

Impact of Er; Cr: YSGG Laser at Different Power Outputs and Hydrofluoric Acid on the Shear Bond Strength and Color Stability of Lithium Disilicate Ceramic and Hybrid Ceramic

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Abstract

Aim Influence of Er; Cr: YSGG laser (ECL) at different power outputs on the color change (ΔE) and shear bond strength (SBS) of lithium disilicate (LDC) and hybrid ceramics (HBC).

Method 48 discs from each LDC discs (Group A) and HBC discs (Group B) were prepared and allocated into three subgroups: Subgroup-1 (HFA), Subgroup-2 (ECL@3.5W), and Subgroup-3 (ECL@7W). ΔE assessment was performed on five samples from each subgroup. One specimen from each group underwent SEM to assess the topographic changes after conditioning. Resin cement was smeared onto the discs of ten samples from each group and thermocycled followed by SBS and failure mode evaluation. ANOVA and post hoc Tukey were used to assess the SBS and color stability ($p < 0.05$).

Result Maximum ΔE was witnessed in the Group A1 (HFA+LDC) treated group. Whereas Group A2 (ECL@3.5W+LDC) samples exhibited minimum ΔE . The highest SBS was observed in the Group A1 (HFA +LDC) pretreated group. Whereas the Group B3 (ECL@7W+HBC) samples revealed the lowest bond strength.

Conclusion LDC performed better than HBC in terms of bond strength and color stability when the same conditioning methods were applied. ECL at the power output of 3.5 W can be considered a suitable alternative to condition HBC and LDC.

Keywords: Lithium disilicate, hybrid ceramics, Er;Cr:YSGG laser, hydrofluoric acid, color change.

I. Introduction

Modern prosthodontics offers a variety of dental materials for the creation and production of fixed dental prostheses ^{1,2}. A wide range of ceramic systems is accessible, each featuring a distinct composition or fabrication technique. Lithium disilicate ceramics (LDC) and hybrid ceramics (HBC) are two widely used materials in clinical settings ³. LDC, introduced in 1998, is composed of lithium oxide crystals embedded within a glassy matrix ^{1,4}. The interlocking arrangement of these crystals impedes crack propagation, thereby enhancing the material's flexural strength. However, because of its inherent brittleness, this material is susceptible to chipping, cracking, and fracture ⁵. Conversely, HBC also exhibits promising mechan-

ical properties by combining the advantages of ceramics and composite resins ⁶. This material is fabricated by incorporating polymers into a porous feldspathic ceramic rich in aluminum oxide, offering numerous benefits such as exceptional fracture resistance ⁷. Despite these advancements, it remains unclear which material is superior in terms of clinical longevity and success.

The intrinsic properties of the material are crucial, as is its ability to bond with natural teeth. Various methods have been employed to prepare different ceramic surfaces. The combination of hydrofluoric acid (HFA) etching and silane coupling agent application remains the benchmark for surface pretreatment ^{8,9}. HFA specifically eliminates the matrix, exposing the crystalline structure and enhancing micromechanical interlocking with resin cement ^{10,11}. As noted by Niuzuma *et al.* ¹² and Alsunbul *et al.* ⁸, the interaction of HFA with the glassy matrix increases the sub-

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strate energy and surface area for silane bonding, resulting in a chemical-mechanical bond at the ceramic-cement interface, thus improving the shear bond strength (SBS). However, HFA's corrosive nature poses risks to human soft tissues, necessitating safer alternatives.

Recently, the Er,Cr: YSGG laser (ECL) has gained popularity among dental professionals as an effective ceramic surface conditioner¹³. Operating at a wavelength of 2 780 nm, ECL is recognized as a painless and non-invasive method for treating dental ceramics¹⁴. Current research indicates that the impact of ECL surface conditioning on the SBS of LDC and HBC on resin cement is controversial^{15,16}. Moreover, laser effects are primarily dependent on the parameters used. Consequently, this study examined various power outputs to assess their influence on the bond strength between LDC, HBC, and resin cement.

