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Polymerization Shrinkage and Depth of Cure of Bulk-Fill Resin Composites and Highly Filled Flowable Resin

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Clinical Relevance

As an alternative to conventional resin composites for direct posterior restorations, the use of new strategic composites that speed up the restorative procedure should be guided by careful attention to case selection and operative procedure.

SUMMARY

The aim of this study was to evaluate the polymerization behavior and depth of cure (DOC) of recently introduced resin composites for posterior use: highly filled flowable composite and composites for bulk fill. A highly filled flowable (G-aenial Universal Flo [GUF]), two bulk-fill flowables (Surefil SDR Flow [SDR] and Venus Bulk fill [VBF]), and a bulk-fill nonflowable composite (Tetric N-Ceram Bulk fill [TBF]) were compared with two conventional composites (Tetric Flow [TF], Filtek Supreme Ultra [FS]). Linear polymerization shrinkage and polymerization shrinkage stress were each measured with custom-made devices. To evaluate DOC, the composite specimen was prepared using a mold with a hole of 4 mm depth and 4 mm internal diameter. The hole was bulk filled with each of the six composites and light cured for 20 seconds, followed by 24 hours of water storage. The surface hardness was measured on the top and the bottom using a Vickers microhardness (HV) indenter. The linear polymerization shrinkage of the composite specimens after photo-initiation decreased in the following order: TF and GUF > VBF > SDR > FS and TBF ($p<0.05$). The polymerization shrinkage stress of the six composite groups decreased in the following order: GUF > TF and VBF > SDR > FS and TBF ($p<0.05$). The mean bottom surface HV of SDR and VBF exceeded 80% of the top surface HV (HV-80%). However, the bottom of GUF and TBF failed to reach HV-

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80%. A highly filled flowable (GUF) revealed limitations in polymerization shrinkage and DOC. Bulk-fill flowables (SDR and VBF) were properly cured in 4-mm bulk, but they shrank more than the conventional nonflowable composite. A bulk-fill nonflowable (TBF) showed comparable shrinkage to the conventional nonflowable composite, but it was not sufficiently cured in the 4-mm bulk.

INTRODUCTION

Because composite has shown a level of success as a restorative material, there have been continuous efforts to improve its physical and mechanical properties and the operating techniques used to apply it.[1-3] Even so, complications related to polymerization shrinkage stress and curing depth still cause significant reluctance to use them. Not only will this polymerization shrinkage stress be trapped within the material itself, but it also will exert forces on the adhesive interfaces of the dentin.[4,5] Consequently, this shrinkage stress could lead to abundant clinical problems such as microleakage, marginal gap formation, recurrent caries, pulpal irritation, and maybe even tooth loss.[2] The decrease in the degree of conversion is also a nuisance, compromising the physical properties and increasing elution of the monomer. It might lead to postoperative sensitivity and result in premature failure of the composite restoration.[6,7]

Various clinical strategies have been suggested to reduce the restorative complications in direct posterior composite restoration; these include an incremental layering technique, the use of a flowable lining layer, and the modulation of the photoinitiation mechanism.[1,8,9] Among them, incremental layering is the standard of care for placement of resin composites in cavity preparations exceeding 2 mm, by virtue of the sufficient exposure of the entire increment to the curing light, as well as the reduction of the volume of the contracting material.[10,11] Despite those strategies having been shown to be effective in improving the longevity of restorations,[11-13] clinicians still desire easier and quicker composite restorations with less shrinkage. Significant advances have been made in composite formulations that target less shrinkage and are more user friendly; these include 1) highly filled flowable, 2) bulk-fill flowable, and 3) bulk-fill nonflowable composites.

Flowable composite was introduced in the 1990s, and it was promoted because it is injectable, which is regarded as a desirable handling property and allows simplification of the placement procedure.[14-16] Typically, flowable composite has a lower filler content and higher volume of resin matrix when compared with nonflowable composite, so the first-generation flowable composite was applied as a cavity liner or Class V restoration due to the low elastic modulus. However, the recent generations of flowable composite (G-aenial Universal Flo [GUF], GC Co, Milford, DE, USA) have higher filler content and are claimed to have improved mechanical properties; thus, they are indicated not only as a cavity liner but also for larger posterior restorations.[17] The latest version of flowable composites for simplifying the restorative procedure is the bulk-filling posterior flowable. Surefil SDR Flow (SDR; Dentsply Caulk, Milford, DE, USA) and Venus Bulk fill (VBF; Heraeus Kulzer GmbH, Hanau, Germany) are intended to be placed and bulk-cured in one increment up to 4 mm. The matrix composition of these two bulk-fill flowables is based on modified urethane dimethacrylate (UDMA). The manufacturer of SDR says that it differs from conventional composites by incorporating stress-decreasing resin technology, which comprises a high molecular weight polymerization modulator in the matrix structure. This unique molecular structure contributes to the delay of the gel point, which represents an increase of viscosity through network formation, and it allows for a greater pregelation-phase time.[18] In terms of the depth of cure (DOC), these new-generation flowable composites showed satisfactory results in 4-mm increments after 20 seconds of photo-polymerization, which is recommended by the manufacturer.[19]

