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Polymerization Shrinkage and Depth of Cure of Bulk Fill Flowable Composite Resins

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Clinical Relevance

Flowable composites had higher shrinkage than the nanohybrid restorative composite that was modified to have a flowable consistency during ultrasonic insertion. All materials tested had significantly less depth of cure than either manufacturers' claims or ISO 4049 scrape test results.

SUMMARY

Objective: **To evaluate polymerization shrinkage and depth of cure of two bulk fill flowable composites, one nanohybrid composite modified to a flowable consistency, and one standard flowable composite, comparing the scraping method to the Knoop hardness test.**

Methods: **Two bulk fill flowable composites, SureFil SDR *flow* (SSF) (Dentsply) and Venus Bulk Fill (VBF) (Heraeus Kulzer), one standard flowable, Filtek Supreme Ultra Flowable (FSUF) (3M/ESPE) (control), and one regular bulk composite that can be made flowable, SonicFill (SF) (Kerr), were used in this study. For polymerization shrinkage (PS), ten 2-mm samples were made for each composite and cured for 20 seconds and shrinkage was measured with a Kaman linometer. For hardness, ten specimens of each composite were made in a 10 × 10-mm mold and cured for 20 seconds; the bottom surface was scraped according to ISO 4049 specification, and the remaining thickness was measured with a micrometer. Hardness samples were prepared at 2-, 3-, 4-, and 5-mm thick ×14-mm diameter, cured for 20 seconds, and polished. After 24 hours of dry storage, a Knoop indenter was applied at 100 g load for 11 seconds. Three readings were made on the top and bottom of each specimen and averaged for each surface to calculate a Knoop hardness value and a bottom/top hardness ratio. One-way analysis of variance and Tukey tests were used to determine significant differences between thicknesses and between test methods for each material.**

Results: **PS values were 3.43 ± 0.51%, 3.57 ± 0.63%, 4.4 ± 0.79%, and 1.76 ± 0.53% for FSUF, SSF, VBF, and SF, respectively. VBF showed**

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significantly greater shrinkage (4.4 ± 0.79%), followed by FSUF (3.43 ± 0.51%) and SSF (3.57 ± 0.63%), which were similar, and SF (1.76 ± 0.53%), which had significantly less shrinkage (*p*<0.05). Values for the scraping method for depth of cure were significantly greater for SSF and VBF (>5.0 mm), followed by SF (3.46 ± 0.16 mm) and FSU (2.98 ± 0.22 mm). Knoop top hardness values (KHN) were: VBF 21.55 ± 2.39, FSUF 44.62 ± 1.93, SSF 29.17 ± 0.76, and SF 72.56 ± 2.4 at 2 mm and were not significantly different at 3-, 4-, and 5-mm thick within each material. Ratios for bottom/top values (depth of cure) for 2, 3, 4, and 5 mm were: VBF 0.80 ± 0.1, 0.78 ± 0.03, 0.67 ± 0.10, and 0.59 ± 0.07, respectively; SSF 0.74 ± 0.08, 0.72 ± 0.08, 0.69 ± 0.18, and 0.62 ± 0.08, respectively; SF 0.82 ± 0.05, 0.68 ± 0.05, 0.47 ± 0.04, and 0.21 ± 0.02, respectively; and FSUF 0.56 ± 0.08 at 2 mm and 0.40 ± 0.08 at 3 mm. The bottom/top ratio was .80 or less at all depths and decreased below 0.70 at 4-mm depth for VBF and SSF, at 3 mm for SF and at 2 mm for FSUF.

INTRODUCTION

Innovative bulk fill composite resins with reported significant flow and low polymerization shrinkage have been marketed. Clinical recommendations suggest that they have greater depth of cure and can be placed in a 4-mm bulk increment and will have adequate polymerization. The stress-decreasing resin technology was created as a new resin system that would minimize shrinkage stress and allow bulk placement. Clinically, this would eliminate the need for incremental placement and curing and reduce the need for material manipulation during insertion. To do this, it was necessary to regulate the overall modulus development while maintaining its polymerization rate and conversion.[1-3]

This new and innovate technology is based on changes in monomer chemistry. Manufacturers introduced this new technology by modifying the Bowen monomer (Bis-GMA: 2,2-bis [4-(2-hydroxy-3-methacryloxypropoxy) phenyl]propane) to create monomers with lower viscosity.[1-5] This new modification could be achieved by incorporating hydroxyl-free Bis-GMA, aliphatic urethane dimethacrylates, partially aromatic urethane dimethacrylate, or highly branched methacrylates.[6] The outcomes of these changes in monomer and composite organic matrix have been shown to reduce polymerization shrinkage stresses over 70%.[1,2,7,8]

