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**Edited by Ritik Kashwani**
**E-mail: docritikkashwani@yahoo.com**
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# Comparison of shear bond strength and microleakage between bioactive restorative material and self-adhesive bulk fill composite - An *in vitro* study

**Mithila Mohan[1,*], D.S Dinesh[1], Bindu Meera John[2], A. Karthikeyan[1], Poorani Durai[1, 2] & Hema Pulidindi[3]**

[1]Department of Conservative Dentistry and Endodontics, Sri Venkateshwaraa Dental College (Affiliated to Pondicherry University), Ariyur, Puducherry, India; [2]Department of Conservative Dentistry and Endodontics, Indira Gandhi Institute of Dental Sciences, Sri Balaji Vidyapeeth (Deemed to be University), Puducherry, India; [3]Department of Conservative Dentistry and Endodontics, KIMS Dental College and Hospital, Amalapuram, Andhra Pradesh, India; *Corresponding author

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**Affiliation URL:**
https://svdcpondy.ac.in/
https://sbvu.ac.in/igids/
https://kimsdental.in/

**Author contact:**
Mithila Mohan - E-mail: mithilamithss@gmail.com; Phone: +91 9400778859
D.S Dinesh - E-mail: drdinesh.d.s@gmail.com; Phone: +91 9443459210
Bindu Meera John - E-mail: bindumeerajohn@igids.ac.in; Phone: +91 9443021768
A. Karthikeyan - E-mail: dr.akarthikeyan@gmail.com; Phone: +91 9943303369
Poorani Durai - E-mail: pooranidurai007@gmail.com; Phone: +91 9087603430
Hema Pulidindi - E-mail: hema.pulidindi2009@gmail.com; Phone: +91 7382484912

**Abstract:**
Achieving reliable resin-dentin bonding with minimal microleakage remains a challenge for contemporary restorative materials. Therefore, it is of interest to evaluate the shear bond strength, microleakage and resin-dentin hybrid layer of a bioactive restorative material, a self-adhesive bulk-fill composite and conventional composites using 64 extracted human teeth. Tetric N-Ceram Bulk-Fill demonstrated the highest shear bond strength (25.90 ± 0.23 MPa) and resin penetration depth (12.91 ± 0.20 µm), with minimal microleakage. SureFil one showed the lowest bond strength and resin penetration, with predominantly severe microleakage (all $p < 0.001$). The findings indicate superior bonding and marginal integrity with the bulk-fill composite compared with the self-adhesive material. Thus, data shows the importance of effective adhesive-mediated resin penetration for durable resin-dentin bonding.

**Keywords:** Shear bond strength, microleakage, bioactive restorative material, bulk-fill composite, confocal laser scanning microscopy

**Background:**
The development of restorative dentistry has focused on materials that provide optimal biological, mechanical and aesthetic properties **[1]**. Resin-based composites permit conservative cavity preparation and improved aesthetics but remain susceptible to polymerization shrinkage, postoperative sensitivity and marginal microleakage **[2].** Glass-ionomer cements provide chemical adhesion and fluoride release but have comparatively lower mechanical and aesthetic properties **[3].** Bulk-fill composites were developed to simplify restoration placement by allowing thicker increments with adequate depth of cure and reduced polymerization stress **[4]**. Bioactive restorative materials have subsequently been introduced with ion-releasing properties intended to support tooth-restoration interactions **[5]**. ACTIVA BioACTIVE Restorative is a dual-cure resin-based material containing bioactive fillers capable of releasing calcium, phosphate and fluoride ions, while SureFil One is a self-adhesive, dual-cure composite designed to reduce bonding procedures **[6]**. Durable adhesion is essential because inadequate bonding may result in gap formation, microleakage and loss of retention **[7]**. Shear bond strength and microleakage assessment, including confocal laser scanning microscopy, provide complementary evaluation of adhesive performance and interfacial integrity **[8]**. Therefore, it is of interest to compare the shear bond strength and microleakage of a bioactive restorative material, a self-adhesive bulk-fill composite and conventional composites.

