

# Influence of High- and Low-Viscosity Resin Cement and Artificial Aging on Bond Integrity and Surface Roughness of Lithium Disilicate Ceramics Pretreated with Ho:YAG Laser and Nano-Hydroxyapatite Coatings

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## Abstract

The influence of a Ho:YAG laser and nano-hydroxyapatite (HAp) coating on the surface roughness ( $Ra$ ) and shear bond strength (SBS) of lithium disilicate ceramics (LDC) bonded to dentin with resin cement of varying viscosity following exposure to thermal aging has been studied. One hundred and fifty-three LDC discs were prepared and randomly allocated into three groups, based on their surface pretreatment. Group 1: 10 % HFA + S, Group 2: Ho:YAG laser, and Group 3: Nano-HAp coating.  $Ra$  was measured using a profilometer and the surface topography was assessed by means of scanning electron microscopy (SEM). Adhesion of prepared LDC discs to the dentinal substrate was performed using high-viscosity and low-viscosity resin cement and the discs were exposed to different storage conditions. Their SBS and failure were assessed with a UTM and stereomicroscope. One-way ANOVA and Tukey post-hoc tests were used for intergroup comparison. Group 2 (Ho:YAG laser) exhibited the highest  $Ra$  scores and Group 3 (HAp coatings) demonstrated the lowest  $Ra$  value. Group 2A (Ho:YAG laser + HIGH) samples achieved the highest SBS values at baseline. However, Group 3A (HAp coatings + HIGH) displayed the lowest bond score. A Ho:YAG laser and nano-HA can be used as an alternative surface conditioning regime with a positive influence on  $Ra$  and SBS of LDC.

*Keywords:* Lithium disilicate ceramics, Ho:YAG laser, nano-hydroxyapatite (HAp) coating, surface roughness, shear bond strength, scanning electron microscopy

## I. Introduction

Lithium disilicate glass-ceramic (LDC) is recognized for its versatility and is highly recommended for various clinical applications, addressing both occlusal stress and aesthetic requirements<sup>1</sup>. Nonetheless, the longevity of ceramic restorations in clinical settings is influenced by various factors, i.e. surface pretreatment, luting resin, and masticatory loading<sup>1,2</sup>. Various ceramic surface treatments have been developed to enhance the bonding of resin cement to LDC surfaces<sup>2</sup>. Traditionally, the bond formed between resin cement and LDC is based on two main mechanisms: micro-mechanical attachment enabled by hydrofluoric acid (HF) etching and chemical bonding accomplished through the use of a silane coupling agent (S)<sup>3</sup>. However, HF acid exhibits caustic properties that pose risks for human use, prompting the initiation of research to establish more suitable alternatives<sup>4</sup>.

Lasering, a non-invasive technique, is increasingly being utilized in a variety of dental procedures<sup>5,6</sup>. Among various lasers used, the Holmium:Yttrium-Aluminum-Garnet (Ho: YAG) laser that operates at a wavelength

of 2.12  $\mu$  has gained widespread acceptance and has been used safely and effectively for the photo conditioning of dentin and different ceramics in clinical settings<sup>7,8</sup>. Alkhudhairy and colleagues, in their *in vitro* analysis, revealed that Ho:YAG-laser-pretreated hybrid ceramics discs exhibited superior surface roughness ( $Ra$ ) and shear bond strength (SBS) compared to other tested groups<sup>9</sup>. Apart from lasers, scientists have tried to incorporate nanotechnology in the field of prosthodontics<sup>10</sup>. Nano-hydroxyapatite (HAp) with a crystal size between 50 and 1 000 nm has become a topic of interest for various dental researchers owing to its morphological and architectural similarities to tooth enamel apatite crystals<sup>11</sup>. Evidence suggests that when nano-HAp particles were used on ceramic surfaces, a uniform and thin coating layer was produced<sup>9,11</sup>. This coating contributes to the enhancement of the SBS of the resin-luting cement used<sup>12</sup>. Currently, there is a scarcity of literature specifically examining the effects of nano-HA on the  $Ra$  and SBS of LDC and hence further inquiry is needed.

