

Impact of Lithium Disilicate Ceramic Surface Modifiers and Resin Cement of Different Viscosities on Surface Roughness and Bond Strength after Artificial Aging: A SEM Assessment

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Abstract

The impact of ceramic surface conditioners and resin cement with different viscosities on the surface roughness (Ra) and strength of the bond of dentin to lithium disilicate ceramics (LDC) at baseline and after aging has been investigated. Dentin and LDC discs were prepared. The discs were conditioned. Group 1: HFA+S, Group 2: SECP and Group 3: Er, Cr: YSGG. Ra and topographic analysis of conditioned discs was performed. Based on high and low viscosities, conditioned discs were bonded with dentin. Bonded samples with low and high viscosity underwent aging while others were not aged. SBS testing with a UTM machine was performed on both aged and non-aged samples and failure analysis was conducted. The means and SD for the SBS and Ra were analyzed using a one-way ANOVA and the Tukey post hoc tests. The maximum Ra score was obtained in specimens conditioned with HF acid-S. Group-2 samples (SECP) displayed the minimum value for Ra . The highest SBS values were attained by Group 1A (HF(S)+High) samples at baseline. The lowest SBS scores were presented by Group 2A (SECP+High) samples after thermal aging. The SBS of LDC is influenced by their microstructure obtained after surface conditioning. Long-term thermal aging causes a decrease in the mechanical performance of LDC discs bonded to dentin using high-viscosity resin cement.

Keywords: Lithium disilicate ceramics, viscosity, thermal aging, surface modification

I. Introduction

In the early 1970s, dental glass ceramics were predominantly used for anterior restorations on account of their excellent aesthetic qualities¹. However, advancements in their strength and toughness have since led to their increased popularity for posterior restorations, as evidenced by previous research^{2,3}. Lithium disilicate ceramics (LDC) have proven to be highly effective as prosthetic materials in dentistry, offering exceptional mechanical and optical properties⁴. However, it is crucial to recognize that the clinical success of ceramic restorations depends on the application of an impeccable luting technique⁵. Various surface modification methods have been proposed to enhance the adhesion between dental ceramics and resin-luting cement⁶.

Among the various conditioners used, a solution of 10 % hydrofluoric (HF) acid followed by a silane coupling agent is regarded as the gold standard for etching the surface of lithium disilicate ceramics (LDC)⁷. Hydrofluoric acid (HF) plays a crucial role in creating the necessary surface roughness (Ra) for cement interlocking⁸. Additionally, the silane compound enhances the chemical bond between the inorganic part of the ceramic and the organic part of the luting cement². However, to streamline the procedure and minimize the health hazards associated with HF acid, alternative options have been explored⁹. Recently, there have been recommendations to use one-step self-etch ceramic primer (SECP) as a potential substitute for HF etching when conditioning LDC⁵. This primer offers a more efficient and rapid application technique, reducing clinical time and minimizing the risk of injury compared to traditional protocols¹⁰.

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Another surface conditioning method that has gained widespread acceptance is laser irradiation. The Er, Cr: YSGG (ECL) laser is highly regarded by orthodontists for its effectiveness in enamel conditioning and reversing the effects of bleached enamel on adhesion¹¹. The results obtained with this laser are quite compelling. ECL has been used for pretreating zirconia and LDC, demonstrating promising outcomes in terms of the integrity of the bond with resin-luting cement^{12,13}. However, data on the impact of these pretreatment protocols on the surface roughness (Ra) and shear bond strength (SBS) of LDC bonded to the dentinal substrate remains limited and requires further investigation.

Apart from the LDC surface pretreatment techniques, the longevity of the adhesive bond is greatly influenced by the type of cement used. Microcracks can be introduced in glass ceramics due to milling and surface treatments, which might weaken their strength when these are used in critical load-bearing areas¹⁴. Given the low damage tolerance of ceramics, it is important for the resin luting agent to effectively penetrate and seal surface defects. This helps to minimize failure by enhancing the overall strength of the ceramic restorations¹⁵. It has been witnessed from past studies that resin cement that is highly filled is more viscous and may vary in terms of its ability to fill flaws created by conditioning protocols as compared to the less viscous types of cement¹⁶. However, data is still dubious regarding the impact of cement viscosity on the strength of the bond between LDC and dentin.