Along with the bulk-fill flowables, the bulk-fill nonflowable composite, Tetric N-Ceram Bulk-fill (TBF; Ivoclar Vivadent, Schaan, Liechtenstein), was recently launched with the claim that it would substitute for not only the conventional nonflowable composite but also for the bulk-fill flowables that are required for the final 2 mm when using the incremental layering technique. According to the manufacturer‘s information, this new composite will achieve full-depth bulk fill up to 4 mm without a superficial capping layer, unlike the bulk-fill flowables. The manufacturer states that TBF contains a shrinkage stress reliever to minimize polymerization shrinkage; this is a modified unique filler partially functionalized with silanes.

Up to now, an incremental layering technique has been the standard procedure in direct posterior composite restorations to reduce polymerization shrinkage stress and achieve adequate DOC.[11,20] Yet, recent advances in composite technology for

Table 1: *Materials, Manufacturers, and Chemical Composition of Matrix and Filler*

Product (Code)	Type	Manufacturer, Batch No.	Matrix System
Tetric N-Flow (TF)	Flowable	Ivoclar Vivadent, Schaan, Liechtenstein N03326	Bis-GMA, UDMA, TEGDMA
SDR (SDR)	Bulk-fill flowable	Dentsply Caulk, Milford, DE, USA 100831	Modified UDMA, EBPDMA, TEGDMA
Venus Bulk Fill (VBF)	Bulk-fill flowable	Heraeus Kulzer GmbH, Hanau, Germany 010100	UDMA, EBPDMA, TEGDMA
G-aenial Universal Flo (GUF)	High-viscosity flowable	GC Co., Milford, DE, USA 1108032	UDMA, Bis-MEPP, TEGDMA
Filtek Supreme Ultra (FS)	Nano-composite (nonflowable)	3M ESPE, St Paul, MN, USA N367463	Bis-PMA, DUDMA, Bis-GMA, TEGDMA
Tetric N-Ceram Bulk Fill (TBF)	High-viscosity bulk-fill composite (nonflowable)	Ivoclar Vivadent, Schaan, Liechtenstein R52452	Bis-GMA, DMA

Abbreviations: Bis-GMA, bisphenol-A diglycidyl ether dimethacrylate; Bis-MEPP, 2,2-Bis (4-methacryloxypolyethoxyphenyl) propane; DUDMA, diurethane dimethacrylate; EBPDMA, ethoxylated bisphenol-A dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] Prepolymer includes monomer, glass filler and ytterbium fluoride.
Note: Material information as supplied by manufacturer.

direct posterior restoration have been targeted as alternatives to the incremental layering technique. Hence, the overall evaluation of the mechanical properties and polymerization shrinkage of various strategic composites including highly filled flowables, bulk-fill flowables, and bulk-fill nonflowable composites is mandatory.

This study evaluated and compared the recently developed resin composites that are targeted as an alternative to composite restoration with the incremental layering technique, regarding their polymerization behavior and DOC. The following null hypotheses were evaluated: 1) There would be no differences in polymerization shrinkage behavior (including shrinkage strain/stress) between bulk-fill composites and conventional composites; and 2) there would be no differences in DOC between tested resin composites.

METHODS AND MATERIALS

Six brands of resin composite materials were analyzed. The chemical composition and manufacturers' information about the materials are listed in Table 1.

Linear Polymerization Shrinkage Measurement

The linear polymerization shrinkage of the composite specimens was measured using a custom-made linometer (R&B Inc, Daejeon, Korea). A fixed amount of composite was pressed between a glass slide and aluminum disk to produce specimens 1.5 mm in thickness and 4.5 mm in diameter. The tip of the linear variable differentiated transducer (LVDT) sensor (R&B Inc) was placed on the center of the glass slide with constant pressure and was set to the zero point. The light-curing unit was positioned on the custom-made light-curing unit station to ensure a constant 2-mm distance from the glass slide. An LED light-curing unit (Bluephase, Ivoclar Vivadent) was used at a light intensity of 700 mW/cm^2. The specimen was then polymerized for 40 seconds. During the light curing, the displacement distance of the disk was measured every 0.5 seconds for 120 seconds; the displacement was caused by the linear shrinkage of the composite material.