In addition to surface pretreatment, a significant aspect influencing the adhesion of indirect restorations to dentin is the selection of luting cement<sup>13</sup>. The effective-

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ness of the luting agent in permeating surface defects created post-surface conditioning is crucial for establishing a seal around the microcracks, which eventually enhances the restorative material bond strength<sup>14</sup>. Nonetheless, limited data exists concerning the influence of different-viscosity resin cement, either high or low, on the integrity of the bond of LDC to the dentinal substrate, so further investigation is required. Another factor that should be considered when assessing bond strength is that immediate SBS without thermal aging does not reflect the true clinical conditions as multiple factors are inflicted when the restoration is placed in the mouth<sup>15,16</sup>. This could result in restoration fatigue, which may influence the physical and mechanical properties<sup>15</sup>.

The current investigation aims to examine the influence of resin cement of varying viscosity on the bond integrity of LDC discs that have been pretreated using distinct conditioning methods. The investigation further evaluates the longevity of the restoration by exposing it to thermal aging processes. It was projected that there would be no significant difference in the *Ra* and bond strength of various-viscosity resin cements used to bond LDC discs to dentin when pretreated with a Ho:YAG laser and nano-HAp coatings as a surface conditioning regimen when compared to the HFA (control). Furthermore, it was also anticipated that the SBS of LDC discs bonded to the dentinal substrate would remain unaffected by artificial aging, irrespective of the viscosity of the cement retained.

## II. Materials and Methods

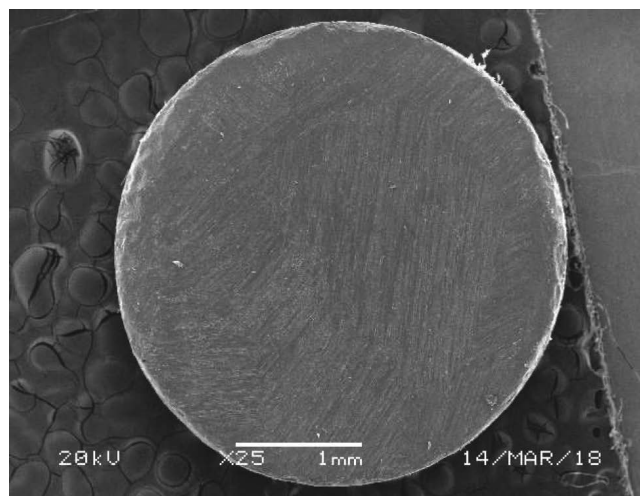
### 1. Dentinal substrate preparation

A total of one hundred and fifty human central incisors were cleaned and preserved in a 0.5% Chloramine-T solution (Explicit Chemicals Pvt Ltd, Pune, India) for one week before preparation of dentin discs. The radicular portion was meticulously sectioned to the level of the cemento-enamel junction (CEJ) using a diamond saw (Pico155, pace Technologies, Tucson, AZ, US). The buccal surface of the crowns was drilled (SBE 1010 Plus, Metabo) resulting in the formation of 2-mm cylindrical discs. The specimens were polished using P500 silicon carbide paper (SiC, Struers, Copenhagen, Denmark) sequentially on a rotational polishing apparatus while under running water<sup>17</sup>.

### 2. LDC disc preparation

Lithium disilicate ceramics (LDC) IPS e-max pressing ingots (Ivoclar Vivadent, Schaan Liechtenstein) were used in the present study. The ingots were meticulously shaped into a cylindrical form utilizing a slow-speed diamond saw in conjunction with water cooling (IsoMet™ 5000 Buehler, IL, USA). The cylindrical rod was meticulously sectioned transversely to create 153 discs, each having a thickness of 2 mm. The measurement was performed utilizing a digital caliper Mitutoyo Digimatic caliper, Michigan, USA). Furthermore, to achieve consistency in the substrate surface, all samples were polished using 600-grit silicon carbide paper (SiC, Struers, Copenhagen, Denmark). The specimens underwent a thorough cleaning process employing ultrasonic tech-

nology (Durasonix 3.2 L Ultrasonic Cleaner, China) for 4 mins<sup>18</sup>. **Fig. 1** The discs were then randomly allocated into three distinct groups, based on the surface pretreatment they received. N = 51



**Fig. 1:** Lithium disilicate ceramics.

**Group 1 (HFA+ S)** The conditioning of LDC discs was performed using 10% HFA (Bisco, Pennsylvania, USA) for 1 min, followed by application of a silane coupling agent (S) (Bisco, Pennsylvania, USA) with the help of a micro brush for 1 min. The residual S was removed with the help of compressed air for 5 seconds<sup>19</sup>.

