Polymerization shrinkage, microhardness and depth of cure of bulk fill resin composites

Fabio Antonio Piola RIZZANTE1, Jussaro Alves DUQUE2, Marco Antônio Húngaro DUARTE2, Rafael Francisco Lia MONDELLI1, Gustavo MENDONÇA3 and Sérgio Kiyoshi ISHIKIRIAMA2

1 Department of Comprehensive Care, School of Dental Medicine, Case Western Reserve University, 2124 Cornell Rd, 44106, Cleveland, OH, USA
2 Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Al Dr. Otávio Pinheiro Brisolla, 9-75, 17012-901, Bauru, SP, Brazil
3 Department of Biologic and Material Sciences, Division of Prosthodontics, University of Michigan School of Dentistry, 1011 N University Ave, Ann Arbor, MI 48109, Ann Arbor, MI, USA
Corresponding author, Fabio Antonio Piola RIZZANTE; E-mail: fap17@case.edu

The present in vitro study assessed the polymerization shrinkage/PS, Knoop microhardness/KHN and depth of cure/DC of 9 different resin composites: Filtek Bulk Fill Flowable (FBF), Surefill SDR flow (SDR), Xtra Base (XB), Filtek Z350XT Flowable (Z3F), Filtek Bulk Fill Posterior (FBP), Xtra Fill (SF), Tetric Evo Ceram Bulk Fill (TBF), Admira Fusion Xtra (ADM), and Filtek Z350XT (Z3XT). PS was assessed with a µ-CT machine, scanning 64 mm³ samples (n=8) before and after 20 s curing. KHN and DC were performed with a microhardness tester (n=8 for each group) right after 20 s light curing, with 3 readings per depth at every 0.5 mm. Low viscosity resin composites showed lower KHN values when compared with high viscosity resins. Z3XT showed the highest microhardness among the tested resin composites. Z3XT and Z3F showed lower DC when compared with bulk fill resin composites. All bulk fill resin composites presented depth of cure higher than 4.5 mm and similar or lower PS than conventional resin composites.

Keywords: Bulk fill resin composite, Depth of cure, Low viscosity resin composite, Composite materials, Micro-computed tomography

INTRODUCTION

Resin composite restorations have become more popular with the development of new adhesive and resin composite systems, as well as new filling techniques. The polymerization shrinkage is an inherent occurrence to resin-based materials, and can generate failures at the adhesive interface. The amount of shrinkage is dependent on the material composition and volume, and the pursuit for low shrinkage resin composites exists for a long time but, since the polymerization process is complex, laboratorial and clinical tests with new resin composite formulations and insertion techniques are necessary. Besides the increase in resin composites filler content, another attempt to reduce the polymerization shrinkage was the substitution of Bis-GMA for other monomers like silorane. This modification resulted in a low shrinkage resin composite with better color stability over time compared with dimethacrylate resin composites, but with inadequate mechanical properties.

Recently, with the development of bulk fill resin composites, the time-costing incremental technique could be substituted by a bulk increment technique. For this, manufacturers claim that the resin composite is able to control the polymerization process as well as ensure a proper depth of cure even when bigger resin composite increments are used. In order to allow larger increments insertion, the molecular base of these resin composites was changed by reduction or substitution of Bis-GMA, resulting in a lower viscosity monomer, and/or using monomers with higher molecular weight, usually based on Bis-EMA, TEGDMA, EBPDMA and UDMA monomers. In addition, incorporation of stress relievers and changes in filler content also helps to control the polymerization shrinkage. Clinically, the volumetric shrinkage can be related with cusp deflection, especially in cavities with high compliance (i.e. low thickness walls) in which the resin composite shrinkage can be directly reflected on the cavity dimensions, as well as in marginal sealing maintenance.

Volumetric shrinkage is a tridimensional phenomenon although being usually assessed through bidimensional methods and conversion/estimation of tridimensional changes. With the development of new technologies such as X-ray micro computed tomography (µ-CT), tridimensional analysis of the volumetric shrinkage might be simpler and reliable.