Polymerization Shrinkage Stress Measurement

The polymerization shrinkage stress of the composite specimens was measured using a custom-made device (R&B Inc). To do so, 0.3 g of the composite was carried to the acrylic disk of the measuring device. The steel rod of the device was positioned 1 mm above the acrylic disk to ensure a constant thickness of the specimen. The specimen was then polymerized for 40 seconds. As it was light cured toward the light source, the polymerization shrinkage stress of the composite specimen was measured by a load cell connected to the metal rod and computer. The data were recorded every 0.5 seconds for 180 seconds.

Depth of Cure by Vickers Microhardness

An opaque poly-acrylic mold (Dentsply Caulk), 4 mm long with an internal diameter of 4 mm, was used to prepare the composite specimens. The mold was placed on a glass slide covered with a Mylar strip, then the composite was filled in bulk for each

Table 1: Extended.

Product (Code)	Filler System	Filler Load (wt%/vol%)	Flexural Modulus (GPa)
Tetric N-Flow (TF)	Barium glass, ytterbium fluoride, and silica	63.8/43	5.3
SDR (SDR)	Barium aluminofluoride borosilicate glass	68/44	5.0
Venus Bulk Fill (VBF)	Barium aluminofluoride borosilicate glass, ytterbium fluoride, and silica	65/38	3.6
G-aenial Universal Flo (GUF)	Silica, strontium glass	69/50	7.95
Filtek Supreme Ultra (FS)	Zirconia/silica	78.5/63.3	11
Tetric N-Ceram Bulk Fill (TBF)	Barium alumino silicate glass, prepolymer filler[a]	80 (including 17% prepolymers)/60	4.5

material. The upper surface of the mold was filled with composite and was covered with a Mylar strip, followed by a glass slide. Then, the specimen was polymerized for 20 seconds, keeping the tip of the light-curing unit in contact with the 1.2-mm-thick glass slide to ensure a constant distance from the specimen. After polymerization, each specimen was removed from the mold. The specimens were stored in distilled water for 24 hours at room temperature. Subsequently, the top and bottom surface hardness of each 4-mm high specimen were measured using the Vickers microhardness instrument (HMV-2, Shimadzu, Kyoto, Japan). The measuring indenter, the Vickers pyramid, was pressed to the composite specimen using a load of 4.903 N for five seconds. The surface Vickers hardness (HV) was measured at three points of each specimen to minimize measurement errors within a specimen. The DOC, usually acknowledged as the thickness of the composite that is adequately polymerized or rather as the depth where HV equals the surface value multiplied by an arbitrary ratio, usually 0.8 (HV-80%), was calculated.[21,22] Therefore, each specimen HV of the lower surface was compared with the upper surface value and was noted when it dropped below HV-80%.

Statistical Analysis

The results of the present study were analyzed by using SAS 9.2 (SAS Inc, Cary, NC, USA). A one-way analysis of variance was applied to examine the significance of the differences in polymerization shrinkage strain occurring in 120 seconds and stress in 180 seconds. Pearson correlation analysis was used to compare the correlation between polymerization shrinkage strain and stress. The differences in microhardness between the top and bottom surfaces within each material were compared using paired *t*-tests. Scheffé and Bonferroni comparison tests were used to isolate statistical significance at the 95% confidence level.

RESULTS

Linear Polymerization Shrinkage Measurement

The patterns of linear polymerization shrinkage of the six composite materials after photo-initiation are presented in Figure 1. The amount of polymerization shrinkage of the composite specimens after photo-initiation decreased in the following order: Groups TF and GUF > VBF > SDR > FS and TBF ($p<0.05$). In all groups, the shrinkage graph curve was steep in the first 20 seconds, which coincides with the polymerization time, followed by a gradual increase.

