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Comparative Effects of Femtosecond, Er, Cr:YSGG, and Ho:YAG Laser Irradiation versus Hydrofluoric Acid Etching on Surface Roughness and Repair Bond Strength of Lithium Disilicate Ceramic

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Abstract

This study comparatively evaluated hydrofluoric acid (HFA) etching, Er, Cr:YSGG laser (ECL), Ho:YAG laser, and femtosecond (FS) laser irradiation on surface roughness (R_a), surface topography, and repair bond strength (RBS) of lithium disilicate ceramic (LDC) bonded to resin composite. A total of 130 rectangular LDC specimens were fabricated and randomly allocated into five groups (n = 26): untreated control (G1), 9.5 % HFA etching (G2), ECL irradiation (G3), Ho:YAG laser (G4), and FS laser (G5). R_a was assessed using a contact profilometer (n = 8), surface morphology by field emission scanning electron microscopy (n = 8), and RBS by a universal testing machine at 0.5 mm/min crosshead speed (n = 10). Failure modes were classified stereomicroscopically. Data were analyzed using one-way ANOVA and Tukey HSD post-hoc test ($p < 0.05$). G5 recorded the highest R_a (1.45 ± 0.12 µm) and RBS (15.12 ± 1.63 MPa), while G1 demonstrated the lowest R_a (0.28 ± 0.04 µm) and RBS (4.82 ± 0.93 MPa). G2, G3, and G5 demonstrated statistically comparable R_a and RBS values ($p > 0.05$). Within the limitations of this *in vitro* study, FS laser and ECL irradiation may offer clinically viable, acid-free options for intraoral LDC repair. Longitudinal clinical validation remains necessary before routine adoption.

Keywords: Lithium disilicate ceramic, surface roughness, repair bond strength, Ho:YAG laser, femtosecond laser, hydrofluoric acid, resin composite

I. Introduction

The last two decades have seen a marked transition toward all-ceramic restorations in contemporary restorative dentistry, with lithium disilicate ceramic (LDC) establishing itself as one of the most clinically adaptable substrate materials currently available [1,2]. Its characteristic microstructure – comprising densely interlocked acicular $Li_2Si_2O_5$ crystals (~ 70 vol%) embedded within a residual aluminosilicate glass matrix – imparts flexural strength values of 360 – 400 MPa and fracture toughness in the range of 2.0 – 3.3 MPa, making it suitable for restorations spanning the full clinical spectrum, from minimally invasive inlays and veneers to full-coverage posterior crowns [3,4]. Nevertheless, cumulative fracture incidences of 3 – 11 % over five-year observation periods have been documented, with posterior segments disproportionately affected. Given that complete prosthetic replacement necessitates progressive hard tissue sacrifice, heightened pulpal injury risk, and substantial patient financial burden [5,6], intraoral composite repair has gained considerable clinical endorsement as the preferred first-line management strategy for structurally intact LDC restorations sustaining localized fractures [7].

The durability of any intraoral ceramic repair is fundamentally contingent upon the quality of the adhesive interface established between the ceramic substrate and the overlying composite [8]. Effective surface conditioning must fulfill two interdependent objectives: generating sufficient surface roughness (R_a) to permit resin micromechanical interlocking within ceramic surface irregularities, and producing reactive silanol groups that enable covalent coupling through bifunctional organosilane chemistry [9]. Hydrofluoric acid (HFA) etching has historically addressed both requirements by selectively dissolving the silica-rich glassy phase of LDC, yielding a characteristically microporous crystalline topography that optimizes resin infiltration depth and silane reactivity [10,11]. As a result, HFA has been considered the reference standard for LDC surface preparation for nearly three decades. However, its clinical application is accompanied by a well-documented and serious risk profile, including pronounced concentration-dependent cytotoxicity, potential for irreversible chemical injury to the oral mucosa and perioral tissues, and notable respiratory

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hazard upon intraoral use [12,13]. These limitations have prompted growing interest in identifying safer, equally effective conditioning alternatives.

