CLINICAL AND LABORATORY EVALUATION OF ADHESIVE FIXATION PROTOCOLS FOR FIXED RESTORATIONS

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ABSTRACT

Aim of the study To compare the performance of different adhesive fixation protocols for indirect ceramic restorations in terms of microtensile bond strength, thermal aging resistance, microleakage, failure modes, postoperative sensitivity, and 12-month clinical retention. **Material and methods** This combined in vitro (n = 120) and prospective clinical study (n = 80) evaluated four adhesive protocols: Group A (total-etch), Group B (universal adhesive in self-etch mode), Group C (self-adhesive resin cement), and Group D (Immediate Dentin Sealing [IDS]). Laboratory specimens underwent 10,000 thermocycles. Baseline and post-aging microtensile bond strength (μTBS), microleakage, and failure modes were assessed. Clinical retention and postoperative sensitivity were evaluated over 12 months. Statistical analysis included parametric and non-parametric tests and chi-square analysis ($\alpha = 0.05$). **Results** Group D showed the highest baseline μTBS (41.63 ± 3.80 MPa), while Group C showed the lowest (22.18 ± 5.40 MPa). After aging, μTBS decreased significantly in all groups ($p < 0.001$), with reductions of 21.77%, 27.41%, 48.40%, and 22.62% in Groups A–D, respectively; differences in reduction were not significant ($p = 0.291$). Severe microleakage was absent in Group D, whereas Group C had the highest Score 2–3 penetration (63.3%; $p < 0.001$) and adhesive failures (70.0%). At 12 months, retention was 100.0%, 95.0%, 90.0%, and 80.0% in Groups D, A, B, and C, respectively ($p = 0.140$). Sensitivity was highest in Group A at 1 month ($p < 0.001$) and resolved by 6 months. **Conclusions** Within the study limitations, the IDS protocol was associated with the highest baseline bond strength, favorable post-aging performance, less severe microleakage, and the highest observed clinical retention. Group C showed the greatest bond-strength reduction and lowest observed retention.

Key words: Immediate dentin sealing; Microtensile bond strength; Microleakage; Clinical retention; Self-adhesive resin cement.

INTRODUCTION

Adhesive fixation of fixed restorations is a key determinant of clinical performance, influencing bond stability, marginal sealing, load distribution, and long-term restoration survival [1–3]. The evolution from conventional etch-and-rinse systems to universal adhesives and self-adhesive materials has expanded treatment options and facilitated preservation of hard dental tissues [3, 4].

Selection of an adhesive protocol depends on the substrate, restoration, operating conditions, surface pretreatment, and expected functional load [5, 6]. Universal adhesives offer simplified application, but their performance remains technique-sensitive. Application mode, adhesive film thickness, solvent evaporation, and polymerization may affect both initial bond strength and long-term stability [7–10].

The restorative substrate and its surface pretreatment are also critical. For glass ceramics and zirconia, bonding effectiveness depends on micromechanical retention, chemical interaction, and the selected luting system [11, 12]. Functional monomers, particularly 10-MDP, may enhance chemical interaction with dental substrates, while degradation of the adhesive

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interface can compromise bond durability over time [13–15].

Although numerous studies have evaluated individual adhesive systems or luting cements, relatively few have compared contemporary adhesive fixation protocols using both laboratory and clinical outcomes. Evidence directly linking bond durability after thermal aging with clinical performance also remains limited. Therefore, comparative evaluation of contemporary adhesive fixation protocols is warranted.

OBJECTIVES

The aim of the study was to compare the laboratory and clinical outcomes of different adhesive fixation protocols for indirect ceramic restorations in terms of microtensile bond strength, resistance to thermal aging, microleakage, failure modes, postoperative sensitivity, and 12-month clinical retention

MATERIAL AND METHODS

Study Design and Ethical Approval

This combined study included an in vitro experiment with 120 specimens (30 per group) and a prospective 12-month clinical follow-up of 80 fixed restorations (20 per group). The protocol was approved by the Bioethics Committee (Protocol No. 5, February 18, 2026) and followed the Declaration of Helsinki. Sample size was calculated using G*Power 3.1.9.7 (Heinrich-Heine-Universität Düsseldorf, Germany; f = 0.35, α = 0.05, power = 0.80), yielding minimum requirements of 27 laboratory specimens and 18 clinical restorations per group.

Study Groups and Experimental Protocols

Four adhesive fixation protocols for indirect ceramic restorations (veneers and inlays/onlays) were investigated (Table I).

