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Influence of the Surface Modification of Lithium Disilicate Ceramics with Er, Cr: YSGG, and Nd:YAG Laser on Surface Properties, Roughness, and Bond Strength Bonded to Light- and Dual-Cure Cements

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Abstract

The influence of hydrofluoric acid (HFA), Er, Cr: YSGG laser (ECL), Nd:YAG laser (NdYL) on the surface roughness (*Ra*), characterization, and shear bond strength of lithium disilicate ceramics (LDC) bonded to light cure (LC) and dual-cure (DC) resin cement was studied. 78 LDC discs were fabricated with the CAD-CAM technique. Samples were categorized into cohorts based on conditioning protocol: Group 1-HFA, Group 2-ECL, and Group 3-NdYL. Assessment of *Ra* and surface characterization was conducted using a profilometer and SEM. Twenty samples from each group were subdivided into two subgroups. Light-cure (LC) (A) and dual-cure (DC) resin cement (B) were built on the LDC, and this was followed by thermocycling. A universal testing machine and stereomicroscope were used for SBS and failure mode assessment. *Ra* and SBS were evaluated via ANOVA with post hoc multiple comparisons. The highest *Ra* was exhibited by Group 1-HFA. Group 3-NdYL-conditioned samples demonstrated the lowest *Ra*. The maximum bond integrity score was recorded for HFA+DC resin cement. The minimum bond strength was noted for NdYL+LC cement. LDC can be effectively conditioned with HFA and bonded with DC resin cement. Due to the drawbacks associated with HFA, it is advisable to seek a superior alternative for the surface conditioning of LDC.

Keywords: Lithium disilicate ceramics, shear bond strength, Er, Cr: YSGG laser, hydrofluoric acid, scanning electron microscopy.

I. Introduction

Dental ceramics are characterized by remarkable attributes such as chemical inertness, compatibility with biological tissue, minimal thermal conductivity, and significant resistance to mechanical stress [1]. Lithium disilicate ceramics (LDC) represent a category of heat-pressed ceramics that exhibit superior fracture toughness and bending strength in comparison to earlier generations of dental materials [2]. Typically, the microstructural composition of LDC comprises two fundamental elements such as silica, which serves as the glass matrix, and lithium oxide (Li_2O) [3]. Although this material exhibits excellent resistance to failure, it is necessary to precondition its intaglio surface before bonding to achieve higher surface roughness (*Ra*) and shear bond strength (SBS) when bonded to resin cement [4]. Therefore, recent advancements in adhesive dentistry have prompted the exploration of various surface treatment methodologies.

Micromechanical conditioning using hydrofluoric acid (HFA) is regarded as a benchmark method, as it enhances the *Ra* of ceramics by generating undercuts and expanding the surface area available for bonding [1, 5]. Consequently, the utilization of a silane coupling agent (S) after HFA pretreatment enhances the wettability of the treated ceramic surface while establishing a covalent chemical bond between the silica constituents of the ceramic restoration and the organic components of the bonding resin [6, 7]. Nevertheless, the process of etching using HFA poses significant risks for both the patient and the dental practitioner, necessitating the complete removal of HFA before the initiation of the bonding procedure [8, 9].

Laser irradiation of ceramic surfaces has attracted considerable interest in recent years. This phenomenon has been the subject of investigation for many years. The Erbium, Chromium: Yttrium, Scandium, Gallium, and Garnet (Er, Cr: YSGG) laser (ECL) is increasingly utilized in various procedures related to dentistry [10 – 12]. Its operation relies on the principle of explosions at the micro level, which leads to both microscopic and macroscopic surface irregularities [10, 13, 14]. Furthermore, neodymium-doped yttrium aluminum garnet (Nd:YAG) lasers (NdYL) characterized by a wavelength of 1 064 nm are employed to enhance the *Ra* of the ceramic surface and thereby improve the surface energy for better adhesion [15, 16]. On the contrary, there is data available that has discovered that the bond integrity scores following the application of ECL and NdYL lasers were found to be comparable to,

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or even lower than, the control (HF) [17]. Nevertheless, the existing data regarding the influence of this laser on the *Ra* and SBS of various resin cements bonded to LDC remains ambiguous, warranting further investigation.

