Effect of filler content on the microtensile bond strength of composite resin and dentin in Class I cavities

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Objective: To investigate the effect of filler content on microtensile bond strength (μ-TBS) in Class I cavities. **Method and Materials:** Experimental composites with filler contents of 80%, 76%, 70%, 60%, and 50% by weight were used. Polymerization shrinkage was measured with Acuvol, and a three-point flexural test was performed to determine flexural properties. For evaluation of μ-TBS, 25 extracted human molars were randomly divided into five groups and Class I cavities were prepared. After filling with one of the experimental composites and curing for 40 seconds, teeth were serially sectioned perpendicular to the cavity floor. Stick-shaped samples were tested with a microtensile tester. Statistical analysis was conducted using ANOVA and Pearson correlation tests. **Results:** Significant correlations were found between filler content and polymerization shrinkage ($r = -0.973$, $P < .05$) and the filler content and Young modulus ($r = 0.891$, $P < .05$). Different filler contents in the experimental composites had no significant effect on μ-TBS or flexural strength. **Conclusion:** In a Class I cavity model, this in vitro study showed that the filler content did not influence the flexural strength of experimental composite resins and had no effect on the microtensile bond strength between composite resin and dentin. *(Quintessence Int 2012;43:e16–e22)*

Key words: composite resins, filler content, flexural modulus, flowable composites, microtensile bond strength, polymerization shrinkage

Recent advances in adhesive dentistry have brought significant changes to the treatment of caries. Large improvements have been made in mechanical and esthetic properties, and direct composite restorations are preferred when treating caries in posterior teeth because of the minimal intervention and cavity preparation required. However, polymerization shrinkage is still a major problem with composite resin. Polymerization shrinkage is induced during the conversion of monomer molecules into a polymer network. During this process, van der Waals and hydrogen interactions are replaced with shorter covalent bonds.[1] The polymerization reaction of light-cured composites induces polymerization contraction stress on tooth structures when a composite resin is bonded to cavity walls.[2] This creates contraction stress, which has the potential to initiate the failure of the composite-tooth interface if the forces of polymerization contraction exceed dentin bond strength. If this occurs, adverse consequences such as postoperative sensitivity, microleakage, secondary caries, and microcracking of the restorative material can result.[3–5]

Several factors have been linked to polymerization shrinkage stress, including filler content, elastic modulus, degree of conversion, and the C-factor of the cavity.

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Table 1 Details of the experimental light-cured composite resins used in the study

Components	Composition	Manufacturer
Resin matrix	Bis-GMA (70 wt%), UDMA (10 wt%), TEGDMA (20%)	Esstech
Filler	Aluminum Silanized amorphous silica, hydrophobed	Schott Degussa
Others	CQ, amine accelerator, BHT	Sigma

Bis-GMA, bisphenol glycidyl methacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; CQ, camphorquinone; BHT, butylated hydroxytoluene.

The *C-factor* is defined as the ratio between the bonded and unbonded surfaces of the composite restoration. It has been shown that a high C-factor value increases polymerization shrinkage stress and decreases bond strength.[6] It is generally thought that shrinkage stress is decreased clinically by using an incremental filling technique by minimizing the C-factor.[3]

The primary role of filler particles is to improve the mechanical properties and wear resistance of composite resins.[3] Since the filler does not participate in the curing reaction, the more filler used, the lower the polymerization shrinkage observed. On the other hand, a positive correlation exists between the elastic modulus and filler content.[7] Therefore, increasing the amount of filler used would simultaneously reduce polymerization shrinkage and increase the modulus of elasticity. Interfacial stress during polymerization is positively correlated with polymerization shrinkage and with the elastic modulus according to Hooke's law.[8] Therefore, in a given resin-based composite matrix, the amount of filler used is a major factor in polymerization contraction stress development.[5] Because the forces act directly at the restoration-tooth interface, volumetric shrinkage stress may have a detrimental effect on bonding strength. It has been shown that polymerization contraction stress also has a detrimental effect on bonding strength at the restoration-tooth interface.[9] Due to the contradictory effects that filler content may have, the effect of filler content on bond strength has been the subject of debate.[10–12] No complete description has yet been given, and some reports have given contradictory results. The objective of the present study was to determine the effects of filler content on microtensile bond strength (μ-TBS) between composite resin and dentin using a Class I cavity model.

