Bonding performance and mechanical properties of flowable bulk-fill and traditional composites in high c-factor cavity models

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Abstract

Objectives: The aim of this study is to evaluate bond strength (BS), shrinkage stress (SS), flexural strength (FS), and elastic modulus (E) of three flowable bulk fill in comparison with conventional composites.

Materials and Methods: Three bulk fill (Filtek Bulk Fill Flow, Surefil SDR, X-tra Base) and three conventional composites (Filtek Z250 XT, Grandioso, Dentsply TPH3) were used. For BS, conical cavities (n = 10) were prepared in bovine dentine and restored with materials and were analyzed through push-out test in a universal testing machine (UTM). For FS/EM, 60 (n = 10) bar specimens (7 mm × 2 mm × 1 mm) were prepared and evaluated with a UTM. SS was measured in UTM coupled to an extensometer (n = 5). The data were statistically evaluated using one-way ANOVA/Tukey tests (P < 0.05).

Results: Conventional composites showed higher E when compared to bulk-fill composites. Regarding FS, they showed similar results, except for (XBF) Xtra Bulk Fill that was inferior. SS and BS of bulk-fill composites were significantly lower and higher than conventional composites, respectively, except for XBF, which showed similar BS to conventional ones.

Conclusions: Flowable bulk-fill composites, except XBF, showed higher BS, lower SS, similar FS, and lower E when compared to conventional ones.

Keywords: Composite; fracture mechanics; light-cured resin composite; push-out bond strength

INTRODUCTION

Improvements in the physical and mechanical proprieties of conventional resin-based composites allow them to be the first choice as direct restorative material in general dentistry. However, the presence of high C-factor in tooth preparations such as those found in Black’s classification (Class I and II), as well as the limited light-curing depth of conventional resin composites, require that these materials must be placed by incremental technique.⁰

In order to simplify the restorative procedures in the posterior segment, bulk-fill resin-based composites were developed. Indeed, this material can be inserted in one single increment of 4–5-mm thickness.¹⁰

Nowadays, moldable and flowable bulk-fill composites are commercially available. Flowable bulk-fill composites are used as base for restorations because their greater flow might promote a better adaptation to the cavity walls, besides decreases the possibility of incorporation of bubbles.³ However, these materials require coverage with conventional restorative composites at the occlusal surface,

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How to cite this article: Freitas Chaves LV, Sousa Lima RX, Azevedo Silva LJ, Bruschi Alonso RC, Geraldeli S, Dutra Borges BC. Bonding performance and mechanical properties of flowable bulk-fill and traditional composites in high c-factor cavity models. J Conserv Dent 2020;23:36‑41.
MATERIALS AND METHODS

Experimental design
This was a laboratory study involving the following response variables: depth of cure, SS, FS, elastic modulus, and dentin BS. The factor under study was the type of resin-based composite resin: three flowable bulk-fill (Filtek Bulk Fill [3M ESPE, St. Paul, MN, USA], Surefil SDR [Dentsply/Caulk, Milford, DE, USA], X-traBase [Voco, Cuxhaven, Germany]) and three conventional resin-based composites (Z250 XT [3M ESPE, St. Paul, MN, USA], TPH 3 [Dentsply/Caulk, Milford, DE, USA], Grandioso [Voco, Cuxhaven, Germany]) were tested. Composition of materials is listed in Table 1.

Push-out dentin bond strength tests
Specimens were produced as previously described by Sousa-Lima, et al. Briefly, 60 bovine incisors with no enamel cracks or structural defects were selected and stored in 0.1% thymol solution at 4°C for 1 week. The roots were removed at the cementoenamel junction (CEJ) using a water-cooled diamond saw coupled to cutting machine (Isomet 1000; Buehler, Lake Forest, IL, USA). A second transverse mesiodistal cut was made 4 mm from the CEJ to obtain a 4-mm thickness slab with a central void corresponding to the pulp cavity. Standardized conical cavities (4.8 mm top diameter × 2.8 mm bottom diameter × 4 mm height) were prepared with Maxicut burs (Komet Inc., Lemgo, Germany) using a handpiece under air-water cooling. The bur was replaced every 10 preparations. The cavities had C-factor of 2.5.