The aesthetic success of dental rehabilitation relies heavily on the color stability of dental materials¹⁷. In the oral environment, various foods and drinks can compromise the integrity of dental restorations, potentially causing aesthetic issues¹⁸. Thus, it is imperative to investigate how different pretreatment methods affect the color change (ΔE) of HBC and LDC discs.

The study was based on the assumption that ECL pretreatment set at two different power outputs would show no significant difference in ΔE and bond strength when compared to HFA in both indirect restorative materials. Moreover, it was projected that the bond strength and color stability of LDC and HBC would be similar, regardless of the conditioning method employed. Thus, the present study aimed to assess the impact of ECL at different power outputs on the color change and SBS of LDC and HBC in resin cement.

II. Materials and Methods

(1) Sample preparation

Group A - LDC discs LDC blocks (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) were manufactured using pressing technology with dimensional parameters of 3 mm in height and diameter. The blocks were cross-sectioned into 48 ceramic plates and water-cooled using a cutting machine (Isomet 4000, Buehler, Berlin, Germany). Subsequently, the plates were rooted in auto-curing acrylic resin. The surface facing upward was polished using 600- and 1 000-grit silicon carbide papers with constant irrigation. The plates were cleaned with 10-% citric acid and placed in an ultrasonic bath for 20 min to eliminate any impurities¹⁷.

Group B - HBC disc Blocks of HBC Cerasmart (GC Corp., Tokyo, Japan) were cut into 48 discs with the same dimensions as for the LDC.

Discs from each group (LDC and HBC) were allocated into six subgroups (A1, A2, A3, B1, B2, and B3) based on the surface conditioning regime used ($n = 16$ for each group).

Subgroup 1 (A1, B1): The samples were etched with 9.5-% HFA (Pulpdent, Watertown, Mass, USA) for 20 seconds, after which they were rinsed and air-dried. This was followed by silane (Bisco, USA) application with the help of a brush for 10–12 secs, followed by careful air-drying¹⁹.

Subgroup 2 (A2, B2): For the ECL (Waterlase; Biolase Technology, Irvine, CA, USA), settings of 3.5 W power output and 2.780 nm wavelength were employed. The laser tip was held approximately 1 mm away from the ceramic surface to deliver a spray composed of 65 % air and 55 % water. Pulses lasted 140 μ s each, with the laser application continuing for 1 min²⁰. *Subgroup 3 (A3, B3):* In this experiment, a 7-W power output of Er, Cr: YSGG laser was employed to condition LDC and HBC, with all other parameters remaining consistent with those used in Subgroup 2¹³.

(2) Color change – ΔE assessment ($n = 5$ from each subgroup)

A reflective spectrophotometer (UV-2401 pc; Shimadzu Co) was used to assess the color change using the CIELAB (Commission International de l'Eclairage) system. The data collection adhered to the illuminant C standard, which simulates the average daylight in the reflectance mode against a black background. The specimen was illuminated at a 0-degree angle through a 7-mm aperture in an integrating sphere. The spectrophotometer was adjusted using a calibration plate, and a 2-mm diameter area in the mid-labial region was focused on all specimens. The L^* , a^* , and b^* coordinates of brightness, red-green, and blue, respectively, constitute this system. The ΔE values were computed using the equation²¹

$$\Delta E: (\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2$$

(3) SEM analysis

One specimen from each subgroup underwent SEM (Carl Zeiss NTS, Oberkochen, Germany) to assess the surface topographic changes after gold sputtering (JFC-1100 fine-coat ion sputter)²². The machine was operated at an acceleration voltage of 15 kV and a vacuum pressure of 105 mbar.