METHOD AND MATERIALS

For this study, five experimental light-cured composites with different filler contents of 80%, 76%, 70%, 60%, and 50% by weight were prepared (Advanced Technology and Materials) (Table 1). The mean filler particle size was 0.7 μm with a range of 0.4 to 3.0 μm. The components were mechanically mixed to produce a homogeneous paste. The resins were cured for 40 seconds using light-emitting diodes (Bluephase 16i, Ivoclar Vivadent) with an intensity of 1,100 mW/cm^2.

Polymerization shrinkage

Polymerization shrinkage was measured using a video-imaging device (Acuvol, Bisco). A 7 ± 2 μl sample (n = 7) of each uncured material was manually shaped into a semisphere and placed on a polytetrafluoroethylene pedestal. Each sample was allowed to sit for 5 minutes before being light cured.

The curing tip was positioned 2 mm above the top of each sample. Volumetric shrinkage was recorded 5 minutes after curing. The volumetric reconstruction and percent change in volume were calculated using the following formulas:

$$volume = \sum_{area} h\pi\left(\frac{w_i}{2}\right)^2$$

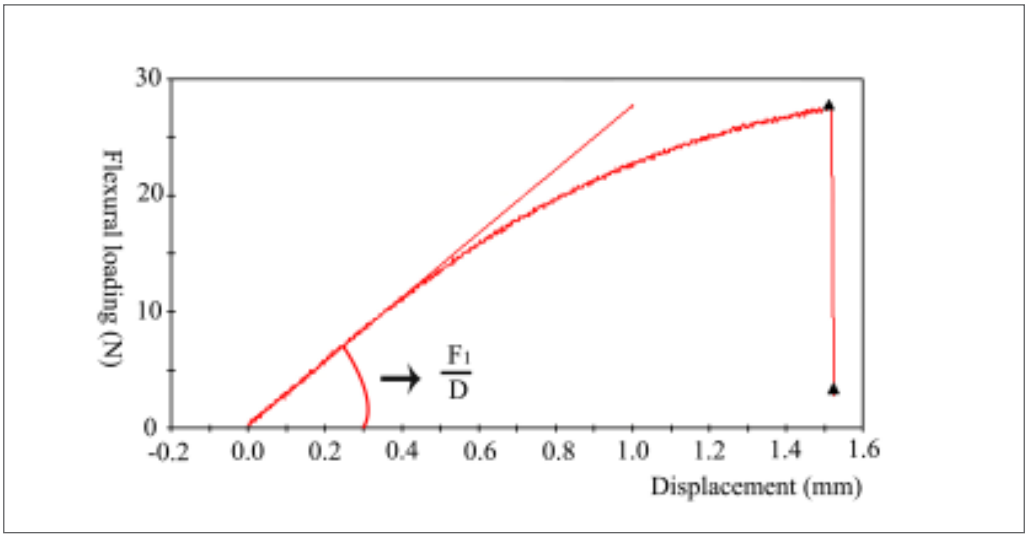

Fig 1 Representative load-deflection graph used to calculate the flexural modulus. This specific sample is the composite with 50 wt% filler content.

Where *M* is the number of the outline divided into horizontal slices; h is the disk height, *w* is the diameter, and *n* represents the number of views, which is equal to 1.

$$\Delta\%P = \left|100\frac{V_1 - V_2}{V_1}\right|$$

Where V_1 is the volume before and V_2 is the volume after polymerization.[13] Each composite was measured seven times.