**Methodology:**
This *in vitro* study was conducted in the Department of Conservative Dentistry and Endodontics, Sri Venkateshwaraa Dental College, Puducherry; shear bond strength (SBS) and confocal microleakage testing were performed at the Alpha Omega Research Institute, Salem. Sixty-four freshly extracted human molars and premolars (32 each), removed for periodontal, orthodontic or impaction-related reasons and free of caries, fissures, discolouration or fluorosis, were collected and stored in 10% formalin. For SBS evaluation, 32 molars were embedded in die stone, sectioned to expose flat dentin and randomly allocated into four groups (n = 8): Group 1 - SureFil One (Dentsply) without adhesive; Group 2 - ACTIVA Bioactive Restorative (Pulpdent); Group 3 - Tetric N-Ceram Bulk-Fill (Ivoclar); and Group 4 (control) - Tetric N-Ceram conventional composite (Ivoclar). Groups 2-4 received a standard etch-and-rinse protocol (37% phosphoric acid; Tetric N-Bond Universal adhesive). Materials were placed in standardized 4 mm-diameter cylindrical moulds and light-cured; bulk-fill material was placed in a single 4 mm increment and conventional composite in 2 mm increments. After 24-hour storage in distilled water at 37°C, SBS was measured using a universal testing machine at a crosshead speed of 0.5-1.0 mm per min and expressed in MPa. For microleakage evaluation, standardized Class II cavities (≈ 2 × 2 × 2 mm) were prepared in 32 premolars, allocated into the same four groups and restored per group protocol. Specimens underwent 1,000 thermocycles (5-55°C), were coated with nail varnish leaving a 1 mm margin and immersed in 0.6% Rhodamine B dye for 48 hours before longitudinal sectioning. Dye penetration and resin-dentin interface characteristics (hybrid layer continuity, resin penetration depth and interfacial gaps) were evaluated by CLSM (Zeiss LSM 510 Meta) at 10 × magnifications. Penetration depth was measured using ImageJ software and microleakage graded from 0 (none) to 3 (severe) using the criteria **[4].** Data were analysed using SPSS version 26. One-way ANOVA with

Bonferroni post-hoc correction was used to compare shear bond strength and resin penetration depth across groups and Fisher's exact test was used for the categorical microleakage scores. A p-value < 0.01 was considered statistically significant.

**Results:**

**Table 1** compares the shear bond strength of the four restorative materials. SureFil One recorded the lowest mean shear bond strength (18.18 ± 0.21 MPa), while Tetric N-Ceram Bulk-Fill recorded the highest (25.90 ± 0.23 MPa); ACTIVA BioACTIVE Restorative (22.51 ± 0.25 MPa) and Tetric N-Ceram Composite (24.10 ± 0.20 MPa) were intermediate. One-way ANOVA revealed a statistically significant difference among groups ($F = 1764.89$, $p < 0.01$). **Table 2** presents the Bonferroni post-hoc pairwise comparisons. Tetric N-Ceram Bulk-Fill showed significantly higher shear bond strength than all other materials and Tetric N-Ceram Composite and ACTIVA BioACTIVE Restorative were each significantly higher than SureFil One (all $p < 0.01$). **Table 3** shows the association between restorative material and microleakage grade. SureFil One exhibited predominantly severe microleakage (75.0%), whereas Tetric N-Ceram Bulk-Fill showed no microleakage in 87.5% of specimens. ACTIVA BioACTIVE Restorative and Tetric N-Ceram Composite mainly showed mild microleakage (75.0% each), with no severe cases in either group. This association was statistically significant (Fisher's exact = 35.412, $p < 0.001$). **Table 4** compares resin penetration depth measured by CLSM. Tetric N-Ceram Bulk-Fill demonstrated the greatest penetration depth (12.91 ± 0.20 µm), followed by Tetric N-Ceram Composite (11.73 ± 0.21 µm) and ACTIVA BioACTIVE Restorative (9.65 ± 0.24 µm); SureFil One showed the least penetration (6.94 ± 0.27 µm) ($F = 1009.51$, $p < 0.001$). Qualitative CLSM analysis showed a discontinuous hybrid layer with interfacial gaps for SureFil One, a moderately continuous hybrid layer for ACTIVA Bioactive Restorative and a highly continuous, gap-free hybrid layer for Tetric N-Ceram Bulk-Fill and Tetric N-Ceram Composite, corroborating the quantitative findings. **Table 5** presents Bonferroni post-hoc comparisons of resin penetration depth, confirming statistically significant differences between every pair of materials (all $p < 0.001$).

**Table 1:** Comparison of shear bond strength (MPa) among restorative material groups (N = 32). One-way ANOVA; *p ≤ 0.05 considered significant.

| Group | Mean SBS (MPa) ± SD | F-statistic | p-value |
|---|---|---|---|
| **SureFil One (n=8)** | 18.18 ± 0.21 | | |
| **ACTIVA BioACTIVE Restorative (n=8)** | 22.51 ± 0.25 | | |
| **Tetric N-Ceram Bulk-Fill (n=8)** | 25.90 ± 0.23 | 1764.89 | <0.01* |
| **Tetric N-Ceram Composite (n=8)** | 24.10 ± 0.20 | | |

**Table 2:** Bonferroni post-hoc comparisons of shear bond strength (MPa) among restorative materials; *p ≤ 0.05 considered significant.

| Reference group | Comparison group | Mean diff. (MPa) | p-value |
|---|---|---|---|
| **Tetric N-Ceram Bulk-Fill** | SureFil One | 7.72 | <0.01* |
| **Tetric N-Ceram Bulk-Fill** | ACTIVA BioACTIVE | 3.39 | <0.01* |
| **Tetric N-Ceram Bulk-Fill** | Tetric N-Ceram Composite | 1.8 | <0.01* |
| **Tetric N-Ceram Composite** | SureFil One | 5.92 | <0.01* |
| **Tetric N-Ceram Composite** | ACTIVA BioACTIVE | 1.59 | <0.01* |
| **ACTIVA BioACTIVE** | SureFil One | 4.34 | <0.01* |

