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Influence of operator experience on the bond strength of etch-and-rinse, self-etch, and universal adhesive systems to dentin

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Abstract

Objective

To evaluate the influence of operator experience on the bonding performance of four adhesive strategies: three- and two-step etch-and-rinse (ER), and two- and one-step self-etch (SE) systems to dentin.

Materials and Methods

Sixty-four caries-free molars were divided according to the operator experience (3YG: third-year undergraduate students; 5YG: fifth-year undergraduate students; 1YR: first-year residents in Operative and Restorative Dentistry) and four adhesive systems. The occlusal third of each crown was removed to obtain a flat, mid-coronal dentin surface, which was standardized using 600-grit silicon carbide paper. Adhesives were applied following the manufacturer's recommendations, and a 4-mm-thick composite resin build-up was placed. Specimens were sectioned into resin-dentin beams (cross-sectional area ≈ $0.8mm^2$) for microtensile bond strength (μTBS) and nanoleakage (NL) evaluation. Data were analyzed using two-way ANOVA and Tukey's post-hoc test (α = 0.05%).

Results

The less experienced group (3YG) generally showed significantly lower μTBS and higher NL compared to more experienced operators ($p < 0.05$), with the exception of the 1-step SE strategy, where experience did not influence bond strength. Within the 3YG and 5YG groups, 1-step SE and 2-step ER approaches yielded the highest μTBS values, while the 1YR group performed consistently across all adhesive systems. Overall, the 1YR group achieved the lowest nanoleakage ($p < 0.01$). Consistently, the 3-step ER strategy resulted in the highest nanoleakage values regardless of operator experience, reinforcing the high technique sensitivity often associated with multi-step systems.

Conclusions

Two-step etch-and-rinse and one-step self-etch adhesives showed higher μTBS values and demonstrated lower sensitivity to operator experience compared with more technique-dependent multi-step systems.

Clinical relevance:

Simplified adhesive protocols may provide more predictable bonding outcomes in teaching environments and for clinicians with limited clinical experience.

INTRODUCTION

Adhesive dentistry has transformed contemporary restorative practice by enabling minimally invasive interventions and preserving sound dental structure [1]. The predictable bonding of restorative materials to enamel and dentin has expanded treatment possibilities, improved esthetic and functional outcomes, and contributed to the long-term maintenance of natural teeth [2, 3]. Continuous advancements in adhesive materials and bonding strategies have sought to enhance clinical reliability while simplifying application protocols [4].

Adhesive procedures comprise two main bonding strategies: etch-and-rinse (ER) and the self-etch (SE) approaches [2]. ER adhesives require prior demineralization of dental substrates using a 32%-40% phosphoric acid before application of the adhesive system. They are classified as a three-step ER system when etching, primer and bonding resin are applied as separate steps, and as a two-step ER system when the primer and bonding resin are combined in a single bottle following a separate etching step [1].

The SE strategy eliminates the need for a separate phosphoric acid conditioning step, as the adhesive bottle contains acidic monomers capable of simultaneously demineralizing and infiltrating the dental substrate. In the two-step SE method, an acidic primer is applied first, followed by a bonding resin. In contrast, one-step SE systems incorporate acidic primer and bonding resin into a single application solution [2].

The latest generation of adhesive systems is known as “universal” adhesive [5, 6]. These materials are referred to as universal because they are designed to be applied using different bonding strategies, ER, SE, or selective enamel etching, while also being compatible with multiple substrates, including enamel, dentin, ceramics, and metals [1, 2, 7]. They contain functional acidic monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), which promote chemical interactions with hydroxyapatite while enabling micromechanical retention [8]. Their simplified application protocol and clinical versatility have contributed to their widespread adoption in daily practice [9].

It is well established that multiple factors influence the longevity of adhesive restorations, including tooth type and location, material properties, operator performance, and socioeconomic, demographic, and behavioral variables [10]. However, the specific impact of operator-related factors remains underexplored.

For instance, the influence of operator experience on dentin bond strength remains a subject of intense controversy in dental literature [11–13], as evidence often conflicts regarding how clinical expertise interacts with different adhesive protocols [14, 15]. Some studies suggest that increased clinical experience correlates with higher bond strength values, showing that specialists or postgraduate students can achieve superior results compared to undergraduate students for both adhesive strategies (ER and SE) [16, 17].

