ORIGINAL RESEARCH

Microtensile Bond Strength of Packable and Flowable Bulk-fill Composite Resin-based Restorative Materials to Dentin

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Abstract

Aim: Comparative assessment of microtensile bond strength of packable and flowable bulk-fill composite resin restorative materials to dentin in a Class II cavity.

Materials and methods: Standardized independent class II tooth preparations were done involving both the proximal surfaces of 126 teeth to prepare 252 specimens. The 252 specimens were divided into seven groups of 36 specimens each as follows: group I—Filtek Z350XT packable conventional composite resin, group II—Filtek Bulk-Fill, group III—Tetric-N-Ceram Bulk-Fill, group IV—everX Posterior highly viscous fiber reinforced bulk-fill, group V—SDR Plus bulk-fill flowable, group VI—Tetric N Flow flowable bulk-fill, group VII—Filtek Bulk-Fill flowable. For group I, the mesial and distal cavities were etched with 37% phosphoric acid for 20 seconds followed by the application of single bond universal bonding agent. The cavities in group I were restored by 2 mm incremental placement of conventional nanofilled composite resin and light cured for 20 seconds. For group II–VII, the cavities were restored by bulk-fill composite resin-based restorative materials in a single increment of 4 mm and light polymerized for 20 seconds. The restored specimens were sectioned using a diamond disc creating a 1 mm thick slabs. The evaluation of the microtensile bond strength (μTBS) was done using the universal testing machine until debonding occurred at the dentin restoration interface.

Results: A statistically highly significant difference was observed in the values among the groups ($p < 0.01$) with the highest values in group II and the least in group VII.

Conclusion: The bulk-fill packable composite resin-based restorative materials were found to have highest μTBS as compared with the other bulk-fill composite resin-based restorative materials.

Keywords: Bulk-fill composites, Microtensile bond strength, Universal testing machine.

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Introduction

The contemporary dental composite resin-based restorative materials have improved mechanical and esthetic properties. The clinical applications of these materials involve a range of restorative treatments from pit and fissure sealants to core build-up materials, anterior esthetic corrections to posterior indirect restorations.[1] A micromechanical bond, which all the direct esthetic composite resin restorations have to tooth substrate,[2] is a prerequisite for the longevity of such restorations. The shrinkage of the composite resins during polymerization induces stresses.[3] These stresses compromise the bond strength and overall bond integrity resulting in microleakage. This further leads to postoperative sensitivity, secondary caries, and irreversible pathological changes in the pulpal tissues.[4] Hence, modifications in the composition, placement techniques, and curing methods have been employed to reduce the shrinkage stresses. Matrix modification, increase in filler loading, filler size distribution, types of filler, initiator–activation modification can be modified by the manufacturer. However, factors controlled by the operator such as control of the intensity of the curing light, curing techniques, application of flowable composite resin on the gingival seat, incremental layering technique and indirect resin restoration also have a significant impact on the amount of shrinkage stress.[5,6] Of all the methods, the incremental placement technique is one of the most effective methods to reduce the effect of polymerization shrinkage stress. However, the incremental placement technique has drawbacks like the inclusion of voids, inter-layer contamination, longer isolation time and increased deformation of restored tooth, and increased deformation of the restored tooth. Moreover, the incremental composite placement technique is time-consuming.[1,4,7] Hence, bulk-fill composite resin-based dental restorative materials have been introduced.