**Group 2 (Ho:YAG laser)** A Ho:YAG laser (StoneLight Holmium Laser System, AMS Inc., Minnetonka, MN, USA) was used to condition the LDC discs in this group along with water coolant for 20 secs. The laser was applied in a pulse mode with laser parameters, i.e. 8 Hz frequency, 6 W power output, and a 2090 nm wavelength<sup>8</sup>.

### Group 3 (Nano-HAp coating)

The HA thermal coating protocol was implemented for LDC pretreatment within this group. A slurry was prepared by integrating 10 g of nano-HA powder (Merck, Germany) into 50 cc of distilled water. 1 g polyvinyl alcohol (Merck, Berlin, Germany) was used as the binder for the suspension. The solution was then heated on a magnetic stirrer at a temperature of 100 °C and a rotational speed of 1000 rpm for 1 min to ensure a homogeneous suspension. The discs were then submerged in the slurry at a 45° angle for 5 seconds<sup>20,21</sup>.

### 3. Ra analysis

*Ra* scores of ten pretreated discs from each group were measured using a profilometer (Wyko, Model NT 1100, Veeco, Tucson, USA). The needle was systematically moved over the geometric center of the specimen, along with two additional points, to precisely record the measurements. The system was re-calibrated before testing. The average was obtained from three readings of each specimen<sup>22</sup>.

### 4. Surface topography analysis

One sample from each group was sputter-coated using a gold-palladium alloy (Bal-Tec SCD 050 Sputter Coater, Bal-Tec AG, Balzers, Liechtenstein). The samples were

then subsequently analyzed under a scanning electron microscope (SEM) (Model MIRA 3, Kohoutovice, Czech Republic) at 15 KV to assess the surface alterations post-treatment at different magnification<sup>23,24</sup>.

### 5. Adhesion of prepared LDC discs to the dentinal substrate

Each conditioning group provided forty discs, which were subdivided into two categories based on the viscosity, i.e. high viscosity (A) and low viscosity (B) of RelyX U200 self-adhesive resin cement (3M ESPE, St Paul, MN, USA) used to bond LDC discs to the dentinal discs  $n = 20$ . Following the removal of excess cement, the specimens were subjected to light polymerization along their margins using LED curing light (Valo, 1 400 mW/cm<sup>2</sup>, Ultradent, Salt Lake City, USA) for 20 secs on each side. All samples were then immersed in distilled water at 37 °C for 24 h<sup>17</sup>.

### 6. Storage regimens

From each subgroup, two cohorts were subsequently generated and kept under separate storage conditions  $n = 10$

#### Storage without thermal aging

In this group, the bonded discs were immersed in distilled water for two weeks at a controlled temperature of 37 °C to ensure that the samples remained at baseline condition.

#### Storage with thermal aging

After cementation, the discs in this group were subjected to an artificial thermal aging process consisting of 10 000 cycles, utilizing a thermocycler (Instron model 4467 tabletop load frame, 825 University Ave, Norwood, MA, USA). The temperature range of the two water baths was maintained between 5 and 55 °C. The duration of immersion in each water bath was 30 secs, with a transfer time of 10 secs<sup>25,26</sup>.

