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Surface Roughness and Shear Bond Strength of Y-TZP Treated via Tri-Biochemical Silica Coating, Femtosecond Laser, and Nano-Hydroxyapatite: An SEM assessment

F. Alkhudhairy*, N.N. Shono

Restorative Dental Sciences Department, College of Dentistry, King Saud University, Riyadh, Saudi Arabia.
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Abstract

AIMS: Efficacy of the recent surface pretreating techniques Tribochemical silica coating (TBC), Femtosecond laser (FS), and Nano-hydroxyapatite (HA) coating in influencing the surface roughness (*Ra*) and shear bond strength (SBS) of yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) bonded to resin luting cement. MATERIALS AND METHODS Eighty-four Y-TZP zirconia discs were prepared followed by grouping based on the conditioning agent (n= 16) Group 1 (APA) (Control), Group 2 (TBC), Group 3 (FS laser), and Group 4 (Nano-HA coating). Surface roughness evaluation was performed on five discs from each group using a profilometer and surface topography was assessed using SEM analysis. Bonding of luting cement on ten specimens in each group was followed by SBS and fracture mode analysis using a universal testing machine and stereomicroscope. One-way ANOVA and post hoc Tukey compared the means of *Ra* and SBS among different tested groups ($p < 0.05$) RESULTS The maximum roughness (1421.26 ± 0.062 μm) and SBS (18.34 ± 0.16 MPa) were exhibited by Group 4 (Nano-HA coating). However, Group 2 (TBC) presented the lowest *Ra* (1 000.57 ± 0.043 μm) and bond strength (14.54 ± 0.09 MPa) CONCLUSION Nano-hydroxyapatite coating and Femtosecond laser can be used as a suitable alternative to air particle abrasion without affecting the physical and mechanical properties of zirconia ceramic.

Keywords: Tribochemical silica coating, femtosecond laser, nano-hydroxyapatite coating, air-particle abrasion

I. Introduction

Yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) as an indirect restoration exhibit exceptional mechanical and aesthetic properties. The transformation toughening characteristic of zirconia stands out from that of other dental ceramics used. However, attaining a strong bond with the resin-luting cement is still challenging [1]. Considerable research has been conducted over the last few years to modify the surface chemistry of zirconia to enhance its shear bond strength (SBS) with the adhesive cement [2]. Air-particle abrasions (APA), also referred to as sandblasting, is considered a gold-standard technique to improve the surface roughness (*Ra*) of zirconia and SBS between luting cement and zirconia [3]. However, it is essential to recognize grit blasting results in a transformation from the tetragonal phase to a monoclinic structure. The alteration may lead to a reduction in the strength of Y-TZP, thereby enhancing its vulnerability to fracture [4]. Therefore, it is necessary to find an alternative surface pretreating method that improves the *Ra* and SBS without affecting the fracture resistance and other mechanical characteristics of the zirconia ceramics.

Tribochemical silica coating (TBC) is a modified air blasting technique used to deposit silica on the surface of zirconia. It utilizes aluminum oxide (Al_2O_3) particles modified with silica as a blasting agent, which not only eliminates surface contaminants but also increases *Ra* [5]. The increase in *Ra* helps with micromechanical retention whereas silica helps with chemical bonding [6]. According to the available indexed literature, the outcomes are dubious regarding its impact on the *Ra* and the integrity of the bond between the resin cement and the zirconia and hence need future investigation [7].

Lasers have been used in the field of dentistry to modify the surface of teeth and different dental materials to improve their mechanical properties [8]. Recently, the Femtosecond laser (FS) has gained significant attention and has been used to micromachine the surface of various materials, i.e. zirconia ceramics without causing any thermal damage [9]. This new laser is capable of ablating materials in thin layers while preserving the material properties [10]. Currently, there is a lack of comprehensive information regarding the feasibility of utilizing this method for zirconia conditioning in dental reconstruction.

Another technique that has gained acceptance in the field of prosthodontics is nanotechnology. Considerable progress in the field has shown the potential uses for nano-hydroxyapatite (HA) as a ceramic surface modifier with crystal sizes ranging from 50 to 1 000 nm [11]. It has been reported in previous studies that nano-HA produces a thin and consistent coating layer on the treated ceramics,

* Corresponding author: falkhudhairy@ksu.edu.sa

which then possess adequate strength. Atri and coauthors, in their lab-based analysis, used nano-HA for zirconia surface modification and reported that it resulted in a comparable SBS to that obtained with APA [12]. However, studies are still scarce and further investigation is required.

The current analysis was founded on the hypothesis that when contemporary conditioning techniques (TBC, FS laser, and nano-HA coating) are used, the *Ra* of Y-TZP will be comparable to that obtained with APA. Furthermore, it is also anticipated that SBS outcomes of zirconia modified using the contemporary regimes will be the same as that of the control specimen. Thus, the existing investigation is aimed at assessing the efficacy of surface pretreating agents in influencing the *Ra* of Y-TZP and the strength of the bond between Y-TZP and resin-luting cement.

