

# Shear bond strength of bulk-fill and nano-restorative materials to dentin

Hakan Colak<sup>1,2</sup>, Ertugrul Ercan<sup>3</sup>, Mehmet Mustafa Hamidi<sup>3</sup>

**Correspondence:** Dr. Ertugrul Ercan  
Email: ertugrul@kku.edu.tr

<sup>1</sup>Department of Restorative Dentistry, Faculty of Dentistry, Zirve University, Gaziantep, Turkiye,

<sup>2</sup>Adjunct Lecturer Department of Conservative Dentistry, College of Dentistry, Ishik University, Erbil City, Iraq,

<sup>3</sup>Department of Restorative Dentistry, Faculty of Dentistry, Kirkkale University, Kirkkale, Turkiye

## ABSTRACT

**Objectives:** Bulk-fill composite materials are being developed for preparation depths of up to 4 mm in an effort to simplify and improve the placement of direct composite posterior restorations. The aim of our study was to compare shear-bond strength of bulk-fill and conventional posterior composite resins. **Materials and Methods:** In this study, 60 caries free extracted human molars were used and sectioned parallel to occlusal surface to expose midcoronal dentin. The specimens were randomly divided into four groups. Total-etch dentine bonding system (Adper Scotchbond 1XT, 3M ESPE) was applied to dentin surface in all the groups to reduce variability in results. Then, dentine surfaces covered by following materials. Group I: SonicFill Bulk-Fill, Group II: Tetric EvoCeram (TBF), Group III: Herculite XRV Ultra, and Group IV: TBF Bulk-Fill, 2 mm × 3 mm cylindrical restorations were prepared by using application apparatus. Shear bond testing was measured by using a universal testing machine. Kruskal–Wallis and Mann–Whitney U-tests were performed to evaluate the data. **Results:** The highest value was observed in Group III ( $14.42 \pm 4.34$ ) and the lowest value was observed in Group IV ( $11.16 \pm 2.76$ ) and there is a statistically significant difference between these groups ( $P = 0.046$ ). However, there is no statistically significant difference between the values of other groups. In this study, Group III was showed higher strength values. **Conclusion:** There is a need for future studies about long-term bond strength and clinical success of these adhesive and bulk-fill systems.

**Key words:** Bulk-fill composite, self-etching adhesive, shear bond strength

## INTRODUCTION

Resin-based composite (RBC) is the most widely used modern dental restorative material. It offers advantages such as excellent esthetics and ease of handling. However, it is also characterized by the risk of complications due to insufficient polymerization of the material and the occurrence of polymerization shrinkage.<sup>[1]</sup> Since photo-polymerized resin composites were introduced, the degree of conversion was acknowledged as vital to the clinical success of these materials.<sup>[2]</sup> Photo-cured resin composites polymerize only to a certain depth. This depends on the penetration of visible light through the bulk of the material.<sup>[3]</sup> It has been shown that the insufficient polymerization may lead to a decrease in the physical/mechanical<sup>[4]</sup> and biological<sup>[5]</sup> properties of resin composites. 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.<sup>[6]</sup>

Therefore, bulk-filling techniques have become more widely used following the development of materials with improved curing,<sup>[7,8]</sup> controlled polymerization

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contraction stresses,<sup>[9,10]</sup> and reduced cuspal deflection. In contrast to, the maximum 2-mm increments recommended for conventional resin composites, manufacturers recommend 4 or 5-mm increments of the bulk-fill resin composites. The use of the bulk-fill technique undoubtedly simplifies the restorative procedure and saves clinical time in cases of deep, wide cavities.<sup>[11]</sup>

Therefore, the aim of this study is to compare the shear bond strength (SBS) of recently introduced two different brands bulk-fill resin composite with the same brand conventional hybrid composites. The null hypotheses to be investigated are that (1) the SBS values of a bulk-fill composite does not differ from that of a conventional hybrid composites (2) the SBS values of Tetric EvoCeram (TBF) and SonicFill (SF) does not show significant difference.