### 7. SBS analysis

The shear bond testing was performed following the ISO/TS 11405:2015 specification. The LDC dentin interface was subject to shear stress in a universal testing machine (UTM (DL-2000, EMIC). A 200- $\mu$ m chisel-shaped head applied load at a crosshead speed of 1 mm/min. The maximum force required to fracture the interface was documented in MegaPascal (MPa)<sup>27</sup>

### 8. Failure mode analysis

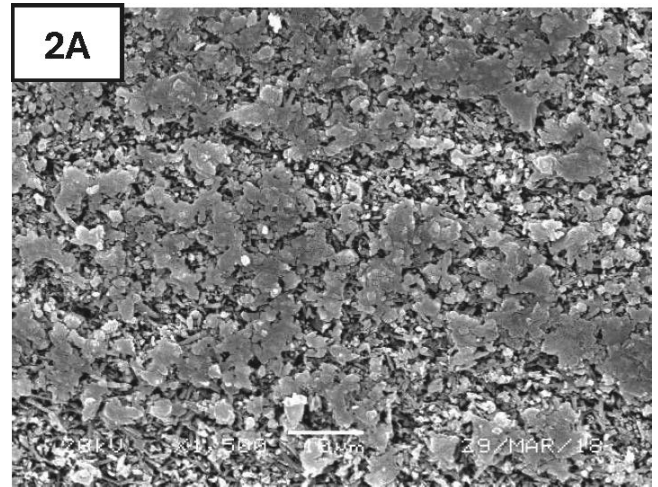
The failure mode was assessed by examining the debonded discs with the help of a stereomicroscope (Wild M3B, Heerbrugg, Switzerland) at a magnification of  $\times 12$ . The failures were categorized into three distinct types: adhesive, cohesive, and admixed.

### 9. Statistical analysis

The SPSS version 22.0 (IBM Corporation, located in Armonk, NY, USA) was used to statistically analyze the SBS and  $Ra$  data. The normality of data was assessed using the Levenes test. One-way ANOVA and Tukey HSD post-hoc test were used for intergroup comparison among tested groups. The statistical significance threshold was set at 0.05.

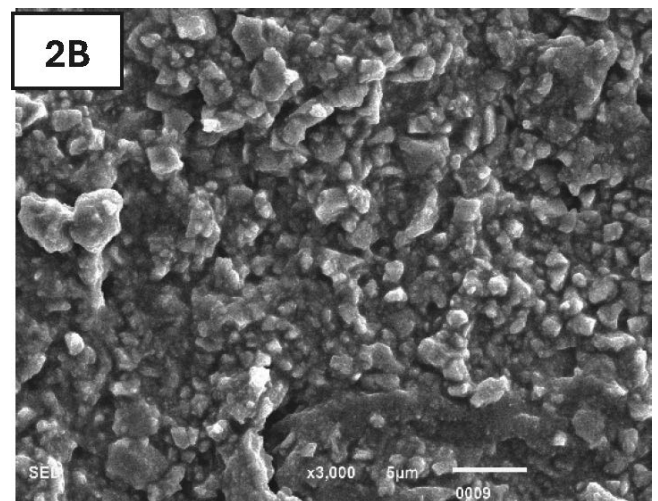
## III. Results

### 1. Surface topography of LDC following surface modification



**Fig. 2 A** Surface modified with HFA+S. The SEM image shows loss of matrix with the needle-like appearance of lithium disilicate crystals.

The surface modification shown in **Fig. 2A** was achieved using HFA+S. The SEM image illustrates a loss of matrix characterized by the needle-like appearance of the lithium disilicate crystals. **Fig. 2B** presents a scanning electron microscope image of the LDC surface modified using a Ho:YAG laser. The appearance of lithium disilicate crystals is prominent, accompanied by the fusion and melting of crystals due to heat. The crystal particles exhibit variability in size. **Fig. 2C** presents the scanning electron microscopy (SEM) image of the LDC disc that has been treated with hydroxyapatite (HAp) coatings. The HAp coating has enhanced the  $Ra$ ; however, the matrix remains undissolved and lacks a prominent crystalline structure.



**Fig. 2 B:** SEM image showing a LDC surface modified with a Ho:YAG laser. The lithium disilicate crystal appearance is prominent along with the fusion and melting of crystals due to heat. The crystal particles are of variable sizes.



