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# Evaluation of the Shear Bond Strength of Three Different Composite Resins Applied to Dentin at Different Thicknesses: An In Vitro Study

## Dentine Farklı Kalınlıklarda Uygulanan Üç Farklı Kompozit Rezinin Makaslama Bağlanma Dayanımının Değerlendirilmesi: In Vitro Çalışma

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**ABSTRACT**

**Aim:** The objective of this study is to evaluate the shear bond strengths of two dissimilar types of bulk fill composites, which use the bulk technique, along with a nanohybrid composite, which uses the incremental technique.
**Methods:** Ninety human molars were extracted for experimentation, with enamel removed to expose the dentin layer. The teeth were divided into nine groups (n=10). Adhesive (Single Bond Universal) was applied to the dentin according to the manufacturer's instructions. Plastic molds with diameters of 3 mm and heights of 2 mm, 3 mm, and 4 mm were used for the composite restorations. Filtek Z550 Nano Hybrid Universal Restorative, Filtek Bulk Fill Posterior Restorative, and Filtek Bulk Fill Posterior Flowable composite resins were applied using different techniques and thicknesses within each group. Shear bond strength was measured with a universal testing machine. Statistical analysis using one-way ANOVA and Kruskal-Wallis H tests ($P$<.05) was performed.
**Results:** Results indicated a significant difference in shear bond strength with increment thickness (2 mm-3 mm / 2 mm-4 mm) for Filtek Bulk Fill Posterior Flowable ($P$=.003). No significant difference was found between the other groups ($P$>.05).
**Conclusion:** These findings suggest that the type of composite resin and the technique used affect shear bond strength, with Filtek Bulk Fill Posterior Flowable being more sensitive to variations in increment thickness compared to Filtek Z550 and Filtek Bulk Fill Posterior.
**Keywords:** Bulk Fill, Bulk Fill Flowable, nanohybrid composite, shear bond strength

**ÖZ**
**Amaç :** Bu çalışmanın amacı, bulk teknik ile uygulanan iki farklı bulk fill kompozitin ve inkremental teknik ile uygulanan bir nanohibrit kompozitin makaslama bağlanma dayanımlarını değerlendirmektir.
**Yöntemler :** Deney için 90 insan molar dişi kullanıldı ve dentin tabakasını ortaya çıkarmak için mine tabakası kaldırıldı. Dişler dokuz gruba ayrıldı (n=10). Dentine, üreticinin talimatlarına uygun olarak adeziv (Single Bond Universal) uygulandı. Kompozit restorasyonlar için çapı 3 mm, yüksekliği 2 mm, 3 mm ve 4 mm olan plastik kalıplar kullanıldı. Filtek Z550 Nano Hybrid Universal Restorative, Filtek Bulk Fill Posterior Restorative ve Filtek Bulk Fill Posterior Flowable kompozitler, her bir grupta farklı teknikler ve kalınlıklar kullanılarak uygulandı. Makaslama bağlanma dayanımı, universal bir test cihazı ile ölçüldü. Tek yönlü ANOVA ve Kruskal-Wallis H testleri ($P$<,05) kullanılarak istatistiksel analiz yapıldı.
**Bulgular :** Filtek Bulk Fill Posterior Flowable için farklı kalınlıklardaki (2 mm-3 mm / 2 mm-4 mm) makaslama bağlanma dayanımında önemli bir fark olduğu ortaya çıktı ($P$=,003). Diğer gruplar arasında anlamlı bir fark bulunmadı ($P$>,05).
**Sonuç :** Bu bulgular, kullanılan kompozit rezin türü ve tekniğin makaslama bağlanma dayanımını etkilediğini, Filtek Bulk Fill Posterior Flowable'ın tabaka kalınlık artışına karşı Filtek Z550 ve Filtek Bulk Fill Posterior'a kıyasla daha duyarlı olduğunu göstermektedir.
**Anahtar Kelimeler:** Bulk Fill, Akıcı Bulk Fill, nanohibrit kompozit, makaslama bağlanma dayanımı

## INTRODUCTION

The incremental technique is widely acknowledged as the conventional approach for the applying of composite resin materials in cavity preparations. patient was referred to the Department of Oral and Maxillofacial This method requires successive layering, with each layer not exceeding a thickness of 2 mm,

