Original Article

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Effect of Acid Etching Durations on Shear Bond Strength of Advanced Lithium Disilicate Ceramics: An In-Vitro Study

Nehal M. Kamal[1]*; Ahmed Farghaly[2]; Karim Awadallah[3]

Abstract:

Introduction: The success of ceramic restorations relies heavily on the bond strength between ceramic and resin composites. Hydrofluoric acid (HF) etching is a critical step in surface treatment, yet its optimal duration for advanced lithium disilicate ceramics like CEREC Tessera remains unclear.

Objectives: This in vitro study aimed to assess how different HF etching times (20, 30, and 60 s) affect the shear bond strength between CEREC Tessera and resin composite substrates. Moreover, it aims toevaluat the influence of etching time on failure modes. **Methodology**: Twenty-four ceramic blocks (1.5 × 11 × 14 mm) were fabricated using CAD/CAM technology and randomly divided into three groups (n=8) based on HF etching duration: Group A (30 s), Group B (20 s), and Group C (60 s). All specimens were subjected to 5% HF etching, silane application, and bonding with dual-cure resin cement under a standardized load. After thermocycling (5,000 cycles), shear bond strength was tested using a universal testing machine. Failure modes were analyzed microscopically. Statistical analysis included one-way ANOVA, Chi-square tests, and normality assessments.

Results: Mean shear bond strengths were 4.84 MPa (Group A), 4.91 MPa (Group B), and 5.32 MPa (Group C). No significant differences were found among groups (p = 0.90). Failure mode analysis revealed predominantly cohesive failures (>80%), with no statistically significant variation across groups (p = 0.39).

Conclusions: Within the limitations of this in vitro study, HF etching times ranging from 20 to 60 s did not significantly alter shear bond strength or failure patterns. The high incidence of cohesive failures indicates strong internal ceramic integrity and effective bonding. Further research is warranted to evaluate long-term stability and clinical applicability.

Keywords: CAD/CAM ceramics, CEREC Tessera, hydrofluoric acid etching, lithium disilicate, shear bond strength, thermocycling, resin composite adhesion.

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[1]* MSc. Student, Fixed Prosthodontics Department, Faculty of Dentistry, Cairo University, Cairo, Egypt
E-mail: Nehal.kamal@dentistry.cu.edu.eg
[2] Professor, Fixed Prosthodontics Department, Faculty of Dentistry, Cairo University, Cairo, Egypt
[3]Lecturer, Fixed Prosthodontics Department, Faculty of Dentistry, Cairo University, Cairo, Egypt

1. Introduction:

Over the past few decades, the field of restorative dentistry has witnessed significant advancements driven by increasing patient demands for esthetics and the rapid evolution of materials science and digital technologies.[1, 2] One of the most notable shifts has been the transition from conventional metal-ceramic restorations to metal-free all-ceramic systems, owing to their superior esthetic integration and biocompatibility.[3] Among these, modern dental ceramics are being continuously modified to emulate the optical and mechanical properties of natural enamel and dentin while offering enhanced fracture resistance compared to traditional ceramics.[4]

Computer-aided design and computer-aided manufacturing (CAD/CAM) technologies have revolutionized restorative workflows, with the CEREC system leading this innovation since its market debut over four decades ago.[5] These technologies facilitate the precise fabrication of ceramic blocks, including reinforced glass ceramics such as lithium disilicate, which strike a balance between esthetic appeal and mechanical strength, especially for posterior restorations where occlusal forces are greater.[6]

CEREC Tessera, a next-generation zirconia-reinforced lithium disilicate glass-ceramic, has emerged as a promising material offering rapid crystallization, high flexural strength, and reliable performance.[7] Like other silica-based ceramics, its bonding efficacy to resin-based materials depends heavily on appropriate surface treatment—most notably hydrofluoric acid (HF) etching.[8] This process creates micromechanical retentive sites by selectively dissolving the glassy matrix and generating surface roughness conducive to resin infiltration.[9, 10]However, the optimal duration of HF etching remains a point of contention, as both under-etching and over-etching may compromise bond integrity or surface morphology.[11]

Typically, HF etching times vary depending on ceramic composition: feldspathic ceramics often require 60 s, whereas lithium disilicate ceramics are recommended for 20 to 40 s.[12] Despite these guidelines, current literature presents inconsistent findings regarding how variations in etching duration influence bond strength, failure modes, and long-term durability.[13] Furthermore, limited data exists specifically for advanced lithium disilicate ceramics such as CEREC Tessera, which possess unique microstructures and may respond differently to surface conditioning.[14]

Given the clinical importance of durable ceramic-resin adhesion, there remains a need to clarify whether altering HF etching durations significantly affects the bond strength of zirconia-reinforced lithium disilicate ceramics. Therefore, the objective of this in vitro study is to evaluate the effect of three HF etching durations (20, 30, and 60 s) on the shear bond strength between CEREC Tessera ceramics and resin composite substrates. It is hypothesized that extended etching times will not result in statistically significant improvements in bond strength under standardized laboratory conditions.