Table I. Study design and group characteristics

Group	Adhesive fixation protocol	Main components	Laboratory stage	Clinical stage
A	Total-Etch	35% phosphoric acid + OptiBond FL + composite resin cement	μTBS microleakage, failure mode	Retention and Schiff sensitivity)
B	Universal Adhesive (Self-Etch)	Single Bond Universal + composite resin cement	μTBS microleakage, failure mode	Retention and Schiff sensitivity
C	Self-Adhesive Cement	RelyX Unicem 2 without separate etching/bonding	μTBS microleakage, failure mode	Retention and Schiff sensitivity
D	Immediate Dentin Sealing (IDS)	Clearfil SE Bond 2 + composite resin cement	μTBS microleakage, failure mode	Retention and Schiff sensitivity

For Group A, dentin was conditioned with 35% phosphoric acid (Ultra-Etch, Ultradent Products, USA) for 15 s, rinsed, bonded with OptiBond FL (Kerr Corp., USA), and luted with RelyX Ultimate (3M ESPE, USA). Group B received Single Bond Universal (3M ESPE) in self-etch mode for 20 s, air-dried for 5 s, light-cured for 10 s, and luted with RelyX Ultimate. Group C was luted directly with RelyX Unicem 2 (3M ESPE) without separate etching or bonding. In Group D, dentin was etched with 35% phosphoric acid for 15 s and immediately sealed with Clearfil SE Bond 2 (Kuraray Noritake Dental, Japan). At final delivery, the pre-sealed surface was air-abraded with 27-μm Al_2O_3 at 1.5 bar, followed by the specified surface conditioning procedure, and luted with RelyX Ultimate.

Restoration Substrate Pretreatment

Lithium disilicate restorations (IPS e.max CAD, Ivoclar Vivadent, Liechtenstein) were etched with 5% hydrofluoric acid for 20 s, ultrasonically cleaned in distilled water for 5 min, dried, and treated with Monobond Plus for 60 s. Monolithic zirconia (Katana Zirconia, Kuraray Noritake) was sandblasted with 50-μm

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Al_2O_3 at 2.0 bar and treated with an MDP-containing ceramic primer (Ceramic Primer Plus).

Laboratory Testing: Microtensile Bond Strength (μTBS), Aging, and Microleakage

Freshly extracted caries-free human third molars were stored in 0.1% thymol at 4 °C for no longer than one month. Mid-coronal dentin was exposed and ground flat with 600-grit SiC paper under water cooling. After the assigned adhesive protocol and resin composite buildup, specimens were sectioned into 1.0 × 1.0 mm² microbeams.

Baseline μTBS was measured using a universal testing machine (ZwickRoell Z010, Germany) at 0.5 mm/min. Artificial aging comprised 10,000 thermocycles between 5 °C and 55 °C, with 30-s dwell and 10-s transfer times. Post-aging μTBS was then measured. Thirty specimens per group were evaluated at both time points, and individual Baseline − Post-aging changes were calculated.

Marginal microleakage was assessed on a dedicated subset after thermocycling and immersion in 2% methylene blue for 24 h at 37 °C. Two calibrated examiners blinded to group assignment scored dye penetration from 0 to 3 using stereomicroscopy (20×–40×). Failure modes were classified as adhesive, cohesive, or mixed at 40× [16,17].

Clinical Evaluation Protocol

Eighty fixed restorations (20 per group) were evaluated prospectively at 1, 6, and 12 months. Veneers and inlays/onlays were assessed using modified FDI World Dental Federation criteria for retention, marginal adaptation, and secondary caries. Postoperative cold-air sensitivity was assessed using the Schiff scale (0 = no response; 3 = severe discomfort requiring discontinuation).

Statistical Analysis

Analyses were performed using IBM SPSS Statistics 26.0 (IBM Corp., USA). μTBS normality was assessed using the Shapiro–Wilk test. Continuous variables are presented as mean ± SD. Baseline and post-aging μTBS were compared using one-way ANOVA with Tukey HSD post-hoc testing; within-group changes were assessed using paired t-tests, and Baseline − Post-aging changes were compared among groups by one-way ANOVA.

Microleakage and Schiff scores were analyzed using Kruskal–Wallis tests with pairwise Mann–Whitney U tests and Bonferroni correction. Retention and failure modes were analyzed using Pearson's χ^2 test. Statistical significance was set at $p < 0.05$.

RESULTS

Microtensile Bond Strength (μTBS) and Thermal Aging Resistance

Baseline μTBS differed significantly among groups ($F(3,116) = 102.98$, $p < 0.001$), with the highest value in Group D (41.63 ± 3.80 MPa) and the lowest in Group C (22.18 ± 5.40 MPa) (Table II).