In addition to surface pretreatments, the influence of various resin cements on the bond strength of LDC is significant. Resin cements are traditionally categorized based on three distinct polymerization methods and their respective applications [1]. Light-cure (LC) and dual-cure (DC) resin cements are employed for the cementation of aesthetic restorative materials, whereas self-cure cement is utilized in areas with limited accessibility, such as posts and cores [18]. Moreover, a higher degree of conversion has been associated with enhanced mechanical properties and bond strength of the resin cement [19] However, the data regarding the impact of these cements, when photo-polymerized using different techniques, on the shear bond strength (SBS) with ceramics conditioned with HF, EYL, and Nd: YL remains inconclusive, necessitating further investigation.

There exists a scarcity of evidence that has examined the different influences of conventional surface treatments and laser applications in conjunction with different types of resin cement on the *Ra* scores and bond strength of LDC. The null hypothesis posited in this investigation is that there will be no substantial difference in the *Ra* values of laser-treated LDC discs when compared to the control. Furthermore, it was anticipated that the SBS of different resin cements adhering to the same conditioning group would exhibit comparable outcomes. Consequently, the objective of the present inquiry was to investigate the influence of varying conditioners (HF, ECL, NdYL) on the bond strength of LDC bonded with different resin cements.

II. Materials and Methods

(1) Specimen preparation

Seventy-eight LDC discs (Emax, Ivocalr Vivadent, Schaan, Liechtenstein), having dimensions of 5 mm diameter and 2 mm height, were fabricated using the CAD CAM technique. The discs were then submerged in an ultrasonic bath filled with distilled water for 10 minutes to ensure cleaning. The discs were rooted in self-cure acrylic resin (DuraLay; Polidental Ltda) using a silicon mold (15 mm diameter and 20 mm height). The samples were meticulously polished and completed using 1000-grit silicon carbide paper. Each of the samples was categorized into three distinct cohorts according to the specific conditioning protocol employed (n = 26) [20].

Group 1 (HFA): 9.5 % HF (IPS® Ceramic Etching Gel-Ivoclar Vivadent, located in Schaan, Liechtenstein) was applied using a micro brush for 2 mins. The discs were washed with pressurized water for 20 seconds to thoroughly eliminate any residual acid from the surfaces of the specimens, and the samples were subsequently dried [1].

Group 2 (ECL): The ceramic samples in this cohort underwent conditioning by means of an ECL (Biolase, Waterlase I-Plus laser (Biolase, Irvine, CA, USA) functioning at a frequency of 50 Hz and set to a power level of 4.5 W, featuring a pulse duration of 230 µs, categorized as a very brief pulse, applied in a non-contact fashion wavelength = 2.78 µm and energy parameter = 300 mJ. T. The laser tip, designated as MZ8, was positioned 2 mm away from the surface of the LDC discs. During this treatment process, the air/water pressure was consistently maintained at a ratio of 65 % to 55 % [21, 22].

Group 3 (NdYL): A Nd:YAG laser (Smartlife, DEKA, Serial No. FA9A41041AQ, Italy) was employed for the pretreatment of the LDC within this particular group. The device was configured to operate in its optimal 532-nm second harmonic generation mode. The diameter of the laser beam measures 8 mm, with a pulse width of 10 ns, and it functioned at a frequency of 10 Hz. The output power of the laser was adjusted to 200 mJ at 532 nm, and the duration of exposure was set for 3 minutes [23].

All specimens were stored in distilled water at 37 °C for 24 hours following the different conditioning protocols.

(2) Assessment of Ra

The *Ra* was measured using a profilometer (Dektak D150; Veeco). For the roughness assessment, five specimens from each experimental group were utilized (n = 5). Three measurements were recorded, one at the center of the specimen and two parallel measurements positioned on the right and left sides of the center. Following this, the average of these readings was computed. The initial findings were expressed in angstroms and subsequently converted into nanometers (nm) [24].

(3) Surface characterization using SEM

Following the surface modification process, the samples were sputter-coated with a layer of gold for 360 seconds to minimize scanning imperfections and image artifacts. These samples were subsequently examined with a scanning electron microscope (SEM) at different magnifications (Vega Tescan TS5136LS, Tescan Orsay, located in Brno-Kohoutovice, Czech Republic) (n = 1) [25, 26].