Bulk fill resin composites can be subdivided into two groups: the materials that can be exposed to the oral environment (usually high viscosity), with greater mechanical properties; and those that should be used as a base/liner (usually low viscosity/flowable), in which the manufacturer recommends a capping layer with conventional resin composite. These characteristics define its indications and different techniques in clinical practice, and can be partially addressed by mechanical tests. In a clinical situation, in addition to polymerization shrinkage, adequate depth of cure also consists in a major concern for use of big increments.

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Table 1 Different groups with respective composition and manufacturers

| Group | Restorative material | Composition (organic matrix, filler percentage in weight, type, and size) | Viscosity, type and increment size | Manufacturer |
|-------|----------------------|-------------------------------------------------------------------------|----------------------------------|--------------|
| ADM   | Admira Xtra Fusion   | Ormocer resin, 84% filler (no specific reference to filler size —based on silicon oxide) | High viscosity, bulk fill, up to 4 mm | Voco, Cuxhaven, Germany |
| FBP   | Filtek Bulk Fill Posterior | AUDMA, UDMA and 1, 12-dodecanol-UDMA, 76.5% filler (0.004 to 0.1 µm —based on silica, zirconia and ytterbium trifluoride) | High viscosity, bulk fill, up to 5 mm | 3M ESPE, St Paul, MN, USA |
| TBF   | Tetric Evo Ceram Bulk Fill | Bis-GMA, UDMA, 78% filler (0.2–0.7 µm average size —based on barium aluminium silicate glass, plus prepolymerized microfilled composite (“isofiller”), ytterbium fluoride and spherical mixed oxide) | High viscosity, bulk fill, up to 4 mm | Ivoclar Vivadent, Schaan, Liechtenstein |
| XF    | X-tra Fil            | Bis-GMA, UDMA, TEGDMA, 86% filler (no reference to filler size —based on barium aluminium silicate glass) | High viscosity, bulk fill, up to 4 mm | Voco |
| ZaXT  | Filtek Z350XT        | Bis-GMA, Bis-EMA, UDMA, TEGDMA, 82% filler (0.004 to 10 µm —based on silica and zirconia) | High viscosity, conventional, up to 2 mm | 3M ESPE |
| FBF   | Filtek Bulk Fill Flowable | UDMA, BISGMA, Bis-EMA, Proclylates resin, 64.5% filler (0.01 to 5 µm —based on silica, zirconia and ytterbium trifluoride) | Low viscosity, bulk fill, up to 4 mm | 3M ESPE |
| SDR   | Surefil SDR flow     | Modified UDMA, EBPADMA, TEGDMA, 68% filler (4 µm average size —barium and strontium alumino-fuoro-silicate glasses) | Low viscosity, bulk fill, up to 4 mm | Caulk Dentsply, York, PA, USA |
| XB    | X-tra Base           | UDMA, Bis-EMA, 75% filler (no reference to filler size —based on barium aluminium silicate glass) | Low viscosity, bulk fill, up to 4 mm | Voco |
| Z3F   | Filtek Z350 flow     | Bis-GMA, Bis-EMA, TEGDMA, 65% filler (0.004–10 µm —based on silica and zirconia) | Low viscosity, conventional, up to 2 mm | 3M ESPE |

In addition, use of bigger increments usually are associated with a higher C-factor, which can increase shrinkage stress development. Manufacturers used different approaches to ensure a proper polymerization in deep cavities such as incorporation of new and more reactive photoinitiators, reduction of resin composite’s opacity (through filler content changes such as increase in particles size or reduction of the mismatch between fillers and organic matrix refractive indexes). The increased translucency for some resin composites can be observed during clinical use and ensures adequate physico-mechanical properties and long term maintenance of restoration.

The objectives of the present study were to assess the microhardness and depth of cure of different resin composites (using a microhardness tester), as well as the polymerization shrinkage using a tridimensional method (µ-CT). The null hypotheses tested were: 1) There would be no differences in surface microhardness when comparing the different resin composites; 2) Bulk fill resin composites would not present higher depth of cure than conventional/non-bulk-fill resin composites and 3) Bulk fill resin composites would not present lower polymerization shrinkage than conventional composites.