Adhesive Cementation

Self-adhesive dual-curing resin cement (RelyX Unicem (Clicker dispenser) (3M ESPE, St. Paul, MN, USA) was applied to 10 discs from each subgroup via a silicon mold (2 mm height and 5 mm diameter), which was placed perpendicular to the flat occlusal surface. The adhesive cement was polymerized for 90 s using a light-curing unit (Elipar 3M ESPE, Seefeld, Germany). The discs were submerged in distilled water while the temperature was maintained at 37 °C. Subsequently, they underwent thermocycling (Ethik Technology, São Paulo, Brazil) for 5 000 cycles, maintaining a temperature in the range of 5 °C and 55 °C²². The submergence time in each water bath was 30 s²³.

(4) SBS test

Bonded discs were subjected to shear loading using a universal testing machine (UTM) (Odeme Dental Research, Brazil) at a speed of 0.5 mm/min. A metal tip shaped like a knife was used for this purpose. The SBS values were determined by dividing the failure load (N) by the bonding area (mm²), with the results documented in MegaPascals (MPa)^{24,25}.

(5) Analysis of failure modes

The failure modes were evaluated using a stereomicroscope (Nikon SMZ745T, Japan) at 40x magnification. Failures are classified as adhesive, cohesive, or admixed.

(6) Statistical analysis

Data normality was assessed using the Shapiro-Wilk test. One-way ANOVA and post hoc tests were used to compare bond strengths. The statistical calculations were conducted using SPSS software version 21 (SPSS Inc., Chicago, IL, USA), with a significance level set at 0.05.

III. Results

(1) Surface characterization of LDC and HBC

Fig. 1 shows various micrographs. A1 shows the LDC exposed to HFA, with arrows indicating needle-like structures and visible silica fusion. B1 displays the HBC after HFA treatment, with arrows pointing to visible barium and glass fillers, along with matrix loss. The A2 SEM image reveals a pronounced glass appearance on the LDC surface when treated with ECL at 3.5 W. Likewise, B2 exhibits barium and glass visibility, matrix loss, and crack propagation in HBC treated with ECL at 3.5 W. In A3, the SEM image illustrates intense glass fusion and thermal damage when exposed to ECL at 7 W (indicated by the arrow). Similarly, HBC treated with ECL at 7 W displayed severe burn areas (shown by arrows) without matrix loss, while barium and glass remained visible.

(2) Color change – ΔE assessment

The ΔE values of the LDC and HBC samples following the application of surface conditioners are shown in Fig. 2. Maximum ΔE was observed in Group A1 (HFA + LDC) (3.95 ± 1.53)-treated group. Whereas, Group A2 (ECL-3.5W + LDC) (1.92 ± 0.65) samples exhibited minimum ΔE . Intergroup comparisons showed that Group A1, Group B1 (HFA + HBC), Group A3 (ECL-7W + LDC), and Group B3 (ECL-7W + HBC) demonstrated no significant difference in ΔE ($p > 0.05$) (Table 1).

(3) SBS assessment

The SBS of LDC and HBC discs bonded to resin cement after using different surface conditioners is shown in Fig. 3. Maximum SBS was observed in Group A1 (HFA + LDC) (11.53 ± 0.32 MPa). Group B3 (ECL-7W + HBC) (9.53 ± 0.27 MPa) samples showed the lowest bond strength. Comparison among the tested groups revealed that Groups A1 and A2 (ECL-3.5W + LDC) (11.49 ± 0.31 MPa) presented no significant difference in bond integrity scores. ($p > 0.05$) Likewise, Group B1 (HFA + HBC) (11.14 ± 0.28 MPa) and Group B2 (ECL-3.5W + HBC) (11.01 ± 0.23 MPa) also exhibited no significant difference in the SBS ($p > 0.05$). However, bond strength scores between Group A3 (ECL-7W + LDC) (9.91 ± 0.21 MPa) and Group B3 (ECL-7W + HBC) (9.53 ± 0.27 MPa) were found to be comparable ($p > 0.05$) (Table 2).

Table 1: ΔE values indicating color alteration in LDC and HBC after exposure to HFA and ECL at various power settings.