This study contributes to the optimization of surface treatment protocols for contemporary CAD/CAM ceramics by providing comparative data on etching duration effects specific to CEREC Tessera. The findings aim to assist clinicians in selecting evidence-based protocols that enhance adhesive performance and ensure the long-term success of indirect ceramic restorations.

2. Materials and Methods

2.1. Study Design

A total of 24 ceramic specimens were randomly allocated into three equal groups (n = 8/group) based on the HF etching duration: 20 s (Group B), 30 s (Group A – manufacturer recommendation), and 60 s (Group C). Randomization was achieved using computer-generated sequences from random.com, and allocation concealment was maintained via opaque, sealed envelopes. Blinding of the assessor was applied throughout testing procedures.

The sample size was calculated to provide 80% statistical power with an alpha level of 0.05, using PS Power and Sample Size software (Version 3.1.6), assuming a medium effect size across three experimental groups.

The protocol adhered to the principles of ethical research conduct and was reviewed and approved by the Research Ethics Committee (REC) of the Faculty of Dentistry, Cairo University, Egypt, on 26/3/2024 (Approval NO: 32-3-24). Ethical approval was also obtained from the Evidence-Based Dentistry Committee (02/05/2023) and the Fixed Prosthodontics Department Board (19/03/2024)

2.2.Materials and Equipment

The ceramic specimens used in this study were CEREC Tessera blocks (Dentsply Sirona, Charlotte, NC, USA), composed primarily of $Li_2Si_2O_5$ (90 wt%), Li_3PO_4 (5 wt%), and virgilite ($Li_{0.5}Al_{0.5}Si_{2.5}O_6$, 5 wt%). The material exhibited a biaxial strength exceeding 700 MPa, a modulus of elasticity of approximately 103 GPa, hardness of 7.37 ± 0.19 GPa, and a fracture toughness of nearly 1.45 ± 0.10 MPa, according to manufacturer data.

Additional materials included Vita Ceramic Etching Gel (5% HF; VITA Zahnfabrik, Germany), BISCO silane primer (BISCO Inc., USA), and PANAVIA SA Luting Multi dual-cure resin cement (Kuraray Noritake Dental Inc., Japan). Equipment utilized included a 4-axis wet milling machine (Dentsply Sirona), Isomet 4000 precision saw (Buehler Ltd., USA), Programat EP3010 ceramic furnace (IvoclarVivadent), and a universal testing machine (Instron Model 3345, Norwood, USA) fitted with a 5 kN load cell.

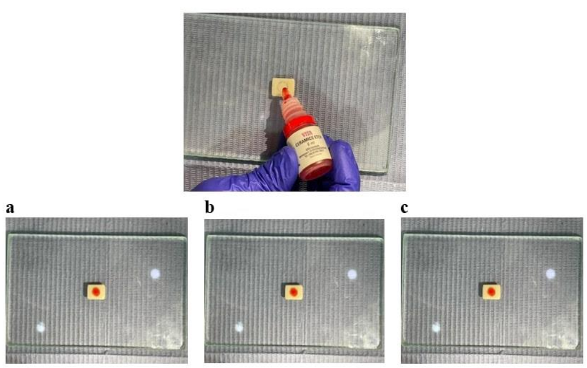
2.3. Specimen Preparation

Ceramic blocks were digitally designed using Blender 3D modeling software and milled to uniform dimensions of 1.5 × 11 × 14 mm (**Figure 1**). The specimens were sectioned using a precision saw under continuous water cooling to prevent thermal damage. Subsequently, all specimens underwent a glaze-firing cycle in a ceramic furnace according to manufacturer-recommended parameters. Post-glazing, specimen thickness was verified using a digital caliper, followed by sequential polishing with silicon carbide papers (#600 to #1200 grit) and ultrasonic cleaning in ethanol, then air-drying.