Laser-based surface modification has emerged as a potentially viable alternative to HFA etching, offering a contactless, chemically inert, and parametrically adjustable ablation approach that eliminates the cytotoxic and biosafety concerns associated with acid-based ceramic conditioning [14]. Among the laser systems investigated, the Er,Cr:YSGG laser (ECL; wavelength: 2.78 μm) is among the most thoroughly characterized in dental hard tissue research. Its ablation mechanism relies on selective absorption of laser energy by hydroxyl groups and residual water within the ceramic microstructure, initiating a hydrokinetic micro-explosive process that produces a heterogeneous micro- and macroretentive surface architecture without inducing significant thermal injury to the underlying material [15,16]. Accumulating evidence supports ECL as a surface conditioner for both zirconia and LDC, with adhesive outcomes that are at minimum comparable to, and in some investigations superior to, those achieved through conventional HFA etching [12,17]. The Ho:YAG laser (wavelength: 2.12 μm), a solid-state system characterized by its substantial tissue penetration depth, compatibility with flexible fused-silica fiber delivery systems, and reliable hemostatic properties, has similarly attracted interest as a ceramic surface conditioning modality [18]. Alkhudhairy *et al.* reported significantly higher shear bond strength (SBS) values for Ho:YAG-laser-conditioned hybrid ceramic specimens relative to other surface treatment modalities, attributing the improvement to the pronounced surface microroughness generated by this system [12]. Despite this preliminary evidence, data characterizing the influence of Ho:YAG irradiation on R_a, surface morphology, and composite repair bond strength of LDC are conspicuously absent from the literature – a knowledge gap that currently limits its evidence-informed adoption in ceramic bonding protocols.

More recently, femtosecond (FS) lasers have attracted substantial scientific interest in dentistry owing to their capacity to deliver ultrashort pulses on the order of 10^{-15} seconds, a property that minimizes thermal diffusion to adjacent structures and enables precision surface modification at the micro- and nanoscale [19,20].While FS laser irradiation has been investigated as a surface conditioner for zirconia with encouraging outcomes, its effect on the R_a, surface topography, and repair bond strength (RBS) of LDC bonded to resin composite remains insufficiently characterized, warranting systematic evaluation [21].

The present study was conducted under the null hypothesis that no statistically significant differences in R_a, surface topography, or RBS would exist among LDC specimens conditioned with HFA etching, ECL, Ho:YAG laser, or FS laser irradiation relative to an untreated control. Accordingly, this investigation aimed to systematically evaluate and compare the effects of these four surface conditioning modalities on the R_a, surface morphology, and RBS of LDC bonded to resin composite.

II. Materials and Methods

A total of 130 rectangular specimens (14 mm × 4 mm × 1.5 mm) were sectioned from a lithium disilicate CAD/CAM block using an automatic precision sectioning machine (IsoMet 5000, Buehler Ltd., Seoul, Republic of Korea) under continuous water coolant (Fig. 1). Sample size was determined *a priori* using G*Power software (version 3.1.9.7; Heinrich Heine University, Düsseldorf, Germany) for a one-way ANOVA design, based on an anticipated effect size of $f = 0.40$ (large), a significance level of $\alpha = 0.05$, and a desired statistical power of $1 - \beta = 0.80$. This calculation yielded a minimum required sample size of eight specimens per group for R_a and surface topography outcomes, and ten specimens per group for RBS testing, consistent with comparable investigations in the ceramic surface conditioning literature [22,23]. One surface of each specimen was sequentially wet-polished with 400- and 600-grit silicon carbide abrasive papers using a grinding and polishing unit (MetaServ 250, Buehler Ltd.) for 15 seconds, and the final specimen thickness was standardized to 1.00 ± 0.01 mm as verified by a digital caliper. All specimens were subsequently ultrasonically cleaned in distilled water, air-dried, and subjected to the crystallization firing cycle in accordance with the manufacturer's recommended protocol.

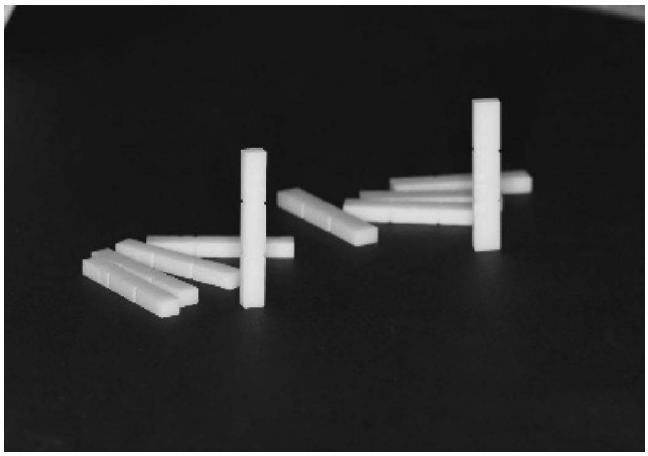

Fig. 1: Lithium disilicate CAD/CAM blocks.