The reduced polymerization shrinkage and higher depth of cure of the bulk-fill composite resin restorative materials enable placement and polymerization of a single increment of up to 4 mm thickness. This helps to speed up the procedure and saves clinical chairside time in cases of wide and deep cavities. The higher depth

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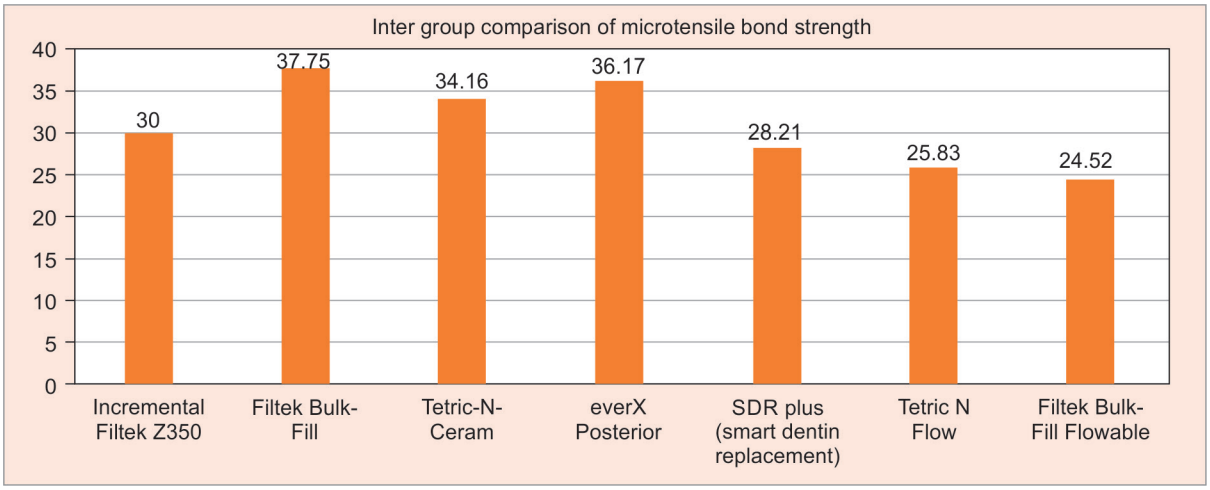

Fig.1: Graphical representation of inter group comparison of mean Microtensile Bond Strength in MPa of all the seven composites

of cure of these materials is attributed to the more translucent photoinitiators which allow the light to pass through much deeper layers.

Stronger the adhesion or the micromechanical bond between the tooth substrate and composite, better will be the resistance offered by the restoration to the stresses generated during resin polymerization and oral function.[8] Hence, microtensile bond strength test was preferred over the conventional bond strength tests. The microtensile bond test enables uniform stress distribution over a much smaller cross-sectional area of one square millimeter.[8] It truly represents the tensile bond strength and hence more reliable.[8] Hence, this study was carried out for the assessment of μTBS of three each flowable bulk-fill and packable high-viscosity bulk-fill resin composites using universal testing machine.

Materials and Methods

A total of 126 human permanent mandibular molars were collected, cleaned, and stored in distilled water until use. The occlusal surfaces of the teeth were polished using polishing disc to get a stable reference point for standard depth level of 4 mm. Two independent class II preparations of standardized dimensions were done involving both the proximal surfaces to obtain 252 specimens. The cavity preparations were done using a high-speed handpiece and diamond burs with air and water coolant. The dimensions of the standardized prepared cavities include buccolingual width of 3.5 mm and occlusogingival depth of 4 mm, the gingival seat margins 1 mm above the CEJ and 3.5 mm of axial wall depth at the gingival seat using a digital Vernier caliper. The 252 specimens were randomly allocated into one control and six experimental groups so as to have 36 specimens in each group, based on the type of composite resin used to restore the class II cavities. All teeth were kept immersed in distilled water till the time the restorations were done. The teeth were then mounted in a block prepared using silicone putty.

Following composite resin-based restorative materials were used in this study: group I—Conventional Nanocomposite (Filtek Z350XT); group II—Packable Bulk-Fill Nanocomposite (Filtek Bulk-Fill); group III—packable bulk-fill nanohybrid composite (Tetric-N-Ceram); group IV—fiber reinforced packable Bulk-Fill everX Posterior); group V—flowable bulk-fill nanocomposite (SDR); group VI — Flowable Bulk-Fill Nanocomposite (Tetric N Flow); group VII — Flowable Bulk-Fill Nanocomposite (Filtek Bulk-Fill Flowable).