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and light curing of composite resin materials from the occlusal direction. This step-by-step process is repeated until the entire cavity is filled.[1] Previous studies have established that the transmitted light energy diminishes as the thickness of the composite resin increases, necessitating a restriction on resin thickness.[2] Consequently, limiting the layer thickness to 2 mm or less is crucial to ensuring optimal light penetration and thereby achieving sufficient polymerization. Inadequate polymerization may lead to suboptimal conversion of composite resin components, resulting in elevated residual monomer levels.[3]

The incremental technique offers a notable advantage in its capacity to mitigate polymerization-induced shrinkage. The reduction of polymerization shrinkage is particularly advantageous because stress development during this process may induce cuspal deformation, potentially giving rise to sensitivity or microcracks in the material or tooth structure.[4] Despite these benefits, the incremental technique is not without its drawbacks. Challenges associated with this method include the potential formation of air gaps, contamination risks, adhesive failures between increment layers, and difficulties in placement, especially in conservative preparations due to limited access. Moreover, the process requires an extended period for the placement and polymerization of each layer, adding a time-related aspect to its considerations.[5,6]

To address the limitations associated with the incremental technique, bulk-fill composites have been launched to the market. Researchers have emphasized several critical attributes for effective bulk-fill composite resin materials, including low polymerization shrinkage, superior adaptation, ease of handling, enhanced physical characteristics, and a substantial depth of cure, typically recommended to be at least 4 mm.[7,8] The concern with polymerization in composite resins with depths of 4 mm and beyond stems from the diminishing intensity of polymerization light as the material depth increases. Achieving successful polymerization in bulk-fill composite resins with layer thicknesses of 4-5 mm, without compromising shrinkage stress, depends on the material composition, which plays a crucial role in determining the overall properties of these materials.[9]

This study aims to evaluate the shear bond strength (SBS) of two distinct types of composite resins using the bulk-filling technique in comparison with a clinically employed nanohybrid composite resin using the incremental technique. The investigation aimed to test two null hypotheses: firstly, that no discernible difference would exist in the bond strengths derived from samples of varying thicknesses within the same composite, and secondly, that no notable difference would exist in the bond strengths derived from samples of uniform thickness composed of different types of composites.

## METHODS

The research protocol used in this study obtained ethical approval from the Ethics Commission for Non-Clinical Scientific Research of Ankara University Faculty of Dentistry (Date: 05.03.2020, Number: 36290600/05). The investigation involved a total of 90 structurally non-deformed, previously unrestored, and non-carious maxillary or mandibular permanent human molar teeth. To ensure cleanliness, the molars underwent a thorough cleaning process with a scaler under tap water to remove calculus and debris. Subsequently, the cleaned teeth were stored in distilled water, with regular changes of water every week, until they were required for the study.

This research used three distinct resin composites, as detailed in Table 1.

**Table 1.** Composites used in the study and their contents

| Resin Composite | Type / Shade | Matrix Content | Filler Load |
|---|---|---|---|
| Filtek Z550 Nano Hybrid Universal Restorative (3M ESPE, St. Paul, USA) Lot number: 7050A2 | Packable Nanohybrid A2 | BIS-GMA UDMA BIS-EMA PEGDMA TEGDMA | 82% by weight 68% by volume |
| Filtek Bulk Fill Posterior Restorative (3M ESPE, St. Paul, USA) Lot number: 4864A2 | Packable Bulk-fill A2 | AUDMA UDMA 1.12-dodecane-DMA | 76.5% by weight 58.4% by volume |
| Filtek Bulk Fill Flowable (3M ESPE, St. Paul, USA) Lot number: 4862A2 | Flowable Bulk-fill A2 | bisGMA UDMA bisEMA Procrylat | 64.5% by weight 42.5% by volume |

Dentin specimens were meticulously prepared by eroding molars off the occlusal surface, revealing the dentin surface. Subsequently, the teeth were placed into epoxy resin inside cylindrical plastic molds. After removal from the molds, the dentin surface of the teeth underwent grinding using 600-grit SiC abrasive papers (Metkon Gripo 2V, Turkey) for 10 seconds to achieve a standardized smear layer.

