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In Vitro Dentin Bond Durability of Additively Manufactured Hybrid Composite Restorations Luted with Preheated Composite: A Comparative Analysis of Fourth- and Sixth-Generation Adhesives

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Abstract

Background: The purpose of this in vitro study was to evaluate and compare the shear bond strength (SBS) and failure mode kinetics of indirect 3D-printed ceramic-reinforced hybrid composite restorations to human tooth dentin using fourth-generation etch-and-rinse and sixth-generation self-etch adhesive systems under a co-curing protocol with a preheated universal composite resin luting material.

Methods and Results: Sound human third molars (N=156) were prepared to expose flat mid-dentin surfaces. Simulated indirect restorations were additively manufactured as composite cylinders (VarseoSmile TriniQ, BEGO) using a Formlabs Form 4B 3D printer. Samples were randomly divided into two experimental groups based on the adhesive system used: Group 1 (n=78, OptiBond FL, fourth generation) and Group 2 (n=78, Clearfil SE Bond 2, sixth generation). Preheated universal composite resin (Filtek Z350, 55°C) was used for luting under a co-curing sequence. Each group was subdivided (n=39) into a baseline subgroup (immediate testing) and a thermocycling subgroup (10,000 cycles, 5°C/55°C). SBS trials were performed at a crosshead speed of 1 mm/min, and debonded surfaces were evaluated under 40× magnification to classify failure modes. Statistical analysis was performed using SciPy 1.11. For continuous data with a normal distribution, inter-group comparisons were performed using Student's t-test. Categorical variables were analyzed using the chi-square test. A P-value <0.05 was considered statistically significant.

At baseline, OptiBond FL (20.7 ± 4.6 MPa) and Clearfil SE Bond 2 (20.6 ± 4.2 MPa) demonstrated equivalent immediate bond strengths ($P > 0.05$) and identical mixed failure profiles. Thermocycling significantly degraded dentin bond integrity across all cohorts ($P=0.000$). However, post-aging evaluation revealed that Clearfil SE Bond 2 (16.8 ± 4.4 MPa) had a significantly higher remaining SBS than OptiBond FL (14.4 ± 4.7 MPa), $P = 0.023$. Microscopic analysis confirmed a complete absence of cohesive failures. Following thermal aging, the OptiBond FL interface underwent an acute intra-group shift toward true adhesive failures (84.6%), whereas Clearfil SE Bond 2 maintained high structural stability with a lower adhesive failure rate (61.5%), $P = 0.022$.

Conclusion. While both adhesive strategies exhibit equivalent immediate performance during preheated composite co-curing, the null hypothesis must be rejected after artificial aging. The sixth-generation 10-MDP-containing self-etch system provides superior hydrolytic stability and interfacial durability compared to the fourth-generation system. Clinically, a two-step self-etch protocol provides a more predictable, chemically driven interface for the long-term cementation of contemporary additively manufactured restorations. (**International Journal of Biomedicine. 2026;16(2):242-247.**)

Keywords: 3D-printed composite • preheated resin composite • 10-MDP • shear bond strength • failure mode

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Introduction

The clinical demand for durable indirect adhesive restorations has led to a paradigm shift from traditional dual-cure resin cements toward preheated universal composite resins for luting procedures. Dual-cure cements, while chemically versatile, frequently exhibit lower filler volume fractions, accelerated chemical degradation, and discoloration over time. Using a highly filled, preheated universal composite resin addresses these drawbacks by significantly reducing structural viscosity to achieve an optimal, micro-thin luting layer while enhancing physical properties and marginal sealing.[1-3]

Crucial to the long-term success of this approach is the integrity of the dentin-adhesive hybrid layer, which must withstand the sudden thermal energy and unique polymerization kinetics of the co-curing technique. Co-curing means that the unpolymerized adhesive layer and the overlying preheated luting composite are light-activated simultaneously. It streamlines clinical protocols but presents a challenging biomechanical scenario.[4,5]