**MATERIALS AND METHODS**

**Study design and materials**

The resin composites were evaluated in 9 levels (nine different materials, Table 1), having as response variables: microhardness and depth of cure through microhardness test, and volumetric polymerization shrinkage through µ-CT analysis.

**Methods**

1. Microhardness and depth of cure
For the Knoop microhardness evaluation, eight
specimens for each group were achieved through insertion of resin composite into a metallic mold designed in a CNC lathe (Sherline 2000, Sherline, Vista, CA, USA) controlled through the Match 3 software (Newflanged solutions, Livermore Falls, ME, USA), with 10 mm length and a central trapezoidal-shaped groove (Fig. 1A).

After resin composite insertion into the groove, a polyester strip was positioned over the top surface to standardize the surface roughness and a metallic cover was put in position (Fig. 1B). The resin composite excess was removed and the specimen was light cured during 20 s through the small window using a LED curing device (LED Blue Star 3, Microdont, São Paulo, Brazil), with wavelength from 420 to 480 nm, irradiance of 1,550 mW/cm² and radiant exposure of 31 J/cm². A Polyvinyl siloxane (PVS) mold was used to standardize the light curing unit in a perpendicular position and in contact with the polyester strip.

Following light curing, the metallic cover and polyester strip were removed (Fig. 1C). The non-polymerized resin composite was removed with aid of a #12 scalpel blade and the top surface of the specimen was analyzed using a Knoop microhardness tester (Micromet 6040, Buehler, Lake Bluff, IL, USA) in all its longitudinal extension through 3 surface readings for each 0.5 mm, separated by 400 µm. The final result for each depth was calculated as the 3 readings mean value. The first reading (surface) was considered as 0.5 mm from the specimen edge (closer to the light curing unit) and the readings were performed until microhardness values lower than 50% (comparing with the initial values) were observed. For each reading, a 50 g force was applied over the specimens' surface during 30 s, with a crosshead speed of 0.5 mm/min. The resulting impression was evaluated with the microhardness tester stereomicroscope with a 10× magnification. The longest diagonal was evaluated and each reading value was determined by automatic calculation. The depth of cure was considered adequate while the reading mean values correspond to a value equal or higher than 80% of the surface readings.

2. Volumetric shrinkage

Four cavities of 4×4×4 mm, were designed in a dual level bi-part Teflon® mold (2 cavities for each mold level). In
order to ensure proper stabilization and alignment of the mold parts, a Teflon® ring holder was also developed (Fig. 2A). The mold parts were designed with aid of the CNC lathe (Sherline 2000, Sherline). Eight specimens of each resin composite were achieved by material insertion into the mold with a total material volume of approximately 64 mm³. High viscosity resin composites were inserted with hand instruments in order to avoid air spaces formation. Low viscosity resin composites were injected directly from their respective syringes.

The resin was inserted into the cavities in a single increment, without bonding agent or any previous surface treatment. The restorative material within the Teflon® mold was scanned in a µ-CT (SkyScan 1174v2, Bruker, Kontich, Belgium), with 50 kVp and 800 µA, with 648×512 resolution. The slice thickness was determined as 16 µm for a scan time of 20 min. Four samples were assessed at each scan and were prepared inside a X-ray dark room and were transported to the µ-CT chamber in a dark storage in order to avoid light interference.

The first scan allowed the assessment of the material volume before polymerization. After the first scanning, the specimens were light cured during 20 s with the LED light curing device. The light curing was performed perpendicular to the specimens' surface, being initiated with the samples located at the second level of the mold (in order to avoid any dimensional changes due to manipulation), followed by the light curing of the specimens located at the first level of the mold. Each specimen was light cured individually during 20 s and a dark paper card was used to avoid polymerization of the adjacent specimen (Fig. 2B). These procedures were followed by a second scan using the above described protocol. All obtained images were rendered in specific softwares [CT-Analyser (CTAn) and CT-volume (CTVol), Bruker] to obtain tridimensional specimen reproduction (Fig. 2E). The volumetric shrinkage was determined as the percentage difference between the initial (before light curing) and final volume (after light curing).