The samples were systematically categorized into nine groups based on the resin composite type and thickness, outlined as follows: Group 1: Filtek Z550 Nano Hybrid Universal Restorative (FZ) 2 mm Group 2: FZ 3 mm Group 3: FZ 4 mm Group 4: Filtek Bulk Fill Posterior Restorative (FBP) 2 mm Group 5: FBP 3 mm Group 6: FBP 4 mm Group 7: Filtek Bulk Fill Flowable (FBF) 2 mm Group 8: FBF 3 mm and Group 9: FBF 4 mm.

The revealed dentin surface was treated with the universal adhesive system (Single Bond Universal) following the manufacturer's instructions. This involved brushing for 20 seconds, allowing to air dry for 5 seconds, and then light curing for 10 seconds. Subsequently, using a clear silicone tube with a 3-mm inner diameter and heights of 2 mm, 3 mm, and 4 mm, incremental composite layers of FZ and bulk increments of FBP and FBF were meticulously applied to the dentin specimens. The composite layers were subjected to light curing from the occlusal aspect for 20 seconds using a light-curing device for each increment layer. The light-curing process for all samples was conducted with a Mini L.E.D. device (Acteon, France), ensuring that the light power density/cm² remained consistently above 1250 mW. This parameter was verified at the commencement and conclusion of each day throughout the specimen preparation phase, using a radiometer. Following the restoration process, all samples were soaked in distilled water and kept at room temperature for a standardized period of 24 hours.

After the storage period, SBS testing was conducted on the samples using a Shear Bond Tester (Bisco, USA), which is a universal testing machine, at a controlled crosshead speed of 0.5 mm/min. The maximum force (Fmax) was recorded, and subsequent calculations were performed to determine the SBS values in megapascals (MPa). The formula used for the calculation was [Fmax (N)/bonding area (mm²)], where the bonding area was determined as approximately 7.1 mm² (calculated based on the formula for the area of a circle: bonding area = $r^2 \times \pi$, with a radius [r] of 1.5 mm). This comprehensive process yielded 10 SBS values per group, which were subsequently subjected to statistical analysis.

**Statistical Analysis**

To assess the distribution of data among the groups and ascertain normality, the Kolmogorov Smirnov test was employed. In cases where a normal distribution with equal variances was confirmed, analysis of variance was applied. For data exhibiting non-normal distribution and disparate variances, the Kruskal Wallis H test was used. After performing the ANOVA test, Tukey's post-hoc test was applied to identify

differences between groups. For comparisons following the Kruskal-Wallis test, the Bonferroni (Dunn) method was utilized. The results were scrutinized at a 95% confidence interval, and statistical importance was determined at $P<.05$. Statistical analyses were conducted using SPSS 20 (IBM SPSS Corp., Armonk, NY, USA).

## RESULTS

The SBS values at different increment thicknesses are presented in Figure 1, whereas the comparisons at identical thicknesses are shown in Figure 2. The mean±SD SBS values (in MPa) for increment thicknesses of 2 mm, 3 mm, and 4 mm are shown in Table 2.

**Table 2.** The mean±SD SBS (in MPa) and *P* values. Different superscript letters indicate statistical significance.

| | 2 mm | 3 mm | 4 mm | *P* |
|---|---|---|---|---|
| Filtek Z550 (FZ) | 9.41±3.83 | 7.18±3.76 | 7.06±6.64 | .125 |
| Filtek Bulk Fill Posterior (FBP) | 9.87±3.81 | 9.80±3.31 | 6.21±4.82 | .129 |
| Filtek Bulk Fill Flowable (FBF) | 11.17±4.38$^{ab}$ | 5.97±4.02$^{a}$ | 4.55±1.85$^{b}$ | .003 |
| *P* | .453 | .069 | .940 | |

Statistical analyses revealed a significant difference in increment thickness (2 to 3 mm and 2 to 4 mm) on SBS for FBF ($P=.003$). However, no significant difference was observed for FZ or FBP ($P>.05$).

The SBS values showed no significant difference ($P>.05$) among the groups for all identical thicknesses.

