

Influence of Surface Preparation Methods on Fracture Behavior of Nanohybrid and Monochromatic Composite Resins: A Stereomicroscopic Analysis

Yüzey Hazırlık Yöntemlerinin Nanohibrit ve Monokromatik Kompozit Rezinlerin Kırılma Davranışı Üzerindeki Etkisi: Bir Stereomikroskopik Analiz

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ABSTRACT

Objectives: This study investigates the effect of different surface preparation methods on the fracture behavior of nanohybrid and monochromatic composite resins.

Materials and Methods: Sixty extracted anterior teeth were embedded in acrylic and divided into two groups to be restored with monochromatic composite resin (Omnichroma, Tokuyama, Japan) and nanohybrid composite resin (Essentia, GC, Japan). Each group was subdivided based on roughening method: acid-etched control, thick/medium-grit disc and Er,Cr: YSGG laser. All teeth were restored using universal adhesive system, subjected to 5000 thermal cycles, and stored in distilled water at 37°C for 24h. Shear bond strength were measured using a universal testing machine (Shimadzu Corp., Kyoto, Japan), modes of failure were evaluated using a stereomicroscope (Leica-M27.5, Leica Microsystems, Germany). The data were analyzed using IBM SPSS V23. Yates Correction and Monte Carlo Corrected Fisher's Exact Test were used to examine the relationship between composites, surface preparation techniques and fracture types. The significance level was set at $p < 0.05$.

Results: Surface roughening improved the bond strength. Adhesive fractures were predominant in most groups, except in the Er,Cr: YSGG laser-treated samples, where mixed fractures were more common. There was no statistically significant difference between composites, fracture types ($p=1$) or between surface preparation techniques and fracture types ($p=0.235$).

Conclusion: Surface preparation methods such as acid etching and mechanical roughening resulted in higher adhesive failure rates, while laser etching led to more mixed failures. Laser etching is a minimal invasive technique with promising results but requires further research to optimize its clinical application.

Keywords: Monochromatic composite resin, nanohybrid composite resin, fracture type, surface preparation techniques, dental enamel.

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Article History

Submitted 17.03.2025

Revised 17.04.2025

Accepted 17.04.2025

Published 30.04.2025

How to cite this article: Yıldız, S., N and Türkmen, C. Influence of Surface Preparation Methods on Fracture Behavior of Nanohybrid and Monochromatic Composite Resins: A Stereomicroscopic Analysis. *European Journal of Research in Dentistry*, 2025;9(1): 49-55. DOI: <http://dx.doi.org/10.29228/erd.93>



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ÖZ

Amaç: Bu çalışma, farklı yüzey hazırlık yöntemlerinin nanohibrit ve monokromatik kompozit rezinlerin kırılma davranışı üzerindeki etkisini araştırmayı amaçlamaktadır.

Gereç ve Yöntemler: Altmış adet çekilmiş anterior diş, akriliğe gömülüp, monokromatik (Omnichroma, Tokuyama, Japonya) ve nanohibrit kompozit rezin (Essentia, GC, Japonya) ile restore edilmek üzere iki gruba ayrılmıştır. Tüm gruplar, yüzey pürüzlendirme yöntemi olarak asitle pürüzlendirme, kalın/orta grenli disk ve Er,Cr: YSGG lazer kullanılarak alt gruplara ayrılmıştır. Tüm dişler, üniversal bir adeziv sistemle restore edilip, 5000 termal döngüye tabi tutulmuş ve 37°C'de distile suda 24 saat bekletilmiştir. Makaslama bağlanma dayanımı, bir üniversal test cihazı (Shimadzu Corp., Kyoto, Japonya) kullanılarak ölçülmüş ve kırılma tipleri stereomikroskop (Leica M27.5, Leica Microsystems, Almanya) ile değerlendirilmiştir. Veriler IBM SPSS V23 ile analiz edilmiştir. Kompozit tipleri, yüzey hazırlık teknikleri ve kırılma türleri arasındaki ilişkiyi incelemek için Yates Düzeltmesi ve Monte Carlo Düzeltmeli Fisher'in Kesin testi kullanılmıştır. Önem düzeyi $p < 0.05$ olarak belirlenmiştir.

Bulgular: Yüzey pürüzlendirmesinin bağlanma dayanımını arttırdığı görülmüştür. Çoğu grupta adeziv kırılmalar baskınken, Er,Cr: YSGG lazerle örneklerde karışık kırılmalar daha yaygın bulunmuştur. Kompozit tipleri ile kırılma türleri arasında ($p=1$) ve yüzey hazırlık teknikleri ile kırılma türleri arasında ($p=0.235$) anlamlı bir ilişki bulunmamıştır.

