™ flow as well as an irradiation time of either 40 s and 6 mm bulk placement or 20 s and 4-mm bulk placement for Venus® bulk fill, is supported by the measured micromechanical values. Therefore, the second hypothesis was rejected.

Assessing FE-SEM pictures (Fig. 2), fillers in Surefil® SDR™ flow are consistently smaller than fillers of Venus® bulk fill. Li et al. claimed that decreasing filler size also means harming depth of cure and compressive strength [41] which however is not evident for the measured bulk fill materials. Further investigations are needed to define the role of the polymerization modulator concerning both, mechanical properties and DC.

The producers' guarantee of placing the RBCs in 4 mm bulks and light curing for 20 s without a loss in DC and mechanical properties seems to be of great interest for customers: it saves time and handling would be very easy. Our results confirm this claim and show no improvement when placing thinner bulks than 4 mm or increasing the irradiation time from 20 to 40 s up to a measurement depth of 4 mm for both RBCs.

Moreover, the 80 %-HV value—presenting the percentage of the relation of bottom to top surface hardness to be 80 % for a properly cured composite [42] and due to Hansen et al. rather important than top surface hardness [43]—was not reached in the 6-mm samples at any of the measured irradiation times. This concludes that both RBCs may be placed in 4 mm bulks without a loss in relevant properties, like mechanical properties or degree of cure.

Besides the factor "RBC", E (η^2=0.46) as well as HV (η^2=0.24) were moderately influenced by "measuring depth". Considering the variation of micromechanical properties with depth (Fig. 3), it has to be noted that HV and E values rise with the depth to a measuring depth of approximately 1.5 mm until then starting to decrease. This behavior is not characteristic for high-filled RBCs [44]. Since the oxygen inhibition layer does not exceed 20–50 μm [45], the initial decrease in mechanical properties can rather be explained by the fact that nonbonded light-cured RBCs may shrink towards the center of the restoration [46]. Kakaboura et al. shared the same thought when evaluating shrinkage strain of light-cured RBCs using X-ray microtomography and a bonded disk method [47]. Therefore, the polymerized bulks could reach lower mechanical values at peripheral surfaces because the volumetric shrinkage in the center of the bulk would be compensated by the flow from the periphery. Moreover, Baroudi et al. explained the increased edge fracture resistance with the lower viscosity of monomers and the reduced particle size of fillers of flowable RBCs [48].

Regarding the results of the Weibull analysis, both materials exerted a high reliability (m value). The high values of m (21.6 and 26.7)—indicating a narrow distribution of values and therefore a small error range—were unexpected as consistently lower values were measured for regular flowable RBCs on the market (6.37–15.23) [49].

Conclusions

A strong influence of the material was statistically proven for all measured properties. A polymerization time of 20 s instead of 40 s as well as placing the RBCs in 4-mm bulks instead of 2-mm bulks neither lowered the micromechanical properties nor DC. Surefil® SDR™ flow showed significantly higher mechanical properties but lower DC values when compared to Venus® bulk fill.

Within the limitations of our study and the experienced high reliability (high Weibull modulus values) and good mechanical properties, a polymerization time of 20 s and bulk placement up to 4 mm can be recommended.

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