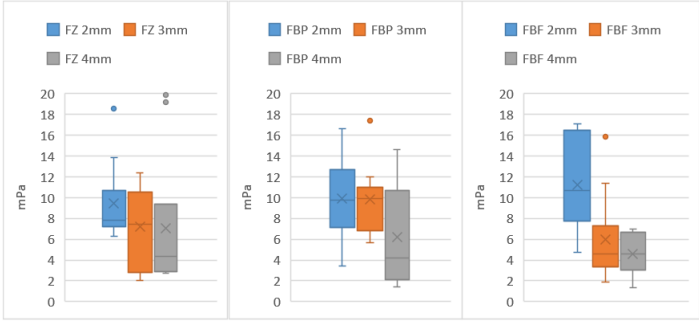



**Figure 1.** Shear bond strength (MPa) at increasing increment thickness.

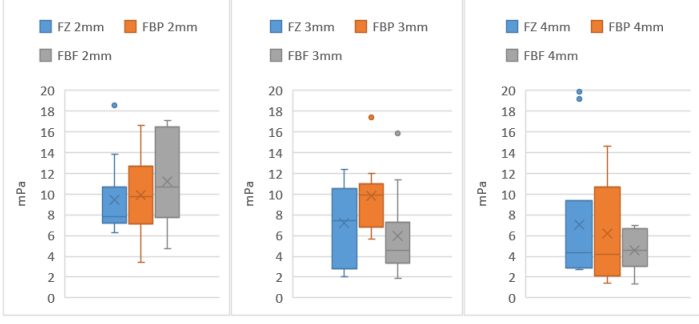



**Figure 2.** Shear bond strength (MPa) at same thickness in different composites.

## DISCUSSION

The incremental technique is a widely accepted method for placing light-curable composite resins in restorative dentistry. However, this approach has drawbacks, including prolonged application times and an increased likelihood of errors. To address these issues, manufacturers have introduced an alternative composite material known as bulk fill, allowing for the cavity to be filled in a single application. This study aimed at evaluate and contrast the SBS between two bulk-fill composite resins and a nanohybrid composite resin. Based on the statistical analyses, the first null hypothesis was partially refuted, while the second null hypothesis was accepted.

The statistical analyses indicated a significant effect of increment thickness on the SBS for FBF, with notable differences observed between 2 mm and 3 mm increments as well as between 2 mm and 4 mm increments ($P=.003$). In contrast, FZ and FBP showed no significant difference in SBS across the same range of increment thicknesses ($P>.05$). Consequently, the first null hypothesis was partially refuted. This suggests that FBF is more sensitive to variations in increment thickness compared to FZ and FBP. One possible reason for this increased sensitivity is the lower viscosity of flowable composites such as FBF, which may lead to greater polymerization shrinkage and stress.[10] Increased polymerization shrinkage could create higher internal stresses within the material, which may impact its bond strength more significantly when thicker increments are used.[11] Additionally, the flowable nature of FBF could result in a less consistent internal structure, further contributing to variations in bond strength with different increment thicknesses. In contrast, the higher viscosity of FZ and FBP may provide a more stable and even internal structure, making their bond strengths less affected by changes in increment thickness. As demonstrated in this study, the noteworthy SBS values observed in samples treated with flowable bulk-fill composite resin pertain to the bond values achieved for the tooth-adhesive-flowable bulk-fill composite applied solely to one surface of the sample tooth. The bonding surface area expands notably in multi-surface cavities characterized by a higher C factor. Importantly, filling cavities with an elevated C factor, indicative of a heightened monomer ratio with a fluid material, in a single application may result in greater shrinkage stresses compared to traditional composites.[10]

Flury et al. assessed the SBS of two packable bulk-fill composites, two flowable bulk-fill composites (one of which was Filtek Bulk Fill Flowable), and a conventional nanohybrid composite applied to dentin at different thicknesses in molar teeth.[12] The SBS values of the nanohybrid composite decreased as the layer thickness increased, and the SBS values at a 2-mm thickness were statistically significantly higher compared to 4 mm and 6 mm thicknesses. However, increasing the layer thickness did not lead to a statistically significant change in bond strength for any of the four bulk-fill materials. In contrast to that study, we found that the bond strength of the nanohybrid composite did not vary significantly at different layer thicknesses, but the flowable bulk-fill composite exhibited a statistically significant difference ($P=.003$).