The current body of research has not yet studied the impact of different surface pretreatments on LDC Ra and SBS of different viscosity resin luting cement at baseline and after artificial thermal aging conditions. Thus, it was hypothesized that there would be no significant disparity in the Ra and bond integrity of different viscosity resin cement to bond LDC to dentin when SECP and ECL are used as a surface pretreatment regime as compared to the control. Furthermore, it was also anticipated that there would be no significant impact of artificial aging on the bond strength scores of LDC discs bonded to the dentinal substrate for both high and low-viscosity cement. Therefore, the existing exploration aimed to investigate the impact of various ceramic surface conditioners and resin cement having different viscosities on the Ra and bond strength of adhesively bonded LDC at baseline and after artificial aging.

II. Material and Method

(1) Dentin disc preparation

The present study followed a checklist for reporting *in vitro* studies (CRIS) guidelines. To prepare the dentin substrate, 150 human tooth incisors were washed and kept in a solution of 0.5 % chloramine at a temperature of 4 °C. The root was severed at the cemento-enamel junction (CEJ) using an Isomet (Buehler, Illinois, USA). The crown buccal surface was then drilled (SBE 1010 Plus, Metabo) to form cylindrical-shape discs with the dimensions of 1.5 mm thickness and 6 mm width. The discs then underwent a manual polishing process using silicon carbide papers of

400 and 1 200 grits (3M, Sumare, Rio de Janeiro, Brazil) to eliminate any irregularities and roughness¹⁷.

(2) LDC disc preparation

LDC (IPS e.max CAD, Ivoclar Vivadent) blocks were meticulously transformed into 153 discs having dimensions of 1.7 mm using an Isomet (Buehler, Illinois, USA) under water-cooling, which was followed by polishing of discs from both sides using a polishing machine (EcoMet/AutoMet 250, Buehler) until a desired thickness of 1.5 mm was achieved, ensuring standardized surfaces. All the LDC discs were then immersed for cleaning purposes in an ultrasonic bath which was filled with distilled water for 5 minutes.¹⁸

Afterward, the intaglio surfaces of the 153 LDC discs were conditioned using different regimes. ($n = 51$)

(3) Ceramic surface conditioning

Group 1

In this group, 10 % HF acid (Condac Porcelana, FGM, Joinville, Brazil) was used to pretreat the LDC discs with the help of a micro brush for 20 secs. They were then washed using air-water spray for 30 seconds followed by air drying. A silane-based coupling agent (Monobond N, Ivoclar, IL, USA) was scrubbed for 15 secs, allowed to react for 45 secs and then air-dried for 30 secs¹⁹ (Fig. 1).

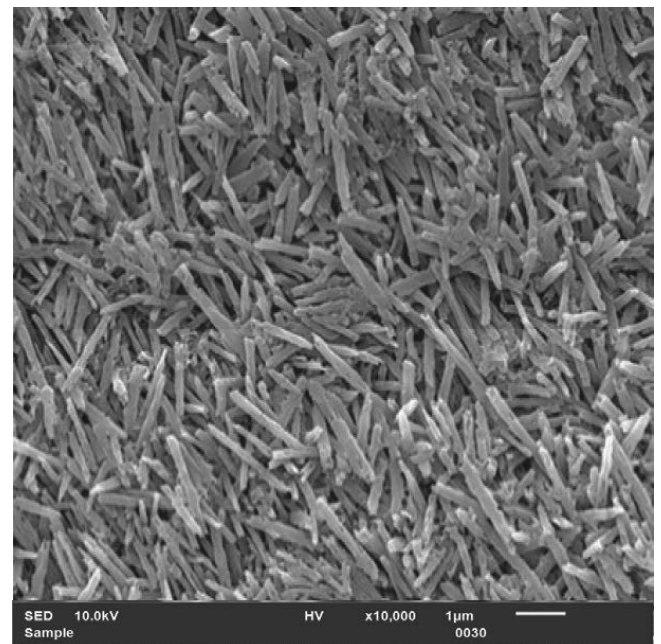


Fig. 1: Lithium disilicate ceramic surface with HF(S) surface conditioning. SEM image shows an open crystalline structure with a needle-like appearance. Small gaps are visible between the needle structure. All needles demonstrate agglomeration.