Flexural modulus and flexural strength

Five kinds of experimental composites were tested for flexural properties according to ISO specifications and related studies.[14] Twelve stick-shaped specimens of each experimental composite (25 mm long × 2 mm wide × 2 mm high) were prepared in a metallic mold, which was positioned on top of a glass slide. A second glass slide was then placed on top of the mold, and gentle pressure was applied to extrude any excess material. Specimens were light cured using overlapped light coverage, as recommended in ISO 4049:2000. Each composite was tested after being exposed to light irradiation for 40 seconds. The specimens were then placed in water at 37°C for 24 hours and subjected to testing in a universal testing machine (Instron model 3367) with a crosshead speed of 1 mm/min until specimen fracture. The specimens were measured using a digital caliper before testing. The flexural modulus and flexural strength were recorded simultaneously.

Flexural strength (σ, MPa) was calculated using the following equation:

$$\sigma = \frac{3FL}{2wh^2}$$

Where *F* is the measured load in Newtons at fracture, *L* is the support span distance (20 mm), *w* is the width (mm), and *h* is the height (mm), as measured with a caliper.

The flexural modulus [*E*, MPa] was calculated using the following equation:

$$E = \left(\frac{F_1}{D}\right)\left(\frac{L^3}{4wh^3}\right)$$

Where (F_1/D) (in Newtons per millimeter) is the gradient measured in the steepest linear portion of the load-deflection curve (as shown in Fig 1). All other parameters are as defined above.

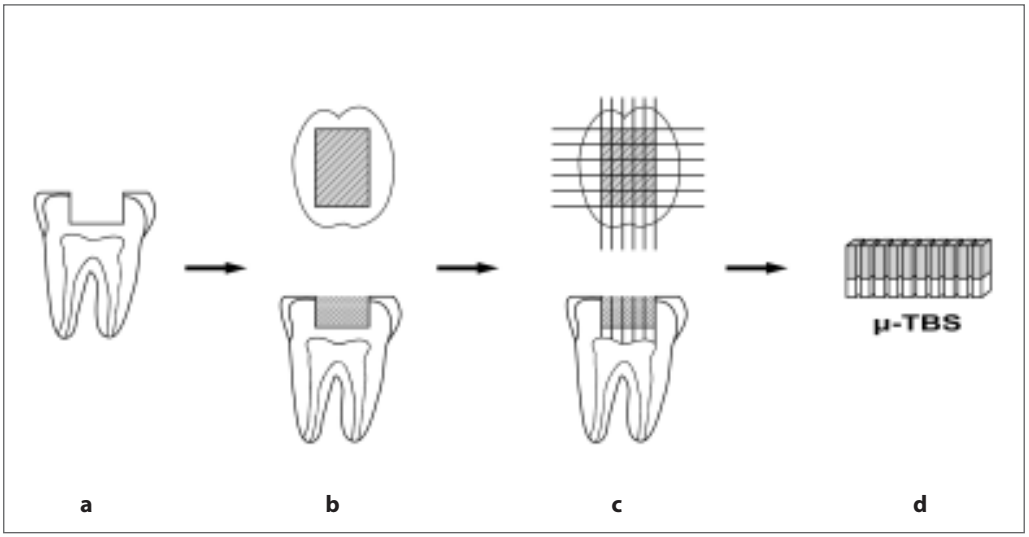

Fig 2 Specimen preparation for microtensile bond strength testing. *(a)* Cavities, 6 × 6 mm mesiodistally and buccolingually wide and 2 mm deep, were prepared in dentin. *(b)* Cavities were restored with adhesive and composite; the view of the tooth after restoration (cross-section and mesiodistal section). *(c)* Teeth were serially sectioned perpendicularly to the cavity floor using a Leica SP1600 saw microtome. *(d)* Stick-shaped specimens with a cross section of 1.0 × 1.0 mm were prepared.