For group I mesial class II cavity, total etching was done for 20 seconds, rinsed for 20 seconds and then dried using chip syringe. Then the Tofflemire matrix band was adapted to the prepared cavity. Adper Single bond universal was applied with an applicator brush and was scrubbed continuously for 20 seconds. It was air-dried for 10 seconds to evaporate the solvent and then light-cured for 20 seconds. The cavity was restored by 2 mm incremental placement of composite resin and light-cured for 20 seconds by placing the tip of curing light at the level of marginal ridge. Then a second increment of 2 mm thickness was placed and light-cured for 20 seconds. The procedure was repeated for distal cavity on the same tooth (Fig. 1)

For groups II–VII, the cavities were restored by packable high-viscosity bulk-fill or flowable bulk-fill resin composites as a 4 mm single increment and cured using LED curing light for 20 seconds by placing the tip of curing light at the level of marginal ridge. To simulate oral conditions, all the specimens were kept in the incubator for 24 h at 37°C until they were subjected to thermocycling. The temperature range of 5–55°C, dwell time 15 s, and an interval time of 10 seconds each was employed for the same. The restored specimens were serially sectioned and sliced further using a diamond disc under water coolant creating an approximately 1mm thick slab. Each slab was trimmed from both sides to obtain rods with standardized bonded surface area of 1 mm^2 of dentin-restoration interface. The standardized rod dimensions were confirmed using digital Vernier caliper. The specimens which showed pre-test failures before testing were discarded and replaced with new specimens. The μTBS was evaluated using computer-based universal testing machine at a cross-head speed of 0.5 mm/min until debonding at the dentin adhesive interface occurred.

$$S = L/A$$

Where S is the bond strength (MPa); L = test load; A = adhesive area (mm^2).

Statistical Analysis

The means and standard deviation for each group were calculated. Inter-group comparison (>2 groups) of μTBS was done using Kruskal–Wallis ANOVA followed by pair-wise comparison using

Table 1: Inter-group comparison of mean microtensile bond strength in MPa of all the seven composites using Kruskal–Wallis ANOVA (n = 36 per group)

Inter-group comparison of mean microtensile bond strength in MPa						
Group	*Mean*	*Std. deviation*	*Std. error*	*Median*	*Chi-square value*	*p-value of Kruskal–Wallis ANOVA*
I	30.00	0.779	0.130	29.97		
II	37.75	0.451	0.075	37.74		
III	34.16	0.594	0.099	34.24		
IV	36.17	0.615	0.103	36.39	244.633	0.000**
V	28.21	0.573	0.095	27.955		
VI	25.83	0.220	0.037	25.795		
VII	24.52	0.358	0.060	24.605		

Mann–Whitney *U* test. For all the statistical tests, $p < 0.05$ was considered to be statistically significant, keeping α error at 5% and β error at 20%, thus giving power to the study as 80%.

Results

The results of the above statistical analysis were tabulated and presented in Table 1. There was a statistically highly significant difference seen for the values between the groups ($p < 0.01$) with the highest values in group II and the least in group VII. Mean was calculated as the measure of central tendency. The μTBS of group II was 37.75MPa. The μTBS of group VII was 24.75 MPa. When the μTBS of incrementally placed nanocomposite (group I) and flowable bulk-fill composites (group V + VI + VII) were compared, a statistically highly significant difference was seen for the values between the groups ($p < 0.01$) with the highest value in group I and lesser in group (V + VI + VII).