Group 2 (SECP)

SECP (Monobond Etch & Prime, Ivoclar Vivadent, Schaan, Liechtenstein) was applied actively on LDC discs for 20 secs. It was then left on the surface for 40 seconds followed by thorough washing and drying using a triple syringe²⁰ (Fig. 2).

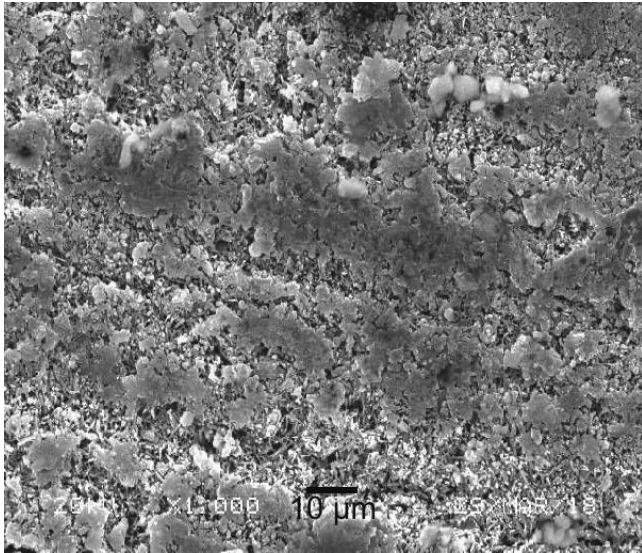


Fig. 2: Lithium disilicate ceramic surface treated with SECP. Loss of superficial surface with no crystal or needle-like appearance.

Group 3 (ECL)

For pretreatment of the LDC surface, an ECL (Millennium Biolase Technology Inc., San Clement, CA, USA) was employed with constant air/water irrigation. Power was set at 7 W, wavelength at 2.78 μm , and frequency at 25 Hz. Two-minute irradiation was performed using an MZ 6- μm tip (0.30 μm) in such a way that the tip was placed perpendicular to the surface being exposed while maintaining a distance of 1 mm (Fig. 3).

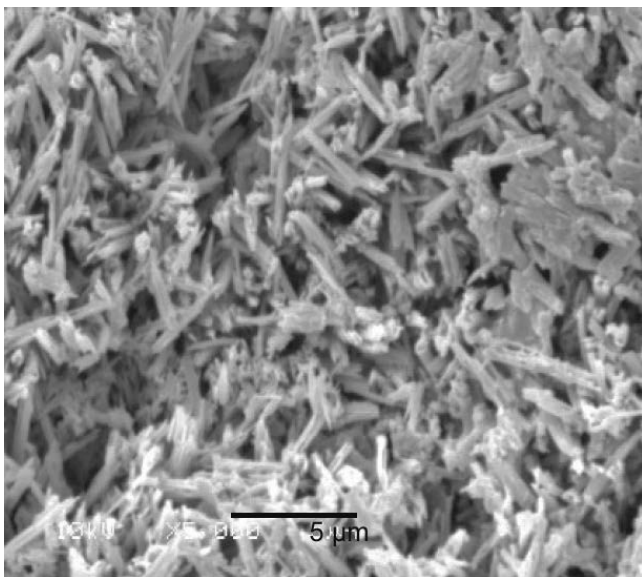


Fig. 3: Lithium disilicate ceramic after treatment with Er, Cr: YSGG laser. SEM image showing needle appearance with an area of lacunae. The needle-like structure shows the fusion of some crystals due to thermal irradiation.

(4) *Ra* analysis

A thorough examination of the *Ra* was conducted on 30 samples, 10 from each conditioning group using a contact profilometer (Mahr Surf M 300c, Mahr GmbH, Germany). Three parallel readings were acquired following the guidelines outlined in the ISO 4287:1997 standard,

which defines the *Ra* and *Rz* parameters. The cut-off value utilized in this study was determined to be 5. Additionally, the cutoff wavelength (λ_C) was established at 0.8 mm, while the sampling length (λ_S) was set to 2.5 μm . When measured from a mean plane, the absolute values of the five highest peaks and five lowest surface valleys are averaged out to provide *Ra*, whereas *Rz* is the average distance between them. The standard unit of measurement is micrometers (μm)²¹.