Conversely, other research indicates that inexperienced operators or students may perform as well as, or even better than, experts, particularly when using simplified or self-etch adhesives [13, 18, 19]. While

certain findings highlight that experts tend to achieve more consistent results with three-step etch-and-rinse systems[20], the high technique sensitivity of these materials often leads to significant inter-operator variability regardless of the professional's background [21, 22]. Notably, despite these various investigations, a critical gap remains.

To the extent of the author’s knowledge, none of the previous studies have simultaneously compared the impact of operator experience across all four primary adhesive strategies, 3-step and 2-step ER, alongside 2-step and 1-step SE systems, within the same experimental model. Therefore, this study aimed to evaluate the influence of operator experience on the micro tensile bond strength (μTBS) and nanoleakage (NL) of different adhesive strategies applied to dentin. The null hypothesis was that operator experience does not affect μTBS and NL values.

MATERIALS AND METHODS

Tooth Selection and Preparation

Sixty-four human molars were included in this study. All stages of tooth selection and preparation were performed by an experienced clinician. Teeth were collected after approval by the local Ethics Committee. After extraction, teeth were disinfected in 0.5% chloramine solution and stored in distilled water until use. The occlusal third of each crown was removed using a diamond saw mounted in a water-cooled precision cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA) to obtain a flat mid-coronal dentin surface. The exposed dentin surfaces were examined under a stereomicroscope (Olympus SZ40, Tokyo, Japan) at 30× magnification to confirm the complete absence of enamel remnants. Subsequently, dentin surfaces were polished with wet 600-grit silicon carbide abrasive paper for 30 seconds to create a standardized smear layer.

Selection of operators

Three groups of dental students: third-year graduate dental students (3YG), fifth-year graduate dental students (5YG), and first-year residents in Restorative and Operative Dentistry (1YR) were selected. Before initiating the experimental procedures, all operators attended the same standardized training session conducted simultaneously. The session consisted of a theoretical lecture (3 hours) addressing the fundamental principles of dental adhesion, including the mechanisms of ER and SE strategies, substrate management, solvent evaporation, and light-curing considerations.

Following the theoretical component, a trained dental materials specialist provided a detailed explanation of the application protocols for each adhesive system evaluated in the study. The step-by-step procedures were reviewed according to the manufacturers’ instructions for both ER and SE application modes. A complete adhesive protocol demonstration was performed on an extracted tooth without enamel tissue to standardize the sequence of adhesive steps, application time, air-drying technique, and light-curing procedure (workshop: 2 hours). Operators were allowed to clarify doubts

during the session to ensure a complete understanding of the protocols and had access to the original manufacturer’s instructions for use, including the printed, step-by-step illustrated application charts supplied with each adhesive system, ensuring standardized protocol execution. All restorative procedures were subsequently performed simultaneously under the specialist's supervision; however, no direct intervention was made during adhesive application.

Experimental Design

Seven experimental groups were established by combining two main factors: “operator experience” (three levels): 3YG, 5YG, and 1YR; and “adhesive system” (four levels): a three-step ER adhesive (3-step ER; Optibond FL, Kerr, Orange, CA, USA); a two-step ER adhesive (2-step ER; Tetric N-Bond Universal applied in the etch-and-rinse mode; Ivoclar, Schaan, Liechtenstein); a two-step SE adhesive (2-step SE; Clearfil SE Bond (Kuraray Noritake, Tokyo, Japan); and a one-step SE adhesive (1-step SE; Tetric N-Bond Universal applied in self-etch mode; Ivoclar, Schaan, Liechtenstein). Detailed product information is provided in Table 1.

Table 1
Application mode according to manufacturer's instructions or experimental conditions.