**Table 3:** Association between restorative material and microleakage score (N = 32). Fisher's exact test = 35.412, $p < 0.001$.

| Group | No leakage n (%) | Mild n (%) | Moderate n (%) | Severe n (%) |
|---|---|---|---|---|
| **SureFil One (n=8)** | 0 (0.0) | 0 (0.0) | 2 (25.0) | 6 (75.0) |
| **ACTIVA BioACTIVE (n=8)** | 0 (0.0) | 6 (75.0) | 2 (25.0) | 0 (0.0) |
| **Tetric N-Ceram Bulk-Fill (n=8)** | 7 (87.5) | 1 (12.5) | 0 (0.0) | 0 (0.0) |
| **Tetric N-Ceram Composite (n=8)** | 2 (25.0) | 6 (75.0) | 0 (0.0) | 0 (0.0) |

**Table 4:** Comparison of resin penetration depth (µm) across restorative material groups (N = 32) using CLSM. One-way ANOVA; *p ≤ 0.05 considered significant.

| Group | Penetration depth (µm) ± SD | F-statistic | p-value |
|---|---|---|---|
| **SureFil One (n=8)** | 6.94 ± 0.27 | | |
| **ACTIVA BioACTIVE (n=8)** | 9.65 ± 0.24 | | |
| **Tetric N-Ceram Bulk-Fill (n=8)** | 12.91 ± 0.20 | 1009.51 | <0.001* |
| **Tetric N-Ceram Composite (n=8)** | 11.73 ± 0.21 | | |

**Table 5:** Bonferroni post-hoc pairwise comparisons of resin penetration depth (µm) between restorative material groups (N = 32); *p ≤ 0.05 considered significant.

| Reference group | Comparison group | Mean diff. (µm) | p-value |
|---|---|---|---|
| **SureFil One** | ACTIVA BioACTIVE | −2.71 | <0.001* |
| **SureFil One** | Tetric N-Ceram Bulk-Fill | −5.97 | <0.001* |
| **SureFil One** | Tetric N-Ceram Composite | −4.78 | <0.001* |
| **ACTIVA BioACTIVE** | Tetric N-Ceram Bulk-Fill | −3.26 | <0.001* |
| **ACTIVA BioACTIVE** | Tetric N-Ceram Composite | −2.07 | <0.001* |
| **Tetric N-Ceram Bulk-Fill** | Tetric N-Ceram Composite | 1.19 | <0.001* |

**Discussion:**

Contemporary restorative dentistry increasingly favours materials that simplify clinical application while maintaining marginal integrity. Polymerization shrinkage can generate interfacial stress, contributing to marginal gaps, microleakage and postoperative sensitivity **[8]**. Bulk-fill composites were developed to reduce technique sensitivity and polymerization stress while allowing thicker increments **[9]**. In the present study, Tetric N-Ceram Bulk-Fill demonstrated the highest shear bond strength and resin penetration depth, with the least microleakage, whereas SureFil One showed the poorest overall performance. Conventional composites bonded with adhesives achieve micromechanical retention through hybrid-layer formation and resin-tag penetration **[10]**. SureFil One is a self-adhesive dual-cure material designed to interact chemically with dentinal calcium without a separate adhesive step **[11]**. Its lower bond strength and greater microleakage in the present study may indicate less effective micromechanical interlocking than adhesive-mediated bonding. Similar findings have been reported, with bulk-fill composites demonstrating better bonding and sealing than self-adhesive restorative materials

**[12]**. ACTIVA BioACTIVE showed intermediate bond strength and microleakage, which may be related to its bioactive ionic matrix and chemical interaction with tooth structure **[13]**. Microleakage is influenced by polymerization shrinkage, shrinkage stress, elastic modulus and cavity-wall adaptation. Greater leakage with self-adhesive materials has also been reported compared with conventionally bonded bulk-fill composites **[14]**. The superior performance of Tetric N-Ceram Bulk-Fill may be associated with its photoinitiator system and favourable depth of cure. The observed relationship between resin penetration, bond strength and microleakage suggests that effective resin infiltration contributes to mechanical retention and marginal sealing. CLSM findings further supported better hybrid-layer formation in adhesive-bonded materials. However, the small sample size and *in vitro* design limit clinical generalizability and larger clinical studies are required to confirm these findings.

**Conclusion:**

Tetric N-Ceram Bulk-Fill showed superior bond strength, resin penetration and marginal sealing, indicating improved resin-dentin adhesion. Conventional Tetric N-Ceram showed reliable bonding, whereas ACTIVA BioACTIVE Restorative and SureFil One exhibited comparatively inferior performance. Effective resin penetration and hybrid layer formation remain critical for durable adhesion, emphasising careful restorative material selection.

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