Polymerization shrinkage is related to the organic and inorganic content of the composite resins. Flowable composites generally contain more organic matrix in order to gain increased flow. Thus, they have greater shrinkage compared to hybrid composites, which have less organic matrix.[9] As a result of the increased resin matrix, flowable composites reduce internal stresses during polymerization shrinkage due to their lower Young's modulus compared to regular packable composites.[10]

As composites are light cured, they convert from a viscous phase into a rigid or solid material and undergo shrinkage. When the composite is placed inside a preparation and subsequently bonds to the tooth surfaces, the polymerized composite develops internal mechanical stress, which is transmitted to the tooth structure-bond interface[11]; if the contraction stress exceeds the bond strength, debonding can occur, resulting in possible marginal leakage, secondary caries, and cohesive tooth fractures at the margins.[12] The cavity configuration or C-factor described by Feilzer and others[13] claims that the stress inside a restoration after it was polymerized is proportional to the ratio of its bonded and nonbonded surfaces, and this C-factor is responsible for the development of the contraction stress in composite resin restorations.

Many laboratory methods such as infrared spectrometry,[14-17] resonance imaging, and Knoop and Vickers hardness[18] have been used to determine the depth of cure of restorative materials. The standard and most common test, the scraping method ISO 4049, has been researched and has been shown to overestimate depth of cure values.[5,14,16,19] When hardness values are obtained, a mean bottom/top ratio (B/T) hardness value is usually determined to establish the depth of cure. This reflects the relative extent of conversion of the deeper surfaces in relation to the top surface. As an accepted minimum standard, many authors have claimed that a ratio of 0.80 is clinically acceptable.[19-22] It has been shown that the value is mostly dependent on filler content and filler size.[22] This is an accurate method to compare the relative extent of cure between different composites at specified depths. Undercured composites can lead to early failure of a restoration and should be avoided in any clinical technique.

The purpose of the present study was to evaluate polymerization shrinkage and depth of cure of three bulk fill flowable composites. Both the scraping method (ISO 4049) and Knoop hardness test were conducted on each material and compared in order to verify the accuracy of the test methodology and to

Table 1: *Materials Used in the Study*

Material	Type	% by Weight	Filler Composition	Manufacturer
Venus Bulk Fill	Nanohybrid	65%	Ba-Al-F silica glass, YbF_3, and SiO_2	Heraeus Kulzer (South Bend, IN, USA)
SureFil SDR *flow*	Urethane dimethacrylate with hybrid glass filler	68%	Barium and strontium, alumino-fluoro-silicate glasses	DENTSPLY Caulk (Milford, DE, USA)
SonicFill	Nanohybrid	83.5%	Barium, aluminum boron, silicate	Kerr Corporation (Orange, CA, USA)
Filtek Supreme Ultra Flowable (control)	Nanocomposite	65%	Ytterbium trifluoride, silica, zirconia/silica clusters	3M/ESPE (St Paul, MN, USA)

better evaluate the suggested depth of cure in the marketing literature of this group of materials.

The two null hypotheses tested were:

- There is no significant difference in polymerization shrinkage and depth of cure between the different bulk fill composite resins.
- There is no significant difference in depth of cure measurements between the ISO 4049 scraping test and the bottom/top hardness ratio test.

METHODS AND MATERIALS

The composites tested in this study are listed in Table 1. SureFil SDR *flow* and Venus Bulk Fill are bulk fill flowable composites. SonicFill is a nanohybrid bulk fill composite that can be placed ultrasonically in a flowable state, and Filtek Supreme Ultra Flowable is a standard flowable used as the control. A light shade was selected for each material (shade A2, except for SureFil SDR *flow*, which was a universal shade) so that light penetration would be optimal and the effect of shading pigments would not be a confounding variable.