## MATERIALS AND METHODS

After the approval of the Ishik University College of Dentistry Ethical Committee with reference number 2014-006 for this study, a sample of 60 extracted human premolar teeth were collected following patients' verbal consent to include their teeth in the study. This paper describes an *in vitro* experimental study that involved 60 freshly extracted human third molars that were without cracks, decay, or any other defects. The teeth were removed from the subjects, the remnant connective tissue was removed and the samples were then stored in a 0.5% chloramine-T solution (Fisher Chemical, Fair Lawn, NJ, USA) for 24 h before being washed with a saline solution and stored in distilled water at room temperature throughout the study period.

Prior to testing, the teeth were cleaned and then the mid-coronal dental was exposed by sectioning the samples using a low-speed diamond disk saw under water coolant (Markus Inc., Michigan, USA). The teeth were rinsed again before being mounted in acrylic resin (2 cm × 3 cm × 5 cm). The surface of the dentin was smoothed using 600, 800, and 1200 grit waterproof polishing papers before the teeth were randomly divided into four groups ( $n = 15$ ).

The total-etch dentine bonding system was utilized on the teeth in all the groups to reduce variability in the results of the investigation. The etch-and-rinse adhesive system Adper Scotchbond 1XT adhesive (3M ESPE, St., Paul, MN, USA) was then used to treat the bonding area of the dentin surfaces in accordance with the manufacturer's instructions.

Two commercial bulk-fill composite systems were tested, and two conventional composite that required 2-mm increments was used as control. Conventional systems used as a control group was chosen from same brand with bulk-fill composite used. Materials used in the study given in Table 1.

- Group I: After adhesive application, the specimens were clamped in the ultradent bonding jig (Ultradent Products; South Jordan, UT, USA), and a column of SF composite resin with 2.0 mm in height and 2.38 mm in diameter was placed by sonic-activation using SF handpiece (Kavo SF System, Kerr, USA). Any excess composite was carefully removed and then cured for 20 s using a 1000 mW/cm<sup>2</sup> intensity light-emitting diode (LED) curing light (Elipar S10; 3M ESPE, Seefeld, Germany)
- Group II: After adhesive application, the

**Table 1: Materials used in this study**

| Group    | Material                                           | Composition                                                                                                                                                                                                          | Manufacturer                            |
|----------|----------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------|
| I        | SF, bulk-fill composite                            | Barium glass, silicon dioxide (5-10%), oxide, chemicals (10-30%), MPS (10-30%), silicon dioxide, EBPDMA (1-5%), bisphenol A bis (2-hydroxy-3-methacryloxypropyl) ether (1-5%), and TEGDMA (1-5%) (filler 83.5% w)    | Kerr, Orange, CA, USA                   |
| II       | TBF, nanohybrid composite                          | Bis-GMA, UDMA, ethoxylated bis-EMA (16.8 weight %); barium glass filler, ytterbium trifluoride, mixed oxide (48.5 weight %); prepolymers (34 weight %); additives, catalysts, stabilizers and pigments (<1 weight %) | Ivoclar Vivadent, Schaan, Liechtenstein |
| III      | Herculite XRV Ultra, nanohybrid composite          | Uncured methacrylate ester monomers, TiO <sub>2</sub> and pigments, MEHQ, BPO, TMPTA and initiators                                                                                                                  | Kerr, Orange, CA, USA                   |
| IV       | TBF Bulk-Fill, Bulk-fill composite                 | Ba-Al-Si glass, prepolymer filler (monomer, glass filler, and ytterbium fluoride) spherical mixed oxide, Bis-GMA, Bis-EMA, UDMA (filler 79-81% w, 60-61% volume)                                                     | Ivoclar Vivadent, Schaan, Liechtenstein |
| Adhesive | Adper Scotchbond 1 XT (2-step total-etch adhesive) | Etchant: 3M ESPE-35% phosphoric acid<br>Primer + bond: BIS-GMA, HEMA, dimethacrylates, polyalchenoic acid, copolymer, ethanol, water 3-8%, initiators                                                                | 3M ESPE, St. Paul, MN, USA              |