Table II. Microtensile Bond Strength (μTBS) Before and After Thermal Aging (n = 30 per group)

Group	Baseline μTBS (MPa)	Post-aging μTBS (MPa)	Reduction (%)
A	35.93 ± 2.90	28.11 ± 3.14	21.77
B	30.76 ± 5.24	22.33 ± 7.59	27.41
C	22.18 ± 5.40	11.45 ± 6.13	48.40
D	41.63 ± 3.80	32.22 ± 5.18	22.62
One-way ANOVA	$F(3,116) = 102.98$, $p < 0.001$	$F(3,116) = 73.99$, $p < 0.001$	—
Change scores	—	—	$F(3,116) = 1.26$, $p = 0.291$

Notes: Values are mean ± SD. Reduction (%) = [(Baseline − Post-aging)/Baseline] × 100. Change scores were compared among groups by one-way ANOVA.

After 10,000 thermocycles, μTBS remained significantly different among groups ($F(3,116) = 73.99$, $p < 0.001$, Fig. 1).

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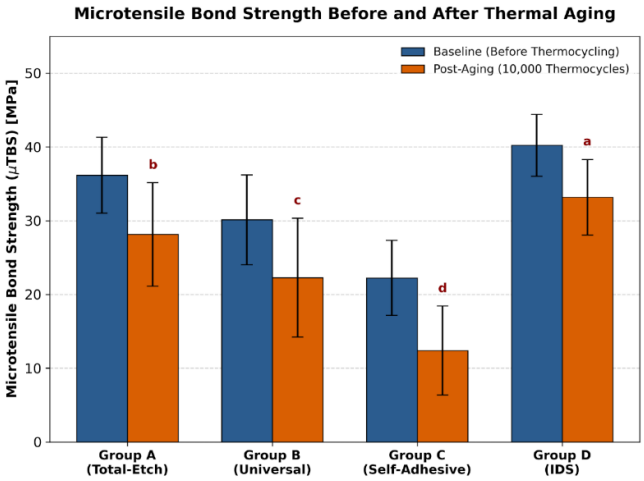


Figure 1. Microtensile bond strength (μTBS) in the study groups before and after thermocycling

Within-group reductions were significant in all groups (A: $t(29) = 12.36$; B: 6.32; C: 7.50; D: 10.01; all $p < 0.001$). Relative reductions were 21.77%, 27.41%, 48.40%, and 22.62% in Groups A–D, respectively. The magnitude of individual change did not differ significantly among groups ($F(3,116) = 1.26$, $p = 0.291$).

Marginal Microleakage Evaluation

Microleakage scores differed significantly among groups (Kruskal–Wallis $H(3) = 42.53$, $p < 0.001$; Table III). After Bonferroni correction, Group D had lower scores than Groups B and C, while Group A had lower scores than C and Group B had lower scores than C (adjusted $p < 0.05$). No significant differences were found between A and B or A and D.

Table III. Score distribution of marginal microleakage following thermocycling (n = 30 per group)

Group	Score 0	Score 1	Score 2	Score 3
A	22 (73.3%)	6 (20.0%)	0 (0.0%)	2 (6.7%)
B	14 (46.7%)	10 (33.3%)	4 (13.3%)	2 (6.7%)
C	5 (16.7%)	6 (20.0%)	11 (36.7%)	8 (26.7%)
D	26 (86.7%)	4 (13.3%)	0 (0.0%)	0 (0.0%)

Note: Data were analyzed using Kruskal–Wallis and pairwise Mann–Whitney U tests with Bonferroni correction; adjusted $p < 0.05$ was considered significant.

Score 3 occurred in 6.7%, 6.7%, 26.7%, and 0% of Groups A–D, respectively. Scores 2–3 occurred in 6.7%, 20.0%, 63.3%, and 0%, respectively.

Failure Mode Analysis

Failure-mode distribution differed significantly among groups ($\chi^2(6) = 29.05$, $p < 0.001$; Table IV). Adhesive failure predominated in Group C (70.0%), whereas cohesive and mixed failures accounted for 80.0% of failures in A and 90.0% in D. Group B showed a more balanced distribution.

Table IV. Distribution of failure modes following thermocycling (n = 30 per group)

Group	Adhesive failure, n (%)	Cohesive failure, n (%)	Mixed failure, n (%)
A	6 (20.0%)	11 (36.7%)	13 (43.3%)
B	12 (40.0%)	6 (20.0%)	12 (40.0%)
C	21 (70.0%)	3 (10.0%)	6 (20.0%)
D	3 (10.0%)	13 (43.3%)	14 (46.7%)

Note: Pearson's χ^2 test: $\chi^2(6) = 29.05$, $p < 0.001$.