Experimental groups and surface treatment protocols

Group 1 (G1): Untreated Control: Specimens received no surface treatment and served as the negative control.

Group 2 (G2): HFA Specimens were etched with 9.5 % hydrofluoric acid gel (Porcelain Etchant, Ultradent Products Inc., South Jordan, UT, USA) applied uniformly to the bonding surface for 20 seconds. It was rinsed thoroughly with water spray for 5 seconds and dried with oil-free compressed air. All procedures were performed under appropriate personal protective measures in a well-ventilated area [24].

Group 3 (G3): ECL Specimens assigned to G3 underwent surface conditioning with ECL irradiation (Millennium, BIOLASE Technology Inc., San Clemente, CA, USA) at the following parameters: wavelength 2.78 μm, output power 1.5 W, and pulse energy 300 mJ, delivered under simultaneous air/water spray coolant to

prevent thermal accumulation at the ceramic surface. The laser handpiece tip was maintained perpendicular to and at a standardized working distance of 1 mm from the specimen surface throughout the irradiation period of 60 seconds. Following irradiation, all specimens were thoroughly rinsed with distilled water for 180 seconds and air-dried with oil-free compressed air before subsequent bonding procedures [10,25].

Group 4 (G4): Ho:YAG Laser Specimens assigned to G4 were conditioned using a Ho:YAG laser system (Stone-Light Holmium Laser System, AMS Inc., Minnetonka, MN, USA) operating at a wavelength of 2 090 nm. Irradiation was delivered in pulsed mode at an output power of 6 W, pulse energy of 0.5 J, and a repetition frequency of 8 Hz, under continuous water cooling throughout the irradiation period to prevent thermally induced microstructural damage to the ceramic substrate. The laser handpiece was maintained perpendicular to and at a standardized working distance of 1 mm from the specimen surface for a total irradiation duration of 20 seconds. All parameters were selected in accordance with previously published surface conditioning protocols [26].

Group 5 (G5): FS laser Specimens assigned to G5 underwent surface conditioning with a femtosecond laser system (Femtopower Compact Pro, Santa Clara, CA, USA) operating at a wavelength of 1 026 nm, pulse duration of 220 fs, and repetition rate of 100 kHz. Surface irradiation was performed at a fixed scan speed of 5 mm/s and a laser fluence of 160 J/cm², delivered in a controlled non-contact scanning mode for a total irradiation duration of 30 seconds [27].

***R_a* measurement** Surface roughness was assessed on eight specimens from each group using a contact profilometer (Mitutoyo SJ-210, Mitutoyo Corporation, Kawasaki, Japan) calibrated before measurements according to the manufacturer's specifications. For each specimen, three measurements were recorded at different locations across the conditioned surface using a 0.8 mm cut-off length and a 4 mm evaluation length, and the mean R_a value (μm) was calculated. All measurements were performed by a single calibrated examiner under standardized conditions to minimize operator variability [28].

Surface topography analysis Representative specimens from each group (n = 8) were sputter-coated with a thin layer of gold-palladium alloy and examined using a field emission scanning electron microscope (FE-SEM; JEOL JSM-7600F, JEOL Ltd., Tokyo, Japan) at accelerating voltages of 10–15 kV. Images were acquired at standardized magnifications of × 500 and × 2 000 to qualitatively assess surface morphological changes induced by each conditioning modality [29].

Composite repair Before the composite application, all conditioned ceramic surfaces across all experimental groups received a standardized adhesive protocol. A silane coupling agent (Monobond Plus, Ivoclar Vivadent, Schaan, Liechtenstein) was applied to each treated surface using a disposable microbrush and allowed to react for 60 seconds, after which residual solvent was evaporated with a gentle stream of oil-free compressed air for 15 seconds in accordance with the manufacturer's instructions. A single coat of dental adhesive resin (Scotchbond Universal Adhesive, 3M ESPE, St. Paul, MN, USA) was subsequently applied to the silanized surface, gently air-thinned for 10 seconds, and light-polymerized for 20 seconds using a calibrated LED curing unit (Elipar S10, 3M ESPE, St. Paul, MN, USA; output intensity: 1 200 mW/cm²). Afterwards, resin composite (Filtek Z250, 3M ESPE, St. Paul, MN, USA) was then applied incrementally onto each treated ceramic surface in 2-mm increments using a hand instrument, with specimen geometry standardized through a split rubber mold of 4 mm internal diameter and 4 mm height. Each incremental layer was light-polymerized for 40 seconds. All fabricated specimens were subsequently stored in distilled water at 37 °C for 24 hours before mechanical testing [24].