Figure(1). Wet milling of ceramic blocks utilizing a CAD/CAM system.

2.4.Surface Treatment Protocol

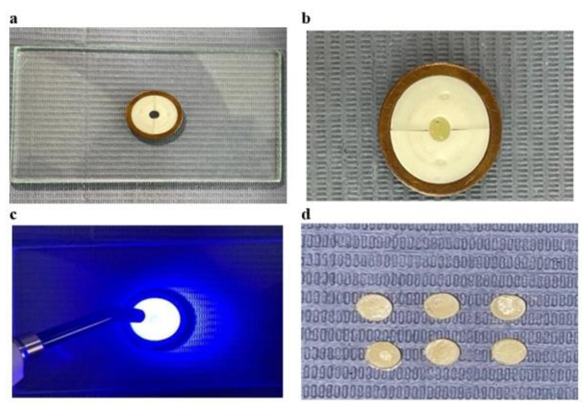
All specimens were cleaned with 70% ethanol and air-dried prior to surface treatment. Etching was performed using 5% HF gel for 20, 30, or 60 s, depending on the group assignment. After etching, samples were rinsed thoroughly and air-dried. A silane coupling agent was applied for 60 s and gently air-dried to enhance chemical bonding with resin cement (**Figure 2**).



Figure(2). Etching of ceramic surfaces utilizing 5% hydrofluoric acid (a30,b20,c60)sec.

2.5. Resin Composite Substrate Fabrication

Composite resin discs (A2 shade) were fabricated using a custom-made cylindrical split Teflon mold (5 mm diameter, 2 mm thickness). The composite was incrementally packed and polymerized using an LED curing unit (Miraj LED.D, Korea; 1400 mW/cm², wavelength 420–480 nm) for 40 s per increment. The discs were then finished using wet silicon carbide paper (320, 600 grit), ultrasonically cleaned in distilled water for 10 min, and air-dried (**Figure 3**).



Figure(3). Production of resin composite substrates.

2.6. Cementation Procedure

A custom-designed stainless steel cementation device was employed to ensure standardized load application. Each ceramic specimen was bonded to its corresponding composite disc using PANAVIA SA Luting Multi resin cement. Cement was applied to the composite surface, and the specimens were assembled in the cementation device with a constant 1 kg vertical load applied for 3 minutes. Excess cement was removed, and the interface was cured using a light-polymerization device (3200 mW/cm²) for 40 s from three directions. The bonded assemblies were finished and polished per the manufacturer's recommendations by a single operator to maintain consistency.

2.7. Thermocycling Protocol

To simulate clinical aging, all specimens underwent thermocycling for 5000 cycles, corresponding to approximately six months of intraoral function. Thermocycling parameters included immersion in water baths at 5°C and 55°C with 25-s dwell times in each bath and a 10-s transfer interval based on established laboratory simulation protocols.

2.8. Shear Bond Strength Testing

Following thermocycling, specimens were mounted in a universal testing machine with the resin-ceramic interface aligned perpendicularly to the direction of applied force. A mono-beveled chisel-shaped metallic rod applied compressive shear load at a crosshead speed of 0.5 mm/min until bond failure occurred. The failure load (N) was recorded, and the shear bond strength (τ) was calculated using the formula:

$$\tau = P/\pi r2$$

Where τ is shear bond strength (MPa), P is the failure load (N), π is 3.14, and r is the radius of the bonded area in millimeters.

2.9. Failure Mode Analysis

Post-fracture, specimens were examined under an electron microscope to determine failure patterns. Failure modes were categorized as adhesive (at the ceramic–cement interface), cohesive (within the composite), or mixed (a combination of both) based on morphological features (**Figure 4**).

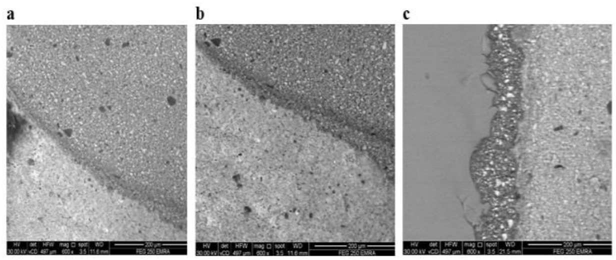


Figure (4). Scanning Electron Microscopy investigation of failure modes at the resin–ceramic contact.