Material (Batch Number)	Manufacturer	Composition	Application mode
OptiBond FL (162830)	Kerr Co., Orange, USA	*Primer*: HEMA, GPDM, MMEP, water, ethanol, CQ and BHT *Adhesive*: Bis-GMA, HEMA, GDMA, CQ, and filler (fumed SiO_2, barium alumi-borosilicate, Na_2SiF_6), coupling factor A174	**Three-step Etch-and-Rinse (3-step ER group)** 1. Apply 37% phosporic acid for 15 s 2. Rinse for 30 s with distilled water 3. Air-drying for 15 s obtaining a moist dentin 4. Two separated layers of primer were applied with a different micro-applicator for 15 s. 5. Mild air-blowing was carried out for 15 s after each application 6. Adhesive layer application and light-cured for 20 s
Clearfil SE Bond (C12840)	Kuraray Noritake Dental Inc., Tokyo, Japan	*Primer*: 10-MDP, HEMA, hydrophilic dimethacrylate, CQ, DEPT, water, ethanol. *Adhesive*: 10-MDP, HEMA, bis-GMA, hydrophobic dimethacrylate, CQ, DEPT, silanized colloidal silica	**Two-step Self-Etch (2-step SE group)** 1. Two separated layers of primer were applied with a different micro-applicator for 15 s. 2. Mild air-blowing was carried out for 15 s after each application 3. Adhesive layer application and light-cured for 20 s
Tetric -Bond Universal (U42904)	Ivoclar, Schaan, Liechtenstein	1. HEMA, 10-MDP, Bis-GMA, MCAP, D3MA, ethanol. Water, higly dispersed silicon dioxide and CQ	**Two-step Etch-and-Rinse (2-step ER group)** 1. Apply 37% phosphoric acid for 15 s

* Based on manufacturers' MSDS. HEMA = hydroxy ethyl methacrylate; GPDM = glycero-phosphate dimethacrylate; MMEP = methacryloyloxy-ethyl-dihydrogen phosphate; CQ = camphorquinone; BHT = butyl hydroxy toluene; Bis-GMA = bisphenol A-glycidyl methacrylate; GDMA = glycerol- dimethacrylate; 10-MDP = 10-methacryloyloxydecyl dihydrogen phosphate; DEPT = N,N-Diethyl-p-toluidine; MCAP =methacrylated carboxylic acid polymer; D3MA =decandiol dimethacrylate.

Material (Batch Number)	Manufacturer	Composition	Application mode
			2. Rinse for 30 s with distilled water 3. Air-drying for 15 s obtaining a moist dentin 4. Two separated layers of adhesive were applied with a different micro-applicator for 15 s. 5. Mild air-blowing was carried out for 15 s after each application and light-cured for 20 s **One-step Self-Etch (1-step SE group)** 1. Two separated layers of adhesive were applied with a different micro-applicator for 15 s. 2. Mild air-blowing was carried out for 15 s after each application and light-cured for 20 s

* Based on manufacturers' MSDS. HEMA = hydroxy ethyl methacrylate; GPDM = glycero-phosphate dimethacrylate; MMEP = methacryloyloxy-ethyl-dihydrogen phosphate; CQ = camphorquinone; BHT = butyl hydroxy toluene; Bis-GMA = bisphenol A-glycidyl methacrylate; GDMA = glycerol- dimethacrylate; 10-MDP = 10-methacryloyloxydecyl dihydrogen phosphate; DEPT = N,N-Diethyl-p-toluidine; MCAP =methacrylated carboxylic acid polymer; D3MA =decandiol dimethacrylate.

Sample size calculation

Sample size calculation was performed using an online tool (www.sealedenvelope.com; accessed March 2022). The calculation was based on previously reported micro tensile bond strength (μTBS) values for sound dentin (43.8 ± 5.3 MPa)[23]. To detect a difference of 8 MPa between groups at a significance level of 5% and a statistical power of 80% (two-sided test), the minimum required sample size was eight teeth per group (n = 8), in accordance with μTBS testing guidelines[23].

Restorative procedures

Adhesive systems were applied according to the manufacturers' instructions and light-cured for 20 s using a light-curing unit (Bluephase NG4, Ivoclar, Schaan, Liechtenstein) at 1200 mW/cm². All teeth were restored using a composite resin build-up (Tetric, A2, Ivoclar, Schaan, Liechtenstein), applied in two 2-

mm increments, each increment light-cured for 40 s 1200 mW/cm² using the same device. The irradiance output was periodically monitored using a radiometer (Bluephase Meter II, Ivoclar, Schaan, Liechtenstein).