RBS testing Each specimen was securely mounted in a custom-designed holding jig and positioned within a universal testing machine (Santam, Model STM-20, Tehran, Iran) to evaluate the RBS at the ceramic-composite adhesive interface. A chisel-edged shear blade was aligned perpendicular to the bonded interface and loaded at a standardized crosshead speed of 0.5 mm/min until interfacial failure occurred. The maximum load at failure was recorded in Newtons (N), and the RBS was calculated in megapascals (MPa) according to the following formula:

Where F represents the maximum load at failure (N), and A denotes the cross-sectional bonded surface area (mm²) of the composite cylinder; r is the internal radius of the standardized rubber mold (r = 2 mm; internal diameter = 4 mm), yielding a fixed cross-sectional bonded surface area of:

$$A = \pi \times (2)^2 = \pi \times 4 = 12.57 \text{ mm}^2$$

The composite cylinder dimensions were thus standardized across all specimens at 4 mm diameter and 4 mm height, and the RBS for each specimen was derived by dividing the recorded failure load (N) by the constant bonded cross-sectional area of 12.57 mm², with all values expressed in megapascals (MPa).

Failure mode analysis All debonded specimens were examined under a stereomicroscope (Dino Lite Pro, AnMo Electronics Corp., Taiwan, ROC) at × 40 magnification to classify failure modes as adhesive, cohesive, and admixed [30].

Statistical analysis Data normality was assessed using the Shapiro-Wilk test. One-way analysis of variance (ANOVA) followed by Tukey's post hoc test was applied for multiple group comparisons when data conformed to a normal distribution. All statistical analyses were performed using SPSS software (Version 26.0, IBM Corp., Armonk, NY, USA), with the level of significance set at $p < 0.05$.

III. Results

R_a outcomes

Table 1 displays the R_a outcomes among the experimental groups. The FS-laser-treated specimens (G5) recorded the highest mean R_a value (1.45 ± 0.12 μm). Whereas untreated samples G1 exhibited (0.28 ± 0.04 μm).

R_a analysis revealed statistically significant differences among all experimental groups (one-way ANOVA, $p < 0.05$). G2 (HFA) (1.42 ± 0.11 µm), G3 (ECL) (1.38 ± 0.14 µm), and G5 (FS laser) (1.45 ± 0.12 µm) presented comparable outcomes of R_a $p > 0.05$, whereas Ho:YAG laser (0.87 ± 0.09 µm) presented significantly lower R_a than the Group 2, 3, and 5, yet higher than Group 1. $p < 0.05$ (Fig. 2).