Clinical Performance and Postoperative Sensitivity (12-Month Follow-Up)

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At 12 months, retention was 95.0% (19/20) in A, 90.0% (18/20) in B, 80.0% (16/20) in C, and 100.0% (20/20) in D; the between-group difference was not significant ($\chi^2(3) = 5.48$, $p = 0.140$; Fig. 2).

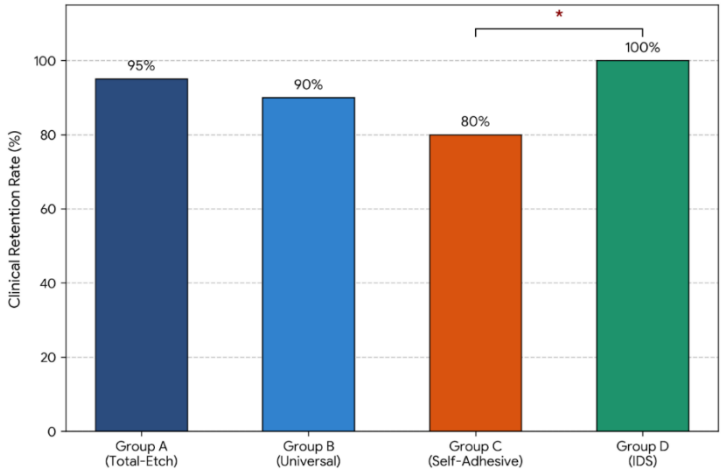


Figure 2. Clinical Retention Rate of Restorations at 12 Months

At 1 month, Schiff scores differed significantly among groups ($H(3) = 35.19$, $p < 0.001$). Mean scores were 1.15 ± 0.48 (A), 0.45 ± 0.51 (B), 0.15 ± 0.36 (C), and 0.20 ± 0.41 (D). After Bonferroni correction, A had higher scores than B, C, and D (adjusted $p < 0.05$), with no differences among B–D. Sensitivity had resolved by 6 months and remained absent at 12 months (Table V).

Table V. Clinical outcomes and postoperative Schiff sensitivity scores over 12 months (n = 20 restorations per group)

Group	12-Month Retention Rate	Schiff Score (1 Month)	(6 Months)	(12 Months)
A	95.0% (19/20)	1.15 ± 0.48	0.00 ± 0.00	0.00 ± 0.00
B	90.0% (18/20)	0.45 ± 0.51	0.00 ± 0.00	0.00 ± 0.00
C	80.0% (16/20)	0.15 ± 0.36	0.00 ± 0.00	0.00 ± 0.00
D	100.0% (20/20)	0.20 ± 0.41	0.00 ± 0.00	0.00 ± 0.00

Note*:* Data are presented as percentages with counts for retention and mean ± SD for Schiff scores. Schiff scale: 0 = no discomfort; 3 = severe discomfort. Kruskal–Wallis analysis was used for 1-month Schiff scores.

DISCUSSION

Mechanistic Considerations of Immediate Dentin Sealing (IDS)

The favorable laboratory performance of IDS may be related to sealing freshly prepared dentin before potential contamination during provisionalization and subsequent procedures [1–4]. A two-step self-etch adhesive applied to freshly cut dentin permits early hybridization, while 10-MDP may interact with hydroxyapatite and contribute to interfacial stability [2–5]. These mechanisms may partly explain the higher absolute μTBS observed for Group D.

Although Group D retained high absolute μTBS after 10,000 thermocycles, Group A showed a slightly smaller numerical reduction (21.77% vs. 22.62%); this difference was not statistically significant. Thus, the findings support higher absolute bond strength with IDS but not a difference in relative bond-strength loss. The study design cannot determine which component of the Group D protocol contributed to its performance.

Total-Etch and Universal Self-Etch Strategies

Group A showed relatively high initial bond strength and favorable observed clinical retention. Phosphoric-acid etching promotes demineralization and collagen exposure, facilitating micromechanical interlocking, but incompletely infiltrated collagen may remain susceptible to hydrolytic degradation and nanoleakage [2,5–7]. Although Group A had the smallest numerical μTBS reduction, between-group differences were not significant. Group B showed an intermediate laboratory response;

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universal adhesive composition and water sorption may influence adhesive-layer permeability and long-term stability [8–10,20].