After cavity preparation, specimens were randomly distributed into 6 groups (n = 10) according to the restorative material [Table 1]. For all groups, the same bonding agent was used. The Single Bond Universal system (3M St Paul, Minnesota, USA) was applied according to the manufacturer’s instructions and photocured for 20 s using a poly wave light-emitting diode (LED) light-curing unit (LCU) (Bluephase G2; Ivoclar Vivadent). The bulk-fill resin-based composites were inserted in a single increment, completely filling the cavity; a polyester strip was placed on the top surface and photoactivated for 20 s. Conventional composites were incrementally inserted; the first a 2-mm increment was inserted and photoactivated for 20 s, then the second 2-mm increment was inserted and photoactivated for 20 s under a polyester strip. After 24 h, all restorations were finished/polished with Sof-Lex Pop On aluminum oxide discs (3M ESPE) and stored in water for 24 h at 37°C before evaluation of the depth of cure and BS.

Push-out BS was evaluated using a universal testing machine (Instron, model 3342; Instron, Norwood, MA, USA) using a 0.001-mm scale at a crosshead speed of 0.5 mm/min.

Table 1: Materials used in this study.

| Material/manufacturer | Resin Matrix | Filler loading |
|-----------------------|--------------|---------------|
| Filtek Z250 XT        | Bisphenol A glycol diether dimethacrylate, diurethane dimethacrylate, bisphenol polyethylene A diglycidyl ether dimethacrylate, triethylene glycol dimethacrylate | Surface-modified zirconia/silica (average size 3µm) and Non-agglomerated/non-aggregated surface-modified silica particles (20nm) - Filler loading: 82 wt% or 68 vol% |
| 3M ESPE, St. Paul, MN, USA | Diurethane dimethacrylate, Substituted dimethacrylate, | Combination of zirconia/silica (size range of 0.01 to 3.5µ) and ytterbium trifluoride filler (size from 0.1 to 5.0) - Filler loading: 64.5 wt% or 42.5 vol% |
| Filtek Bulk Fill Flow | Bisphenol A polyethylene glycol diether dimethacrylate, Bisphenol A diglycidyl ether dimethacrylate, Benztriizol, triethylene glycol dimethacrylate, ethyl 4-dimethylaminobenzoate | |
| 3M ESPE, St. Paul, MN, USA | Bisphenol a diglycidyl ether dimethacrylate, triethylene glycol dimethacrylate, bisphenol A polyethylene glycol diether dimethacrylate | Glass ceramic filler (average size 1µm) and functionalized silicon dioxide nano-particles (20-40nm) - Filler loading: 89 wt% |
| TPH 3 | Bisphenol a polyethylene glycol diether dimethacrylate, aliphatic dimethacrylate | Filler loading: 75 wt% |
| Dentsply/Caulk, Milford, DE, USA | Barium glass silanized borosilicate aluminum, barium fluoride aluminum borosilicate silanized glass, silica - Filler loading: 79 wt% or 63 vol% | |
| Surefil SDR | Polymericizable dimethacrylate resins, polymericizable trimethacrylate resins, polymericizable urethane dimethacrylate | Barium glass and Strontium glass - Filler loading: 68 wt%, 45 vol% |

*Data obtained from Manufacturers Brochures and Material safety data sheet (MSDS).
An acrylic device with a central hole was adapted to the base of the machine to position the specimens with the bottom surface (2.8 diameter) facing up. A rounded probe was adapted to the testing machine, and a compressive force was applied with a crosshead speed of 0.5 mm/min to the bottom surface of the restoration until restoration displacement. The results (in Kgf) were divided by the bonded area (57 mm²) and transformed to MPa.