Sonuç: Asit ile pürüzlendirme ve mekanik pürüzlendirme gibi yüzey hazırlık yöntemleri, daha yüksek adeziv başarısızlık oranlarına yol açarken, lazer ile pürüzlendirme daha fazla karışık kırılmaya neden olmuştur. Lazer ile pürüzlendirme, minimal invaziv bir teknik olup ve umut verici sonuçlar sunmaktadır, ancak klinik uygulamasını optimize etmek için daha fazla araştırma gerekmektedir.

Anahtar Kelimeler: Monokromatik kompozit rezin, nanohibrit kompozit rezin, kırılma tipi, yüzey hazırlık yöntemleri, diş minesini.

INTRODUCTION

Restorative dentistry aims to preserve tooth structure, restore function and esthetics, prevent bacterial leakage, and enhance the patient's overall well-being through conservative approaches. These goals align with the philosophy of minimally invasive dentistry (MID), which emphasizes preserving healthy dental structures while achieving optimal clinical outcomes. MID has evolved with advancements in adhesive technologies and dental materials, enabling conservative treatment options such as direct resin composite restorations. These approaches are not only less invasive but also yield more esthetic and natural results, shifting the focus from Black's philosophy of "extension for prevention" to "prevention of extension" (Turkun, 2023)."

Among minimally invasive restorative materials, composite resins are the most commonly preferred due to their versatility and clinical success (Ricketts & Pitts, 2009). The success of composite resin restorations largely depends on the bond quality formed between dental hard tissues and the restorative material, which plays a crucial role in ensuring long-term durability (Milia et al., 2012). Achieving a strong bond between enamel and the restorative material is closely tied to the surface preparation method employed (Atoui et al., 2010). Traditionally, enamel is etched with phosphoric acid as part of total-etch adhesive systems, creating micromechanical retention by forming resin tags approximately 6-12 μm in length. However, whether this method alone is sufficient remains a topic of debate, leading to the exploration of alternative techniques to enhance enamel surface energy further (Silverstone et al., 1975).

In this study, nanohybrid and monochromatic composite resins were selected due to their distinct mechanical and esthetic properties. Nanohybrid composites, characterized by their small particle sizes (5-75 nm) and nanocluster fillers, exhibit exceptional surface smoothness, mechanical durability, and compatibility with various surface preparation methods. These attributes make them suitable for anterior and posterior restorations (García et al., 2006;

Radz, 2011; Simos, 2011). Monochromatic composites utilize "smart chromatic technology" to adapt to the natural color of surrounding teeth, ensuring a seamless appearance without the need for shade selection. This technology not only reduces clinical application time but also minimizes procedural errors, providing efficient and esthetic results (Eliezer et al., 2020).

Surface preparation plays a critical role in achieving optimal bond strength. Techniques such as phosphoric acid etching, coarse/medium-grit disks, and Er,Cr: YSGG lasers have been employed to enhance the interaction between composite resins and enamel surfaces. Acid etching has been a cornerstone in restorative dentistry since its introduction by Buonocore in 1955. While extensively studied, most research has focused on variables such as acid type, concentration, and application techniques (Triolo Jr et al., 1993; Wang et al., 1994). However, limited attention has been given to the influence of surface textures created by rotary instruments on bond strength (Jung et al., 1999). Recent advancements, such as laser etching, have gained traction due to their ability to create irregular enamel surfaces with open dentin tubules, ideal for adhesive bonding. Lasers also offer a painless, vibration-free alternative to traditional methods, making them highly attractive for routine use (Visuri et al., 1996; Karandish, 2014).

This study focuses on the fracture types observed after different surface preparation methods, with a particular emphasis on their analysis through stereomicroscope. The study aims to compare various surface roughening techniques, including conventional methods like phosphoric acid etching and alternative approaches such as discs and laser etching. The fracture patterns were analyzed statistically to determine the influence of surface preparation on the fracture behavior of nanohybrid and monochromatic composite resins. The findings are expected to contribute valuable insights into the development of more effective and minimally invasive surface treatment techniques in restorative dentistry. The null hypotheses of the study are: (1) There is no significant difference in the fracture types between the surface roughening techniques,

(2) There is no significant difference in the fracture types between nanohybrid and monochromatic composite resins, and (3) Surface roughening techniques have no significant interaction effect on the fracture behavior of nanohybrid and monochromatic composite resins.