Bis-EMA: Bisphenol-A polyethylene glycol diether dimethacrylate, Bis-GMA: Bisphenol-A diglycidyl ether dimethacrylate, EBPDMA: Ethoxylated Bisphenol-A-dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate, UDMA: Urethane dimethacrylate, MPS: 3-(trimethoxysilyl) propyl methacrylate, TMPTA: Trimethylolpropane triacrylate, HEMA: 2-Hydroxyethyl methacrylate, SF: SonicFill, TBF: Tetric EvoCeram, TiO<sub>2</sub>: Titanium dioxide, MEHQ: 4-methoxyphenol, BPO: Benzoyl peroxide

specimens were clamped in the ultradent bonding jig (Ultradent Products; South Jordan, UT, USA), and a column of conventional hybrid composite resin TBF (Ivoclar, Vivadent, AG) with 2.0 mm in height and 2.38 mm in diameter was placed. The increments were placed into the preparations. Two millimeters thickness and each layer were polymerized with 1000 mW/cm<sup>2</sup> intensity LED curing light for 20 s (Elipar S10, 3M ESPE, Germany)

- Group III: Herculite XRV Ultra (Kerr Hawe, CA, USA) universal nanohybrid dental composite was used. The procedures were the same as those in Group I
- Group IV: A high viscosity TBF Bulk-Fill viscosity (Ivoclar Vivadent, USA) was placed to the preparation about 4 mm thickness and polymerized with the same LED curing light. The procedures were the same as those in Group I.

Once prepared, the specimens were stored in an incubator at 37°C in 100% humidity for 24 h before the SBS of the resin bond was tested using a universal testing machine (Esetron, Ankara, Turkey) at a crosshead speed of 1 mm/min. The SBS of the composite resin to dentin was recorded in Newtons and calculated in MPa, the cross-sectional area of the composite build-up was taken into account.

Two separate examiners observed the mode of failure in each specimen under a stereomicroscope (NZ.1902-P; Euromex, Arnhem, Netherlands) at ×20 magnification and failure modes were classified as either adhesive (failure at the dentin/composite interface), cohesive (failure within the resin composite or dentin), or mixed (partial adhesive/partial cohesive fracture).<sup>[12,13]</sup>

The statistical evaluation was performed with SPSS software for Windows (version 20, SPSS, Chicago, Illinois, USA). The SBS values were nonnormally distributed, as was shown by Shapiro-Wilk test. Therefore, nonparametric tests were performed for pairwise comparisons among groups (Kruskal-Wallis and Mann-Whitney U tests). The confidence level was set to 95% ( $P < 0.05$ ).

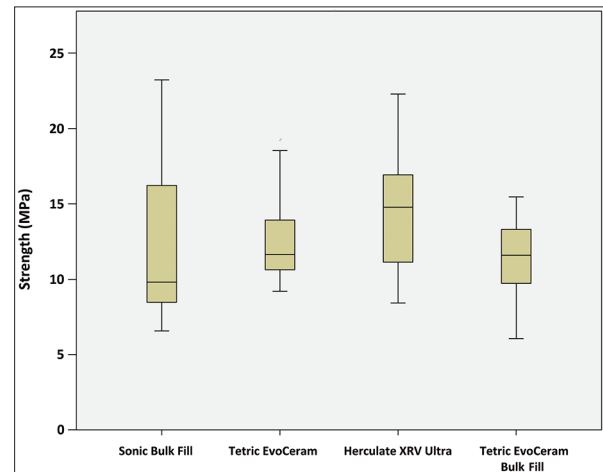
## RESULTS

Mean values and standard deviations of all groups are shown in Table 2 and Figure 1. The highest mean SBS ( $14.42 \pm 4.34$  MPa) was recorded for Herculite XRV Ultra nanohybrid composite (Group III) bonded to dentin specimen, while the lowest mean

**Table 2: Mean SBS values of groups in this study**

| Composite           | Mean±SD    |
|---------------------|------------|
| SF Bulk-Fill        | 12.19±5.48 |
| TBF                 | 12.59±2.97 |
| Herculite XRV Ultra | 14.42±4.34 |
| TBF Bulk-Fill       | 11.16±2.76 |
| <i>P</i> *          | 0.170      |