**Shrinkage stress**

Briefly, poly(methylnethacrylate) rods (5 mm diameter × 28 mm or 13 mm height) were used as substrate for composites. The bonding surfaces of these rods were sandblasted, treated with methyl methacrylate monomer (JET Acrilico Auto Polimerizante, Artigos Odontológicos Clássico, São Paulo, Brazil) and covered by an unfilled bonding agent (ScotchBond Multi-Purpose Plus Adhesive; 3M ESPE), which was light-cured using a polywave LED LCU (Bluephase G2; Ivoclar Vivadent) for 10 s. The 28-mm rod was attached to the top of the machine and the 13-mm cylinder to the bottom (through a stainless steel device). This device has a hole that allows the LCU tip to be adapted in contact with the base of the 13-mm rod. The distance between treated surfaces of the rods was 4 mm for bulk-fill composites and 2 mm for conventional composites. Resin-based composites were inserted into the empty space and shaped into a cylinder according to the rod diameters and treated areas (n = 5).

An extensometer (model 2630-101, 0.1 mm resolution; Instron, Norwood, MA, USA) was attached to the rods to monitor specimen change on its height and provide feedback to the testing machine to keep the height constant. The load cell provided the corresponding force necessary to counteract the polymerization shrinkage force to maintain the specimen’s initial height. Contraction force development (N) was monitored for 20 min from the beginning of photoactivation, and maximum nominal stress (expressed in MPa) was calculated by dividing the maximum force value by the cross-sectional area of the rod. For bulk-fill composites, SS was evaluated only for 4-mm bulk increments. For conventional composites, SS was evaluated in two situations: (1) First increment: 2-mm increment placed directly between the rods; (2) Second increment: 2-mm increment placed between the rod and a prepolymerized 2-mm increment inserted in the 28-mm rod. The sum of SS of the first and second increments was considered for statistical analyses.

**Flexural strength and elastic modulus**

For these tests, bars-shaped specimens (7 × 2 × 1 mm) of the materials (n = 10 each) were prepared using a polyvinyl siloxane mold (Express XT; 3M ESPE, St. Paul, MN, USA). All composites were inserted in a single increment and photocured for 20 s using a polywave LED LCU (Bluephase G2; Ivoclar Vivadent, Schaan, Liechtenstein). The light-curing tip has a diameter of 10 mm, which allowed a single exposure to cure the specimens. After light curing, specimens were stored in dark at 37°C for 24 h before testing.

FS and elastic modulus (E) were evaluated using a universal testing machine (Instron, model 3342, Norwood, MA, USA) with a three-point bending design (distance between supports of 5 mm, cell load of 500 N, compressive loading at crosshead speed of 0.5 mm/min until fracture). The Bluehill 2 software (Illinois Tool Works, Inc., Glenview, IL, USA) was used to calculate FS and E, considering the dimensions of the specimens. Each specimen size was individually determined with a digital caliper (Mitutoyo, Brazil).

**Statistical analysis**

After being tested for normality by the Shapiro–Wilk test, the data obtained from each test were submitted to one-way ANOVA and Tukey’s test, with a global significance level of 95% (α = 0.05). Statistical analysis was performed using ASSISTAT 7.7 (Campina Grande, Brazil) Software.

**RESULTS**

Table 2 shows the results of dentin BS, SS, FS and elastic modulus (E) of all composites. Filler content and volumetric shrinkage (VS) were obtained from the manufacturer’s information (Technical Product Profiles) and are included in Table 2 to facilitate comparisons among the materials.

Regarding dentin BS, the flowable bulk-fill composite (FTB) Filtek Bulk Fill showed the highest BS, followed by Surefil...
Sdr Bulk (SDR), both showed BS significantly higher than all other tested materials. The lowest BS values were observed for GRAN, XBF, and TPH, which showed no significant difference among each other.

Regarding the SS, all flowable bulk-fill resin-based composites showed lower values than conventional composites. All flowable bulk-fill composites showed similar SS values, and all conventional composites showed similar SS values among them.

Regarding the FS, in general, the conventional resin-based composites showed higher FS than flowable bulk-fill resin-based composites, except for FTB that showed similar FS to conventional resin-based composites Z250 and GRAN. When the conventional resin-based composites Z250, GRAN, and TPH were compared, no difference was observed. Among flowable bulk-fill resin-based composites, XBF showed FS significantly lower than FTB, while SDR showed intermediate results, neither different from XBF nor from FTB.