Measurement of μ-TBS

Twenty-five sound first and second molars were randomly divided into five groups. As shown in Fig 2, tooth cusps were ground away under running water to expose a plane surface. Cavities with a 6 × 6–mm mesiodistal and buccolingual width and 2-mm depth were prepared in the dentin using a diamond bur (TF-13, MANI), and finished using a superfine bur (TF-21EF, MANI). The cavities were evaluated using an optical microscope (ZOOM-630E, Shanghai Changfang Optical Instrument) to check for defects. The cavities were coated with a one-step self-etching adhesive (Clearfil S^3 Bond, Kuraray Medical; lot 00056A) and cured for 10 seconds. They were filled with one of the five experimental composites by bulk filling and cured for 40 seconds. After storage in water at 37°C for 24 hours, the specimens were serially sectioned perpendicular to the cavity floor using a Leica SP1600 saw microtome (Leica), yielding stick-shaped specimens with a cross section of approximately 1.0 × 1.0 mm. There was no failure during sample preparation. Before the test, each stick was checked to exclude samples with defects; the length of dentin was less than 2 mm. The μ-TBS samples of each group after the check were 30 (80 wt% filler content group), 26 (76 wt% filler content group), 25 (70 wt% filler content group), 25 (60 wt% filler content group), and 28 (50 wt% filler content group). These stick-shaped specimens were mounted on a Micro Tensile Tester (Bisco) using cyanoacrylate adhesive and stressed to failure in tension at 1 mm/min. The μ-TBS was expressed in MPa and calculated by dividing the fracture load (F) by the bond area (S). The equation is:

$$\mu\text{-TBS} = F\ (N)/S\ (mm^2).$$

Statistical analysis

The Shapiro-Wilk test was used to evaluate the normal distribution. Continuous normally distributed variables were reported as mean ± standard deviation (SD). Statistical analysis was performed using SPSS for Windows 11.5 (IBM). Data for polymerization shrinkage, microtensile bond strength, flexural modulus, and flexural strength of experimental composites were subjected to one-way analysis of variance (ANOVA) followed by post hoc least significant difference tests to determine the differences between composites. The Pearson correlation coefficent

Table 2 Polymerization shrinkage, microtensile bond strength, flexural strength, and Young modulus (mean and SD) of composites

Filler content (wt%)	Polymerization shrinkage (%)	Flexural strength (MPa)	Young modulus (GPa)	Microtensile bond strength (MPa)
80	2.52 ± 0.22[a]	82.16 ± 6.41[a]	5.23 ± 0.70[a]	21.92 ± 5.61[a]
76	3.14 ± 0.12[b]	83.64 ± 6.07[a]	4.67 ± 0.47[b]	21.59 ± 4.07[a]
70	3.61 ± 0.10[c]	84.04 ± 6.66[a]	4.19 ± 0.27[c]	21.25 ± 6.35[a]
60	4.43 ± 0.24[d]	84.81 ± 5.44[a]	3.12[d] ± 0.46[c]	20.79 ± 4.99[a]
50	4.96 ± 0.06[e]	83.40 ± 6.54[a]	2.69 ± 0.32[e]	21.74 ± 5.71[a]

SD, standard deviation. Different superscript letters indicate statistical difference among composites with different filler content ($P < .05$).

was conducted between the factors of filler content and other parameters. The level of significance was set at .05.

RESULTS

Data including polymerization shrinkage, microtensile bond strength, flexural modulus, and flexural strength of the experimental composites under various conditions are listed in Table 2. The polymerization shrinkage decreased with increasing filler loading. The correlation index between filler content and polymerization shrinkage of composites was –0.973 ($P < .05$). On the other hand, the Young modulus increased with increased filler content. The correlation index between filler content and the Young modulus of the composites was 0.891 ($P < .05$). According to the results of the one-way ANOVA, the experimental composites with different volumes of filler were not significantly different in their flexural strength and μ-TBS ($P > .05$). The correlation index between the filler content and the flexural strength of the composites was –0.067 ($P = .612$). The correlation index between the filler content and the μ-TBS of the composites was 0.023 ($P = .796$).