(4) Subgroups based on resin cement

Twenty samples from each group were subsequently subdivided into two distinct subgroups. Light-cured Variolink Esthetic LC (VLC; Ivoclar Vivadent Ltda, Barueri, SP, Brazil) (A) and dual-cured Variolink N (VN; Ivoclar Vivadent Ltda, Barueri, SP, Brazil) (B). Two clear cylindrical molds (Saint Gobain Performance Plastic, Miami Lakes, FL, USA) measuring 2 mm in height and 2 mm in internal diameter were positioned on the LDC disc surface. Resin cements were mixed following the guidelines provided by the manufacturer. A #5 exploratory probe (HuFriedy, Chicago, IL, USA) was utilized to apply the resin cement within the molds. Each resin cement cylinder was subjected to light-curing using the Coltolux 4 (Coltène, Whaledent, Mahwah, USA) light-curing unit.

(5) Storage and thermocycling of the specimens

All the samples were immersed in distilled water at room temperature, specifically maintained at 37 °C for 24 hours. Following this, the specimens were subjected to thermocycling in a thermocycling apparatus, alternating between temperatures of 5 °C and 55 °C for a cumulative total of

10 000 cycles, with each temperature bath allowing a dwell time of 30 seconds and transfer time of 5 secs [27].

(6) SBS analysis

Specimens were tested for maximum failure loads before fracture on a universal testing machine (Instron 5965 Material Testing System) at a cross-head speed of 0.5 cm/min. A gradually increasing load was applied perpendicularly along the interface using a conical, round-ended metal probe. The applied force was measured in MegaPascals (MPa) [28]

(7) Failure analysis

The evaluation of the fracture type was conducted with the aid of a stereomicroscope (Hirox-KH-7700). The failures were categorized into three distinct types: adhesive failures occurring at the junction of the ceramic and resin, cohesive failures manifesting within the resin cement itself, and admixed failures that exhibited characteristics of both resin cement and the ceramic-resin interface.

(8) Statistical analysis

The normality of the data was assessed using the Kolmogorov-Smirnov test. All the collected data were systematically compiled utilizing the Statistical Package for the Social Sciences (SPSS-Version 20, based in Chicago, IL, USA) software, and the gathered data were evaluated employing one-way analysis of variance (ANOVA) along with *post hoc* multiple comparisons ($p < 0.05$).

III. Results

(1) Surface Characterization following different conditioning regimes via SEM

Fig. 1 A: The SEM micrograph of LDC conditioned with HFA displays a loss of matrix, resulting in a glass-like surface and increased surface roughness (*Ra*). Similarly, the SEM image in Fig. 1 B of LDC that has been treated with Er, Cr: YSGG laser shows insufficient micro-depths, which are attributed to either excessive damage to the matrix phase or crystals or the formation of a heat-damaged layer. In contrast, the SEM image of LDC treated with an Nd:YAG laser reveals a surface that has been glazed, melted, and resolidified, with the micrograph highlighting the formation of globules and the presence of cracks Fig. 1C.