Regarding $E$, the conventional resin-based composites Z250 and GRAN showed the highest values and significantly higher than all the flowable bulk-fill resin-based composites. Among flowable bulk-fill resin-based composites, XFB showed $E$ higher than FTB, while SDR showed intermediate results, neither different from XBF nor from FTB.

**DISCUSSION**

In this study, the null hypothesis tested that flowable bulk-fill and conventional resin-based composites would not have differences in dentin BS, and mechanical properties were rejected once significant differences among the materials were observed for BS, SS, FS, and $E$.

While conventional resin-based composites were polymerized in two 2-mm increments, flowable bulk-fill resin-based composites were polymerized in a single 4-mm increment. In this way, respecting the manufacturer’s directions, bulk fill and conventional composites show similar efficacy of polymerization in depth. Greater translucency increased photoinitiator content, or an additional photoinitiator type may be the factors responsible for effective depth of cure of bulk-fill composites even in 4-mm depth.

In this study, all composites showed $E$ lower than. However, our results corroborate with Randolph et al.\textsuperscript{[9,10]} in the sense that composites with higher filler loading showed higher $E$. The conventional composites Filtek Z250XT and Grandioso, which has the highest filler loading of the tested composites, showed the highest $E$ values and were significantly higher than TPH and all the flowable bulk-fill composites ($P < 0.05$), as observed in Table 2.

Among the flowable bulk-fill composites, the same pattern was observed and the composite with the highest filler content X-tra Base showed the $E$ higher than Filtek Bulk Fill Flow, while Surefil SDR showed intermediate results. Other studies, which also compared flowable bulk-fill composites, also showed lower $E$ for these materials when compared to conventional composites.\textsuperscript{[11]} Despite this, it is important to reassure that the flowable bulk-fill composites used in this study are indicated as cavity base, acting as a substitute of dentin. Therefore, the reduced stiffness is not a problem since a restorative composite with higher stiffness would be used over it. Still, despite the lower elastic modulus, bulk-fill composites showed FS compatible with clinical use.\textsuperscript{[11,12]}

FS has been extensively evaluated since it is a clinically relevant property for restorative materials developed to restore high-stress-bearing areas.\textsuperscript{[13]} In this study, all the tested composites showed FS higher than the limit of 80 MPa established in ISO 4049/2009 for polymer-based restorative materials indicated for restorations involving occlusal surfaces.\textsuperscript{[14]} As a matter of fact, that is in accordance with recent findings in the literature.\textsuperscript{[7]}

As it was also observed by Leprince et al.,\textsuperscript{[15]} in general, the mechanical properties of the bulk-fill composites were mostly lower compared with the conventional high viscosity material. In this way, the veneering of a bulk-fill composite restoration using a conventional composite material is essential due their lower cohesive strength and stiffness, properties extremely important for restoration of high occlusal stress areas.

When analyzing the bulk-fill resin-based composites FS findings, X-tra Base showed the lowest value (89.82 MPa), and this was statistically inferior to Surefil SDR (115.75 MPa) and Filtek Bulk Fill (135.98 MPa). The reduced FS of X-tra Base may be related to the composition of its organic phase. This material contains mainly BisEMA and it is associated with a dimethacrylate aliphatic differing from the other resin-based composites, which are mostly based on BISGMA. Previously reported findings indicate that composites containing BisEMA presented inferior FS when compared to the ones that contain BisGMA.\textsuperscript{[16]} Due to its higher molecular weight, BisEMA (568.708 g/mol) has relative lower mobility during the polymerization reaction. Such behavior makes difficult the contact of the monomers with free radicals and decreases the conversion degree. It can be speculated that it probably this affected mainly the physical-mechanical proprieties, such as FS.\textsuperscript{[15]}

On the restorative technique with resin composites, due to the polymerization shrinkage, tensions are generated and can be transmitted to the tooth/restoration interface. If these tensions are too high, they might cause ruptures at the
adhesive layer and/or the resin-dentin interface, reducing BS, increasing microleakage, elevating postoperative pain, etc. In the present study, the conventional composites demonstrated elevated SS values, when compared to bulk-fill composites [Table 2].