\*Kruskal-Wallis test. SD: Standard deviation, SBS: Shear bond strength, SF: SonicFill, TBF: Tetric EvoCeram



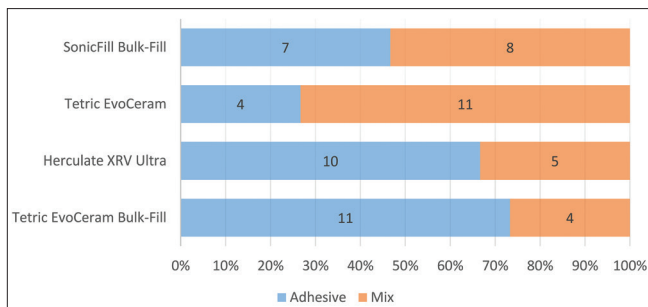
**Figure 1:** Bar graph show mean shear bond strength values of groups in this study

SBS ( $11.16 \pm 2.76$ ) was recorded for TBF Bulk-Fill composite (Group IV). This difference between Group III and IV found statistically significant ( $P = 0.046$ ). Although Group III showed higher bond strength than Group I and Group II, this differences was not found statistically significant ( $P$  values between Group III and I, Group III and II was found as 0.135 and  $P = 0.272$ , respectively).

Between SF ( $12.19 \pm 5.48$ ) and TBF ( $11.16 \pm 2.76$ ) bulk-fill composites, the mean bond strength of SF was found higher than that of TBF; however, no statistically significant differences were observed between two bulk-fill composites.

Although overall bulk-fill composites showed lower bond-strength when compared with conventional ones, a significant difference was only observed between Group III (Herculite XRV Ultra) and Group IV (TBF Bulk-Fill). Furthermore, no significant difference was observed between two conventional composite Herculite XRV Ultra and TBF ( $P = 0.272$ ).

The group failure modes were evaluated and are shown in Figure 2. Regarding mode of failure, adhesive mode of failure represented mostly



**Figure 2:** Distribution according to the type of fracture of the groups

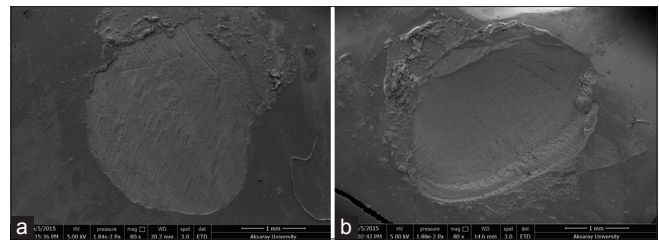
observed in Group III (Herculite XRV Ultra) and Group IV (TBF Bulk-Fill), while mix fractures observed in Group I (SF) and Group II (TBF). No cohesive type fractures were observed in any group. Furthermore, two samples from bulk-fill groups were evaluated under scanning electron microscopy to see the failing surfaces [Figure 3].

## DISCUSSION

The aim of this study was to investigate and compare the SBS of bulk-fill resin composites and conventional resin composites. The bulk-fill composites SF and TBF, although showed lower SBS values than the conventional composite, a significant difference was only observed between Herculite and TBF. Thus, the first null hypothesis was partially rejected.

Agarwal *et al.*<sup>[14]</sup> evaluated the cervical marginal and internal adaptation of posterior bulk-fill resin composites of different viscosities, before and after thermocycling. They found that SF was showed significantly better gap-free margins when compared TBF Bulk-Fill. The inferior adaptation to dentine observed in Group III TBF Bulk-Fill when compared with the other experimental groups could be attributed to the restricted flow of the material in the cavity. However, researchers did not find any difference between conventional composites and SF.<sup>[14]</sup>

Previous studies found that samples tested with bulk-fill resin composites demonstrate a better depth of cure than those treated with conventional resin composites.<sup>[11]</sup> However, in this study, there was no significant difference found between the two bulk-fill systems, despite the fact that SF demonstrated higher SBS than TBF. Thus, the second null hypothesis was accepted. A study by Alrahlah *et al.*,<sup>[15]</sup> also found that SF and TBF demonstrated similar depth of cure with no significant difference between them ( $P > 0.05$ ). SF has a higher filler content than TBF. During the process of SF, sonic energy is applied through a special hand