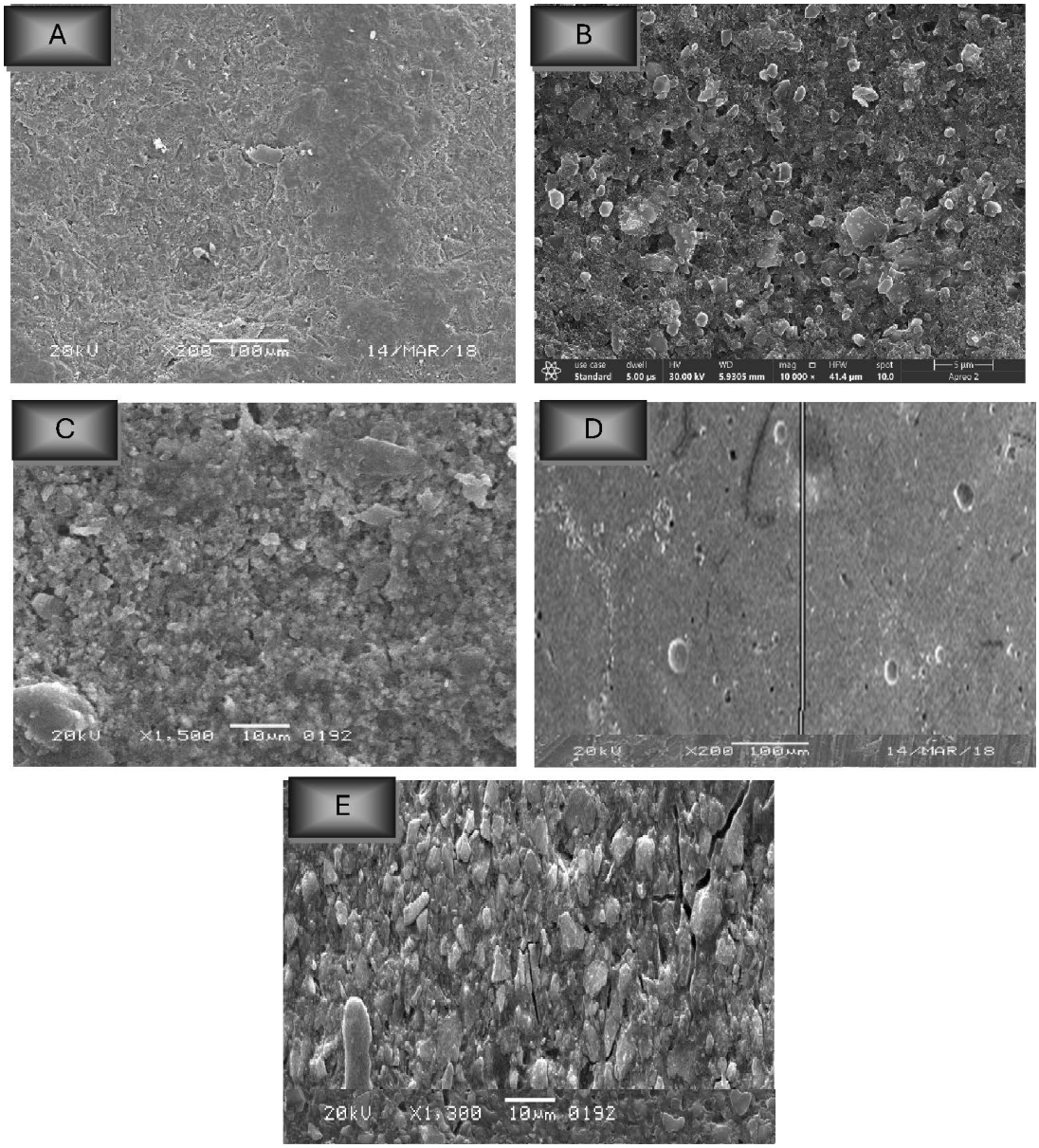

Fig. 2: Scanning electron micrographs of lithium disilicate ceramic surfaces: (A) untreated control, (B) HFA etching, (C) Er, Cr: YSGG laser, (D) Ho: YAG laser, (E) femtosecond laser.

Table 1: R_a outcomes among the experimental groups.

#	Groups	Treatment	Mean R_a ± SD (µm)	Min (µm)	Max (µm)	Sig.†
1	G1	Untreated control	0.28 ± 0.04	0.19	0.37	c
2	G2	HFA	1.42 ± 0.11	1.18	1.65	a
3	G3	ECL	1.38 ± 0.14	1.07	1.69	a
4	G4	Ho:YAG laser	0.87 ± 0.09	0.64	1.08	b
5	G5	FS laser	1.45 ± 0.12	1.18	1.72	a

R_a = arithmetic mean surface roughness; SD = standard deviation.
† Different lowercase letters indicate statistically significant differences between groups (one-way ANOVA with Tukey HSD post-hoc test, $p < 0.05$

Table 2: Repair the shear bond strength (SBS, MPa) of resin composite bonded to lithium disilicate ceramic across experimental surface treatment groups.