MATERIALS AND METHODS

This study received ethical approval from the Clinical Research Ethics Committee of Marmara University Faculty of Dentistry (Protocol No: 2023/138). The authors affirm that there are no financial or personal conflicts of interest associated with this research. All necessary signed consents from all participants are properly taken.

The materials utilized in the study are presented in Table 1.

Table 1. Materials used in the study

Material	Type	Composition	Manufacturer
Omnichroma	Monochromatic Composite Resin	UDMA, TEGDMA, Zirconia, Silica (68% by weight)	Tokuyama Dental, Tokyo, Japan
Essentia Dark Enamel	Nanohybrid Composite Resin	BisEMA (10-25 wt%), TEGDMA (2-5 wt%), UDMA (1-2.5 wt%), BisGMA (1-2.5 wt%), Nanoclusters	GC Corporation, Tokyo, Japan
G-Premio Bond	Adhesive	MDTP, 4-MET, MDP, Acetone, Dimethacrylate Monomers, Silanized Silica Filler	GC Corporation, Tokyo, Japan
Sof-Lex Discs	Surface Preparation Disk	High-strength granules with medium/thick grit	3M Dental Products, USA
Spident Acid Etchant	Acid	37% Phosphoric Acid, Thickeners	Spident Co., Ltd., Korea

Teeth selection and restoration protocol

Sixty extracted human anterior teeth obtained from the maxillary and mandibular regions due to periodontal diseases were utilized in this study. Prior to experimentation, the teeth were preserved in a 0.1% thymol solution. Only teeth with no prior restorations, carious lesions, or hypomineralization defects were selected. To ensure standardization, the buccal surfaces of the crowns of the teeth were prepared sequentially with 600, 1000, and 1200 grit silicon carbide waterproof sandpaper. The polishing device (Isomet, Buehler, USA) was digitally controlled, with a speed range adjustable between 20-600 revolutions per minute, and water cooling was applied to achieve flat surfaces. The teeth were divided into two primary groups: Group A for monochromatic composite resin restoration (Omnichroma, Tokuyama Dental, Tokyo, Japan) and Group B for nanohybrid composite resin restoration (Essentia Starter Kit Syringe, GC, Japan). Each primary group was further subdivided into four subgroups (n=10 for each) based on the surface roughening method employed:

- Acid etching: A 37% phosphoric acid solution (FineEtch, Spident Co. Ltd., Incheon, Korea) was applied for 20 seconds and subsequently rinsed with distilled water.

- Thick/medium-grit disc abrasion: SofLex discs (3M Dental Products Division, St. Paul, Minn., USA) were used to roughen the enamel surface. The procedure was performed according to the manufacturer's instructions, with a speed setting of 10000 rpm, at a pressure of 0.2 N, and with a power of 10 W. The enamel surface was abraded for 20 seconds per tooth, ensuring consistent abrasion in a circular motion. The device was operated with water cooling to prevent excessive heat buildup during the procedure.
- Erbium, Chromium: Yttrium Scandium Gallium Garnet (Er, Cr: YSGG) laser: The enamel surface was roughened by applying laser energy using an Er, Cr: YSGG laser (Shimadzu Corporation, Kyoto, Japan). The device was set according to the manufacturer's instructions, with a power of 1.5 W, 65% air, and 55% water cooling. Energy was applied in a 5x5 mm area on the enamel surface by making circular movements at a 140-microsecond pulse interval and a pulse frequency of 20 Hz using a sapphire tip with a 750 µm diameter. The application was carried out for 15 seconds. During the application, the sapphire tip was held 2 mm away from the enamel surface, ensuring homogeneous scanning of the enamel surface.

In this study, six experimental groups were used. These groups are as follows: **A1**: Control group restored with monochromatic composite resin; **A2**: Group restored with monochromatic composite resin and roughened with thick/medium-grit discs; **A3**: Group restored with monochromatic composite resin and roughened with Er,Cr:YSGG laser; **B1**: Control group restored with nanohybrid composite resin; **B2**: Group restored with nanohybrid composite resin and roughened with thick/medium-grit discs; **B3**: Group restored with nanohybrid composite resin and roughened with Er,Cr:YSGG laser.

Following surface preparation, teeth surfaces were restored using the corresponding composite resins. A universal adhesive (G-Premio Bond, GC, Japan) was applied selectively to the enamel following the manufacturer's guidelines and cured using an LED device (Valo Grand, Ultradent Products, USA). The curing unit operated at a broad wavelength spectrum of 395-480 nm with an intensity of 1000 mW/cm² for 20 seconds. Composite resin materials were placed in increments of 2 mm within silicone molds (2 mm in height and diameter) and cured for the duration recommended by the manufacturer.