Micro tensile Bond Strength Test (μTBS)

Restored teeth were stored in distilled water at 37°C for 24 h. Specimens were sectioned longitudinally using a cutting precision machine (Isomet 1000, Buehler, Lake Bluff, IL, USA) and subsequently rotated 90° to obtain resin–dentin bonded sticks with a cross-sectional area of approximately 0.8 mm², measured with digital calipers (Digimatic Caliper, Mitutoyo, Tokyo, Japan). Each tooth yielded 25–30 resin–dentin bonded sticks. The resin–dentin bonded sticks were assigned as follows: three resin–dentin–bonded sticks per tooth from each experimental group were used for nanoleakage test, while the remaining resin–dentin bonded sticks were subjected to μTBS testing.

After 24 hours of storage, the resin-dentin specimens were attached to a Geraldeli microtensile device using cyanoacrylate adhesive (IC- Gel, bSi, Atascadero, CA, USA) and subjected to a tensile testing in a universal testing machine (AG-I, Shimadzu Autograph, Tokyo, Japan) at a crosshead speed of 0.5 mm/min until failure. μTBS values (MPa) were calculated by dividing the failure load (KgF) by the specimen's cross-sectional area (mm^2) [23, 24].

Failure modes were classified under stereomicroscope (Olympus SZ40; Tokyo, Japan) at a magnification of 100× as: cohesive (C: failures occurring solely within dentin or composite resin), adhesive (A: failures at the resin-dentin interface), and mixed (M: combination of adhesive and cohesive failure). Specimens with premature failure (PF) were assigned a bond strength value of 4.0 MPa and included in the tooth mean calculation [25].

Nanoleakage (NL)

For each group, three resin–dentin–bonded sticks per tooth were immersed in an ammoniacal silver nitrate solution for 24 h in darkness. Afterward, specimens were rinsed with distilled water and immersed in photo-developing solution under fluorescent light for 8 h^{27}. Specimens were polished using 2500-grit SiC paper, followed by 1-μm and 0.5-μm diamond pastes (Diamond Polish, Ultradent, South Jordan, UT, USA). After ultrasonic cleaning and air-drying, specimens were mounted on stubs and sputter-coated with carbon-gold (MED 010, Balzers Union, Liechtenstein). Silver deposition at the resin–dentin interface was analyzed using a field-emission scanning electron microscope (VEGA 3 TESCAN, Shimadzu, Tokyo, Japan) operating in backscattered electron mode.

Three images per resin-dentin bonded stick were captured: one at the center and two at 0.3 mm to the right and left of the center. Nanoleakage percentage along the adhesive and hybrid layers was quantified using ImageJ software (v1.53, National Institutes of Health, USA) [26].

Statistical Analysis

For statistical analysis, the mean μTBS and NL values of all resin-dentin-bonded sticks from the same tooth were calculated; therefore, the experimental unit was the tooth (n = 8).

Normality (Shapiro–Wilk test) and homogeneity of variance (Levene's test) were verified. Data were analyzed using two-way ANOVA (operator experience × adhesive strategy) for both μTBS (MPa) and nanoleakage (%), followed by Tukey's post hoc test. The significance level was set at 5%. Statistical analyses were conducted using SPSS®, version 25.0 (SPSS Inc., Chicago, IL, USA).

RESULTS

Micro Tensile Bond Strength (μTBS)

Figure 1 illustrates the distribution of failure modes. Cohesive failures were rare (only 2% of all specimens), while mixed failures accounted for 7% of the total. Most mixed failures occurred in the 1YR and 5YG groups. The majority of specimens showed adhesive or adhesive/mixed failures; however, the 3YG group showed a higher proportion of purely adhesive failures.

Two-way ANOVA detected a significant interaction between operator experience and adhesive strategy) for all dependent variables (p = 0.03). μTBS (MPa) values are presented in Table 2. Overall, the bonding interfaces created by the less experienced 3YG group showed significantly lower μTBS values compared with those created by the other operator groups in most bonding approaches (p < 0.001). The only exception was the 1-step SE approach, in which operator experience did not significantly influence the results.

Table 2
Mean micro tensile bond strength values (MPa ± standard deviation) for resin-dentin bonded sticks across all experimental groups.