Polymerization Shrinkage

Ten disk-shaped samples of each composite material approximately 5 mm in diameter and 1.5 to 2 mm thick were fabricated. The uncured composite was dispensed in a Teflon ring with a glass slide on the bottom which was coated with a separating medium to allow the composite to shrink free of surface adhesion. A lubricated flat aluminum target parallel to the glass slide covered the open side of the sample. Once in position, the composite specimens were polymerized for 20 seconds using the SmartLite iQ2 (Model No. 200, DENTSPLY Caulk) curing unit with a wavelength range between 450 and 475 nm and a power of 800 mW/cm^2. This value was verified every five samples to corroborate adequate polymerization. This analysis was performed with a Kaman linometer (KADA, Kaman Instrumentation, Colorado Springs, CO, USA),[23] where the arrangement consists of a noncontact displacement transducer with the sensor placed in a vertically oriented quartz tube. The holder allows the placement of the curing light approximately 1 mm away from the composite. The linear shrinkage data were recorded as change in length (ΔL), and the percentage of shrinkage was calculated using the following formula: $Lin\% = \Delta L/(L + \Delta L) \times 100$. Here, ΔL is the displacement recorded in microns, and L is the thickness of the specimen in microns after polymerization. The $Lin\%$ is then converted mathematically to a volumetric value using the formula:

$$Vol\% = 3Lin\% - 0.03(Lin\%)^2 + 0.0001(Lin\%)^3$$

Depth of Cure

For the ISO 4049 scraping method, depth of cure was calculated by fabricating ten specimens of each composite in a 10 $\times$ 10 mm mold and light curing for 20 seconds using the SmartLite iQ2 curing unit. After the specimen was removed from the mold, the bottom surface was scraped to remove soft unpoly-

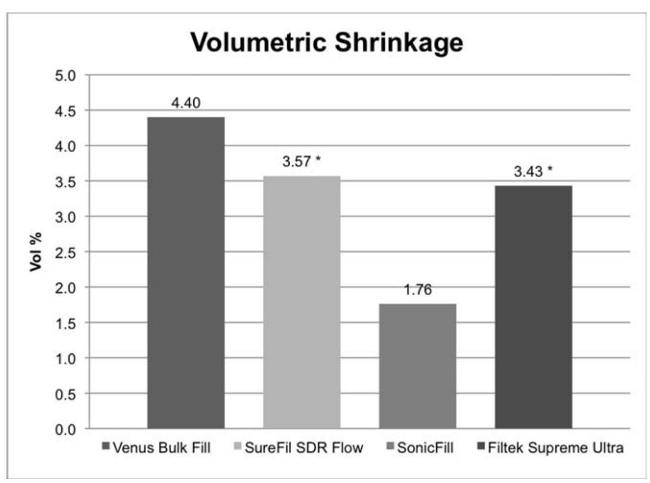

Figure 1. *Mean volumetric shrinkage values for each material tested. Values with an (*) were not significantly different.*

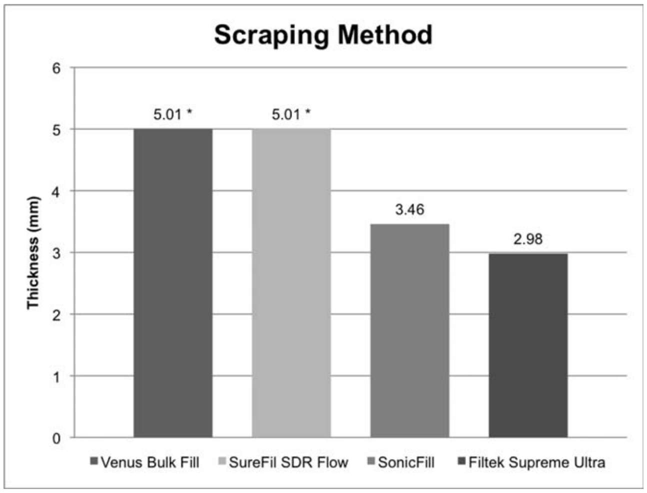

Figure 2. *Mean scraping method values for each material tested. Values with an (*) were not significantly different.*

merized material using a plastic spatula according to ISO 4049, and the remaining thickness of composite was measured three times with a micrometer (Mitutoyo America, Aurora, IL, USA), averaged and divided by two.