To replicate temperature changes in the oral environment, the specimens underwent thermal cycling using a thermocycler (SD Mechatronik Thermocycler, SD Mechatronik; Westerham, Germany) between 5°C and 55°C for 5000 cycles. Each temperature was maintained for 30 seconds, by ISO/TS 11405 standards. Following thermal cycling, the specimens were polished again and kept in distilled water at 37°C for 24 hours before testing.

For shear bond strength, all specimens were tested using a universal testing machine (Shimadzu Corporation, Kyoto, Japan) with the Trapezium X (Shimadzu Corporation, Kyoto, Japan) program, applying an approach speed of 1 mm/min. The samples were placed on supporting metal pieces and

fixed in place to prevent movement. A 0.5 mm thick and rounded separating flat-tipped connected to the machine was positioned parallel to the tooth surface, perpendicular to the composite resin and enamel bonding surface, at the closest distance to the enamel surface without making contact. The separating tip was applied at a crosshead speed of 1 mm/min until bond failure occurred in the specimens.

The fracture types (adhesive, cohesive, and mixed) were analyzed using a stereomicroscope (Leica M27.5, Heerbrugg, Switzerland) at 25x magnification.

- Adhesive failure: Detachment occurring at the interface between the resin and enamel.
- Cohesive failure: Fracture happening within the composite material or enamel structure.
- Mixed failure: A combination of both adhesive and cohesive types of fractures.

Statistical analyses

The data were analyzed using IBM SPSS V23 software. Yates Correction and Monte Carlo Corrected Fisher's Exact Test were used to examine the relationship between composite materials, surface preparation techniques, and fracture types. The analysis results are presented as percentages. The significance level was set at $p < 0.05$.

RESULTS

This research investigated the effect of various surface roughening methods on the bond strength of monochromatic and nanohybrid resin composites to dental enamel. Statistical analysis and visual evaluations were conducted to support the findings (Table 2 and 3).

Table 2. Percentage of fracture types based on composite materials

Fracture Types	Composite Resins		Total	Test Statistic	p
	Monochromatic	Nanohybrid			
Adhesive	19 (63.3)	18 (60)	37 (61.7)	0.000	1.000 ^a
Cohesive	0 (0)	0 (0)	0 (0)		
Mixed	11 (36.7)	12 (40)	23 (38.3)		

Table 3. Percentage of fracture types based on all study groups

Fracture Types	Groups						Total	Test Statistic	p
	A1	A2	A3	B1	B2	B3			
Adhesive	7(70)	8 (80)	4 (40)	8 (80)	6 (60)	4 (40)	37 (61.7)	6.798	0.235 ^a
Cohesive	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)		
Mixed	3 (30)	2 (20)	6 (60)	2 (20)	4 (40)	6 (60)	23 (38.3)		

Group A: monochromatic composite resin restorations

In the control group (A1), where the enamel was acid-etched and restored with monochromatic composite resin, the stereomicroscopic evaluations revealed a relatively smooth enamel surface due to the absence of surface roughening. This smooth surface resulted in lower bond strength compared to the groups where surface roughening was performed.

In the A2 group, where the enamel was roughened using thick/medium grit discs and restored with monochromatic composite resin, stereomicroscopic evaluations revealed a uniformly roughened surface. This surface roughness facilitated stronger adhesion between the composite resin and the enamel. Adhesive fractures were the most common failure mode, occurring in 80% of the specimens, while mixed fractures were observed in the remaining 20%.

In the A3 group, where the enamel was roughened using Er, Cr: YSGG laser and restored with monochromatic composite resin, stereomicroscopic evaluations revealed deep and irregular surface roughness. This surface treatment significantly enhanced the bond strength between the composite material and enamel (Fig 1,2 and 3).

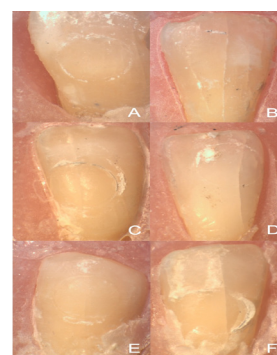


Fig 1. Representative stereomicroscope images of fracture surfaces obtained after shear bond strength testing, illustrating enamel surfaces treated under different surface roughening conditions (25x). (A) Acid-etched enamel surface restored with monochromatic composite resin (Control Group). (B) Enamel surface roughened with thick/medium-grit discs and restored with monochromatic composite resin. (C) Enamel surface treated with Er, Cr: YSGG laser and restored with monochromatic composite resin. (D) Acid-etched enamel surface restored with nanohybrid composite resin (Control Group). (E) Enamel surface roughened with thick/medium-grit discs and restored with nanohybrid composite resin. (F) Enamel surface treated with Er, Cr: YSGG laser and restored with nanohybrid composite resin.