GROUPS	3-step ER	2-step SE	2-step ER	1-step SE
3YG	42.7 ± 4.1 Cd	45.3 ± 4.0 Bc	46.2 ± 4.6 Bb	50.1 ± 4.5 Aa
5YG	47.2 ± 3.9 Bb	47.4 ± 4.3 Ab	50.6 ± 3.6 Aa	50.3 ± 4.0 Aa
1YR	52.8 ± 4.5 Aa	49.3 ± 3.3 Aa	52.1 ± 3.9 Aa	51.7 ± 3.8 Aa
(*) Means followed by the same uppercase letters within column and lowercase letters within the row are not significantly different (two-way ANOVA and Tukey's test; p < 0.05).				
3YG: third-year graduation students, **5YG**: fifth-year graduation students, **1YR**: first-year residents in Operative and Restorative Dentistry; **ER**: etch-and-rinse, **SE**: self-etch.				

The 1YR group achieved significantly higher μTBS values than the 5YG group only when 3-step ER was used (p = 0.03). For the remaining adhesive systems, no significant differences were observed between 1YR and 5YG operators.

When adhesive strategies were compared within the 3YG group, the 1-step SE group demonstrated significantly higher μTBS values than the 2-step ER group ($p = 0.03$), the 2-step SE group ($p = 0.02$), and the 3-step ER group ($p < 0.001$). Within the 5YG group, no significant differences were found between 1-step SE and 2-step ER groups ($p = 0.99$), and both showed significantly higher μTBS values than 2-step SE and 3-step ER approaches ($p = 0.04$). In the 1YR group, no significant differences were detected among adhesive strategies ($p = 0.97$).

Nanoleakage analysis (NL)

Two-way ANOVA detected a significant interaction between operator experience and adhesive strategy ($p = 0.03$). NL (%) values are shown in Table 3.

Table 3
Means of silver nitrate deposition (% ± standard deviation) for resin-dentin bonded sticks across all experimental groups.

GROUPS	3-step ER	2-step SE	2-step ER	1-step SE
3YG	24.7 ± 1.3 Cd	18.3 ± 1.2 Cc	14.2 ± 0.8 Cb	10.1 ± 0.6 Ba
5YG	20.3 ± 1.4 Bd	16.4 ± 0.9 Bc	11.6 ± 1.2 Bb	9.1 ± 0.9 ABa
1YR	8.8 ± 0.8 Abc	7.3 ± 0.9 Abc	7.1 ± 0.6 Aab	8.1 ± 0.7 Abc
(*) Means followed by the same uppercase letters within column and lowercase letters within the row are not significantly different (two-way ANOVA and Tukey's test; $p < 0.05$).				
3YG: third-year graduation students, **5YG**: fifth-year graduation students, **1YR**: first-year residents in Operative and Restorative Dentistry; **ER**: etch-and-rinse, **SE**: self-etch.				

Significant differences in silver nitrate uptake were observed between adhesive strategies and between operator experience groups ($p < 0.001$). The 3YG group exhibited the highest NL values ($p = 0.02$), while the 1YR group showed the lowest values ($p < 0.001$). No significant difference in the NL values was observed between the 5YG and 1YR groups ($p = 0.80$).

When bonding approaches were compared within each operator experience group, 3-step ER consistently demonstrated the highest NL values, regardless of operator experience. In the 3YG and 5YG groups, 3-step ER values were significantly higher than those of the 2-step SE group ($p < 0.001$), which in turn were higher than those of the 1-step SE ($p < 0.001$) and 2-step ER ($p < 0.001$). Within the 1YR group, no significant difference in NL values was observed between the 3-step ER and 1-step SE groups ($p = 0.118$), nor between the 2-step SE and 2-step ER groups, the latter two presenting the lowest nanoleakage values.

Figure 2 shows representative SEM photomicrographs for all experimental groups. Silver nitrate infiltration, indicated by a white hand, was observed in all groups. Greater silver deposition within the hybrid layer was evident in specimens prepared by less experienced operators (3YG and 5YG) compared with those prepared by first-year residents (1YR).

DISCUSSION

The present study showed a significant interaction between operator experience and adhesive strategy for both μTBS and NL. Significant differences were also observed among adhesive strategies within each operator groups. Therefore, the null hypothesis was rejected. These findings indicate that the complexity of the bonding protocol significantly modulates the influence of operator experience on bonding properties to dentin.