Investigated groups	Δa	Δb	ΔL	ΔE (Mean $\pm$ SD)	p -value!
Group A1: HFA + LDC	0.123	3.543	3.371	3.95 ± 1.53^a	<0.05
Group B1: HFA + HBC	0.345	1.214	3.131	3.80 ± 1.22^a	
Group A2: ECL (3.5 W) + LDC	0.352	1.335	3.187	1.92 ± 0.65^b	
Group B2: ECL (3.5 W) + HBC	0.399	1.811	2.331	1.83 ± 0.71^b	
Group A3: ECL (7 W) + LDC	0.491	1.546	2.012	3.68 ± 0.31^a	
Group B3: ECL (7 W) + HBC	0.333	1.321	1.923	3.91 ± 0.57^a	

!ANOVA

Hydrofluoric acid (HFA), Er, Cr: YSGG laser (ECL)

Different superscript small alphabets denote statistically significant differences (Tukey Post Hoc Multiple Comparison test)

Table 2: Comparative analysis of LDC and HBC SBS after exposure to HFA and ECL under varying power conditions.

Tested groups	Mean $\pm$ SD MPa)	p -value!
Group A1: HFA + LDC	$11.53 \pm 0.32^*$	<0.05
Group B1: HFA + HBC	$11.14 \pm 0.28^*$	
Group A2: ECL (3.5 W) + LDC	$11.49 \pm 0.31^*$	
Group B2: ECL (3.5 W) + HBC	$11.01 \pm 0.23^*$	
Group A3: ECL (7 W) + LDC	$9.91 \pm 0.21^{**}$	
Group B3: ECL (7 W) + HBC	$9.53 \pm 0.27^{**}$	

!ANOVA

Hydrofluoric acid (HFA), Er, Cr: YSGG laser (ECL)

Different asterisks denote statistically significant differences (Tukey Post Hoc Multiple Comparison tests)