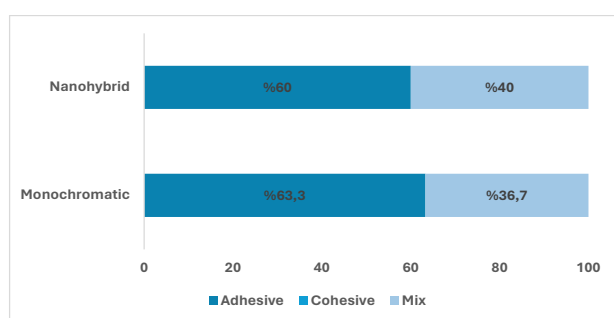


Fig 2. Proportions of fracture types based on composite materials

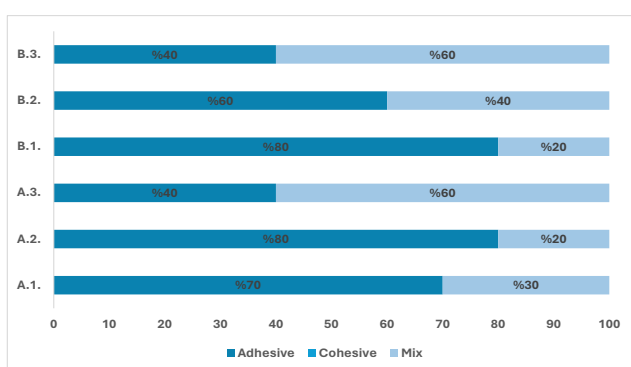


Fig 3. Proportions of fracture types based on all study groups

Group B: nanohybrid composite resin restorations

In the control group (B1), where the enamel was acid-etched and restored with nanohybrid composite resin, the lack of surface roughening resulted in a lower bond strength compared to the roughened groups. The predominant failure mode was adhesive fracture, observed in 80% of the samples, while mixed fractures occurred in the remaining 20%.

In group B2, where the enamel was roughened with thick/medium grit discs and restored with nanohybrid composite resin, the roughened surface resulted in improved adhesion. Adhesive fractures occurred in 60% of the specimens, indicating enhanced bond strength compared to the control group.

In group B3, where the enamel was roughened using Er, Cr: YSGG laser and restored with nanohybrid composite resin, the deep and irregular surface roughness created by the laser treatment significantly enhanced the adhesion between the composite material and enamel.

Fracture types based on composite resins

The fracture types observed in both nanohybrid and monochromatic composite resins were categorized into three groups: adhesive, cohesive, and mixed. No statistically significant relationship was found between the composite materials and fracture types ($p=1$).

The adhesive failure was observed in 63.3% of the monochromatic composite resin group and 60% of the nanohybrid composite resin group. Cohesive failure was not observed in any of the groups. The mixed failure was observed in 36.7% of the monochromatic composite resin group and 40% of the nanohybrid composite resin group.

Fracture types based on surface preparation techniques

There was no statistically significant difference between surface preparation techniques and fracture types ($p=0.235$). The adhesive failure rates were as follows: 70% in Group A1, 80% in Group A2, 40% in Group A3, 80% in Group B1, 60% in Group B2, and 40% in Group B3. No cohesive failure was observed in any group. The rates of mixed fracture types were: 30% in Group A1, 20% in Group A2, 60% in Group A3, 20% in Group B1, 40% in Group B2, and 60% in Group B3.

Overview of results

The fracture type analysis revealed that adhesive fractures were the predominant failure mode across most groups, except for Er, Cr: YSGG laser-treated samples, which displayed a higher proportion of mixed fractures. These results highlight the importance of surface preparation methods in enhancing both the bond strength and longevity of restorative materials.