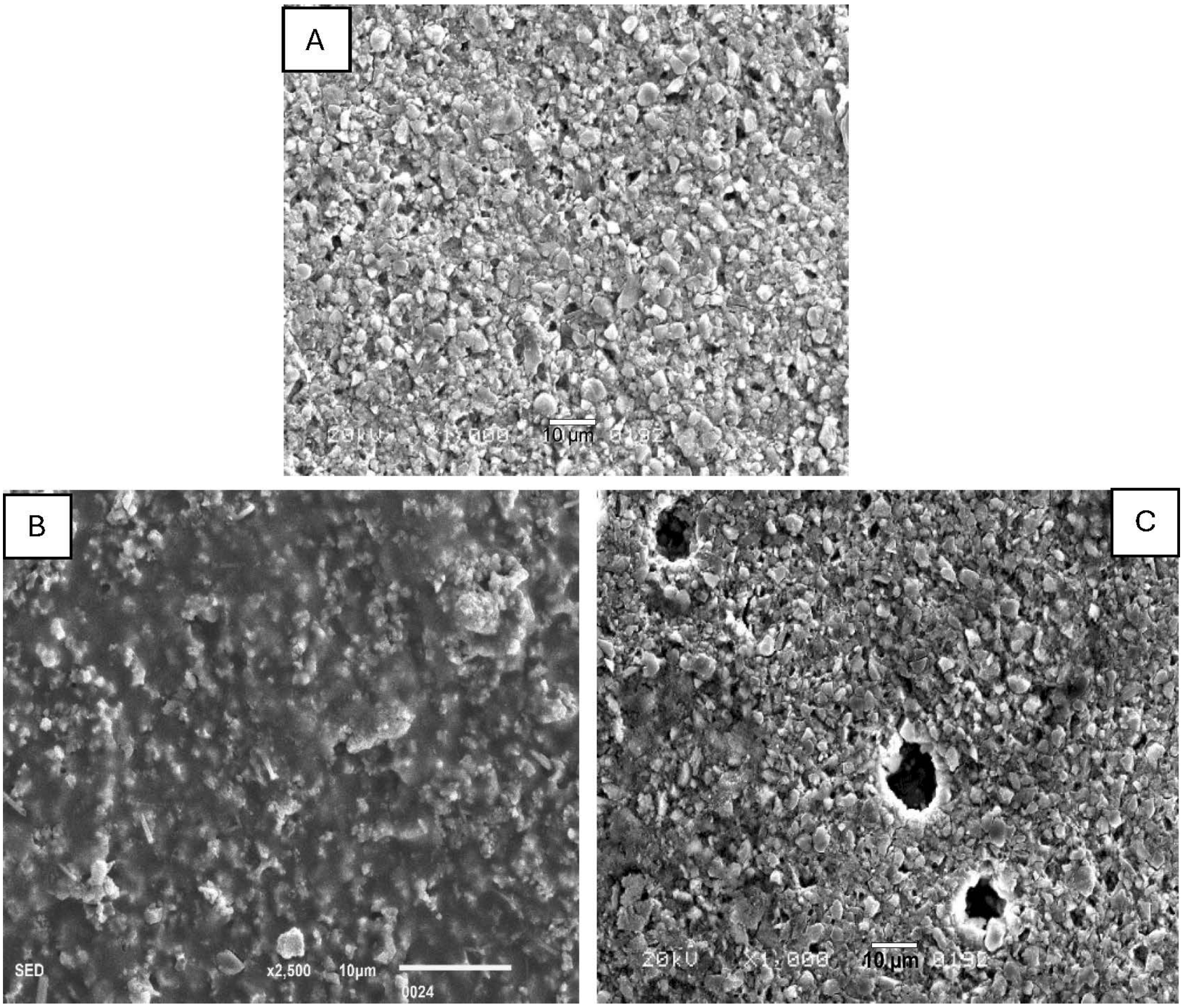

Fig. 1: Fig. 1A the SEM micrograph of LDC treated with HFA shows matrix reduction, resulting in glass-like appearance and increased surface roughness (*Ra*). Fig. 1B's SEM image of LDC exposed to the Er,Cr:YSGG laser reveals insufficient micro-depths, possibly due to matrix phase damage, crystal damage, or heat-damaged layer formation. The SEM image of LDC treated with the Nd:YAG laser shows a glazed, melted, and resolidified surface, with globules and cracks forming Fig. 1C.

(2) *Ra analysis*

The mean and SD of roughness among all tested groups are delineated in Table 1. The highest mean *Ra* score was exhibited by Group 1 (HFA) (1 041.53 ± 0.023 µm)-pretreated discs. Conversely, Group 3 (NdYL) (856.27 ± 0.035 µm)-conditioned samples demonstrated the lowest *Ra* outcomes. A comparative analysis revealed that Group 2 (ECL) (879.66 ± 0.021 µm) and Group 3 presented no statistically significant difference in *Ra* scores ($p > 0.05$)

Table 1: *Ra* of LDC after conditioning with different surface pretreatment protocols.