For Knoop hardness, a split brass mold was used to prepare composite samples 14 mm in diameter and 2, 3, 4, and 5 mm in thickness. Ten specimens of each thickness were fabricated for each composite. Under low light conditions, composite was applied to fill each hole in the brass mold. A thin layer of Al-Cote Septarating Agent (Dentsply, York, PA) was placed on a clean glass slide (1 mm thick), which was then placed on top of the composite to create a flat surface. Samples were light cured for 20 seconds through the glass slide using the SmartLite iQ2. Each sample was then removed from the mold and polished at the top and at the bottom surface with 2400-grit silicon carbide paper (Mager Scientific, Dexter, MI, USA), followed by a 9-μm polycrystalline diamond suspension (Micro Star 2000, Inc, Ontario, Canada) to remove the resin-rich layer and develop a polished surface to make the indentation more visible in the microscope. After a dark storage period of 24 hours, a Tukon 2100B testing machine (Wilson Instruments, Northwood, MA, USA) was calibrated, and a Knoop diamond indenting tool was applied three times with a 100 g load to the top and the bottom of each sample surface with a dwell time of 11 seconds.

The following formula was used to calculate the Knoop hardness number, which is the ratio of the load applied to the area of the indentation: $KHN = L/(I^2Cp)$. In this formula, L is the applied load in kilograms, I is the length of the long diagonal of indentation (mm), and Cp is the constant related to the area of the indentation (0.070).

One-way analysis of variance (ANOVA) was used to determine a significant difference among materials for all three variables. Tukey multiple comparison test was then used to determine significant differences between the means ($p<0.001$).

RESULTS

Polymerization Shrinkage

The mean values for volumetric shrinkage (Figure 1) for Venus Bulk Fill, SureFil SDR *flow*, Filtek Supreme Ultra Flowable, and SonicFill were 4.40% (±0.79), 3.57% (±0.63), 3.43% (±0.51), and 1.76% (±0.53), respectively. Tukey test revealed that SonicFill had the lowest shrinkage value ($p<0.001$), and Venus Bulk the highest shrinkage value. SureFil SDR *flow* and Filtek Supreme Ultra Flowable were not significantly different.

Scraping Method

Figure 2 represents the mean depth of cure values using the scraping method: Venus Bulk Fill 5.01 mm (±0.02) and SureFil SDR *flow* 5.01 mm (±0.03) both demonstrated full depth of cure at 5 mm, while both Filtek Supreme Ultra Flowable 2.98 mm (±0.11),

Table 2: *Means and Standard Deviations for Top and Bottom Surface Knoop Hardness Values*[†]

Material	Top Hardness					Bottom Hardness				
	2 mm	3 mm	4 mm	5 mm	*p*-value	2 mm	3 mm	4 mm	5 mm	*p*-value
Venus Bulk Fill	21.6 (2.40)*	21.3 (1.02)*	23.5 (3.25)*	23.0 (2.34)*	0.13	17.0 (2.35)*	16.6 (0.70) [a]*	15.6 (2.09)*	13.5 (0.74) [a]	<0.001
SureFil SDR *flow*	29.1 (0.77)*	29.4 (1.16)*	29.7 (3.97)*	31.5 (1.42)*	0.11	21.5 (2.53) [a]*	21.1 (1.98)*	19.8 (2.782)*	19.4 (2.74)*	0.21
SonicFill)	72.6 (2.40)*	72.3 (3.20)*	72.4 (2.11)*	71.1 (2.64)*	0.60	59.4 (2.85)	48.9 (2.06)	34.0 (1.88)	15.1 (1.29) [a]	<0.001
Filtek Supreme Ultra Flowable	44.6 (1.93)*	42.8 (3.07)*	—	—	0.13	24.7 (3.32) [a]	16.9 (3.13) [a]	—	—	<0.001

[†] *Column values with the same letter are not significantly different at* p*<0.001; row values with the same symbol (*) are not significantly different.*

Table 3: *Means and Standard Deviations for Hardness Ratio Values (B/T)*†

Material	2 mm	3 mm	4 mm	5 mm	*p*-value
Venus Bulk Fill	0.80 (0.10) a*	0.78 (0.03) a*	0.67 (0.10) a	0.59 (0.07) a	<0.001
SureFil SDR *flow*	0.74 (0.08) a*	0.72 (0.08) a*	0.69 (0.18) a*	0.62 (0.08) a*	0.09 (NS)
SonicFill	0.82 (0.05) a	0.68 (0.05) a	0.47 (0.04)	0.21 (0.02)	<0.001
Filtek Supreme Ultra Flowable	0.56 (0.08)	0.40 (0.08)	—	—	<0.001

Abbreviations: NS, not significant.
† *Column values with the same letter are not significantly different at* p<0.001; *row values with the same symbol (*) are not significantly different.*

and SonicFill 3.46 mm (±0.08) were significantly lower.