Self-Adhesive Resin Cement

Group C showed the lowest baseline μTBS, greatest numerical reduction after thermocycling, highest proportion of Score 2–3 microleakage, and most frequent adhesive failure, indicating less favorable dentin–cement interfacial behavior under the conditions studied. This pattern may be related to limited interaction with unconditioned dentin and relatively shallow demineralization and infiltration in simplified self-adhesive approaches [4,9,18]. However, the present study cannot establish a specific molecular mechanism. Although Group C had the lowest observed 12-month retention, between-group differences were not statistically significant and should therefore be interpreted as observed rather than definitive clinical differences.

Marginal Integrity and Substrate Pretreatment

Group D showed the most favorable microleakage distribution, with the highest proportion of specimens without detectable leakage and no Score 3 specimens, whereas Group C had the highest proportion of Score 2–3 specimens. Dye penetration indicates interfacial sealing under experimental conditions but should not be considered a direct surrogate for secondary caries or restoration failure [11].

Standardized substrate pretreatment reduced variation related to restorative material: hydrofluoric acid etching and silanization for lithium disilicate, and airborne-particle abrasion with MDP primer for monolithic zirconia [11–14,19].

Postoperative Sensitivity

At 1 month, cold sensitivity was highest in Group A and lower in the other groups. This may be consistent with the hydrodynamic mechanism of dentinal sensitivity, whereas lower early sensitivity with IDS may reflect its proposed dentin-sealing effect [1,2,6,8,21]. These findings do not establish that IDS prevents sensitivity. By 6 and 12 months, Schiff scores were 0.00 ± 0.00 in all groups.

Limitations and Future Research

The 12-month follow-up cannot establish long-term restoration survival, and 20 restorations per clinical group limits the ability to detect retention differences. Occlusal loading, parafunctional activity, cement-film thickness, and tooth position were not stratified. Thermocycling provides an accelerated aging model but cannot reproduce the full mechanical, thermal, chemical, and biological complexity of the oral environment.

Future studies should include larger multicenter cohorts, longer follow-up, and standardized assessment of occlusal loading and restoration location. Follow-up of 36–60 months or longer and non-destructive methods such as optical coherence tomography may provide additional information on restoration survival and interfacial degradation.

CONCLUSIONS

Based on the combined in vitro and 12-month clinical findings:

1. The IDS protocol with a two-step self-etch MDP-containing adhesive (Group D) showed the highest baseline (41.63 ± 3.80 MPa) and post-aging (32.22 ± 5.18 MPa) microtensile bond strength, no severe microleakage, and the highest observed 12-month clinical retention (100%).

2. The total-etch protocol (Group A) showed high baseline bond strength (35.93 ± 2.90 MPa), a 21.77% reduction after thermal aging, and 95% clinical retention. It also produced the highest 1-month postoperative sensitivity (Schiff score 1.15 ± 0.48), which resolved by 6 months.

3. Group B, using a universal adhesive in self-etch mode, showed a 27.41% reduction in bond strength after thermal aging and 90% clinical retention at 12 months.

4. Group C showed the greatest bond-strength reduction (48.40%), the highest proportion of Score 2–3 microleakage (63.3%), predominantly

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adhesive failures (70.0%), and the lowest observed clinical retention (80.0%).

5. Within the limitations of this laboratory and 12-month clinical investigation, the IDS-based protocol was associated with favorable laboratory interfacial characteristics and the highest observed clinical retention. However, between-group differences in bond-strength reduction and clinical retention were not statistically significant

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Author Contributions

All authors contributed to the study design, data collection, analysis, and writing of the manuscript. All authors have read and approved the final version of the manuscript.

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Institutional Review Board Statement

The clinical component of this study was conducted in accordance with the ethical principles of the Declaration of Helsinki. The study protocol was reviewed and approved by the Bioethics Committee (Protocol No. 5, February 18, 2026).

Informed Consent Statement

Written informed consent was obtained from all participants prior to enrollment in the study.

Clinical Trial Registration

Not applicable.

Data Availability Statement

The datasets generated and analyzed during the current study are available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare that they have no conflicts of interest relevant to this study.

AI Use Statement

Generative artificial intelligence (ChatGPT, OpenAI) was used only for language editing and improvement of manuscript readability under the full supervision of the authors. The AI tool did not generate, analyze, or interpret research data. All scientific content, data analysis, conclusions, and final manuscript approval were performed exclusively by the authors.

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Figure legends

Figure 1. Microtensile bond strength (μTBS) of the study groups before and after 10,000 thermocycles. Bars represent mean ± SD. The figure presents baseline and post-aging μTBS values for each adhesive protocol. Statistical comparisons between adhesive protocols and changes after thermal aging were performed using the statistical procedures described in the Methods and Results sections.

Figure 2. Cumulative retention rate (%) of ceramic restorations over the 12-month clinical evaluation period across the different adhesive fixation protocols.

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