Tested groups	*Mean ± SD (µm)*	*p- value!*
Group 1: HFA	$1\,041.53 \pm 0.023^{*}$	*< 0.05*
Group 2: ECL	$879.66 \pm 0.021^{**}$	
Group 3: NdYL	$856.27 \pm 0.035^{**}$	

! ANOVA
Hydrofluoric acid (HFA), Er, Cr: YSGG laser (ECL), Nd: YAG laser (NDYL)
Different superscript asterisk characters denote statistically significant differences (Post Hoc Tukey)

(3) *SBS analysis*

The SBS of resin cement following pretreatment with various conditioning agents is presented in Table 2. The maximum bond integrity score was recorded for Group 1B (HFA + DC resin cement) (10.85 ± 0.24 MPa).

On the other hand, the minimum average bond strength was noted for Group 3A (NdYL + LC cement) (7.82 ± 0.14 MPa). A comparative evaluation among the experimental cohorts revealed that Group 2A (ECL + LC cement) (7.78 ± 0.11 MPa) and Group 3A (NdYL + LC cement) (7.82 ± 0.14 MPa) exhibited no statistically significant differences in the bond strength parameter ($p > 0.05$). Likewise, Group 2B (NdYL + LC cement) (8.07 ± 0.17 MPa) and Group 3B (NdYL laser + DC cement) (8.11 ± 0.19 MPa) also demonstrated no significant difference in bond strength outcomes. ($p > 0.05$) Nevertheless, Groups 1A (HFA + LC cement) (9.23 ± 0.12 MPa) and 1B exhibited significantly dissimilar bond strength values ($p < 0.05$)

Table 2: SBS of LDC bonded to two different resin cements after pretreatment with different conditioning agents

Investigated groups	*Mean ± SD (MPa)*	*p-value!*
Group 1A: HFA + LC cement	9.23 ± 0.12^{c}	*< 0.05*
Group 1B: HFA + DC resin cement	10.85 ± 0.24^{d}	
Group 2A: ECL + + LC cement	7.78 ± 0.11^{b}	
Group 2B: ECL + DC cement	8.07 ± 0.17^{a}	
Group 3A: NdYL + LC cement	7.82 ± 0.14^{b}	
Group 3B: NdYL laser + DC cement	8.11 ± 0.19^{a}	

! ANOVA
Hydrofluoric acid (HFA),
Er, Cr: YSGG laser (ECL), Nd: YAG laser (NDYL)
Different superscript small alphabets denote statistically significant differences (Post Hoc Tukey)

(4) *Failure type analysis*

In the examination of different failure modes Fig. 2, it became evident that the LDC discs conditioned with ECL and NdYL exhibited admixed types of failures. Conversely, the discs that were pretreated with HFA primarily showed a cohesive type of failure most frequently.

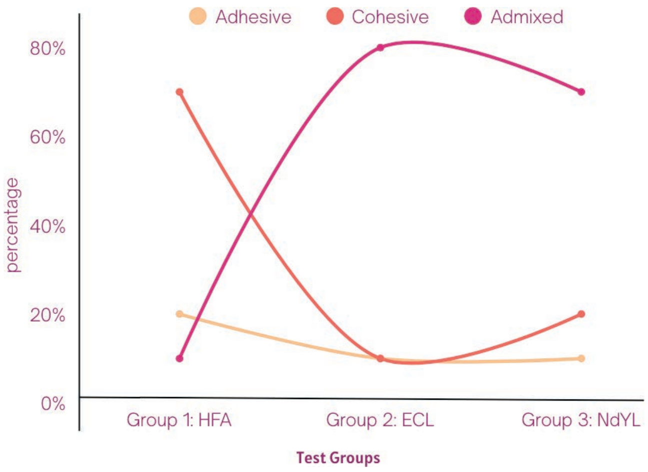

Fig. 2: Percentage of failure types in different experimental groups.

IV. Discussion

The present analysis was based on the hypothesis that there would be no significant difference in the *Ra* values of laser-treated LDC discs when compared to the control group. Additionally, it was also anticipated that the SBS of different resin cements adhering to the same conditioning group would exhibit comparability. Consequently, the objective of this study was to investigate the influence of varying surface treatments on the bond strength of LDC with different resin cements. The findings derived from this research indicated that the utilization of 4.5-W ECL and 4.5-W NdYL lasers to prepare the surface of LDC did not enhance the SBS when juxtaposed with the HFA conditioning, thereby rejecting the primary hypothesis put forth. In contrast, the secondary hypothesis was similarly dismissed, as the DC resin cement demonstrated superior bond strength performance in comparison to its LC resin cement counterpart.