DISCUSSION

In recent years, restorative dentistry has witnessed significant advancements in adhesive technologies and surface preparation methods, aimed at enhancing the bond strength of resin composites to dental hard tissues. One such advancement is laser etching, which has emerged as a promising alternative due to its precision, minimal invasiveness, and ability to create optimal surface morphologies. Despite the established efficacy of traditional acid etching techniques in achieving micromechanical retention, the demand for improved adhesion in complex clinical scenarios has driven the exploration of alternative or complementary methods. Surface preparation methods, including mechanical roughening (using rotary instruments such as discs), chemical etching (with phosphoric acid), and laser etching (Er,Cr:YSGG lasers), each offer distinct advantages and limitations. However, their combined effects on adhesion remain insufficiently explored. This study investigates the influence of different surface preparation methods on the fracture behavior of nanohybrid and monochromatic composite resins. The study aims to compare conventional surface roughening techniques such as phosphoric acid etching with alternative approaches, including rotary instrumentation (such as discs) and laser etching. Through statistical analysis of fracture patterns, the study seeks to evaluate the impact of surface preparation on the fracture behavior of these composite resins. The findings are expected to contribute to the development of more

effective and minimally invasive surface treatment strategies in restorative dentistry, improving both the longevity and performance of dental restorations.

In our study, we found that adhesive failure was predominant in the acid etching and mechanical roughening (discs) groups, which is consistent with the findings of Al Habdan et al. (2021). They observed higher adhesive failure rates in groups using acid etching and mechanical roughening techniques. However, we found that mixed failure was more commonly observed in the laser-etched groups, which suggests that laser etching might induce different surface morphology changes compared to traditional methods. These changes could affect bond strength and failure modes. Al Habdan et al. (2021) also reported similar findings, linking laser etching's effect on bond strength to the surface changes it induces. While laser etching offers a less invasive approach, further research is required to fully understand its influence on adhesive bond strength, particularly when considering variations in surface morphology (Al Habdan et al., 2021).

Our results are also in align with Bilgrami et al. (2022), who similarly reported high adhesive failure rates in their study on composite bonding. Bilgrami et al. (2022) observed that different surface treatment methods impacted the bond strength and failure modes, supporting our own findings that acid etching and mechanical roughening lead to higher adhesive failure (Bilgrami et al., 2022).

Additionally, we observed that the mixed failure mode was more common in the laser-etched groups, while adhesive failure was predominant in the acid-etched and mechanical roughening groups. This observation is in agreement with Sibai et al. (2022), who reported similar trends. Specifically, Sibai et al. (2022) noted that laser treatments led to mixed failure in most groups, while the group treated with Single Bond Universal self-etch adhesive showed complete debonding (adhesive failure). Our findings support their observation that acid etching enhances bond strength compared to self-etch adhesives, with laser etching producing a rough surface but not improving bond strength as much as acid etching (Sibai et al., 2022).

These differences in failure modes (adhesive vs. mixed) can be attributed to the surface morphology changes induced by various surface preparation techniques. Further studies are needed to investigate the long-term clinical implications of different surface treatments, especially in terms of their influence on bond strength and failure modes.

CONCLUSION

This study demonstrates that surface preparation methods, such as phosphoric acid etching and mechanical roughening, result in higher adhesive failure rates, while laser etching predominantly leads to mixed failures. Laser etching provides a less invasive approach with promising results but still requires further research to optimize

its use in clinical applications. The study highlights the importance of surface morphology changes in bond strength and failure modes and suggests that different surface treatments should be explored further to enhance the durability and longevity of composite resin restorations.

Limitations and Future Perspectives

Although this study presents encouraging results, it has certain limitations. As an in vitro investigation, it does not fully simulate the complex dynamics of the oral environment. Future in vivo studies are needed to assess factors such as salivary contamination, occlusal forces, and thermal changes. Furthermore, additional research should focus on examining the combined effects of laser and acid etching under various laser parameters. Establishing standardized protocols could improve the clinical feasibility and consistency of laser-based surface preparation methods. In addition, the universal adhesive system utilized in this study with selective enamel etching may have influenced the results. Investigating alternative adhesive systems could offer valuable perspectives for refining bonding techniques. Moreover, further research is recommended to assess the impact of different surface roughening methods on the fracture types resulting from bond strength. While this study used stereomicroscopy for the analysis, advanced imaging techniques such as SEM could also be employed.

Acknowledgments

This study was supported by the Scientific Research Projects Coordination Unit of Marmara University under grant number 11114. The authors extend their gratitude for the support and resources that facilitated this study.

Conflicts of Interest

The authors have no financial interest in any companies or products mentioned in this article.

Ethical Approval

This study received ethical approval from the Clinical Research Ethics Committee of Marmara University Faculty of Dentistry (Protocol No: 2023/138).

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