**Figure 3:** Scanning electron microscopy evaluation of dentin surfaces after shear testing in bulk-fill composite used groups: (a)  $\times 80$  magnification of an adhesive failing mode in Group IV (Tetric EvoCeram) (b)  $\times 80$  magnification of a mix failing mode in Group I (SonicFill)

piece to increase the flowability and to further ease the packing of the composite.<sup>[16]</sup> It was stated that the good depth of cure observed in the SF may be due to a refractive index matching between the resin and filler, which enhances light transmission. A reduction in the refractive index differences between resin and filler improves the degree of conversion,<sup>[17]</sup> increases the depth of cure and increases color shade matching.<sup>[18]</sup> The slightly higher bond strength that the SF exhibits in comparison to the TBF could be attributed to the properties of the SF.

According to, the data collected in the present study, TBF Bulk-Fill exhibited similar SBS values as the other conventional resin composites. This finding is aligned with the outcomes of previous studies. In a study by Benetti *et al.*,<sup>[11]</sup> TBF Bulk-Fill exhibited a higher depth of cure than the conventional resin composite. Furthermore, a higher depth of cure has been previously reported for bulk-fill resin composites,<sup>[19,20]</sup> and the differences between the two materials have been attributed to improvements in their initiator system<sup>[19]</sup> and increased translucency.<sup>[19,21]</sup> In an alternative study,<sup>[11]</sup> SF exhibited a depth of cure that was statistically similar to that of the conventional resin composite TBF. In addition, reported that the use of high viscosity bulk-fill resin composites with reduced polymerization contraction (SF and TBF Bulk-Fill) resulted in a similar gap formation as the conventional resin composite. This finding is partially in agreement with the results of the current study.

In the present study, the TBF and TBF Bulk-Fill systems exhibited statistically similar SBS values. This may be because they exhibit very similar mechanical properties and consistency.<sup>[22,23]</sup> As confirmed in different *in vitro* studies, bulk-fill RBCs might be cured in larger increments, as the degree of cure and the micromechanical properties can be maintained within 4-mm layers at an irradiation time of up to 20 s.<sup>[24]</sup> Thus, layering two consecutive 2-mm

increments with TBF or one 4-mm increment with TBF Bulk-Fill could produce similar mechanical properties as conventional filling techniques. According to the TBF Bulk-Fill approach, the increased depth of cure was realized by adding a new initiator in addition to the camphoroquinone/amine initiator systems; that is, ivocerin, as opposed to reducing the filler amount and increasing the filler size as per the process employed with the majority of bulk-fill materials.<sup>[22]</sup> The efficiency of the initiator is confirmed by the increased depth of cure in TBF Bulk-Fill compared with its regular nanohybrid RBC pendant TBF because the chemical composition and the filler systems in both materials are comparable.

Orlowski *et al.*<sup>[25]</sup> compared under *in vitro* conditions, marginal sealing of four different bulk-fill materials composite restorations of class II. They found that SF showed better marginal sealing than TBF. In addition, they also found that a higher marginal integrity and lower penetration of dye in fillings inserted using a sonic-activation condensing device were shown when compared with manual condensation. Orlowski<sup>[25]</sup> stated that statistically significant better marginal integrity of flowable tested materials, SF compared with TBF Bulk-Fill may be due to their flow consistency during application. Peutzfeldt and Asmussen showed that the degree of fluidity when applying the composite material influences the marginal adaptation; increased fluidity of the composite makes it adhere better to the walls of the cavity.<sup>[26]</sup>

## CONCLUSION

Although this study has a number of limitations, the results do indicate that the application of bulk-fill composite results in acceptable SBS that is comparable to that achieved via conventional RBCs. As such, bulk-fill composites may represent reliable alternatives to conventional composites. This could be of potential benefit to dental technicians because bulk-fill composites are simpler than conventional composites and can be applied more efficiently. However, further studies are required in this area to better understand how the bond strengths of these adhesive systems behave under clinically acceptable conditions.

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## Conflicts of interest

There are no conflicts of interest.

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