2.10. Statistical Analysis

Statistical analysis was conducted using SPSS software (Version 26.0, IBM Corp., Armonk, NY, USA). Descriptive statistics (mean ± standard deviation) were calculated for each group. The normality of data distribution was verified using the Kolmogorov–Smirnov and Shapiro–Wilk tests. Intergroup comparisons of shear bond strength were performed using one-way analysis of variance (ANOVA), followed by post hoc Tukey testing when appropriate. The chi-square test was used to analyze differences in failure modes among groups. A significance level of $p < 0.05$ was considered statistically significant.

3. Results

Primary Outcome: Shear Bond Strength

The normality of the shear bond strength data was verified using the Kolmogorov–Smirnov and Shapiro–Wilk tests for each group. All p-values exceeded 0.05, indicating that data were normally distributed across the three etching duration groups (**Table 1, Figure 5**).

Group C (60 s) recorded the highest mean shear bond strength (M = 5.32 MPa, SD = 1.87), followed by Group B (20 s; M = 4.91 MPa, SD = 1.89) and Group A (30 s; M = 4.84 MPa, SD = 1.67). Despite these numerical differences, one-way ANOVA revealed no statistically significant differences between groups ($F = 0.102$, $p = 0.900$), confirming that variations in etching time did not significantly impact bond strength under the tested conditions (**Table 2**).

Secondary Outcome: Mode of Failure

Post-debonding examination under stereomicroscopy revealed a predominance of cohesive failure across all groups. Group A demonstrated 80% cohesive and 20% adhesive failures, Group B exhibited 80% cohesive and 20% mixed failures, and Group C showed 100% cohesive failures. The chi-square test found no statistically significant difference in the distribution of failure modes among the three groups ($\chi^2 = 4.15$, $p = 0.39$; **Table 3, Figure 6**).

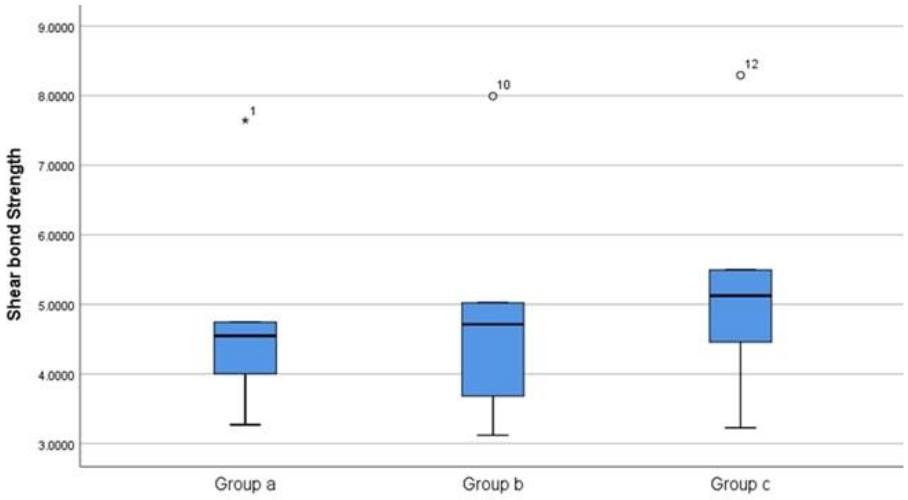


Figure (5). Box plot illustrating shear bond strength among experimental groups.

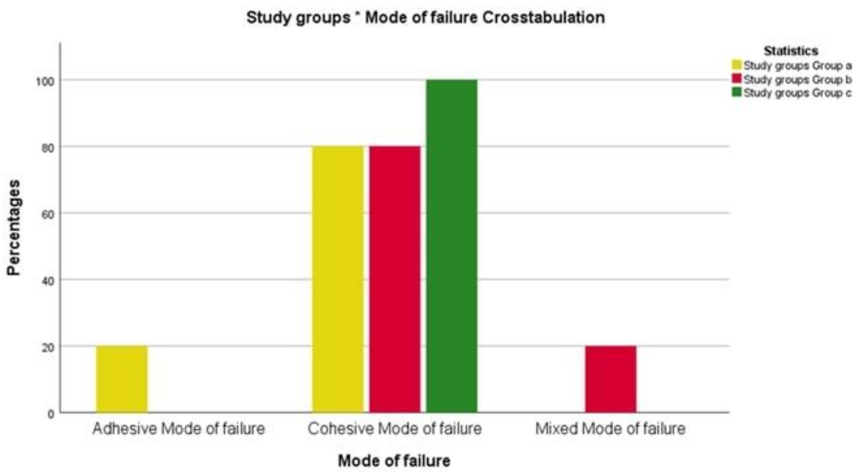


Figure (6). A cluster bar chart showing the percentages of failure types.