The choice of dental adhesive strategy remains highly debated. Fourth-generation etch-and-rinse systems involve separate phosphoric acid etching, a hydrophilic primer, and a hydrophobic bonding resin layer, which acts as a robust mechanical buffer against immediate composite shrinkage forces. In turn, two-step sixth-generation self-etching adhesive systems eliminate the separate rinsing step by using a self-etching primer followed by an adhesive layer. This simplifies clinical placement and minimizes technique sensitivity, though their acidic monomers remain prone to phase separation and subsequent water treeing when subjected to rapid polymerization heat.[6,7]

To mitigate the rapid volumetric shrinkage associated with preheated universal composites, using prefabricated indirect restorations is a promising approach, as it drastically reduces the overall volume of unpolymerized resin within the tooth cavity. Historically, the fabrication of indirect restorations has evolved from traditional laboratory manual layering to subtractive CAD/CAM milling and, more recently, to additive manufacturing. Contemporary 3D-printed ceramic-reinforced hybrid composite resins (such as VarseoSmile Crown plus, BEGO) represent a breakthrough in this evolutionary trajectory, offering high-precision definitive restorations via vat polymerization.[8] These additively manufactured materials provide notable clinical benefits, including excellent dimensional adaptation, simplified chairside workflows, minimized material waste, and an antagonist-friendly mechanical buffering effect.[9] Consequently, they offer a highly promising future in restorative dentistry as a sustainable, cost-effective alternative to permanent single-unit restorations and conservative indirect treatments.[10] However, achieving predictable adhesive cementation for these contemporary printed restorations remains a highly challenging aspect of the clinical procedure.

Although several studies have evaluated the shear bond strength (SBS) of conventional polymer cements, limited data exist on how the distinct chemistries of fourth- and sixth-generation adhesive systems perform under rapid, high-temperature co-curing when a preheated composite serves as the luting material for indirect restorations.[11-13]

Therefore, the purpose of this in vitro study was to evaluate and compare the shear bond strength of indirect-printed composite restorations to human tooth dentin using fourth- and sixth-generation adhesive systems during co-curing with a preheated universal composite. The null hypothesis tested was that there is no statistically significant difference in SBS to dentin between the two adhesive generations when utilized in a preheated composite luting protocol.

Materials and Methods

Sound human third molars extracted for orthodontic reasons were selected and stored in a 0.1% thymol solution. Sample preparation required that the roots of the teeth be removed and the crowns be sliced mesiodistally into two halves. The obtained tooth slabs were embedded in self-curing acrylic resin blocks. The buccal and lingual enamel was removed using a low-speed diamond saw under constant water cooling to expose a flat, uniform mid-dentin surface. The dentin surfaces were polished with 600-grit silicon carbide paper for 60 seconds to create a standardized smear layer.

Simulated indirect restorations were additively manufactured in the shape of cylinders utilizing a ceramic-reinforced hybrid composite resin (VarseoSmile TriniQ, BEGO, Bremen, Germany). The specimens were printed using a Low Force Stereolithography (LFS) 3D printer (Formlabs Form 4B, Formlabs, Somerville, MA, USA) utilizing a flexible build platform (Build Platform Flex) at a standardized layer thickness of 50 µm. Following completion of the print cycle, the cylinders were automatically washed in high-purity isopropyl alcohol (99%) (IPA) using an automated washing unit (Form Wash, Formlabs) for 3 minutes, rinsed with fresh IPA, and thoroughly air-dried. Final post-curing was performed in a validated UV light-curing chamber (Form Cure, Formlabs) in accordance with the manufacturer's instructions for use to ensure complete monomer conversion. The finalized composite cylinders presented a height of 2.4 ± 0.1 mm and a diameter of 2.36 ± 0.02 mm. Immediately prior to adhesive application and luting, the bonding butt surfaces of the cylinders were airborne-particle abraded (sandblasted) with 27µm Al_2O_3 particles at a pressure of 2.0 bar, rinsed with water, and air-dried.