Polymerization stress has a strong correlation with shrinkage and an inverse correlation with elastic modulus. When the compliance of the entire in vitro testing system is high, polymerization SS is more determined by polymerization shrinkage than the elastic modulus. On the other hand, when high C-factor cavities are considered, the low compliance of the tooth substrate could intensify the influence of the composite’s elastic modulus on polymerization stress.

Boaro et al. (2010) found that a low compliance system, as used in this study, increases the influence of composite stiffness. Hence, considering this experimental design, SS is mainly affected by filler content, due to the relation between filler content and stiffness. The higher the filler content, the higher the stiffness, the higher the SS. Flowable bulk-fill composites showed elastic modulus lower than conventional composites, as described before in this study. With low E and high flexibility during the initial phase of polymerization, flowable bulk-fill composites showed significant reduction on SS when compared to conventional composites [Table 2]. The stress reduction observed herein corroborates with the results of El-Damanhoury and Platt. There might be other mechanisms responsible for reduction of SS in bulk-fill composites. For example, the appearance of new monomers, likewise the insertion of rheological modulators, such as urethane dimethacrylate, is associated with decrease on tensions generated by the polymerization phenomenon.

Finally, BS was evaluated through push-out test. Usually, the push-out test is used to evaluate the BS of endodontic cement. However, in the present study, the push-out test was adapted to evaluate the BS of restorative composites, as it was described by Sousa-Lima et al. Other BS tests such as shear, tensile, microshear, and microtensile evaluations are usually carried out to evaluate the BS of restorative composites to different substrates.

However, these tests are generally performed on flat surfaces, where the C factor is very low and SS is not orientated to the bonding interface. Hence, the great advantage of push-out test is the possibility of evaluating enamel and/or dentin BS in high C-factor tooth preparations (≈2.2, in this study). This preparation allows high-stress development directed toward the bonding area so that the entire bonding area is submitted to compressive loading at the same time, allowing the BS to be evaluated in a cavity. Alonso et al. found an inverse relationship between push-out BS and internal adaptation of composite restorations. In addition, the reliability of the push-out test in this study was confirmed by the low variability of data [Table 2].

In this test, it was observed that the bulk-fill composites Filtek Bulk Fill and Surefil SDR showed significantly higher BS than other tested composites, what can be related with the lower E and lower SS showed by these materials, corroborating the findings of Braga et al. (2005) who found that composites with lower SS resulted in higher BS values.

The stress reduction of Surefil SDR has been already reported by Goracci et al. Surefil SDR features a photoactive group embedded in urethane-based methacrylate monomers and capable of interacting with camphorquinone. Such interaction can modulate curing and control stress development. The modulator is supposed to increase monomer flexibility and thus to contribute to polymer matrix relaxation, evidently leading to lower shrinkage force. Furthermore, the high-molecular weight of the monomer is responsible for low polymerization shrinkage of Surefil SDR. Finally, the polymerization of Surefil SDR occurs at a slower rate when compared with conventional resin composites. The delay in the gelation phase allows more viscous flow of the material toward bonded surfaces of the tooth preparation and thus relieve part of the polymerization-induced shrinkage forces.

Among the flowable bulk-fill composites, X-tra Base showed the lowest dentin BS. Although X-tra Base shows the lowest VS (2.54% according to the manufacturer) and SS similar to other bulk-fill composites, it is the highest filled with all the resin-based composite material tested. This high filler content may have caused a significant increase in stiffness, which may have increased gap formation during polymerization, jeopardizing the BS.

Finally, although flowable bulk-fill composites have shown several advantages in laboratory studies, it remains to be seen if these advantages would be associated with higher clinical longevity and better performance of composite restorations.

**CONCLUSIONS**

Within the limitations of the present study, it is possible to conclude that, in general, bulk-fill composites polymerize effectively in 4 mm increments, present lower SS, higher BS and lower stiffness than conventional restorative composites, although the mechanical strength is lower.

**Acknowledgments**

This work was supported in part by grants from the National Council for Scientific and Technological Development (CNPq) under Grant (402132/2016-5).
Financial support and sponsorship
Nil.

Conflicts of interest
There are no conflicts of interest.

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