Polymerization Shrinkage Stress Measurement

The patterns of shrinkage stress of the six composite materials after photo-initiation are shown in Figure 2. The polymerization shrinkage stress of six composite groups decreased in the following order:

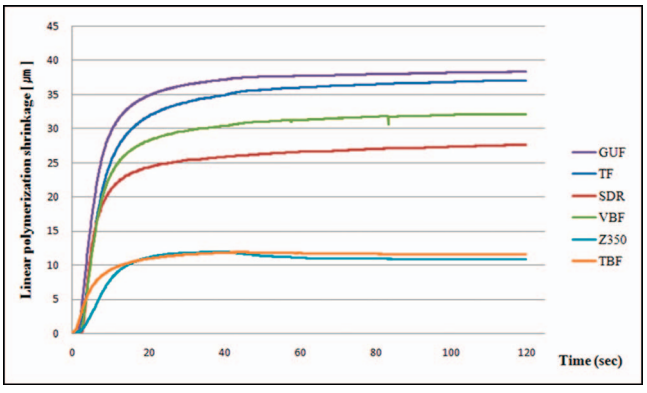

Figure 1. *Comparison of the linear polymerization shrinkage, average curves (n=8).*

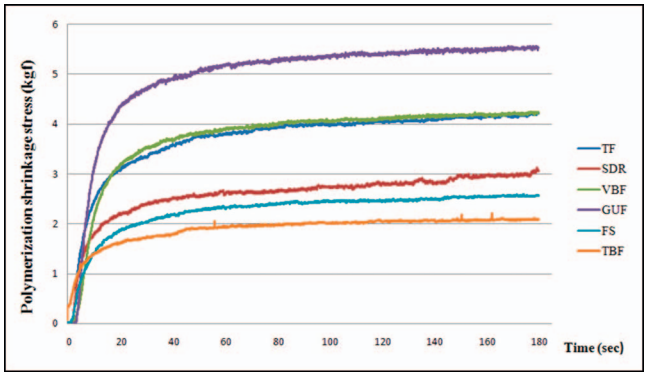

Figure 2. *Comparison of the polymerization shrinkage stress, average curves (n=8).*

Groups GUF > TF and VBF > SDR > FS and TBF ($p<0.05$).

Pearson correlation analysis showed that with respect to the shrinkage strain, shrinkage stress correlated significantly ($r=0.890$). The mean amount of linear shrinkage and the polymerization shrinkage stress for each test group are summarized in Table 2.

Depth of Cure by Vickers Microhardness

The statistical analysis of HV on the top and the bottom surfaces and the bottom-to-top ratio for each test group are presented in Table 3. All of the composites except VBF showed significantly lower HV values for the bottom compared with the top surface ($p<0.05$). Among the bulk-fill composites, the bottom surface HV of SDR and VBF, which were the bulk-fill flowables, exceeded HV-80%. However, TBF and GUF, which were bulk-fill nonflowable and highly filled flowable, respectively, failed to reach HV-80%.

Table 2. *Mean (Standard Deviation) Amount of Linear Shrinkage and Polymerization Shrinkage Stress for Each Test Group (n=8)*

Material	Linear Shrinkage (μm)	Shrinkage Stress (kgf)
TF	35.75 (2.71)A[a]	4.02 (0.37)A
SDR	25.36 (1.49)B	3.08 (0.16)B
VBF	32.14 (1.75)C	4.34 (0.35)A
GUF	38.38 (1.28)A	5.59 (0.41)C
FS	11.13 (1.15)D	2.48 (0.15)D
TBF	11.57 (1.48)D	2.09 (0.20)D

Abbreviations: FS, Filtek Supreme Ultra; GUF, G-aenial Universal Flo; TBF, Tetric N-Ceram Bulk Fill; TF, Tetric N-Flow; VBF, Venus Bulk Fill.

[a] *The measured linear shrinkage and the shrinkage stress were within 120 and 180 seconds, respectively. Different small cap letters indicate statistical differences between the groups (p<0.05). The Pearson correlation coefficient between shrinkage strain × stress was 0.890 (r=0.890, p=0.000).*

Table 3. *Comparison of Vickers Surface Hardness (HV) of Top and 4-mm Bottom, as well as Depth of Cure (HV-80%), for Each Test Group (n=8)*

Material	Top	4-mm Bottom	Bottom-to-Top Ratio
TF	35.36 (4.62)A	26.36 (6.90)B	0.74
SDR	32.14 (1.42)A	30.28 (1.73)B	0.94
VBF	30.55 (1.17)A	29.95 (1.16)A	0.98
GUF	48.54 (5.39)A	23.75 (1.51)B	0.49
FS	87.30 (6.41)A	67.21 (4.96)B	0.77
TBF	49.05 (3.82)A	37.83 (5.73)B	0.77

[a] *The samples were light polymerized for 20 seconds and stored for 24 hours at room temperature in distilled water. HV is detailed in mean and standard deviations. Different small cap letters indicate statistical differences between the top and 4-mm-depth bottom HV (p<0.05). A ratio of bottom-to-top surface microhardness over 0.80 indicates adequate DOC.*

DISCUSSION

In the current study, the polymerization shrinkage behavior and mechanical properties of recently introduced resin composites including highly filled flowables, bulk-fill flowables, and bulk-fill nonflowable composite were investigated and compared with conventional resin composites.