Statistical analysis

After Shapiro-Wilk normality test, all data were analyzed through one-way Anova followed by Tukey’s test. For all the statistical analysis, 5% was adopted as significance level (p<0.05). In addition, linear correlation tests were performed based on the manufacturers’ information about the filler content in order to estimate its association with the depth of cure and volumetric shrinkage for both high and low viscosity resin composites.

RESULTS

All low viscosity resin composites showed lower values for surface microhardness (FBF presented the lowest value) than high viscosity resin composites. Among the high viscosity materials, Z3XT presented the highest microhardness values, followed by XF, FBP/TBF, and ADM. For depth of cure (80% of initial microhardness), conventional resin composites showed lower depth of cure when compared with the bulk fill resin composites. All bulk fill resin composites presented depth of cure values higher than 4.5 mm. SDR and XB showed the highest values (Table 2).

The initial measured volume and shrinkage after light curing are listed in Table 3. All groups presented similar initial volumes, with low standard deviation. All high viscosity bulk fill resin composites showed lower volumetric shrinkage when compared with Z3XT and Z3F. All low viscosity bulk fill resin composites showed results similar to Z3XT and Z3F. Z3F presented the highest shrinkage values while XF presented the lowest shrinkage values.

In the Fig. 3, it is possible to observe a moderate correlation between microhardness and filler content (R²=0.5708). Figure 4 shows a strong correlation between filler content and volumetric shrinkage for all groups (R²=0.6918), which is even stronger when Z3XT is not considered (R²=0.9081).

Table 2  Surface microhardness (Knoop) and depth of cure (80% of initial microhardness)

| Group | Surface microhardness (KHN) | Depth of cure (80%)(mm) |
|-------|-----------------------------|-------------------------|
| ADM   | 37.36 (5.15) D              | 5.44 (0.62) BC          |
| FBP   | 49.60 (2.40) C              | 5.00 (0.46) C           |
| TBF   | 50.89 (5.17) C              | 4.88 (0.44) C           |
| XF    | 74.34 (10.70) B             | 5.38 (0.69) BC          |
| Z3XT  | 89.37 (6.77) A              | 2.63 (0.23) E           |
| FBF   | 16.21 (2.28) E              | 5.63 (0.35) BC          |
| SDR   | 22.05 (2.07) E              | 6.94 (0.42) A           |
| XB    | 31.95 (2.66) D              | 6.13 (0.69) B           |
| Z3F   | 33.31 (3.15) D              | 3.63 (0.23) D           |

Different letters mean statistically significant difference between each material in the same column (inter-groups comparison, p<0.05)
Table 3  Initial volume (mm$^3$) and volumetric shrinkage (%)

| Group | Initial volume (mm$^3$) | Volumetric shrinkage (%) |
|-------|-------------------------|--------------------------|
| ADM   | 61.18 (2.01) A          | 1.24 (0.18) AC           |
| FBP   | 61.47 (1.07) A          | 2.19 (0.47) B            |
| TBF   | 61.12 (1.68) A          | 1.75 (0.12) BC           |
| XF    | 62.23 (1.10) A          | 0.84 (0.36) A            |
| Z3XT  | 60.88 (1.07) A          | 3.07 (0.61) A            |
| FFB   | 60.17 (2.23) A          | 3.34 (0.6) DE            |
| SDR   | 61.94 (1.51) A          | 3.36 (0.62) DE           |
| XB    | 63.31 (2.23) A          | 3.11 (0.16) DE           |
| Z3F   | 62.52 (1.17) A          | 3.84 (0.23) E            |

Different letters mean statistically significant difference between each material in the same column (inter-groups comparison, $p \leq 0.05$)