#	Groups	Surface Treatment	Mean SBS ± SD (MPa)	Min (MPa)	Max (MPa)	Sig.†
1	G1	Untreated control	4.82 ± 0.93	2.9	6.7	c
2	G2	HFA	14.56 ± 1.74	10.8	17.6	a
3	G3	ECL	13.89 ± 1.82	9.4	16.9	a
4	G4	Ho:YAG laser	8.73 ± 1.21	5.9	11.2	b
5	G5	FS laser	15.12 ± 1.63	11.4	18.3	a

One-way ANOVA

† Different lowercase letters indicate statistically significant intergroup differences (Tukey HSD post-hoc, $p < 0.05$)

RBS

Table 2 displays the RBS outcomes among all the experimental groups. The FS-laser-treated specimens (G5) recorded the highest mean bond strength value (15.12 ± 1.63 MPa), whereas untreated samples G1 exhibited (4.82 ± 0.93 MPa) displayed the lowest bond strength. RBS analysis revealed statistically significant differences among all experimental groups (one-way ANOVA, $p < 0.05$). G2 (HFA) (14.56 ± 1.74 MPa), G3 (ECL) (13.89 ± 1.82 MPa), and G5 (FS laser) (15.12 ± 1.63 MPa) presented comparable outcomes of bond integrity. $p > 0.05$. whereas Ho:YAG laser (8.73 ± 1.21 MPa) presented significantly lower RBS than Groups 2, 3, and 5, yet higher than Group 1.

Failure modes

Failure mode outcomes among the tested groups are shown in Fig. 3. G2, G3, and G5 exhibited cohesive failures the most, whereas G4 displayed the most admixed fractures, and G1 showed adhesive failure most frequently.

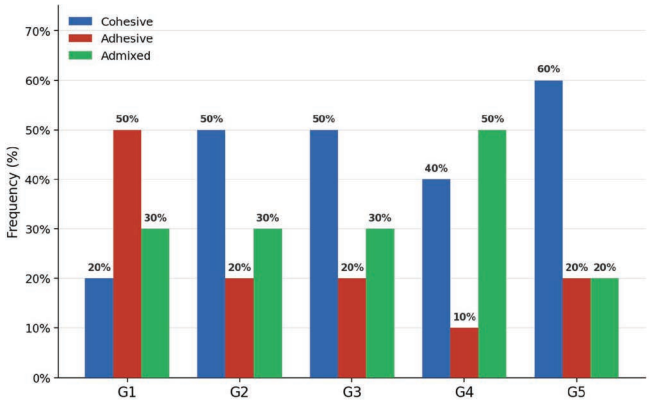

Fig. 3: Failure mode distribution across groups.

IV. Discussion

The present study was designed on the null hypothesis that no significant differences would exist in R_a and surface topography among LDC specimens conditioned with HFA etching, ECL, Ho:YAG laser, or FS laser compared to an untreated control. It was further hypothesized that no significant difference in the RBS of LDC to composite would be observed across the evaluated conditioning regimes. Based on the outcomes, it was observed that all the groups presented significantly different yet higher R_a, surface topography, and RBS outcomes than the control, thus rejecting both the tested hypotheses. Shear bond strength testing is one of the most widely employed mechanical assays in dental biomaterials research. It quantifies the force per unit area required to dislodge a bonded material from a dental substrate under a shearing load. Its enduring popularity stems from a convergence of methodological simplicity, clinical relevance, and interpretive versatility.

The FS laser yielded the highest R_a and RBS values among all experimental groups. This outcome is attributable to the ultrashort pulse duration characteristic of FS laser systems operating in the picosecond-to-femtosecond range, which enables precise surface ablation while effectively eliminating collateral thermal damage to the surrounding ceramic matrix. These findings are consistent with those reported by Erdur and associates, who demonstrated that FS-laser-conditioned ceramic surfaces exhibited the highest R_a values, characterized by distinct ablation craters, selective matrix degradation, and a uniform microroughness profile [31]. Similar morphological features were observed in the SEM micrographs of the present study. The resultant surface architecture is expected to enhance micromechanical interlocking at the ceramic-composite interface, directly contributing to the superior RBS recorded in this group [10]. Corroborating evidence was further provided by Garcia-Sanz *et al.* [32] who reported that FS laser conditioning of LDC surfaces yielded significantly greater R_a and SBS values relative to all other evaluated modalities. These collective findings underscore the clinical translational potential of FS laser irradiation as a superior and biologically safe surface conditioning alternative for LDC repair protocols. Nevertheless, its influence on the broader mechanical integrity of the ceramic substrate including flexural, tensile, and compressive strength warrants systematic investigation before definitive clinical recommendations can be established.