Discussion

The recently introduced bulk-fill resin composites have newer proprietary resins, additional modulators, novel photoinitiators, unique fillers. Owing to the nano-sized filler particles, the bulk-fills can be closely compacted in the resin matrix to improve the physical properties (i.e., wear resistance, compressive strength, and tensile strength) of the material. The bulk-fill resin composites speed up the restoration process by enabling up to 4 mm increments to be cured in one step, with reduced polymerization shrinkage and without negatively affecting the macro mechanical properties. Flury et al.[9] stated that bulk-fill composites facilitate enhanced speed of clinical procedures compared with the conventional composite. Furthermore, according to the review by Chesterman et al.[10] the bulk-fill composites eliminate the possibility of voids between composite layers and avoid inter-layer contamination which is a drawback of incrementally placed conventional composites.

From the results of the present study, the packable bulk-fill composite resin restorative materials have shown the highest mean μTBS among all the test groups. The presence of a modified resin matrix and other monomers incorporated into the bulk-fills lower the shrinkage stress and enhances the μTBS. Filtek bulk-fill posterior (3M) consists of aromatic dimethacrylate (AUDMA), additional fragmentation molecules (AFM), urethane dimethacrylate (UDMA) and 1,12-dodecane dimethacrylate (DDMA). The inclusion of these monomers into the polymerization mixture enables the network to rearrange and get adapted during and/or after the polymerization.[11] This rearrangement accommodates the shrinkage without developing significant stresses at the tooth-restoration interface.[11] It also contains additional zirconia filler and this substitution of glass fillers with zirconia/silica fillers (2.5 and 5.0 wt%) is said to improve mechanical properties, such as flexural strength and fracture toughness.

Tetric N-Ceram Bulk-Fill composite resin restorative materials contain a special patented filler, which is a unique shrinkage stress reliever. It also utilizes the initiators: camphor quinone with an additional acyl phosphine oxide, and novel photoinitiator Ivocerin – a dibenzoyl germanium derivative.[12] These additional modifiers facilitate and impart the appropriate properties to be used as bull fill material. The short-fiber reinforced everX Posterior had superior properties due to the stress transfer from the matrix to the fibers depending on the fibers' length and diameter. The short-fiber fillers prevent crack propagation and prevent restoration of fracture. They also increase strength and contribute to reduction of polymerization shrinkage. Garoushi et al.[13] investigated the inclusion of short fibers and its impact on the properties. The study showed improved physical properties due to the incorporation of short fibers. Similar results demonstrating the superior value of high-viscous bulk-fill composites have been obtained by Mandava et al.,[3] Taneja S et al.,[4] and Kazemi D et al.[14] A systematic review Akah M et al.[15] also concluded that bulk-fill resin composites could elucidate and resolve the inherent problems associated with polymerization shrinkage and the degree of conversion in the contemporary resin composites. Especially when used in deep posterior cavities, the bulk-fill materials have added advantage due to reduced shrinkage stress over the contemporary packable composite resins. Figueiredo Reis et al.[16] and Nikolaenko et al.[17] have shown contradictory results to the present study. In their study, bulk-fill technique (depth: 4 mm) led to low dentin adhesion at the cavity floor. The contradictory results can be attributed to substrate composition-related parameters and their properties, factors related to specimen preparation, and composite resin restorative material used.[18] The Figueiredo Reis et al.[16] stated that maximum polymerization at the depth of the cavity was possible only due to incremental layering technique. It assures uniform curing and reduces the C-factor to about 1.3.

When comparing incrementally placed nanocomposites and flowable bulk-fill composites, there was a statistically highly significant difference seen for the values between the groups ($p < 0.01$) with the highest value in the incrementally placed nanocomposite. These findings can be attributed to the higher matrix and lower filler amount in bulk-fill flowable composites when compared with conventional nanocomposites. Kim et al.[19] in their study concluded that it was primarily the morphology of the filler particles and the amount of filler loading that improved the mechanical and physical properties of the resin-based composites. Reduced filler content of flowable bulk-fill composites results in

an increase in the polymerization shrinkage and depletion in the mechanical properties when compared with conventional hybrid composites.[20] Flowable bulk-fill materials are more susceptible to high wear rate due to reduced filler content and reduced filler-polymer matrix ratio, increased interparticle distance. These factors also result in an increase in the shrinkage stresses to potentially debond the material from dentin during polymerization.[21]