Omran et al. examined how layer thickness influenced SBS using two different bulk-fill composite resins, a fiber-reinforced composite resin, and a hybrid composite resin.[13] As the layer thickness of the composite increased, the bond strength values decreased statistically significantly. Similar to our study, no statistically significant variation was observed in SBS values with increased layer thickness, except for the flowable bulk-fill composite. Furthermore, consistent with our results, no significant variations in bond strength were found among different composite resin groups at the same thicknesses.

No significant difference was observed in SBS values between the different groups at identical thickness levels ($P>.05$), leading to the acceptance of the second null hypothesis. This indicates that, when applied at the same thickness, all three types of composite resins exhibited similar bonding strengths to dentin. This may be due to

changes and developments in the organic resin structure. Methacrylate composites tend to shrink during polymerization, varying with the monomers used. Filtek™ Bulk Fill Posterior Restorative incorporates two new methacrylate monomers designed to reduce polymerization stress. AUDMA, an aromatic dimethacrylate with high molecular weight, decreases the number of reactive groups in the resin, which helps in controlling volumetric shrinkage and stiffness, thereby mitigating polymerization stress. The other monomer, an addition-fragmentation monomer (AFM), forms cross-links and cleaves during polymerization, allowing stress relief.[14] Low-viscosity monomers with low molecular weights can increase resin hardness due to higher conversion and cross-linking but also lead to higher shrinkage and stress. The resin system of Filtek™ Bulk Fill Flowable Restorative combines four high-molecular-weight monomers: BisGMA, BisEMA, Procrylat, and UDMA. Procrylat, similar to BisGMA but with lower viscosity, lacks hydroxyl groups, reducing viscosity due to decreased hydrogen bonding.[15] Despite the lack of significant differences in SBS values, considering the practical attributes of each composite resin is essential.

Çolak et al. [16] assessed the SBS of two different bulk-fill composites and two conventional nanohybrid composites applied to dentin at a 4-mm thickness in premolar teeth. The nanohybrid composite group exhibited statistically significantly higher bond strength values compared to the one bulk-fill composite group. No differences were found among the other groups. Tavangar et al. evaluated the SBS of a condensable bulk-fill composite, a flowable bulk-fill composite, and a conventional composite applied to dentin at a 4-mm thickness in premolar teeth.[17] The bulk-fill composites had statistically significantly lower bond strength values compared to the conventional composite. These two studies differ from ours since we observed no significant variation in SBS values among the different composite resin groups at the same thicknesses. Possible reasons for this discrepancy could be the use of different adhesive systems and composite resins.

**Limitations**

Although this study offers important insights into the SBS of different composite resins, several limitations should be considered. First, the use of a single adhesive system may limit the generalizability of the results because different adhesive systems could interact differently with the composite resins and dentin surfaces. Second, the sample size of 90 human molars, although adequate for initial testing, may not capture the full range of variability inherent in a larger population. Third, the use of extracted teeth, while a common practice in dental research, might not perfectly replicate in vivo conditions, potentially affecting the external validity of the results. Additionally, no aging procedures were applied in the SBS test, which may limit the understanding of the long-term stability of the bond strength values. Furthermore, variability in operator technique, even with standardized protocols, could introduce inconsistencies in sample preparation and application of the composite resins. These factors collectively highlight the need for further research, including larger, more diverse sample sizes, various adhesive systems, and potentially in vivo studies to confirm and extend these findings.

## CONCLUSION

The increment thicknesses of 2 mm to 3 mm and 2 mm to 4 mm significantly affected the SBS of flowable bulk-fill compoesite (FBF), as indicated by the p-value of 0.003. However, the increment thickness did not significantly influence the SBS of the nanohybrid composite (FZ) and the packable bulk-fill (FBP) composite, with p-values greater than 0.05. Additionally, no significant statistical variations were observed between the remaining experimental groups. From a clinical perspective, these findings suggest that the type of composite resin and the technique employed have differential impacts on SBS, with FBF being more sensitive to variations in increment thickness compared to FZ and FBP. These insights can aid clinicians in making more informed choices, ultimately enhancing the durability and success of restorative treatments.

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