The flowable composites SDR and VBF, which are intended to bulk-fill, showed lower polymerization shrinkage than the conventional flowable composite. The nonflowable composites, TBF and FS, presented no significant differences in polymerization shrinkage (Figures 1 and 2). Thus, the first null hypothesis was partially rejected.

The two nonflowable composites (TBF and FS) showed significantly lower linear polymerization shrinkage compared with the flowable composites (Figure 1). In general, the flowable composite had a lower inorganic filler content and higher volume of resin matrix as compared with the nonflowable composite, and it usually exhibited a greater amount of polymerization shrinkage.[23] The flowable composites intended for bulk filling (SDR and VBF) showed lower linear shrinkage than the conventional flowable composite (TF) (Figure 1). SDR exhibited the least linear polymerization shrinkage in the tested flowables for 120 seconds, with an average of 25.36 μm (Table 2), although its shrinkage was greater than that of the two nonflowable composites. This might be attributed to the modified polymer chains of the bulk-fill flowables, which are very flexible in the pregelation phase.[24] This highly stress-relieving internal monomer might delay the gel point, which could allow more time to compensate for the shrinkage; consequently, polymerization shrinkage would be reduced.[18,19]

Polymerization shrinkage induces shrinkage stress during the curing of the resin composites. In our study, a strong correlation was observed between the shrinkage strain and stress (Table 2). The magnitude of the polymerization shrinkage stress has been found to be dependent on volumetric polymerization shrinkage and polymer elastic modulus,[9,25] whereas polymerization shrinkage is related to the degree of conversion and initial reactive group concentration.[4,26] Generally, increasing the filler load in the resin matrix results in reduction of overall shrinkage of the composite due to the reduced availability of the monomer for the curing reaction. But it also may result in a high elastic modulus of the material, which can lead to high shrinkage stress.[14,27] The present results of the shrinkage stress test showed an inverse relationship between the filler load and flexural modulus (Table 1). Concerning the polymerization shrinkage stress, which relies on the volumetric shrinkage and elastic modulus, the order of the stress value of our study was similar to the order of the estimated shrinkage strain value multiplied by each material's flexural modulus. These results coincide with those of a previous study, which hypothesized that a relevant influence of the material's stiffness on stress development was present.[28]

Of the flowable composites, SDR showed the least polymerization shrinkage stress, although it has a relatively high filler load and elastic modulus (Table 1). The modified matrix containing the shrinkage modulator might incorporate it to control the polymerization kinetics. Among the tested bulk-fill materials, TBF, which is a bulk-fill nonflowable composite, showed the least polymerization shrinkage stress. Several factors might have affected the results. First, this material contained a shrinkage stress reliever, which is a special filler functionalized with silane.[29] The manufacturer stated that the shrinkage stress reliever features a lower modulus of elasticity so that it acts like a microscopic spring, attenuating the forces generated during shrinkage.[29] Second, the material included prepolymerized fillers. Resin composites typically show a relatively low elastic modulus with the use of prepolymerized filler particles.[30]

Microhardness has been suggested as a way to examine the DOC of photo-activated resin composite. According to Bouschlicher,[31] a value over 0.80 in bottom-to-top surface microhardness indicates adequate DOC. The HV values are highly dependent on the size, weight, and volume of the filler particles as well as on the chemical composition of the composite when the test instrument produces larger indentations than the size of the filler.[32] Consequently, the HV values in our study present the average microhardness of the fillers and matrix, and for this reason, the HV value should not be considered a mechanical property and should be compared only within the same material.

In our study, the bottom surface HV values of SDR and VBF, which are bulk-fill flowables, exceeded HV-80%. The HV values of the bottom-to-top ratio of TBF, a bulk-fill nonflowable, and GUF, a highly filled flowable, were less than 0.80 (Table 3). Thus, the second null hypothesis was partially rejected.