(5) *Surface topography analysis*

One specimen from each surface conditioning group underwent surface topography analysis. To accomplish this, the specimens were coated with a thin layer of gold using a gold-sputtering device. The discs were then subjected to analysis using a scanning electron microscope (SEM) (JSM 610LA; JEOL Ltd) at different magnifications²¹.

(6) *Bonding procedures*

Forty discs from each conditioning group were obtained and were further divided into two subcategories based on the application of high- (A) and low- (B) viscosity dual-curing resin cement (Variolink N, Ivoclar) $n = 20$. The catalyst of each viscosity cement was mixed in equal parts with the cement base and carefully applied on the surfaces of the ceramic discs using transparent Tygon tubes. The intaglio surface of the dentin discs was subjected to etching using a 35 % phosphoric acid solution (K-etchant syringe gel, Kuraray Noritake Dental Inc., Okayama, Japan) for 15 secs. Subsequently, the discs were rinsed and air-dried. The discs were then smeared with an adhesive (Excite F DSC, Ivoclar, Variolink system) for a precise period of 10 seconds following the guidelines provided by the manufacturer. The LDC discs were carefully attached to the substrate discs ensuring a consistent load of 2.5 N for 10 mins. The excess resin cement was then carefully removed, and the assemblies were cured under LED curing light (Radical LED curing light, SDI; Bayswater, Australia). The light was irradiated for a total of five exposures, with each exposure lasting 20 secs. The light was directed in five different positions: 0°, 90°, 180°, 270°, and on top⁵.

(7) *Storage regimens*

Each subgroup was further divided into two cohorts and were exposed to different storage conditions. $n = 10$

(a) *At baseline (no aging)*

The specimens in baseline condition did not undergo any aging process. They were stored in distilled water for a period of 7 to 14 days at a temperature of 37 °C.

(b) *Artificial aging*

The samples in this group were stored in distilled water at a temperature of 37 °C for 180 days. Additionally, the samples underwent 10 000 thermal cycles in a thermocycler (Nova Ética, São Paulo, Brazil). Each cycle consisted of 30-secs baths in a cold and hot bath maintained at a temperature of 5 °C and 55 °C, respectively, with a transfer time of 5 secs²⁰.

(8) SBS testing

The SBS of all specimens was evaluated using a universal test machine ((Kratos IKCL 3-USB, Kratos Equipamentos Industriais, Cotia, SP, Brazil) at a crosshead speed of 1 mm/min. The load was applied using a stainless-steel load applicator with a diameter of 5 mm. A 1 KN load cell was connected to the test device to measure the applied load. F is the force at which the specimen fails, measured in Newtons (N), and A represents the interfacial area of the cross-section, measured in square millimeters (mm²)⁵.

The equation $SBS=F/A$ calculates the SBS R in Megapascals (MPa)

(9) Fracture mode analysis

A stereomicroscope (Olympus, Shinjuku, Japan) at 40x magnification was used to identify the failure pattern, categorized into three distinct types: cohesive, adhesive, and admixed.

(10) Statistical analysis

The study was conducted using the IBM SPSS Statistics software program, version 20.0. Data normality was assessed using the Levenes Test. The means and standard deviation (SD) of the SBS and Ra were analyzed using a one-way ANOVA and the Tukey post hoc tests. A significance level of $\alpha = 0.05$ was set for all statistical tests.

III. Results

(1) Surface topography of LDC via SEM

Fig. 1: LDC with HF(S) Conditioning. SEM micrograph of the LDC surface after HF(S) conditioning reveals an open, crystalline structure characterized by needle-like formations. Inter-needle gaps were evident. The needles

exhibit agglomeration. Fig. 2: LDC surface treated with SECP. SEM micrograph of the LDC surface following SECP treatment shows a loss of the superficial layer, resulting in a surface devoid of the crystalline, needle-like structures observed in the HF(S) treated sample Fig. 3: LDC after treating with Er, Cr: YSGG laser. SEM micrograph of the LDC surface treated with Er, Cr: YSGG displayed a needle-like morphology, interspersed with lacunae. Evidence of crystal fusion, likely attributable to the thermal effects of the laser irradiation, is observed.