For μTBS, operator experience had a greater impact when more complex adhesive protocols were used. The 3-step ER system exhibited the most pronounced operator-dependent pattern, with 3YG showing lower μTBS than 1YR. In contrast, the 1-step SE strategy was not significantly affected by operator experience. Within the 1YR group, μTBS values were comparable across adhesive strategies, suggesting that increased clinical training may attenuate the influence of procedural complexity [16, 17, 20, 27].

The increased sensitivity observed with certain adhesive strategies appears to be related to the total number of independent clinical steps in the protocol. In some systems, this includes a separate phosphoric acid etching step followed by rinsing and substrate management. These ER strategies require careful timing, thorough rinsing, removal of excess moisture, and careful substrate handling, thereby adding more time-sensitive clinical actions.

In the 3-step ER system, primer and adhesive are applied separately after acid conditioning, which increases the number of steps, the number of bottles to handle, and the time required for application. Each stage requires opening, dispensing, closing, and properly applying different components in a strict sequence. In contrast, the 2-step ER strategy keeps the etch-and-rinse method but combines primer and adhesive into a single step after acid etching, reducing the number of independent application steps while still maintaining the phosphoric acid etch. Although the presence of phosphoric acid alone did not consistently lead to lower bonding performance, its inclusion as a separate clinical stage adds an additional procedural step to the overall protocol. The results suggest that operator influence is more closely related to the total number of independent stages than to the etching approach itself.

The 2-step SE strategy showed moderate performance, removing the need for separate phosphoric acid conditioning while keeping multiple application stages. In this method, an acidic primer is applied and air-dried to evaporate the solvent, followed by a separate adhesive layer. It maintains sequential execution and timing control without the ER step. Conversely, the 1-step SE system integrates conditioning and bonding into a single procedure, reducing the need for sequential stages and potentially minimizing cumulative technique-related deviations. When adhesive protocols involve more bottles and sequential time-dependent steps, inter-operator variability may increase due to cumulative technique-sensitive actions [20, 28]. This may explain the differences observed, which were reflected not only in absolute μTBS and NL values but also in the performance across experience levels.

In this matter, the literature shows conflicting results about how operator experience affects adhesive outcomes [11–13]. This variability may be partially explained by methodological heterogeneity across

studies. Differences in the adhesive categories evaluated, the number of application steps, substrate conditions, operator training level, and degree of prior familiarity with the materials may substantially affect outcomes [13, 18]. Studies assessing simplified 1-step adhesives may report minimal operator-dependent effects [21, 22], whereas investigations involving multi-step ER protocols often demonstrate greater inter-operator variability [20]. In addition, differences in outcome measures, such as shear bond strength [14, 16, 22], tensile bond strength [20], marginal gap width [21], and microleakage [14], may further contribute to inconsistent findings.

Besides operator-related variability, inherent differences among adhesive strategies likely contributed to the observed variations in μTBS and NL. Differences in solvent composition [29, 30], hydrophilicity, and functional monomer chemistry can influence hybrid layer formation, solvent evaporation efficiency, and interfacial permeability [31, 32]. For instance, strategies that require strict control of solvent evaporation and air thinning may be more susceptible to performance inconsistencies if application procedures are not carefully standardized [33]. Therefore, differences among adhesive methods might arise not only from operator execution but also from their procedural and compositional variations.

Limitations of this study include the inherent constraints of *in vitro* testing research, which limit direct extrapolation to clinical scenarios. In addition, the short-term outcomes preclude assessment of long-term adhesive interface, considering the different adhesive systems and operator experience level. Nevertheless, the findings suggest that simplified one-bottle adhesive approaches may decrease variability between operators without compromising effectiveness when properly executed.

CONCLUSIONS

Within the limitations of this in vitro study, operator experience influenced dentin bonding performance depending on the adhesive strategy employed. Simplified one-bottle adhesive approaches (2-step ER and 1-step SE) demonstrated comparable μTBS and NL outcomes across different experience levels, indicating less operator-dependent variability. In contrast, strategies involving multiple sequential clinical stages/bottles, such as the 3-step ER or 2-step SE, showed greater sensitivity to differences in operator experience.

Declarations

Conflict of Interest

The authors declare that they have no conflict of interest.