DISCUSSION

Changes in both organic and inorganic matrixes can have influence on resin composites mechanical and physical properties. Although several resin composites are classified as bulk fill materials, these materials show very heterogeneous behaviors, being important to assess their properties\textsuperscript{26}. Very heterogeneous results could be observed for the surface microhardness test, even when comparing the high viscosity bulk fill resin composites. Such results are concerning because the high viscosity resin composites are indicated to restore and be exposed at occlusal surfaces since they might present adequate mechanical properties to clinically endure the occlusal and masticatory challenges. Conventional Z3XT (89.37±6.77) and bulk fill XF (74.34±10.7) showed the highest microhardness values and the highest filler contents according with the respective manufacturers' information. TBF (50.89±5.17) and FBP (49.60±2.40) showed similar microhardness values. Interestingly, despite also being a high viscosity resin composite, ADM presented a low microhardness value (37.36±5.15). Such differences for ADM might be explained by the differences in the Ormocer\textsuperscript{®} matrix, which is based on organically modified ceramics (organic polymers linked with the inorganic matrix), instead of being based on methacrylates\textsuperscript{27}, as well as in its modified filler content (based only on silicon oxide). The lower mechanical properties for Ormocer-based resin composites were reported by other authors\textsuperscript{28,29}. The results for Z3XT and TBF are similar to the ones reported by Rodriguez et al.\textsuperscript{30}.

As expected, microhardness' results for lower viscosity resin composites were lower when compared with high viscosity resin composites\textsuperscript{22,31}, with Z3F (33.31±3.15) and XB (31.95±2.66) showing the highest
values, followed by SDR (22.05±2.07) and FBF (16.21±2.28). Despite presenting similar filler content as FBF and SDR22, Z3F (65%)23 showed similar microhardness values as XB, which presents a higher filler content (75%)26,32. This might be explained by the use of nanoparticles and nanoclusters in Z3F, claimed by the manufacturer to increase the resin composite’s resistance, due to a better distribution and interaction of the particles. The presented results clearly show the manufacturers’ indications for clinical use. The high viscosity resin composites showed very heterogeneous results, but definitely higher than the low viscosity resin composites, which might need a capping layer. The heterogeneous values observed for high viscosity resin composites can rely partially on the lower elastic modulus observed for some of the bulk fill resin composites, associated with the different filler contents and organic matrix composition21,22,34-36. Thus, it might be important to consider each resin composite according with each clinical indication.

An increase in microhardness values is expected as the filler content increases22,30,31,35,37. In the present study, considering the filler content reported by the manufacturers, it was possible to observe a tendency of moderate correlation between microhardness and filler content, showing an increase in the microhardness as the amount of filler increases, especially when comparing low and high viscosity resin composites (R²=0.5708, Fig. 3). It should be noted that, probably the correlation was not higher due to the different organic matrix of ADM, and the higher filler content of XB. Although XB is a low viscosity resin composite, it presents high microhardness values for this class of resin composites (Table 2). Since the different resin composites showed very different values for microhardness, the first null hypothesis was rejected.

Using the microhardness evaluation, it was also possible to determine the resin composites’ depth of cure, through longitudinal test, being considered adequate when values equal or higher than 80% are achieved (comparing with the first readings mean values/upper surface readings)25,38. All the tested bulk fill resin composites showed adequate polymerization at least up to 4.5 mm; being even higher for low viscosity bulk fill resin composites (at least 5 mm). The conventional resin composites presented adequate depth of cure up to 2.63 mm (Z3XT) and 3.63 mm (Z3F), which explains why they should not be used in big increments. Similar results were described in the literature22,25,30,36-39. Since all bulk fill resin composites showed adequate polymerization at least up to 4.5 mm (versus 3.5 mm from conventional Z3F and 2.5 mm from Z3XT), the second null hypothesis was rejected (difference between conventional and bulk fill resin composites).

Another interesting finding with the longitudinal microhardness is the increase in the microhardness values up to 2 mm before starting to decrease. This was reported before in the literature and, since the oxygen inhibited layer corresponds to 20–50 µm and was avoided in the present study (polymerization in contact with polyester strips), such behavior can be related with the shrinkage of the resin composite towards the center in non-bonded models. The shrinkage towards the center of the specimen might result in a denser polymer than on the edges, explaining the results observed in the present study and in the literature22,29.