A total of 156 dentin samples were randomly allocated into two experimental groups based on the adhesive system used. In Group 1 (n=78), a fourth-generation etch-and-rinse adhesive (OptiBond FL [OFL], Kerr Corp., Orange, CA, USA) was applied. In Group 2 (n=78), a sixth-generation two-step self-etch system (Clearfil SE Bond 2 [CSEB2], Kuraray Noritake Dental, Tokyo, Japan) was utilized.

To eliminate solvent-trap-induced micro-voids, all applied adhesives were thoroughly air-dried with a gentle, continuous air stream until a completely still, glossy film was achieved. The clean, sandblasted butt surfaces of the prefabricated composite cylinders were coated with their respective adhesive bonding resins and then thoroughly air-dried in the same manner. The adhesive layers on both the dentin and the composite columns remained unpolymerized prior to the luting stage.

For luting, a highly filled universal resin composite (Filtek Z350, 3M ESPE, St. Paul, MN, USA) was utilized as the material of choice. It was preheated incrementally on a commercial heating panel up to 55 ± 3°C. The temperature of the heated composite was verified using a thermal imaging camera (UNI-T UTi260A, Uni-Trend Technology, China). A precise amount of the preheated composite was placed directly onto the adhesive-coated butt surface of the composite cylinder. To avoid rapid heat dissipation, the cylinders were immediately transferred to a bonding clamp holding the fixed tooth sample and driven into a plastic mold to a predetermined depth. This controlled insertion guaranteed a standardized uniform luting composite thickness. Constant seating pressure on the cylinders was maintained with a 1-mm-thick transparent plastic shield and the light-emitting tip of a Valo X light-curing unit (Ultradent Products Inc., South Jordan, UT, USA), which was kept in tight contact with the shield during photoactivation.

Each main adhesive group was further subdivided into two equal subgroups (n=39) based on the artificial aging parameter. In the baseline subgroup, SBS testing was conducted immediately without preliminary artificial aging. In the thermocycling subgroup, all samples underwent 10,000 thermal cycles in alternating water baths at 5°C and 55°C. The dwell time in each bath was 10 seconds, and the transfer time between baths was 5 seconds. SBS trials were performed using an Ultra Test Machine (Ultradent Products Inc., South Jordan, UT, USA). The load was applied at a constant crosshead speed of 1 mm/min until failure occurred. The maximum peak values were recorded in pounds and converted into megapascals (MPa).

Following shear testing, the debonded dentin surfaces were examined under a stereomicroscope at 40× magnification. To simplify the assessment and maximize practical relevance for clinicians and scientists, failure modes were classified into three distinct categories based on the percentage of adhesive remnants left on the substrate surface: Type I (Adhesive), characterized by less than 25% substrate coverage; Type II (Mixed), presenting between 25% and 75% substrate coverage; and Type III (Cohesive), demonstrating greater than 75% substrate coverage. This standardized thresholding eliminated ambiguous boundary definitions between mixed and pure failure types.

Statistical analysis was performed using SciPy 1.11. Baseline characteristics were summarized as frequencies and percentages for categorical variables and as mean ± standard deviation (SD) for continuous variables. For continuous data with a normal distribution, inter-group comparisons were performed using Student's t-test. Categorical variables were analyzed using the chi-square test. A *P*-value <0.05 was considered statistically significant.

Results

Shear bond strength (SBS) analysis (Table 1) revealed comparable baseline performance for OptiBond FL (20.7 ± 4.6 MPa) and Clearfil SE Bond 2 (20.6 ± 4.2 MPa), showing no statistically significant differences between the two adhesive strategies immediately after luting (*P* = 0.920). However, artificial thermal aging via thermocycling significantly degraded the integrity of the dentin bond in both primary experimental groups (*P* = 0.000). In the post-aging scenario, Clearfil SE Bond 2 (16.8 ± 4.4 MPa) performed significantly better than OptiBond FL (14.4 ± 4.7 MPa), indicating greater resistance to hydrolytic breakdown (*P* = 0.023).