2. Ra evaluation

The Ra scores of LDC discs following conditioning with various pretreating agents are detailed in Table 1. The specimens categorized under Group 2 (Ho:YAG laser) exhibited the highest Ra scores ( $1160.13 \pm 1.1 \mu\text{m}$ ). In contrast, the LDC discs in Group 3 (HAp coatings) demonstrated the lowest Ra value ( $1090.32 \pm 1.2 \mu\text{m}$ ). The results from the intergroup comparison analysis indicated that Group 1- (HFA) ( $1152.12 \pm 1.6 \mu\text{m}$ ) and Group 2- and Group 3-pretreated discs demonstrated comparable Ra outcomes ( $p > 0.05$ )

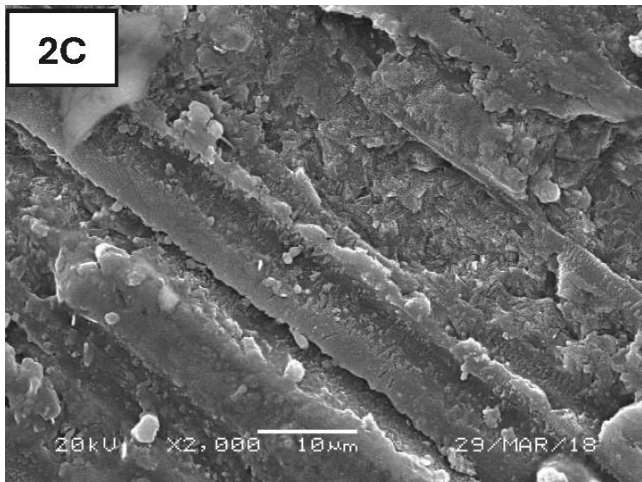


Fig. 2 C: SEM of the LDC disc treated with HAp coatings. The HAp coating has improved the Ra but the matrix is undissolved and there is no prominent crystalline structure.

Table 1: Surface Ra of LDC restoration after pretreatment with different surface conditioners.

Investigated groups	Mean ± SD (µm)
Group 1: HFA (S)	1152.12 ± 1.6 <sup>a</sup>
Group 2: Ho: YAG laser	1160.13 ± 1.1 <sup>a</sup>
Group 3: HAp coatings	1090.32 ± 1.2 <sup>a</sup>

Hydrofluoric acid (HFA), Silane (S), Holmium:Yttrium-Aluminum-Garnet (Ho: YAG) laser, Nano-hydroxyapatite (HAp) coatings

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

3. SBS estimation at baseline and aging

The bond strength of LDC discs subjected to various surface conditioning treatments and bonded to dentin with luting cement of different viscosities, measured at baseline and following thermal aging, are presented in Fig. 3. Group 2A (Ho: YAG laser + HIGH) ( $8.6 \pm 0.2$  MPa) samples achieved the maximum SBS at baseline storage conditions. However, Group 3A (HAp coatings + HIGH) ( $6.9 \pm 0.5$  MPa) demonstrated the minimum bond score. Comparison among tested groups revealed that Group 1A (HFA (S) + HIGH) ( $8.5 \pm 0.3$  MPa), Group 1B (HFA (S) + LOW) ( $8.4 \pm 0.2$  MPa), 2A, 2B (Ho:YAG laser + LOW) ( $8.4 \pm 0.1$  MPa), Group 3A (HAp coatings + HIGH) ( $8.3 \pm 0.2$  MPa) and Group 3B (HAp coatings + LOW) ( $8.2 \pm 0.1$  MPa) displayed comparable scores for SBS ( $p > 0.05$ )