(2) Ra analysis

Table 1 shows the Ra scores of LDC discs after different conditioning regimes. The maximum Ra score was obtained in Group 1 specimens (HF acid-S) ($1033.23 \pm 1.3 \mu\text{m}$). Nevertheless, Group 2 samples (SECP) displayed the minimum value of Ra ($870.43 \pm 1.5 \mu\text{m}$). In an intergroup comparison analysis, it was discovered that Group 1 and Group 3 (ECL) ($993.12 \pm 2.1 \mu\text{m}$) treated discs presented comparable Ra outcomes. ($p > 0.05$) (Fig. 4).

Table 1: Ra after the use of different conditioning regimes on LDC.

Conditioned LDC groups	Mean \pm SD (μm)
Group A: HF(S)	1033.23 ± 1.3^a
Group B: SECP	870.43 ± 1.5^b
Group C: ECL	993.12 ± 2.1^a

Hydrofluoric acid (HF), Silane (S), Self-etch ceramic primer (SECP), Er, Cr: YSGG laser (ECL)

Different superscript alphabets denote statistically significant differences! (Post Hoc Tukey)

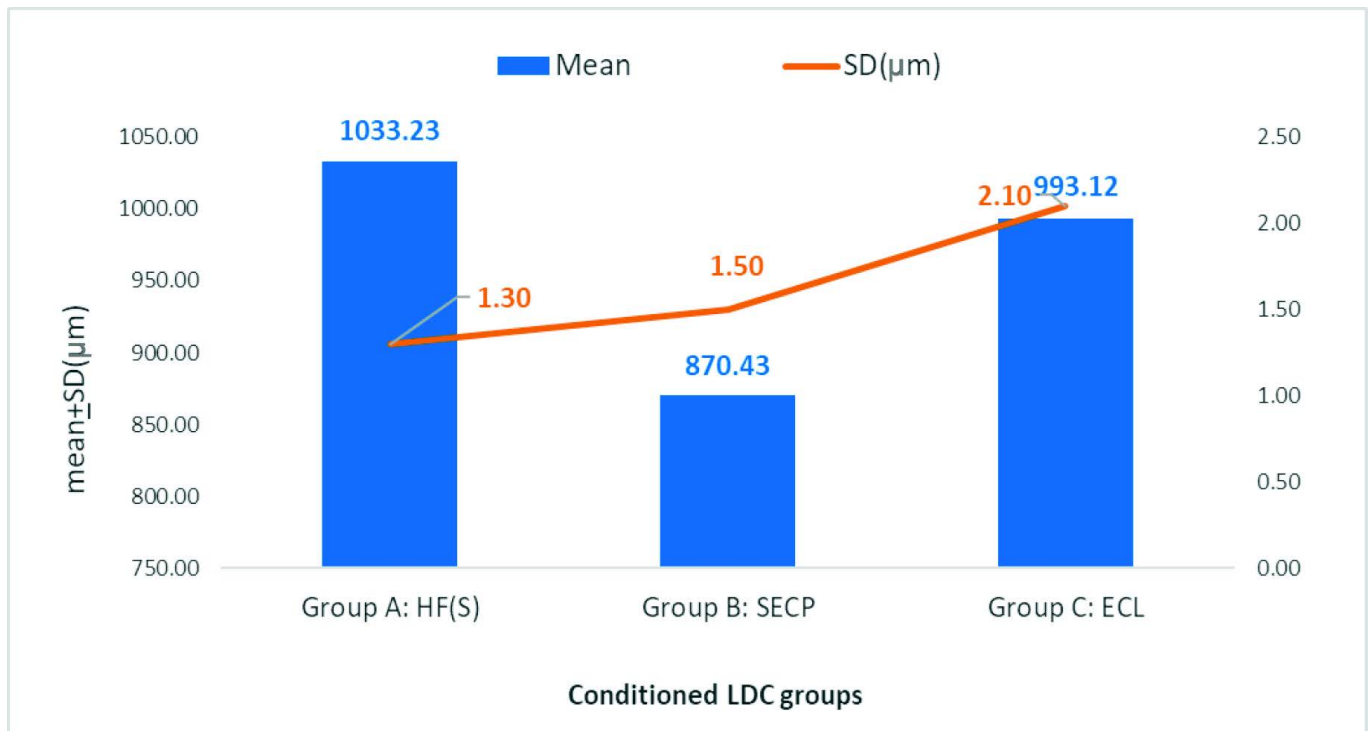


Fig. 4: Surface Ra after the use of different conditioning regimes on LDC.