Except for the TBF, which relies on a new dibenzoyl germanium compound (Ivocerin), which absorbs light between 370 and 460 nm and is claimed to be more reactive, the other bulk fill resin composites show the same camphorquinone/amine initiator system, probably relying in changes in the filler contents and higher translucency for improvements on the depth of cure22,26,34,35,41,43. A more translucent resin composite can be achieved through reduction in the filler content, use of bigger particles size and study of the interaction between the fillers and organic matrix refractive indexes21,22. The increased translucency of the bulk fill resin resin composites can be observed in clinical situation, in which the necessity of a capping layer comes from both mechanical and esthetical properties.

Another important question when polymerizing big increments consists in the polymerization shrinkage. Usually, volumetric shrinkage is assessed through bidimensional measurements and conversion of values estimating the volume16,17. With the use of new technologies such as µ-CT, it is possible to assess the real volumetric shrinkage of the resin composites19. It can be observed that the conventional resin composites and the low viscosity bulk fill resin composites showed similar volumetric shrinkage (around 3.2%). The highest value was observed for the low viscosity Z3F (3.84±0.23%), while the high viscosity bulk fill resin composites showed lower but heterogeneous results, ranging from 0.84±0.36% (XF) up to 2.19±0.47% (FBF). Such results are in agreement with the literature, which reported values between 1–3% for high viscosity resin composites and up to 6% for low viscosity composites30. In a recent study by Yu et al.39, the results for SDR and TBF were similar to the results of the present study despite the different methods, validating the use of µ-CT. It is important to note that even some bulk fill resin composites show similar shrinkage values when compared with the conventional Z3XT (3.07±0.61), this is not necessarily related with an increased shrinkage stress since it depends also on the elastic modulus and development, polymerization kinects, among other factors38,42-44.

It is interesting to observe that the low viscosity bulk fill resin composites showed similar shrinkage values when compared with the conventional high viscosity resin composite. This fact demonstrates that the mechanisms used in the bulk fill resin composites in order to reduce the shrinkage, such as introduction of monomers with higher molecular weight (reducing the number of reactive sites per volume)45 and increase in filler content, were able to effectively reduce the shrinkage46. In fact, the results of the present study show a tendency of strong correlation between filler content and volumetric shrinkage (R²=0.6918, Fig. 4). Similar results were reported by Al sunbul et al.46. Z3XT can
be considered as an outlier and this can be explained by the lower shrinkage observed for all high viscosity bulk fill resin composites (Table 3) and, repeating the linear regression analysis without Z3XT resulted in R²=0.9081. Since the shrinkage values varied widely, the third null hypothesis was also rejected.

The use of µ-CT for tridimensional volumetric shrinkage assessment consists in a reliable and simpler test when compared with other methods such as linear shrinkage assessment and estimation of the tridimensional changes, dilatometer based methods, among others. In addition, the use of a Teflon® mold instead of a tooth cavity avoids possible interferences (“bonding”) between the resin composite and the adjacent walls/structures.

All bulk fill resin composites presented equal to lower polymerization shrinkage when compared with conventional resin composites. These properties, associated with the increased depth of cure are interesting for clinical application, with possibility of easier and faster restoration placement. Nevertheless, the low microhardness values for some resin composites can be concerning in some clinical situations, especially regarding occlusal cavities. Thus, further studies are necessary assessing the restorative properties of such resin composites.

CONCLUSIONS

Considering the limitations of the present study, it is possible to conclude:

- The surface microhardness is widely variable between the tested resin composites. No bulk fill resin composite achieved the same surface microhardness as Filtek Z350XT.
- All tested bulk fill resin composites showed proper depth of cure up to at least 4.5 mm being indicated for bulk placement, and presenting higher depth of cure than conventional resin composites.
- All tested bulk fill resin composites showed similar or lower volumetric shrinkage when compared with conventional resin composites.

CONFLICTS OF INTEREST

The authors report no conflicts of interest.

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