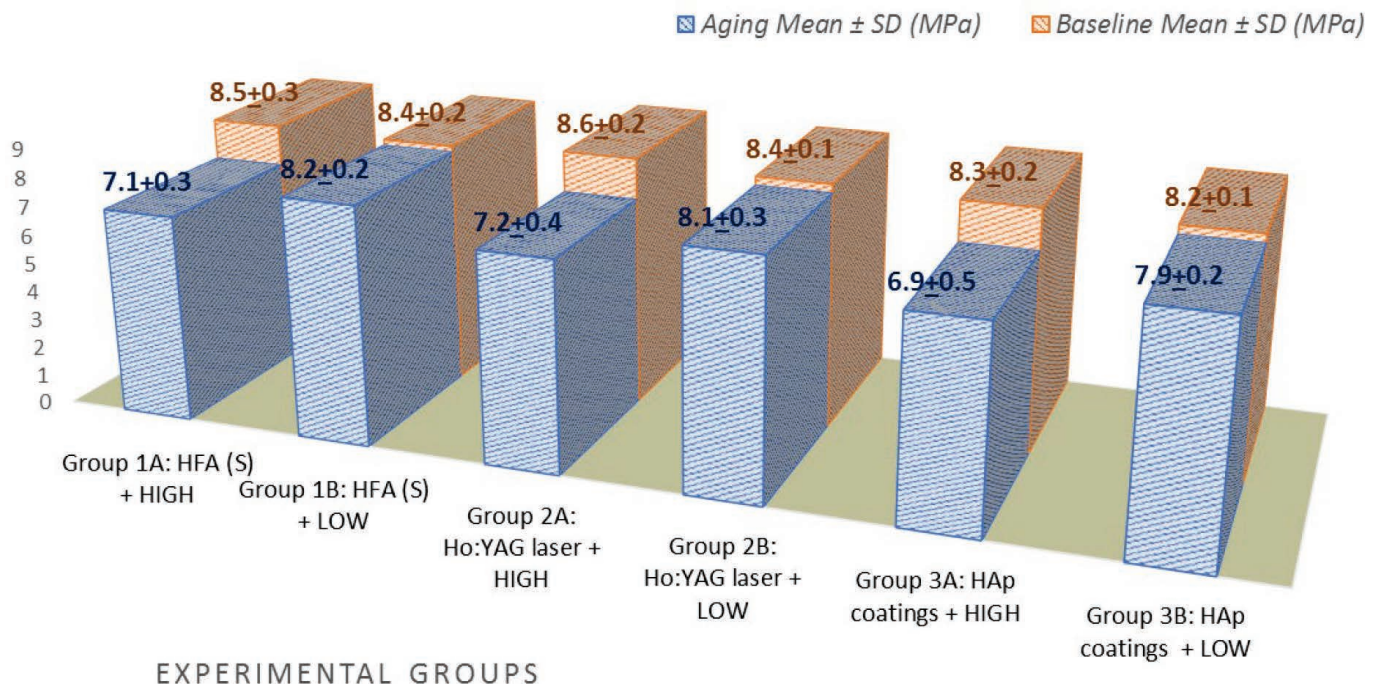


Fig. 3: SBS of LDC discs pretreated with different surface conditioners and bonded with dentin using resin luting cement of varying viscosities at baseline and after thermal aging.

4. SBS estimation after thermocycling

After thermocycling, it was identified that Group 1A (HF(S) + HIGH) ( $6.8 \pm 0.2$  MPa and Group 2A (HF(S) + LOW) ( $7.2 \pm 0.4$  MPa) and 3A ( $6.9 \pm 0.5$  MPa) presented comparable scores for bond strength. ( $p < 0.05$ ). However, Group 1B (HFA (S) + LOW) ( $8.0 \pm 0.2$  MPa), Group 2B (Ho:YAG laser + LOW) ( $8.1 \pm 0.3$  MPa) and Group 3B (HAp coatings + LOW) ( $7.9 \pm 0.2$  MPa) pretreated samples exhibited comparable outcomes for bond integrity ( $p > 0.05$ )

5. Failure mode analysis

The admixed failure was prominent in all the groups at baseline conditions. Whereas, after thermal aging a combination of adhesive and admixed failures was most commonly observed with high-viscosity cement and admixed in low-viscosity resin cement (Fig. 4).