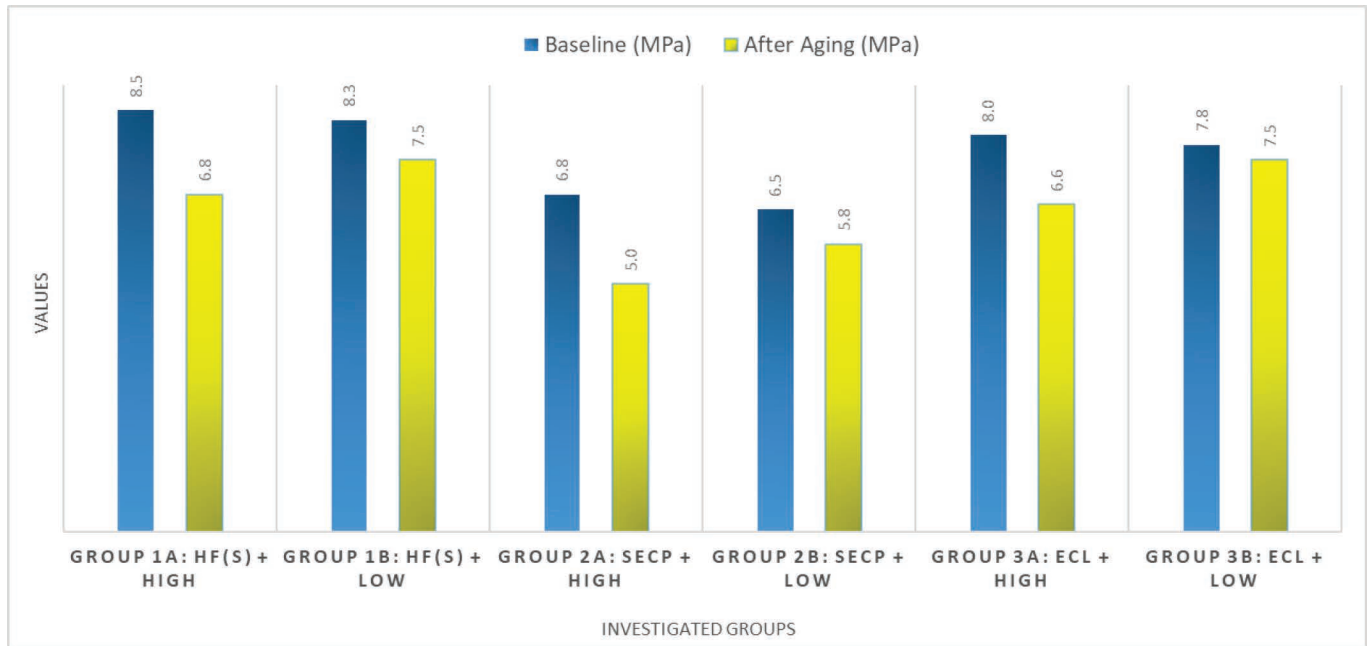


Fig. 5: SBS of LDC discs pretreated with different surface conditioners bonded with resin cement of different viscosities at baseline and after thermal aging.

(3) SBS analysis at baseline

Fig. 5 presents the SBS of LDC discs pretreated using different surface conditioners bonded with resin cement of different viscosities at baseline and after thermal aging. The highest SBS values were attained by Group 1A (HF(S) + HIGH) (8.5 ± 0.4 MPa) samples at baseline. However, the lowest SBS scores were presented by Group 2A (SECP + HIGH) (5.2 ± 0.1 MPa) samples after thermal aging. Intergroup comparison analysis at baseline unveiled that Group 1A, Group 1B (HF(S) + LOW) (8.3 ± 0.2 MPa), Group 3A (ECL + HIGH) (8.0 ± 0.2 MPa) and Group 3B (ECL + LOW) (7.8 ± 0.3 MPa) displayed comparable outcomes for bond integrity (*p* > 0.05). Likewise, Group 2B (SECP + LOW) (6.5 ± 0.3 MPa) and Group 2A (SECP + HIGH) (6.8 ± 0.4 MPa) revealed comparable outcomes for bond strength. (*p* > 0.05)