Surface Hardness: Knoop Hardness

The means and standard deviations for top and bottom hardness using the Knoop hardness indentation for each material at 2-, 3-, 4-, and 5-mm thickness are represented in Table 2. Top hardness values for each material revealed no significant differences at all four thicknesses. For bottom hardness values, Venus Bulk Fill decreased significantly between 4- and 5-mm thickness. SureFil SDR *flow* was the only material that showed statistically similar values at all four thicknesses tested. SonicFill had a significant decrease at 2, 3, 4, and 5 mm. Filtek Supreme Ultra Flowable showed a significant difference between 2 and 3 mm and was too soft to test at both 4 and 5 mm.

Bottom/Top Hardness Ratio (Depth of Cure)

Table 3 represents the means and standard deviations for B/T ratio at different thicknesses. For reference, a perfectly cured material would have a ratio of 1.0. For Venus Bulk Fill, B/T ratio was .80 or less at all depths, with a significant decrease between 3 and 4 mm. B/T ratio values for SureFil SDR *flow* were 0.7 or less, and the decreases at each depth were not statistically significant. For SonicFill, B/T ratio was 0.80 or less with a significant decrease at all depths. For Filtek Supreme Ultra Flowable, B/T ratio values were significantly lower compared to the other materials (<0.6) at both 2- and 3-mm thicknesses, and the material was too soft to measure at 3 and 4 mm.

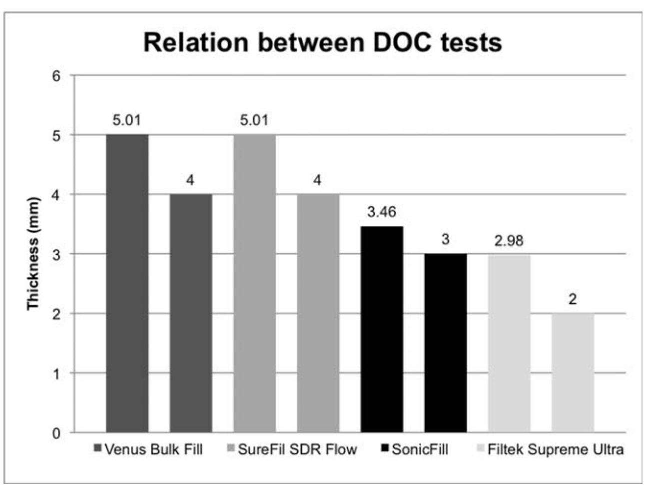

Figure 3. *Graphic representation of relation between depth of cure tests. The left bar for each material represents the scraping method value in mm. The right bar represents the critical depth in mm where the bottom to top hardness ratio decreases below 0.70.*

Relation Between Depth of Cure Tests

Depth of cure values from the scraping method and Knoop hardness were compared to determine if an association existed between the two tests (Figure 3). Venus Bulk Fill and SureFil SDR *flow* both cured to 5 mm depth using the scraping test, but were at least 1 mm less when tested using hardness tests and only a 70% cure. SonicFill and Filtek Supreme Ultra Flowable cured to lesser depths but also were about 1 mm less using the hardness tests. All B/T hardness values were significantly lower than those obtained by the scraping method.

DISCUSSION

The results for this study document that the three flowable materials show greater shrinkage (3.3-4.4 vol%) than the nanohybrid material, SonicFill (1.8%). A recent study reported volumetric shrinkage of 3.1% for SureFil SDR *flow*,[1] which is in agreement with this study. SonicFill is actually a full body composite resin with an estimated filler content of 83.5% by weight and 69% by volume and thus is expected to have less shrinkage than a flowable material with higher resin content.[7] Shrinkage values have a direct relationship with the amount of organic matrix in the material.[24] A study by Herrero and others[23] in 2005 found that not only the size and amount of filler content were important in determining the shrinkage, but the shape of the particles can also generate a different outcome. The distinct advantage of a material like SonicFill is that it has a consistency that is similar to a flowable when it is being placed, and then it has the properties of a hybrid after it polymerizes.