The restorative technique can also affect the integrity of the adhesive layer, especially in cavities with a high C-factor. Restorations fabricated with a single increment, in a cavity with a high C-factor, lead to higher contraction stresses and disruption of the adhesive layer. Additionally, the materials may not get adequately polymerized in deeper areas. The findings are in congruence with the study performed by Nikolaenko et al.[17] He reported that bond strength increased when a vertical layering technique was used. Bulk placement of conventional or contemporary packable composite resin may not allow sufficient light penetration to deeper layers for adequate polymerization of the materials.

In the present study, group II (Filtek Bulk-Fill Posterior, 3M) showed the highest value for μTBS of 37.75MPa. Similar results demonstrating the superior value of Filtek bulk-fill posterior have been obtained by Mandava et al.[3] His study showed the highest bond strength with Filtek bulk-fill composites and the values were of statistically significant difference compared with that of Tetric EvoCeram Bulk-Fill ($p < 0.003$) and X-tra fil bulk-fill ($p < 0.001$) composites.

Among the flowable bulk-fill composites, Group V (Smart Dentin Replacement) showed the highest value for μTBS of 28.21 MPa. That can be attributed to patented stress-decreasing technology, which results in a low shrinkage stress rate and better cavity wall adaptation. The unique composition results in a lower modulus of elasticity – which helps the material to withstand masticatory loads and helps to reduce the marginal gap formation of the tooth restorative interface.[22,23] The formulation of the SDR influences its surface energy and wettability characteristics which are higher than those of the other composites used.[24] Van Ende et al.[25] reported that in cavities with high C-factor, bulk-fill flowable composites exhibited higher wettability and hence better internal adaption than methacrylate-based flowable bulk-fills. Similarly, Moorthy et al.[26] reported that bulk-fill composites have higher cavity adaptation than methacrylate-based composites.

Hence, based on the results obtained in the present study, the following conclusions could be reached that the μTBS for the tested high-viscosity bulk-fill resin-based composites were high compared with that of the tested incrementally placed conventional nanocomposite and flowable bulk-fill composites. Therefore, high-viscosity bulk-fill composites may be able to substitute the time-consuming contemporary packable composite resin restorative materials which employ incremental placement techniques. In the μTBS study, one of the important factors to be evaluated is the number of pre-test failures.[18,27] The pre-test failures can be associated with poor adaptation of materials during the insertion/condensation of the composites in the cavity before polymerization.[18,27,28] This could be the possible reason for the high variability in bond strength results and emphasizes the need for further investigation. More studies describing the combined use of a flowable composite and a viscous composite resin molded together in an unpolymerized state, followed by the final polymerization of both materials (snow plough technique) need to be done with respect to bulk-fill composites. Also, it is important to emphasize that the results of this in vitro study cannot be directly extrapolated to all clinical application due to the substrate variation.[18,27,29] Therefore, further studies should be conducted to evaluate different parameters including other mechanical properties in order to establish the superiority of high-viscosity bulk-fill composites over incrementally placed composites.

Conclusion

Based on the results obtained under experimental conditions and taking into account the limitations of this study, it can be concluded that:

- The Bulk-fill high-viscosity composites were found to have highest micro tensile bond strength as compared with the other resin-based composites, that is, incrementally placed Nanocomposite and Bulk-fill flowable composite.
- The μTBS demonstrated by incrementally placed nanocomposite was higher when compared with flowable bulk-fill composites.
- Among the high-viscosity bulk-fill composites Filtek Bulk-Fill Posterior showed the highest value for μTBS.
- Among the flowable bulk-fill composites Smart Dentin Replacement (SDR) showed the highest value for μTBS.

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