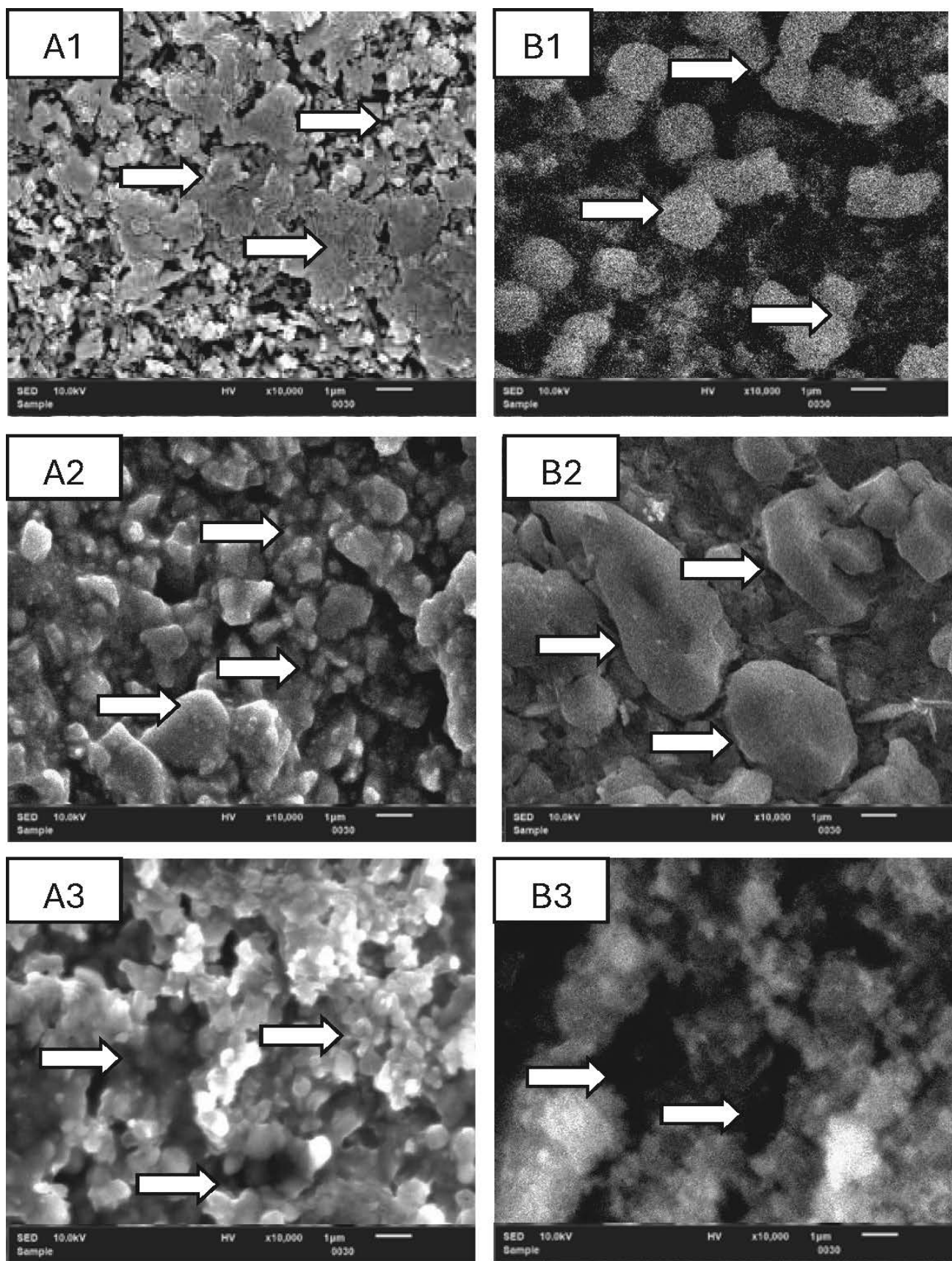


Fig. 1: A1 micrograph depicts LDC treated with HFA. Arrows show a needle-like appearance and the fusion of silica is also evident. B1 shows HBC treated with HFA. Arrows show barium and glass fillers visible with loss of matrix. SEM image of A2 shows a prominent glass appearance when the LDC surface is treated with ECL@3.5 W. Similarly, the B2 image shows barium and glass visible with loss of matrix and crack propagation when HBC treated with ECL@3.5 W. SEM image of A3 shows aggressive glass fusion and thermal burn when treated with ECL@7 W (Arrow). Similarly, HBC treated with ECL@7 W shows hostile areas of burn as shown by arrows with no loss of matrix and visible barium and glass.