Even though SonicFill had the lowest volumetric shrinkage, further investigations are needed to determine shrinkage stress. An assumption can be made that even though SureFil SDR *flow* had higher volumetric shrinkage compared to SonicFill, stress can be lower due to the new modulator incorporated in the chemistry, which slows the rate of polymerization and reduces stress.[1] It is important to understand that a significant reduction in reaction rate does not necessarily correspond to reduction in contraction stress.[25]

In the present study, depth of cure values using the scraping method were greater for the two modified flowable materials that are marketed with depth of cure values that support bulk fill techniques than the nanohybrid or the standard flowable materials. These results are in accordance with a recent study where scraping method values for the same bulk fill materials were found to be higher than the control flowable composite resins.[5] The higher depth of cure of the bulk fill materials may be due to the incorporation of more efficient initiator systems and higher translucency of composites.[5] Even though SonicFill is considered to be a bulk fill composite, the true depth of cure was less than 4 mm in this study. It has been demonstrated that filler size and content in dental composites may reduce light penetration and is directly related to depth of cure.[14,17] The presence of pigments in shaded composite materials should also have an effect on depth of cure because pigments are opaque particles that will limit light penetration and reduce the degree of polymerization at greater depths within a cavity preparation.

Knoop hardness indentation is an actual measurement of surface hardness compared to the estimated values from the scraping method in ISO 4049. For each of the materials tested, top hardness values revealed no significant differences at the four thicknesses, as seen in Table 2. This result is in accordance with a previous study,[26] and this outcome is expected since the light source is being applied at the top surfaces of all composites. For bottom hardness values, all materials decreased as the thickness of the samples increased, which can be expected. Venus Bulk Fill showed a statistically significant decrease at 4- and 5-mm thickness. Numerous studies have defined depth of cure based on hardness measurements performed on the top and bottom surface of a light-cured resin composite specimen and agree that a ratio of 0.80 may be used as a critical minimum acceptable threshold value.[16,19,22] All B/T values for the materials in this study were less than the 80% critical value for depth of cure, and their use in bulk fill situations greater than 3-mm depth should be questioned.

As seen in Table 3, top and bottom hardness values revealed that SureFil SDR *flow* had statistically similar results at 2, 3, 4, and 5 mm. In comparison, Venus Bulk Fill showed a B/T ratio of 0.80 at 2 and 3 mm but had a significant decrease at 4 and 5 mm. These results are in accordance with a study done by Czasch and Ilie[3] in 2012 where it was shown that SureFil SDR *flow* was significantly harder than Venus Bulk Fill. It is important to state that in the present study even though SureFil SDR *flow* had similar results at the different depths, the B/T ratio at 2 mm was less than 0.80. Composition in the chemistry of SureFil SDR *flow* is based on a modification of triethylene glycol dimethacrylate, which is found to have more flexible side groups, reducing and forming a more homogeneous polymer network.[8]

The difference in hardness values for the materials tested can also be a result of filler content. It has been reported that decreasing filler size can affect depth of cure, while increasing filler volume can improve the hardness.[16] Overall, if the filler content of a composite resin is increased, the mechanical properties will improve.[16] SonicFill had the highest filler content of the materials tested (83.5%), but the depth of cure significantly decreased at 3, 4, and 5 mm. A possible reason might be the lack of light penetration through the composite at increasing depths because a high percentage of the wavelengths are absorbed near the top surface of the composite and not available to excite co-initiators at greater depths. Further research is needed to identify the role of the polymerization modulator on mechanical properties of these new bulk fill materials.

CONCLUSIONS

Within the limitations of this *in vitro* study the following conclusions can be stated:

1. SonicFill had significantly lower shrinkage values ($p<0.05$) than all of the other materials. Filtek Supreme Ultra Flowable and SureFil SDR *flow* were higher but not significantly different. Venus Bulk Fill showed significantly higher shrinkage values than the other materials tested.
2. Using the scraping method (ISO 4049 specification), Venus Bulk Fill and SureFil SDR *flow*

showed significantly higher depth of cure values than Filtek Supreme Ultra Flowable and SonicFill.
3. For top hardness measurements, all materials exhibited similar values, regardless of thickness.
4. For bottom hardness measurements, all materials showed a decrease in hardness with increasing depth.
5. The bottom/top ratio was 0.80 or above for only SonicFill at 2-mm depth. It decreased below 0.70 at 4-mm depth for Venus Bulk Fill and SureFil SDR *flow*, at 3-mm depth for Sonic Fill, and at 2-mm depth for Filtek Supreme Ultra Flowable.
6. The scraping method significantly overestimated depth of cure values for all materials, and this could influence clinical outcomes.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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