Table 1: Representing the normality tests values

Tests of Normality							
	Study groups	Kolmogorov-Smirnov[a]			Shapiro-Wilk		
		Statistic	df	Sig.	Statistic	df	Sig.
Shear bond Strength	**Group a (30sec.)**	0.323	5	**0.095**	0.856	5	**0.215**
	Group b (20sec.)	0.275	5	**0.200***	0.889	5	**0.354**
	Group c (60 sec.)	0.263	5	**0.200***	0.933	5	**0.614**

* The significance level is greater than 0.05.

Table 2 : Representing the descriptive analysis and significance value between the groups.

	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Min.	Max.	F-value	P-value
				Lower Bound	Upper Bound				
Group a (30sec.)	4.84	1.67	0.74	2.77	6.91	3.27	7.64	**0.102**	**0.9**
Group b (20sec.)	4.91	1.89	0.84	2.56	7.25	3.12	7.99		
Group c (60sec.)	5.32	1.87	0.84	2.99	7.65	3.23	8.29		

* The significance level is less than or at the 0.05.

Table 3: Representing the percentages of the mode of failure between the study groups.

Study groups	Mode of Failure			Total	X^2	P-value
	Adhesive	Cohesive	Mixed			
Group a	**20%**	**80%**	**0%**	**100%**	**4.15**	**0.39**
Group b	**0%**	**80%**	**20%**	**100%**		
Group c	**0%**	**100.0%**	**0%**	**100%**		

4. Discussion:

The progression of ceramic materials and adhesive technologies has significantly influenced restorative dentistry, with an increasing preference for metal-free, esthetically pleasing, and durable restorations.[15] Among various ceramic systems, zirconia-reinforced lithium disilicate ceramics—especially advanced formulations such as CEREC Tessera—have gained prominence for their superior mechanical properties and compatibility with digital workflows.[16, 17] However, the success of these restorations largely hinges on the integrity of the adhesive interface, which is critically influenced by surface treatment protocols, particularly HF etching.[10]

Given the evolving material compositions and clinical demands, the optimal HF etching duration for newer ceramics like CEREC Tessera remains a point of clinical uncertainty.[18]The present study investigated the impact of three etching durations—20, 30, and 60 s—on the shear bond strength (SBS) of CEREC Tessera bonded to resin composite. The goal was to determine whether the manufacturer-recommended 30-s etch could be optimized or streamlined for clinical efficiency without compromising adhesion quality.

The study found no statistically significant differences in SBS among the three tested groups ($p = 0.90$) despite numerically higher values in the 60-s group. This result suggests a plateau in the effect of etching duration on bonding efficacy—wherein increasing the etching time beyond 20 s does not yield additional mechanical or chemical bonding benefits. Cohesive failure was the predominant failure mode across all groups, indicating strong interfacial adhesion and suggesting that failure occurred within the bulk of the material rather than at the adhesive interface.

This pattern aligns with previous studies, such as those by Straface et al.[19] and Behnaz et al.[20], which also observed no substantial benefit from etching durations longer than 20–30 s when using HF concentrations of 5–10%. These studies collectively suggest that the surface of lithium disilicate ceramics reaches optimal micromechanical and chemical bonding capacity relatively early in the etching process.[21] Similarly, Kagalkaret al.[22]and Ullah et al.[23]confirmed that extending etching time beyond 20–30 s, even at higher HF concentrations, offers diminishing returns and can risk over-etching, which may degrade the ceramic microstructure or impair resin infiltration.

CEREC Tessera's unique microstructure, composed of dual crystalline phases of lithium disilicate and virgilite embedded in a zirconia-reinforced glass matrix, likely influences its interaction with HF acid.[24] Unlike traditional lithium disilicate, which may benefit from longer etching to expose crystalline structures, the virgilite phase enhances crack resistance and may contribute to a more rapid saturation of surface reactivity.[25] This may explain the observed plateau in SBS across etching durations.