The surfaces that were treated with a solution of 10 % HFA combined with a silane coupling agent exhibited the highest SBS values observed in the present study. This finding corroborates earlier research conducted by Lyann *et al.* [29] The processes of HFA etching followed by silanization contributed to the micromechanical and chemical bonding mechanisms found in silica-based ceramics, as described by Chaharom and colleagues [30]. This also enhances the surface free energy, microporosity, and surface area of the material, which subsequently reduces the contact angle and improves the wettability of resin cement with ceramics [31]. The surface characteristics of the LDC discs identified by means of SEM that underwent pretreatment exhibited noticeable alterations, resulting in the formation of grooves and pores that elucidate the *Ra* values and bond strength results.

NdYL and ECL have been employed for the surface conditioning of various dental materials [11]. Numerous *in vitro* investigations have assessed the SBS following the surface preparation of ceramic restorations utilizing different lasers and power settings. However, the present findings indicated that ECL and NdYL irradiation did not influence the adhesion strength with resin cement when juxtaposed with the control samples [32]. A reduction in the bond strength of ceramic surfaces treated with ECL may be ascribed to insufficient micro-depths generated by excessive damage to the matrix phase or crystals, or to the formation of a heat-damaged layer [8, 33]. Similarly, the available literature revealed that even a short exposure of NdYL resulted in a surface that looks glazed, melted, and resolidified, while sometimes also causing the formation of globules and the occurrence of cracks [34]. Similar findings were noted on the surfaces of the irradiated samples during this study, highlighting distinct cracks, indications of melting, and signs of re-solidification. Furthermore, it was also stated previously that as the intensity of the NdYL energy intensified, craters were surrounded by globules [35]. This aligns with the results of the laboratory-based analysis conducted by Akpinar *et al.* [36] They reported that the SBS levels were inadequate for clinical application when NdYL was used at 4 Watts. SEM images of laser-conditioned discs generated a smooth surface topography lacking retentive micromechanical features. All these observations validated the rationale that may explain the bond integrity scores and *Ra* scores in these groups [36].

According to the available literature, it can be stated that DC resin cements are advantageous in situations where light penetration is limited, such as with thicker or more opaque materials, when compared to LC resin cements [37]. Regarding cement type, it can be observed that DC cement performed better than LC resin cement. This finding can be explained by research analysis conducted by Mondal and Mazumdar [38]. They have discovered that DC cement provides higher adhesive strength of LDC veneers when compared to LC resin cement [38]. This finding can further be explained by the fact that achieving a high degree of conversion (DC) is essential for strong and durable bonds. Past evidence has stated that DC resin cement shows a higher DC than light-cured cement.

It is essential to thoroughly examine the various fracture modes present in studies focused on adhesion. The quality of the bond should not be determined solely by analyzing the data related to bond strength [20]. The mode of failure could yield significant insights regarding potential clinical limitations. In this specific research endeavor, a clear and direct relationship was noted between the various failure modes and the results obtained from bond strength assessments. It can be observed that samples treated with HFA displayed a cohesive failure pattern with the highest frequency. Conversely, samples conditioned with ECL and NdYL showed an admixed failure.

The current research presented specific limitations that must be acknowledged. While established protocols were followed during the fabrication of samples, however, the characteristics of the specimen‘s surface could have been influenced by the manufacturing process. Furthermore, how the materials were handled throughout production might have affected the final results. Moreover, since this investigation was performed *in vitro*, it was not able to consider all the variables that could exist in the oral environment, including factors like fatigue and fluctuations in pH levels. Considering the limitations previously mentioned, the findings of the present study should be generalized with caution, and additional laboratory-based and *in vivo* research are necessary to validate the results of the current analysis.

V. Conclusions

Lithium disilicate ceramics can be effectively conditioned and bonded using hydrofluoric acid and dual-cure resin cement. Nonetheless, due to the drawbacks associated with hydrofluoric acid, it is advisable to seek a superior alternative for surface conditioning.

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