HFA- and ECL-pretreated LDC discs presented comparable scores for R_a and RBS to that of FS-laser-conditioned samples which were significantly higher than the control. This aligns with the outcomes of previous research conducted by Alshahrani *et al.* [22] and Khuld

al Ali [33]. According to their assertion, HFA etching functions by selectively dissolving the glassy matrix phase of the LDC specimen, thereby exposing the underlying rod-like lithium disilicate crystal morphology [22,33]. This preferential dissolution of the amorphous silica-rich matrix leaves the crystalline phase intact and protruding, creating a characteristic interlocking microstructure typified by a series of microscopic peaks and valleys across the ceramic surface, as was observed in the SEM micrographs [34]. The resulting topography substantially increases the effective surface area available for bonding and promotes micromechanical interlocking with subsequently applied repair material. Past evidence has reported that with higher HFA concentrations (7.5 % and 10 %), greater glassy matrix removal and exposure of lithium disilicate crystals are observed. This increased surface area correlated with higher bond strength to composite resin [35]. Furthermore, the significantly promising outcomes after ECL pretreatment can be attributed to it mechanism of action. This work is in concordance with the findings of *in vitro* analysis conducted by Alkhudhairy and associates [36]. ECL works on the principle of micro-explosion during tissue ablation, resulting in micro- and macroscopic irregularities on the irradiated surface [16,37]. In essence, the ECL increases R_a of LDC through a dual mechanism: (i) hydrokinetic photomechanical ablation driven by instantaneous vaporization and micro-explosive expansion of surface-adsorbed water molecules energized at 2 780 nm, and (ii) photothermal micro-ablation of the glassy matrix phase [38,39]. Together these mechanisms selectively disrupt the matrix, expose the crystalline architecture, and generate an ablation-crater topography whose R_a is directly tunable through laser parameter optimization. This finding is further supported by SEM analysis of the treated LDC surfaces with ECL conditioner.

The Ho:YAG laser demonstrated intermediate conditioning efficacy, yielding R_a and RBS values significantly lower than those recorded for HFA, ECL, and FS-laser-treated specimens, yet significantly superior to the untreated control a finding that positions it as a moderately effective, albeit suboptimal, surface conditioning modality for intraoral LDC repair. The mechanistic basis for this intermediate performance may be elucidated through the dual-laser analogy proposed by Albakri and colleagues, who characterized the Ho:YAG laser as exhibiting ablation properties intermediate between those of CO_2 and Nd: YAG laser systems [40]. This spectral duality has direct implications for ceramic surface modification efficacy. The intermediate R_a and RBS outcomes observed for Ho:YAG laser in the present study likely reflect this spectral dichotomy: sufficient ceramic matrix interaction to exceed baseline surface energy, yet inadequate to generate the microtopographic complexity characteristic of CO_2-analogous or hydrokinetic ablation mechanisms. Further systematic investigation employing optimized Ho: YAG laser parameters is therefore warranted.

Regarding failure mode, cohesive failure predominated in HFA, ECL, and FS laser-treated groups, an outcome directly attributable to the superior interfacial bond strength generated by these conditioning modalities. When the adhesive interface exceeds the cohesive strength of the bonded materials, fracture propagates within the composite or ceramic substrate rather than at the interface itself, a failure pattern widely regarded as indicative of clinically optimal bonding efficacy. Ho:YAG laser, on the contrary, presented admixed failures the most, whereas untreated control displayed adhesive failures. The predominance of admixed failure in the Ho:YAG laser group reflects a moderately developed adhesive interface sufficiently strong to partially resist interfacial debonding yet inadequate to redirect fracture propagation entirely within the substrate [41,42]. The exclusively adhesive failures recorded in the untreated control are consistent with the inherently weak interfacial bonding characteristic of unconditioned ceramic surfaces, as previously corroborated in the literature [43].

The present study carries inherent limitations attributable to its *in vitro* design, precluding direct extrapolation to the complex biomechanical environment of the oral cavity. Findings are specific to the evaluated ceramic substrate, laser systems, and composite material, and may not be universally applicable across alternative material combinations. The effect of laser irradiation on the bulk mechanical properties of LDC, including flexural and compressive strength, was not investigated, despite evidence that laser-induced thermal gradients may alter ceramic microstructure under functional loading. Prospective randomized clinical trials incorporating long-term functional loading and patient-specific variables are therefore warranted to validate the translational applicability of these findings.

V. Conclusions

Femtosecond laser and Er, Cr: YSGG laser irradiation represent evidence-based, biologically safer alternatives to conventional HFA etching for intraoral LDC repair protocols. However, more studies are required to confirm this outcome.

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