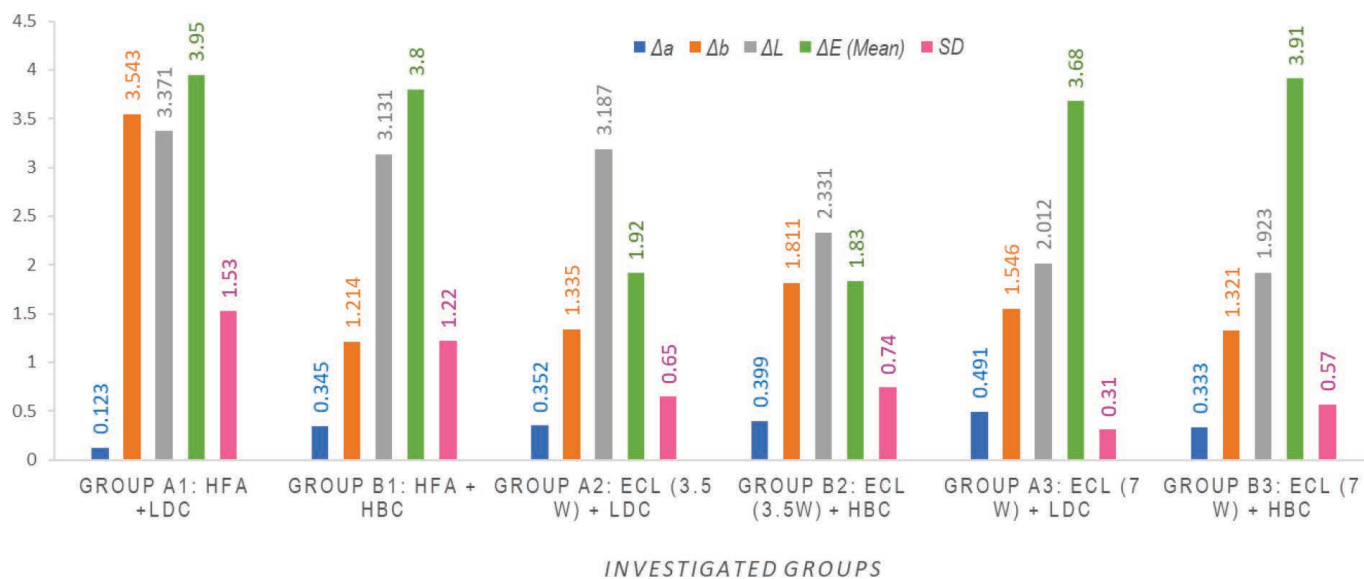


Fig. 2: Color change (ΔE) of LDC and HBC after using HFA and ECL at different power outputs.