DISCUSSION

To investigate the effect of filler content on microtensile bond strength, cavities with uniform size and a C-factor of 2.3 were prepared in this study, since a higher C-factor would have caused greater shrinkage stress. In our previous study,[15] the microtensile bond strength of one- and two-step self-etching adhesives to dentin planes were compared in vitro. The results showed that the two-step self-etching adhesive (Clearfil SE Bond) had a higher bond strength than one-step systems (such as Adper Prompt [3M ESPE], Clearfil S^3 Bond, and Xeno III [Dentsply]). Clearfil SE Bond and Clearfil S^3 Bond have been used extensively in our clinical work. In this study, Clearfil S^3 Bond, which has a lower bond strength than Clearfil SE Bond, was chosen as the adhesive to decrease the impact of the adhesive.

Curing rate is determined by the light intensity applied to the composites.[16] Increasing energy density leads to significant increases in the degree of conversion and polymerization shrinkage, without affecting the polymerization rate.[17] To minimize the influence of curing rate, a curing unit with a power density of 1,100 mW/cm^2 was applied.

In this study, Acuvol was used to test the polymerization shrinkage of composites. It is a video-imaging device used in several

previous studies.[13,18,19] The results obtained in this study indicate that polymerization shrinkage of composites decreases when a high filler content is used. This is consistent with previous studies and can be attributed to the decreased number of resin molecules available to form a network. According to one previous study, filler size and filler shape influence the Young modulus.[20] Filler of uniform size and shape was therefore used in this study. The results show that the Young modulus increases with increased filler content, as observed previously.[7]

Due to the contradictory effects of filler content on polymerization shrinkage and the Young modulus, the effect of filler content on bonding strength was also explored. In previous studies, two different viewpoints have been expressed regarding the relationship between filler content and bonding strength. The results reported by Miyazaki et al suggest that bonding strength increases with increased filler content, due to decreased polymerization shrinkage.[21] On the other hand, other studies have reported that composites with lower filler content (such as flowable composites) have improved microtensile bond strength with the dentin. For example, Durafill (Heraeus Kulzer), which has 56 wt% filler content, presented a higher μ-TBS in Ilie et al's study.[22] The results demonstrate that less viscous materials caused a better wetting of the dentin and resulted in a bonded surface with fewer defects. However, neither marginal adaptation nor bonding strength were improved in restorations lined with flowable composite in other studies.[12,23] The differences between these studies may be attributable to the different experimental materials and methods because in addition to filler content, the C-factor, the composition of the resin matrix, the placement technique, and the photoactivation method used may all influence microtensile bond strength.[6,24]

In this study, uniform cavities with a C-factor of 2.3 were prepared, and all composites were made of the same component type and content with the exception of the filler content to minimize the impact of differences in the composition of commercial composites. No significant positive correlation between filler content and μ-TBS was found, although volumetric shrinkage was negatively correlated with filler content. This latter result may be due to reduced polymerization shrinkage with increased filler content; however, the elastic modulus also increased with filler content. Interfacial stress during polymerization was found to be positively correlated with polymerization shrinkage and elastic modulus (Hooke's law).[3] In Condon's study,[5] composites with a lower filler content were shown to be less likely to generate high levels of shrinkage stress. Using a low-shrinkage composite resin in clinical cases is not always favorable due to its higher elastic modulus and shrinkage stress.[25,26] In a study that evaluated a low-shrinkage silorane composite and a conventional methacrylate-based composite, the results showed that there was no statistical difference in μ-TBS when flat surfaces were evaluated. When bonding to the cavity bottom, the μ-TBS of both composites decreased. The μ-TBS of the bulk-filled, low-shrinkage silorane composite showed a statistically significant difference when compared with composite bonding to a flat surface.[1]

Flexural strength is among the most important properties of polymer-based materials and has been widely studied.[27,28] According to ISO specification 4049, the flexural strength of composites used should be 80 MPa for an occlusion restoration and 50 MPa for other purposes. In the present study, the flexural strength of all experimental composites was over 80 MPa.

CONCLUSION

Within the limitations of this in vitro study, using a Class I cavity model, it was possible to conclude that the filler content did not influence the flexural strength of the experimental composite resins and had no effect on the microtensile bond strength between composite resin and dentin. The decrease in the filler content of the resin did not improve the bond strength to dentin, although it provided increased volumetric shrinkage and reduced modulus of elasticity of the tested material.

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