Some studies, such as those byAdali et al.[26]and Nagabhooshanam et al.[27]found increased surface roughness with prolonged etching. These improvements did not consistently translate into higher bond strength. This highlights the important distinction between surface topography and adhesive efficacy; enhanced roughness does not guarantee improved micromechanical interlocking if the ceramic's structural integrity is compromised.[28]

Furthermore, the consistent application of silane following HF etching is a critical variable. In this study, a single-component pre-hydrolyzed silane was used, which has been shown to offer reliable coupling performance when used properly. The consistency in silane application likely contributed to the high cohesive failure rates observed, indicating robust silane-mediated chemical adhesion.[29]

From a clinical perspective, the results support a conservative HF etching protocol of 20 s for CEREC Tessera when using a 5% HF concentration and a compatible silane-resin system. Shorter etching times offer several practical advantages: reduced chairside time, lower risk of over-etching, enhanced procedural control, and minimized exposure to HF acid for both patient and operator safety.[30] The ability to standardize a 20-s protocol could improve workflow efficiency without compromising adhesive performance.

This recommendation is further reinforced by the failure mode analysis, which showed a dominance of cohesive rather than adhesive failures, particularly in the 20-s group. Such failure patterns indicate that bond strength exceeded the cohesive strength of the substrate or resin cement, underscoring the efficacy of the surface treatment at even the shortest duration tested.[31]

Despite these promising findings, several limitations must be acknowledged. Firstly, the in vitro nature of the study limits direct clinical extrapolation. Although thermocycling was performed to simulate six months of aging, intraoral conditions involve more complex variables, including masticatory fatigue, pH fluctuation, salivary enzymes, and biofilm formation.

Sly, only one HF concentration (5%), one ceramic system (CEREC Tessera), and one adhesive cement were evaluated. The generalizability of the results to other ceramics or adhesive systems remains uncertain. Additionally, the absence of surface characterization tools such as scanning electron microscopy (SEM) or atomic force microscopy (AFM) limits our ability to directly correlate surface morphology with bond strength outcomes. Including such analyses would enhance the mechanistic understanding of etching-induced changes and provide a clearer rationale for the observed SBS trends.Furthermore, only shear bond strength testing was used. Although widely accepted, this method presents limitations such as stress concentration at the loading point and non-uniform force distribution. Complementary tests like microtensile bond strength (μTBS) or finite element modeling could provide a more nuanced assessment of the bonded interface under functional loads.

Future studies should investigate the combined influence of etching time and HF concentration to develop more refined surface treatment protocols. Additionally, testing across various brands of lithium disilicate or zirconia-reinforced ceramics could help determine whether material-specific etching recommendations are warranted. Incorporating different resin cements, silane systems, and adhesive strategies (e.g., universal adhesives with MDP monomers) would also improve the translational value of findings.

The application of long-term aging protocols, including extended thermocycling and mechanical fatigue, should be prioritized to validate the long-term stability of the bond. Furthermore, surface morphology characterization using SEM, profilometry, and contact angle goniometry is strongly recommended to elucidate the microstructural changes induced by HF etching.

5. <u>Conclusion:</u>

This study investigated the effect of varying HF etching durations—20, 30, and 60 s—on the shear bond strength of CEREC Tessera, an advanced lithium disilicate ceramic. Results demonstrated no statistically significant differences in bond strength among the groups, with the 20-s protocol performing comparably to longer durations. These findings suggest that a 20-s etch may suffice for effective resin-ceramic bonding, optimizing clinical workflow while preserving material integrity. However, further research is needed to expand upon these results and to tailor etching protocols based on ceramic composition, adhesive system, and long-term clinical performance.

Ethical Approval and Consent to Participate

The protocol adhered to the principles of ethical research conduct and was reviewed and approved by the Research Ethics Committee (REC) of the Faculty of Dentistry, Cairo University, Egypt, on 26/3/2024 (Approval NO: 32-3-24). Ethical approval was also obtained from the Evidence-Based Dentistry Committee (02/05/2023) and the Fixed Prosthodontics Department Board (19/03/2024). As this was an in vitro study, no human participants were involved, and informed consent was not applicable.

Consent for Publication

Not applicable.

Availability of Data and Materials

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

Competing Interests

The authors declare that they have no competing interests.

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Authors' Contributions

All authors contributed to the conceptual design, sample preparation, experimental procedures, data collection, and manuscript writing. All authors read and approved the final manuscript and agree to be accountable for all aspects of the work.

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