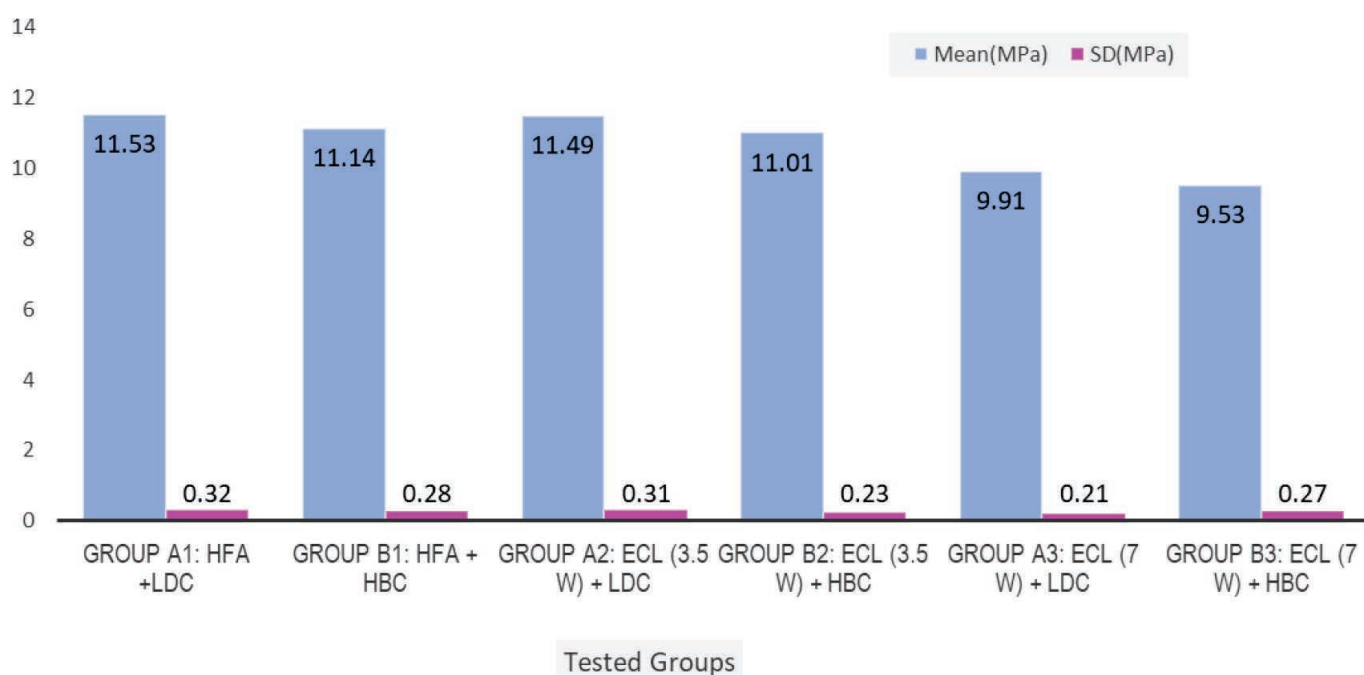


Fig. 3: SBS of LDC and HBC after using HFA and ECL at different power outputs.

(4) Failure mode assessment

The failure modes among the various groups are presented in Fig. 4. Groups A1, B1, A2, and B2 predominantly exhibited cohesive failures. In contrast, Groups A3 and B3 had the most admixed failures.

IV. Discussion

This research was conducted under the hypothesis that ECL pretreatment at two distinct power levels (7 W and 3.5 W) would not yield significant variations in ΔE and bond strength when compared to HFA for indirect restorative materials. Additionally, it was anticipated that LDC and HBC would exhibit comparable bond strength and color stability, irrespective of the conditioning tech-

nique used. The results of the current study revealed that the initial hypothesis was partially confirmed, while the second prediction was entirely disproven, as the HBC discs demonstrated significantly different performance than the LDC discs. A UTM was employed for SBS testing because this method enables the evaluation of a specific variable while keeping other factors constant^{15,26}.

The findings showed that discs subjected to HFA and ECL treatments at 3.5 W power demonstrated similar SBS within their respective material categories. However, increasing the power output to 7 W decreases the bond strength. The favorable outcomes observed in HFA-treated discs are consistent with previous research findings^{27,28}. Studies have reported that HFA creates a surface pattern of pores and grooves on discs, varying in