After aging, it was observed that the SBS decreased significantly for samples bonded using high-viscosity resin cement. A comparison of the different groups after artificial aging revealed that Group 1B (HF(S) + LOW) (7.5 ± 0.3 MPa) and Group 3B (ECL + LOW) (7.5 ± 0.2 MPa) samples demonstrated comparable outcomes for bond integrity. Similarly, Group 1A (HF(S) + HIGH) (6.8 ± 0.2 MPa) and Group 3A (ECL + HIGH) (6.6 ± 0.1 MPa) presented significantly lower scores than Group 1B and 3B. yet were comparable to each other. For Group 2A (SECP + HIGH) (5.0 ± 0.1 MPa) and Group 2B (SECP + LOW) (5.8 ± 0.2 MPa) significantly lower outcomes were established (*p* < 0.05).

(4) Failure mode analysis

It was identified from the outcomes of the present inquiry that at baseline storage conditions, HF acid- and ECL-treated discs bonded using high and low-viscosity cement mostly presented cohesive failure patterns. Whereas SECP-treated groups exhibited all three types of failures. However, after thermal aging, it was established

that groups bonded with low-viscosity cement and pretreated using ECL and HF acid predominantly displayed cohesive failure patterns while for the rest of the other groups all types of failure could be established (Fig. 6).

IV. Discussion

The present inquiry was based on the assumption that there will be no significant disparity in the *Ra* and bond integrity of different viscosity resin cement to bond LDC to dentin when SECP and ECL are used as surface pretreatment compared to the control. Furthermore, it was also anticipated that there would be no significant impact of artificial aging on the bond strength of LDC discs bonded to the dentinal substrate for both high and low-viscosity cement. The results of the present study led to the partial acceptance of the primary null hypothesis as the *Ra* score and SBS of the treated group were significantly lower for both high- and low-viscosity cement as compared to HF acid. Whereas the second stated supposition was partially rejected as the negative influence of artificial aging was only observed in high-viscosity resin cement.

The findings of the current research discovered that HF-acid- and ECL-treated LDC discs presented comparable scores for *Ra* and SBS when bonded with different viscosity cements. This aligns with the outcomes of previous research conducted by Tarek *et al.* ²² According to their assertion, HF acid works by dissolving the matrix of the LDC sample and exposes the rod-like morphology with the interlocking microstructure in the form of peaks and valleys ²². Similarly, they also described that ECL irradiation operates utilizing thermo-mechanical ablation, thus resulting in the generation of micro-roughness. The findings were supported by SEM analysis of the treated LDC surfaces with both conditioners ²². Another *in vitro* analysis affirmed the generation of hexafluorosilicates as a result of the chemical reaction between the HF acid and the glass matrix. During a chemical reaction, the