Ethical Approval

This article does not contain any studies with human participants or animals performed by any of the authors. Human tissue (teeth) was obtained with written informed consent, in accordance with a local ethics-approved protocol.

Informed Consent

For this type of study, formal consent is not required.

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Author Contribution

C.P.: Conceptualization, Investigation, Methodology, Visualization, Writing – original draft. C.F.: Investigation, Writing – review & editing. C.A.: Conceptualization, Project administration, Writing – review & editing. A.L.: Supervision, Writing – review & editing.

Data Availability

The data supporting the findings of this study are not publicly available but can be obtained from the corresponding author upon reasonable request.

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Figures

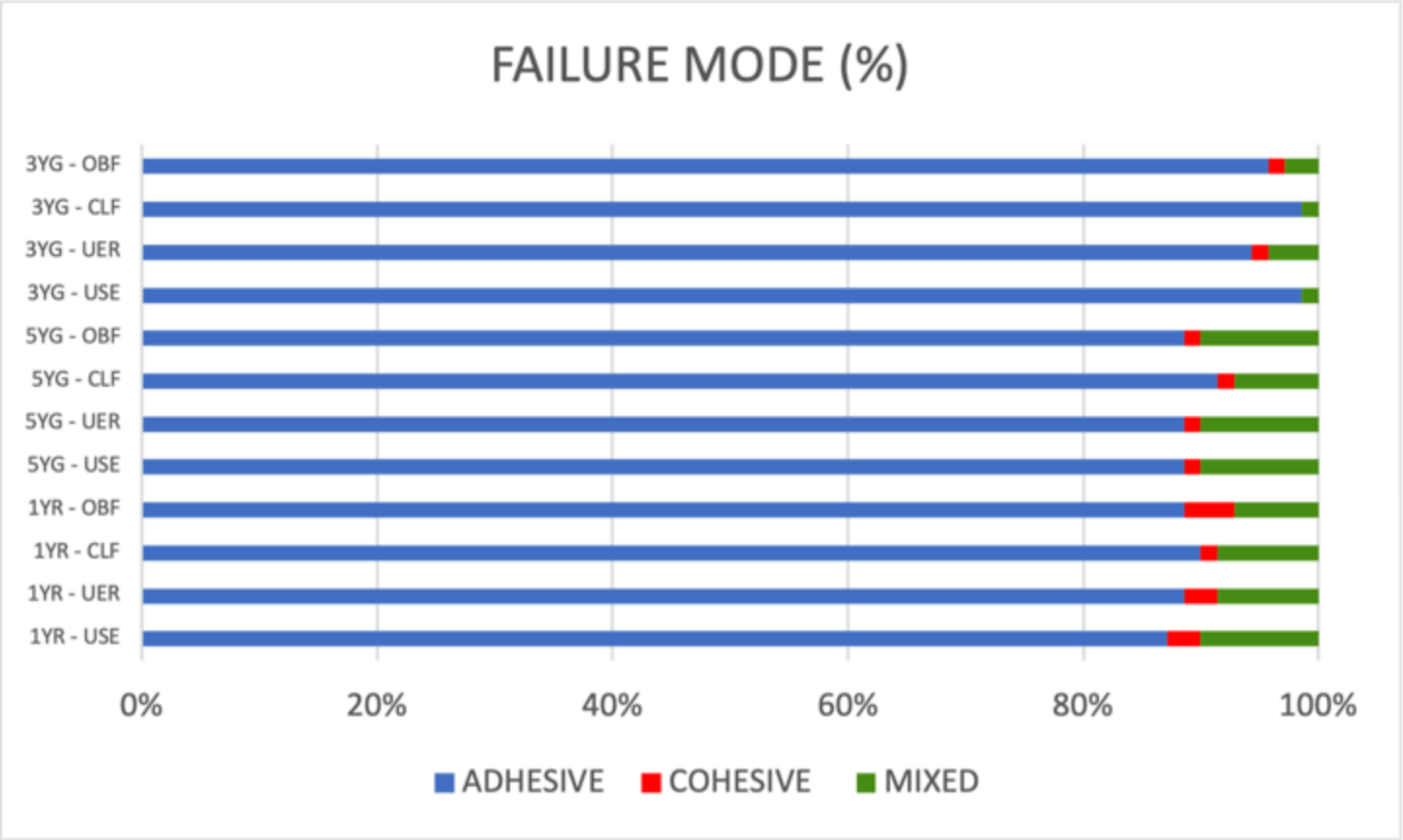


Figure 1

Distribution of failure modes across all experimental conditions

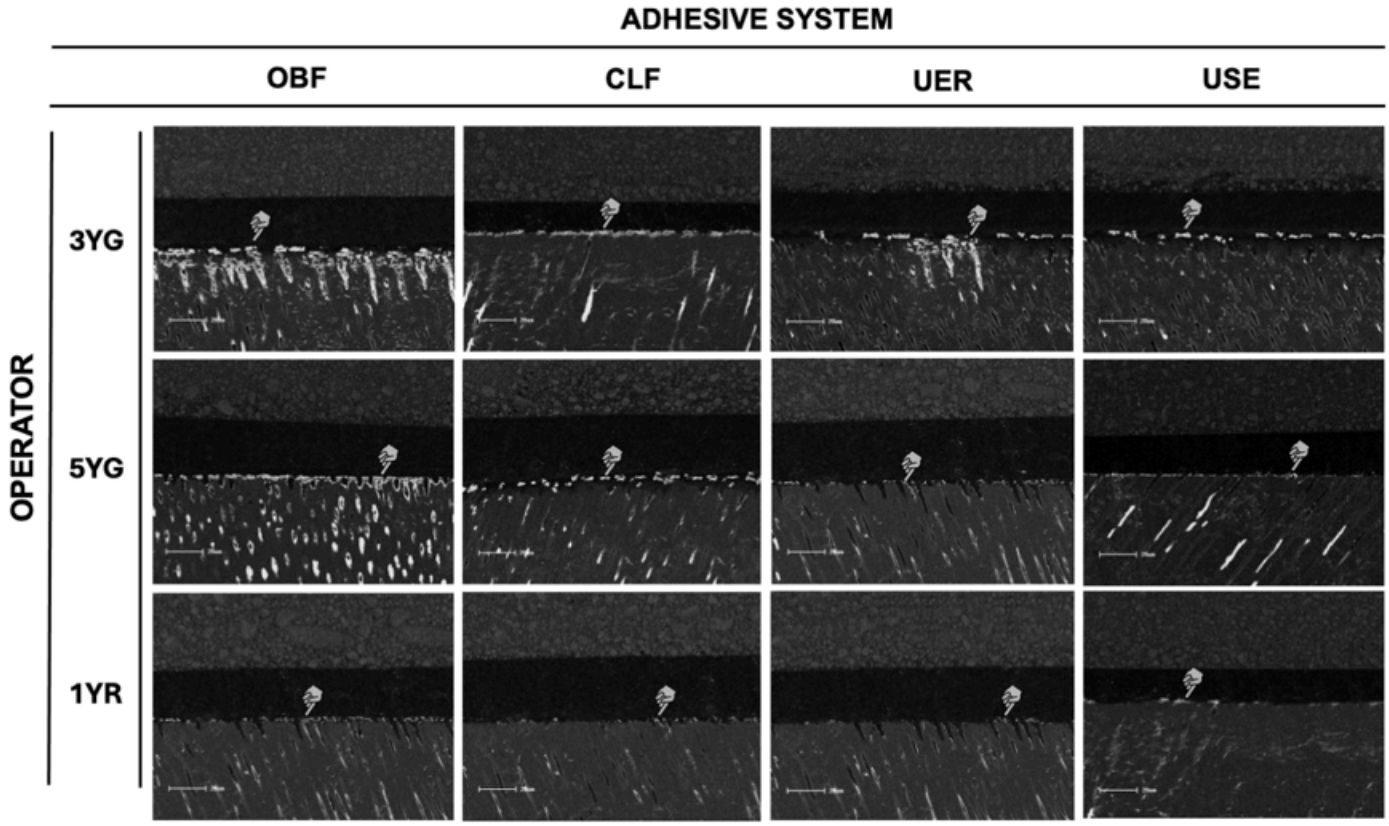


Figure 2

Representative backscatter SEM images of the adhesive interface for all experimental groups. Silver nitrate infiltration (indicated by arrows) was observed within the hybrid layer in all groups.