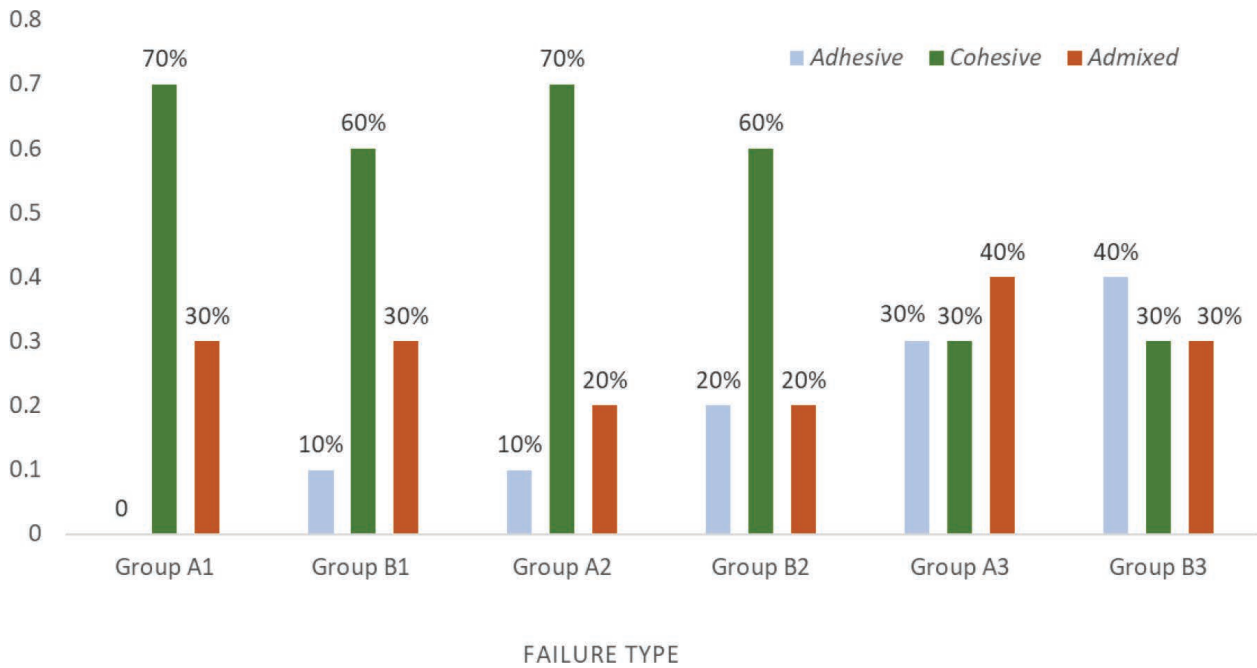


Fig. 4: Failure mode among various tested groups.

width and depth, by selectively dissolving the glass matrix and crystals. This process enhances the surface roughness, significantly improving interlocking and consequently increasing the SBS. Another investigation revealed that HFA impacts the outermost layer of the material, which is typically more prone to degradation²⁹. The roughening of the surface leads to an increase in surface energy, which in turn improves the wetting of the surface by resin cement, ultimately resulting in higher bond scores³⁰.

The acceptable performance of ECL at 3.5 W power output can be attributed to its ability to erode the surface through heat generation, thereby increasing the surface roughness³¹. However, the findings of this study also demonstrate that as the power increases, the average bond strength decreases. This observation aligns with laboratory research conducted by Kursoglu *et al.* Their findings suggest that higher power levels lead to reduced bond strength owing to the degradation and weakening caused by thermal vaporization of the substrate surface³². This process creates a heat-affected ceramic surface layer that forms weak bonds with the resin cement³³.

A significant difference in the SBS of the LDC and HBC groups was observed across all conditioning groups. The type of ceramic material substantially affected the bond strength of the resin cement, leading to the rejection of the second hypothesis. This variation may be attributed to differences in particle size, crystal structure, and manufacturing processes of the ceramic systems³⁴. IPS Emax ceramic consists of a glass-ceramic material with 70 % crystal volume embedded in a glass matrix, characterized by small grains measuring about 1.5 μm ¹⁵. The superior SBS of LDC compared to HBC can be explained by the micro-porosities created when the glass phase in LDC dissolves³⁵. In contrast, Cerasmart contains barium and glass fillers with a lower crystal volume, resulting in decreased surface roughness and, consequently, weaker bond strength.

The color difference was assessed using the CIE L*a*b* method, with a suggested threshold for visually noticeable but physiologically acceptable color change set at $\Delta E < 2.25$ ³⁶. Investigations into the impact of pretreatment on color stability revealed that both HFA and ECL at 7 W caused perceivable color alterations in the materials. In contrast, ECL at 3.5 W did not produce visible color changes in HBC and LDC discs. This can be attributed to the removal of the vitreous phase of the ceramic during the HFA acid etching process^{15,37}. The significant disparity between the two laser groups can be explained by the heat generated at higher power settings (7 W), which alters the chemical properties of LDC and HBC discs. Conversely, ECL at 3.5 W demonstrated color stability with fewer irregularities and non-uniformity, supporting its clinical application³⁸. Additionally, HBC exhibited more pronounced color changes compared to LDC, consistent with the findings of an *in vitro* study by Kilinc and Turgut³⁰. They attributed this greater color change in HBC to its polymer component.

The current *in vitro* study has certain limitations. Primarily, the lab-based nature of the study limits its generalization to clinical settings. Only two laser parameters were used to condition the LDC and HBC discs, which may have limited the outcomes obtained. Furthermore, atomic force microscopy (AFM) was performed to better understand the topography of the modified LDC and HBC.

V. Conclusions

LDC performed better than HBC in terms of bond strength and color stability when the same conditioning methods were applied. ECL at a power output of 3.5 W can be considered a suitable alternative to HBC and LDC as it displays comparable bond strength to hydrofluoric acid without causing color change.

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