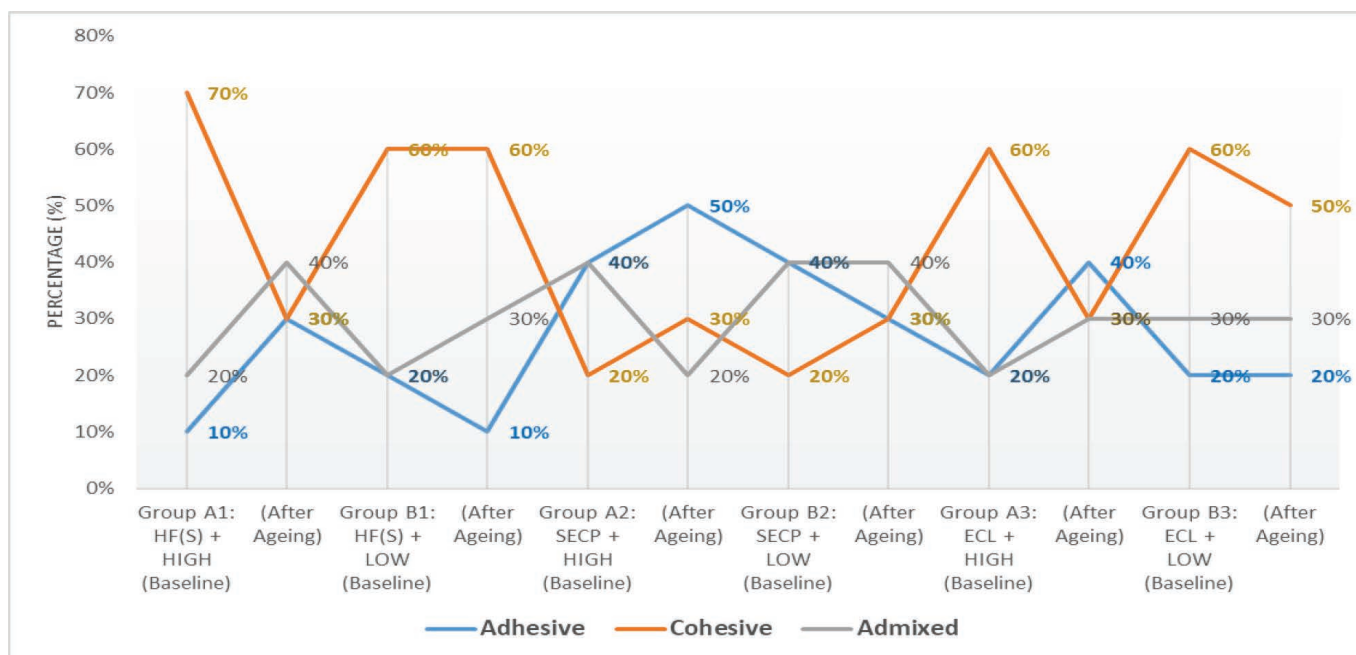


Fig. 6: Distribution of failure modes among the different studied groups.

surface texture of the LDC samples roughens, leading to an overall increase in surface energy^{23,24}. The improved mechanical behavior displayed by these groups can be elucidated on the basis of past explorations which have revealed that an increase in Ra directly increases the resin cement seeping within the microstructure, forming resin tags^{25,26}. SECP, on the other hand, exhibited lower Ra and bond integrity scores than the other investigated groups. This is in line with the outcomes of laboratory-based research conducted by Tribst *et al.*²⁷ and Lopes *et al.*²⁸. The authors of these lab-based investigations have established that SECP that consisted of ammonium polyfluoride and a trimethoxy-propyl methacrylate being a mild etchant alters the surface topography of LDC specimens less significantly thus reducing the SBS²⁹. However, data regarding the impact of SECP on the bond strength of indirect ceramics are controversial and require further investigation.

Concerning the influence of resin cement viscosity on the SBS of LDC discs at baseline storage conditions and after thermocycling, a noteworthy finding from the current investigation is that different viscosities of the resin cement did not have a significant influence on the bond strength of LDC discs at baseline without thermal aging. This is in concordance with the outcomes of research conducted by Barbon *et al.*¹⁶. Contrary to this, a study conducted by Dapieve and coauthors within the same field of research discovered variations in filling defects when resin cement with varying viscosities and surface treatments were employed. The most striking contribution to emerge from this research is the possibility of predicting survival probabilities of restoration from shear data after thermal aging³⁰. However, when samples were subjected to the aging thermal cycles, a deleterious effect was observed on the mechanical performance of high-viscosity resin cement. This outcome can be explicated by the fact that high-viscosity cement has a higher filler content

and less monomer which might get swelled or disintegrate after being exposed to the thermal cycle in distilled water, affecting SBS^{31,32}. However, there is only limited research available that has studied the influence of resin cement with different viscosity on the strength of the bond between LDC discs and dentin before and after artificial aging, hence further investigation is needed.

Despite the interesting results obtained in this study, it is crucial to highlight that this study possesses inherent limitations that could potentially restrict the generalizability of the data. The *in vitro* approach and use of a simplified geometry for specimens not using the anatomic designs are the major limitations as these would have impacted the outcomes. Another drawback is related to the non-standardization of the cement thickness as it influences the stress distribution and ultimately the bond strength. A single laser and specified parameters and ceramic composition restrict the extrapolation of the findings and necessitate further investigations.

V. Conclusions

The shear bond strength of lithium disilicate ceramics is strongly influenced by their microstructure obtained after surface conditioning. Long-term thermal aging causes a decrease in the mechanical performance of LDC discs bonded to dentin using high-viscosity resin cement.

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