IV. Discussion

The present exploration was based on the supposition that there would be no significant difference in the Ra and bond strength of resin cements of varying viscosity used to bond LDC discs to dentin when pretreated with a Ho:YAG laser and HAp coatings as a surface conditioning regimen when compared to the HFA. Furthermore, it was also anticipated that the SBS of LDC discs to the dentinal substrate would remain unaffected by artificial aging, irrespective of the viscosity of the cement employed. The results of the existing investigation demonstrated that the Ra score and SBS of the Ho:YAG-laser-

and Hap-coatings-pretreated discs were notably comparable to the control when bonded with both high- and low-viscosity resin cement, thereby entirely refuting the primary null hypothesis. In addition, it must be noted that the second hypothesis was only partially rejected as the groups that used high-viscosity resin cement to attach LDC discs to the dentin were the only ones to experience the negative consequences of artificial aging.

Before the application of resin cement, specific surface conditioning procedures may significantly influence the bond strength by providing a rough substrate for micromechanical interlocking<sup>28,29</sup>. The Ho:YAG-laser-treated samples displayed the highest scores for Ra and bond integrity. This outcome aligns with the findings of laboratory-based analysis performed by Albakri and colleagues<sup>8</sup>. Albakri explained that the Ho: YAG laser demonstrates characteristics akin to both CO<sub>2</sub> and Nd:YAG lasers<sup>8</sup>. Past research has revealed that, when used as a ceramic surface conditioner, a Nd:YAG laser increases Ra via the creation of surface cracks<sup>30</sup>, Furthermore, the application of CO<sub>2</sub> laser irradiation also plays a significant role in enhancing the LDC Ra by facilitating thermomechanical melting. The phenomenon of melting induces an expansion of the surface area of LDC, while the process of solidification is characterized by a swift contraction<sup>31</sup>. This leads to surface crack formation as a result of stress generation induced by sudden alterations in the material's physical conditions<sup>31</sup>. The presence of these fissures contributes to an enhancement in the SBS of the resin cement based on mechanical interlocking.