Table 1.

Shear bond strength values (MPa) and statistical comparisons between experimental groups under immediate and aged conditions.

Adhesive group	Baseline subgroup (n=39)	Thermocycle subgroup (n=39)	*P*-value (aging effect)
Group 1 (OFL)	20.7 ± 4.6	14.4 ± 4.7	0.000
Group 2 (CSEB2)	20.6 ± 4.2	16.8 ± 4.4	0.000
P-value (adhesive effect)	0.920	0.023	

Visual analysis of the debonded surfaces revealed no cohesive failures across all treatment modalities. Because this failure mode did not occur, it was excluded from further description and statistical analysis. The distribution of debonding patterns was presented in Table 2 as adhesive and mixed failure modes, which together accounted for 100% of the evaluated samples.

At baseline, no statistically significant differences in the distribution of failure modes were detected between the OFL and CSEB2 groups (*P* > 0.05). Both systems demonstrated a balanced mix of pure adhesive and mixed failures. However, artificial aging induced a highly significant divergence between the two adhesive strategies.

Following 10,000 thermal cycles, a significant intragroup shift occurred in the OFL group (*P* = 0.013): the proportion of cases with purely adhesive failure rose sharply to 84.6%. Conversely, the CSEB2 group remained remarkably stable, with no significant structural shift after aging (*P* = 0.366), maintaining a high proportion of mixed failures (38.5%). Thus, following thermal aging, the interface in the OptiBond FL group demonstrated a high rate of adhesive failures (84.6%), whereas Clearfil SE Bond 2 maintained high structural stability with a lower frequency of adhesive failures (61.5%); *P* = 0.022.

Consequently, the post-aging comparison between the two primary adhesive systems revealed a highly significant difference, objectively confirming the superior structural durability of the CSEB2 interface over long-term thermal stress.

Table 2.
Distribution of failure modes and statistical significance among experimental groups.

Primary adhesive group	Subgroup (n=39)	Adhesive failure n (%)	Mixed failure n (%)	Inter-Failure mode significance (*P*-value)
Group 1 (OFL)	Baseline subgroup	23(59.0)	16 (41.0)	0.114
	Thermocycling subgroup	33 (84.6)	6 (15.4)	0.000
	Intra-Failure mode significance (*P*-value)	0.013	0.013	
Group 2 (CSEB2)	Baseline subgroup	20 (51.3%)	19 (48.7%)	0.820
	Thermocycling subgroup	24 (61.5%)	15 (38.5%)	0.043
	Intra-Failure mode significance (*P*-value)	0.366	0.366	

Discussion

Modern minimally invasive dentistry dictates a paradigm shift toward maximum tooth preservation, utilizing biomimetic materials to recreate the natural dentin-enamel junction. In indirect restorative approaches, the creation of a durable multi-layered interface relies on interfacial co-polymerization between distinct resin components.[5] Consequently, evaluating both macroscopic bond-strength parameters and microscopic failure kinetics is paramount for understanding the clinical longevity of these hybrid complexes.

In the present study, the baseline SBS testing demonstrated comparable immediate bonding efficacy between the fourth-generation etch-and-rinse (OFL) and sixth-generation self-etch (CSEB2) groups ($P > 0.05$). This initial equivalence was supported by statistically similar failure profiles, which exhibited a predominance of Type II interfacial failures localized strictly at the dentin-adhesive boundary. This baseline phenomenon is driven by a stark biomechanical mismatch: the highly resilient, 3D-printed composite cylinders possess a significantly lower elastic modulus (4 GPa) than the rigid, highly filled universal luting composite (Filtek Z350 XT, 14 GPa).[2,8] Under monotonic loading, the compliant printed substrate acts as an elastic buffer, undergoing micro-deformation and transferring destructive kinetic energy downward, where it concentrates on the rigid dentin floor.[14]