The favorable DOC results of SDR and VBF might be attributed to the translucent matrix being highly conducible to light transmission and the incorporation of a functional photoactive group in the methacrylate matrix.[33] Previous studies[19,34] reported that bulk-fill flowables exhibited large filler size with dominant polygonally shaped features compared with conventional flowable resin composites, as seen with a scanning electron microscope. The filler load was slightly increased, but the filler-matrix interface was assumed to be decreased, due to the bigger size of the filler particle. Hence, it allows more curing light to transmit through the composite and improve the DOC.

TBF, a bulk-fill nonflowable composite, also contains a translucent filler and matrix that allow the light to pass through the material.[29] In addition, it includes Ivocerin (Ivoclar Vivadent), which is described as a germanium-based photo-initiator. According to the manufacturer, Ivocerin has a higher photo-curing activity than camphorquinone, due to its higher absorption in the region between 400 and 450 nm.[35,36] Furthermore, it can be used without the addition of an amine as coinitiator and forms at least two radicals able to initiate the radical polymerization; thus, it is more efficient than camphorquinone/amine systems with only one radical having that capability.[36,37] However, in our study, TBF presented no difference in the HV value of the bottom-to-top ratio compared with the conventional nonflowable composite, FS (Table 3). A recent study[7] was consistent with our result; it investigated the DOC of several resin composites including TBF using the ISO 4049 method and 80%-HV depth method. The author reported that the TBF specimens showed a low DOC, calculated from the bottom-to-top surface microhardness; this might have resulted in the hardness of TBF, which dropped drastically after the measurement of the superficial surface (0.1 mm).

Although the bulk-fill flowables, SDR and VBF, are indicated for restoration in bulk up to a 4 mm thickness, the manufacturers commonly recommend that these materials be covered with a 2-mm-thick capping layer by using conventional nonflowable composites.[24,37] This step is mandatory not only for reinforcing the surface hardness but also for preventing subsequent water sorption of the composite material. Recent research indicated that the composites intended for bulk fill, including SDR and VBF, are more susceptible to water deterioration in comparison with conventional composites, causing creeping deformation of the composites.[38] SDR and VBF commonly incorporate UDMA instead of bisphenol A-glycidyl methacrylate (Bis-GMA), and the matrix contents are increased to control the consistency.[18] A high-content UDMA matrix exhibits low viscosity that contributes to the void-free bulk-fill restoration. Yet it is also known as a high water-sorptive composite matrix compared with Bis-GMA or triethyleneglycol-dimethacrylate (TEGDMA).[39] The absorbed moisture may expand the matrix and induce crazing and hygroscopic expansion; it could elute the residual monomers, resulting in dimensional change of the composite restoration, and weaken the mechanical properties.[40,41] Furthermore, the results of the present study showed that the polymerization shrinkage of bulk-fill flowables was higher than that of the conventional nonflowable composite. Considering these limitations, their use as the first increment in Class II restorations as a dentin and proximal enamel replacement might result in poorer physical properties than restoration with a conventional composite. To minimize the configuration factor and restorative water sorption, the inner core of the cavity should be filled with a bulk-fill flowable first, and the placement of the conventional nonflowable composite on the outer capping layer would result in better restorative integrity in a Class II restoration. Until now, no study has been available on their use in Class II restorations without a capping layer, so this needs further investigation.

GUF was not intended to be placed in one bulk-increment, but was targeted to alternate with the resin composite for mild to moderate cavity restoration with flowable composite texture. GUF has been promoted for its unique consistency, called "injectable composite," with a glossy surface similar to that of microfill resin composite.[42] The manufacturer suggested that it could be applied not just as a cavity liner or for a small cavity but also for a larger cavity or stress-bearing area in a posterior tooth because its mechanical properties are comparable with those of conventional nonflowable composites. However, the polymerization shrinkage of GUF exceeds not only that of nonflowable composites but also that of flowable composites (Table 2). Regarding DOC, GUF exhibited the lowest HV value at the bottom surface (Table 3). Thus, its use as the alternative for conventional resin composites in an extensive posterior cavity could cause restorative failure, and it is relevant to limit the clinical indications with careful consideration.

Despite the limitations of this study, bulk-fill flowables (SDR and VBF) were properly cured in 4-mm increments, but they showed more shrinkage than conventional nonflowable composite. The bulk-fill nonflowable (TBF) showed comparable shrinkage to that of conventional nonflowable composite, but it was not sufficiently cured in 4-mm increments. The highly filled flowable (GUF) revealed, due to its polymerization shrinkage and DOC, its limitation as an alternative to conventional nonflowable composite. Further study of real restorations and long-term clinical evaluation are required for final evaluation of the suggested results.

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Conflict of Interest

The authors 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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