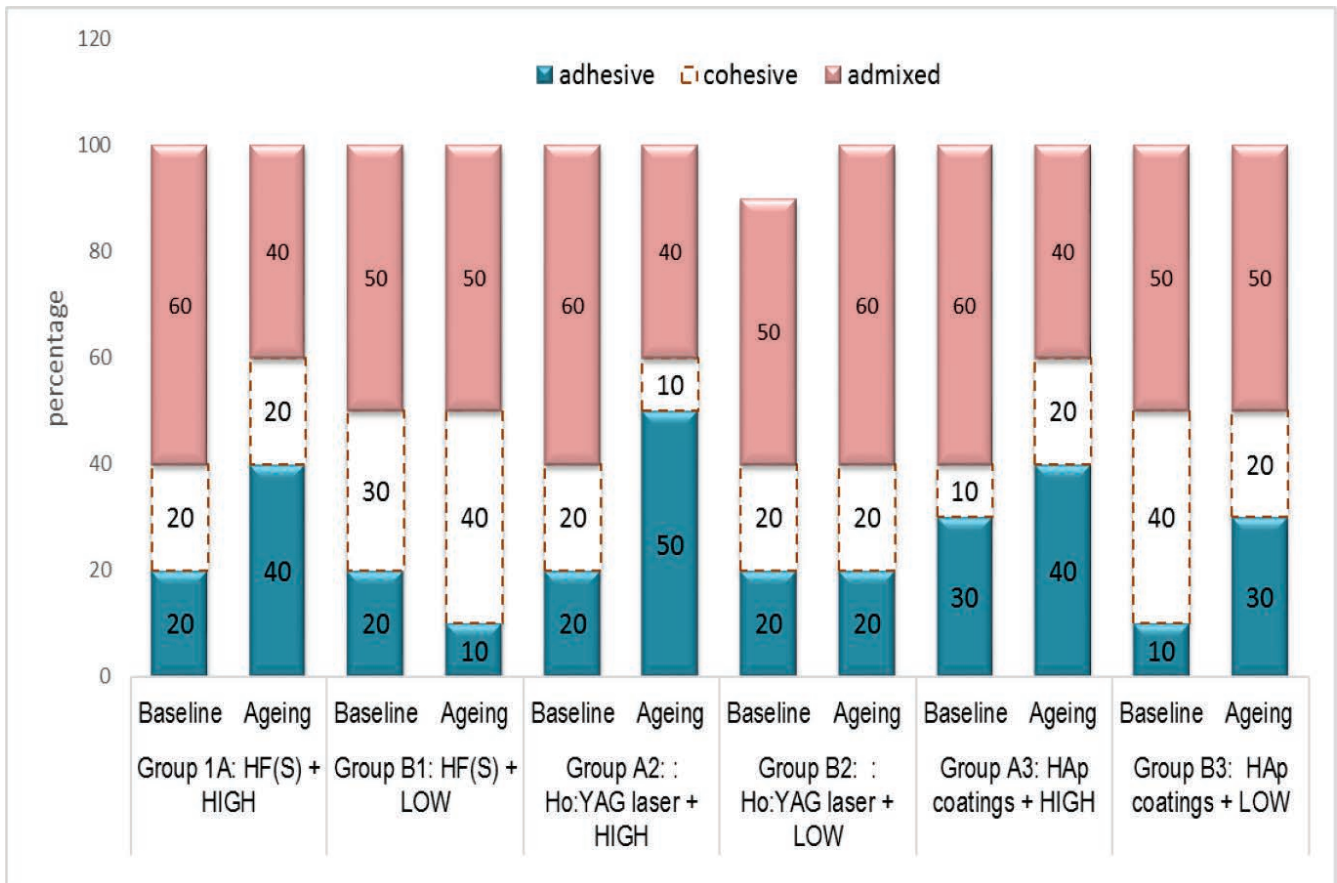


Fig. 4: Distribution of failure modes among different study groups.

Additionally, among all tested groups nano-HA coating displayed the lowest scores for  $Ra$  and SBS, however, this difference was not statistically significant and comparable to the control. The existing research utilized the thermal coating method, which offers superior mechanical properties and practical applicability. HAp coating has been used for a very long time in implant dentistry to increase the surrounding cell attachment to the implant surface<sup>32–34</sup>. The satisfactory  $Ra$  outcomes when LDC discs are modified with HAp coating are consistent with the findings from a laboratory-based analysis conducted by Jung and coworkers<sup>35</sup>. Furthermore, in their SEM analysis, Sagsoz and colleagues revealed that micro-retentive areas had been created on ceramic surfaces which may have significantly contributed to enhancing the mechanical bond strength<sup>36</sup>. The positive impact of HFA on LDC  $Ra$  and SBS with luting cement cannot be denied as various authors have indicated that surface treatments with HFA should be conducted when a ceramic surface has a glassy matrix including silica or silicates since silicon tetrafluoride is formed initially<sup>36</sup>. This silicon tetrafluoride reacts with HFA to produce hexafluorosilicates, which interact with hydrogen protons to generate tetra-fluorosilicate acid, a compound that can be eliminated by water. This reaction results in micro retentions on the ceramic surface that allow for the penetration of cement agents<sup>37</sup>.

The outcomes for bond strength are affected by various factors during thermal aging, such as temperature, dwell time, and total cycles<sup>38</sup>. Thermal aging experiments indicated that thermocycling negatively impacts the SBS of high-viscosity resin cement in all conditioning groups, in contrast to low-viscosity resin cement. This phenomenon can be attributed to the elevated filler content and reduced monomer concentration in these cements, leading to the swelling and subsequent disintegration of the bond as a result of repeated abrupt temperature fluctuations<sup>39</sup>. Nonetheless, the existing literature is scarce regarding the impact of artificial aging on the SBS of luting cement of different viscosity bonded to LDC discs, indicating a clear need for additional research in this area.

While the findings from existing analysis are noteworthy, it is necessary to acknowledge the integral limitations that may affect the generalizability of the data. The limitations encompass the dependence on an *ex vivo* methodology and the application of a simplified geometric representation for specimens, which diverges from anatomical designs. Another drawback relates to the absence of standardization in cement thickness, which influences the stress distribution and, consequently bond strength. Different laser parameters must be used to assess the impact of the Ho:YAG laser.

## V. Conclusions

A Ho:YAG laser and nano-HA can be used as an alternative surface conditioning regime with a positive influence on mechanical SBS and  $Ra$  outcomes. Furthermore, high-viscosity luting cement is more affected after thermal aging.

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