This mechanical stress concentration is further exacerbated by thermal and optical attenuation kinetics during the immediate curing phase. When the preheated (55°C) Filtek Z350 is applied, its initial contact with the room-temperature Bego cylinder surface is highly effective due to the shared resin-to-resin chemical affinity. However, immediate contact with the underlying dentin substrate results in rapid thermal dissipation within the tooth mass.[11] This localized cooling abruptly spikes the luting composite's viscosity, micro-mechanically compromising its adaptation to the unpolymerized adhesive film at the dentin floor.[3,12] Furthermore, during photoactivation, the curing light intensity attenuates exponentially as it propagates through the 2.4-mm-high printed cylinder and the intervening luting matrix.[15] The resulting reduction in total radiant exposure at the deepest interface yields a lower degree of conversion, establishing the under-polymerized dentin floor as the primary locus for structural cleavage under peak baseline loads, thereby explaining why both groups performed identically prior to aging.

Following 10,000 thermal cycles, a distinct divergence in bond durability emerged, with CSEB2 exhibiting significantly greater resistance to hydrolytic degradation than OFL. This long-term stability is fundamentally rooted in the chemical interaction of the 10-MDP monomer within CSEB2.[1,6] While water is mandatory for self-etch ionization, this bonding occurs dynamically during the active 20-second scrubbing phase. Subsequent air-drying stabilizes these self-assembled, water-insoluble 10-MDP–Ca nanolayers by eliminating volatile solvents without disrupting the newly formed ionic chemical anchor.[16] Additionally, the localized heat from the preheated luting composite acts as a thermodynamic catalyst, increasing molecular mobility and consolidating this interface prior to vitrification.[2]

This pre-established chemical foundation fundamentally dictates how the aging networks tolerate polymerization contraction stress. In the OFL group, which lacks primary chemical stabilization and relies solely on a micromechanical hybrid layer,[14] severe polymerization contraction forces easily rupture the vulnerable dentin interface over time.[17] This acute internal strain manifests after thermocycling as a statistically significant shift in failure kinetics, culminating in a predominant distribution of true adhesive failures (84.6%).

Conversely, the pre-formed 10-MDP–Ca ionic bonds in the CSEB2 group serve as robust structural anchors that buffer polymerization stress and resist hydrolytic cleavage. According to Yoshida et al.,[18] this mechanism may well explain the high clinical longevity of adhesive bonds produced by 10-MDP-based adhesives.This superior durability highlights the critical role of the hydrophilic dihydrogen phosphate functional group within the 10-MDP monomer.[6,7] By creating a stable chemical link rather than a purely mechanical one, this interaction opens highly promising avenues for biomimetic tooth restoration, successfully absorbing functional stresses while preserving the structural integrity of the treated dentin substrate.

Conclusion

Within the limitations of this in vitro laboratory study, it can be concluded that while both adhesive generations exhibit equivalent immediate bonding performance when using a co-curing technique with preheated universal composite, the null hypothesis must be rejected after thermocycling. Artificial thermal stress significantly compromises the long-term dentin bond integrity of both strategies; however, the sixth-generation 10-MDP-containing self-etch system (Clearfil SE Bond 2) demonstrates significantly superior hydrolytic stability and interfacial durability compared to the fourth-generation etch-and-rinse system (OptiBond FL).

Clinically, the use of a two-step self-etch protocol provides a more predictable, chemically driven interface for the cementation of contemporary 3D-printed ceramic-reinforced hybrid composite restorations, offering promising prospects for the longevity of biomimetic indirect treatments.

Ethical Statement

The study was approved by the Ethics Committee of the Institute of Medicine RUDN (Protocol Number: 29, dated 06.20.2024). Written informed consent was obtained from all participants prior to the processing of their teeth.

Author Contributions

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All authors have approved the final article.

Conflict of Interests

The authors have declared no conflict of interest.

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