II. Materials and Methods

Eighty-four Y-TZP zirconia discs (IPS e.max ZirCAD) having dimensional parameters of 4 × 4 × 4 mm were prepared by means of cutting with a slow-speed saw (Isomet Buehler, Lake Bluff, IL, USA). The formed discs were placed in an ultrasonic cleaner (Cristofoli Ultrasonic Cleaner, 2008, Campo Mourão, PR, Brazil) filled with ethanol (96 %) for 10 mins and air-dried. Grouping was performed based on the conditioning technique applied (n = 16 each group)

Group 1 (APA) (Control) APA was performed using Al_2O_3 particles (50 µm)(Mega OX, Megablast, Brazil) and sandblasting apparatus (Talleres Mestraitua, Spain), keeping the blasting pressure at 2.5 bar. A distance of 10 mm from the discs was maintained and blasting was performed for 15 secs. The discs were cleaned with an ultrasonic cleaner as described previously.

Group 2 (TBC) The zirconia discs in this group were pretreated with silica-modified aluminum oxide (Rocatec Soft sand, 3M) at a pressure of 2 bars for 15 seconds, with the tip being kept 5 cm away from the surface. The discs were cleaned with an ultrasonic cleaner as described earlier.

Group 3 (FS laser) Y-TZP discs underwent pretreatment with a FS laser (Coherent Quantronix-Integra-C system (Hamden, CT, USA). The laser parameters were set to a wavelength of 800 nm, with an output power of 200 mW for 12 mins at a repetition rate of 1 kHz. The laser beam with a diameter of 6 mm was directed onto the sample surfaces through a 75-mm plano-convex lens [13].

Group 4 (Nano-HA coating) A slurry was prepared by dissolving 10 g of HANPs powder (Merck, Germany) with particle size less than 100 nm in 50 cc of distilled water. Subsequently, 1 g of polyvinyl alcohol (Merck, Germany) was incorporated as a suspension binder. This mixture was then subjected to heating on a magnetic stirrer at 1 000 rpm and 100 °C for 1 min to ensure a uniform solution. The zirconia discs were then positioned in the slurry at a 45° angle for 5 seconds.

(1) Surface Roughness evaluation

Ra assessment was performed with a profilometer (Contour GT, Bruker, CA, USA). The center 2 mm area of each disc is specified for running a profilometer and taking three readings. The mean *Ra* for the specific sample was established by computing the mean of the three readings.

(2) Bonding of luting cement

The adhesive primer (Clearfil Ceramic Primer Plus) was applied using an applicator on 44 samples. A Teflon mold having dimensions of 2 x 2 mm was placed on the disc surface perpendicular to its horizontal axis. The mold was filled with resin-based cement (Panavia V5) (Kuraray Noritake Dental, Tainai, Niigata, Japan), which had been prepared by mixing its two pastes following the instructions provided by the manufacturer. The cement was photo-polymerized using an LED curing unit (New Light VL-II, GC, Tokyo, Japan) for 1 min. The specimens were then kept in deionized water at 37 °C for 7 days.

(3) Thermal aging of samples

To simulate 1-year clinical service in an *in vitro* setting, thermal aging was performed using a thermocycler (Mechatronik, Feldkirchen-Westerham, Germany). A total of 10 000 cycles were completed, with one cycle consisting of immersing discs in two water baths maintained at temperatures of 5 °C and 55 °C respectively. The dwell time in each bath was 30 secs with a transfer time of 5 secs.

(4) SBS assessment

A universal testing machine (UTM) (Autograph AG-X 20kN, Shimadzu, Kyoto, Japan) was used to assess the bond strength scores. The resin cement and zirconia interface was loaded in a perpendicular direction at a crosshead speed of 0.5 mm/min until debonding. The applied load was measured in Megapascal (MPa).

(5) Surface topography assessment using SEM and failure mode assessment

The morphology of the zirconia discs that had undergone surface conditioning and bonding with resin cement was assessed by means of scanning electron microscopy (LEO-1530VP; GmbH, Oberkochen, Germany) at different magnifications following gold sputter coating at an acceleration of 0 kV. To assess the failure mode, a stereomicroscope (Carl Zeiss Jena GmbH, Göttingen, Germany) was used with different magnifications. The samples were categorized into adhesive, cohesive, and admixed.

(6) Statistical analysis

All statistical analyses were performed using the SPSS version 28.0 (SPSS, Chicago, IL, USA). One-way ANOVA and post hoc Tukey tests were conducted to test the means of *Ra* and SBS for the different tested groups ($p < 0.05$).

III. Results

(1) SEM evaluation

Fig. 1: Microscopic Characterization of Y-TZP Coatings(A) Scanning electron microscopy image of Y-TZP coated with Nano-HA. The coating exhibits a distinctive rose-petal-like morphology (arrows). (B) Topographic analysis of the Nano-HA coating reveals a highly ordered surface characterized by well-defined, regular patterns in both the horizontal and cross-sectional planes.

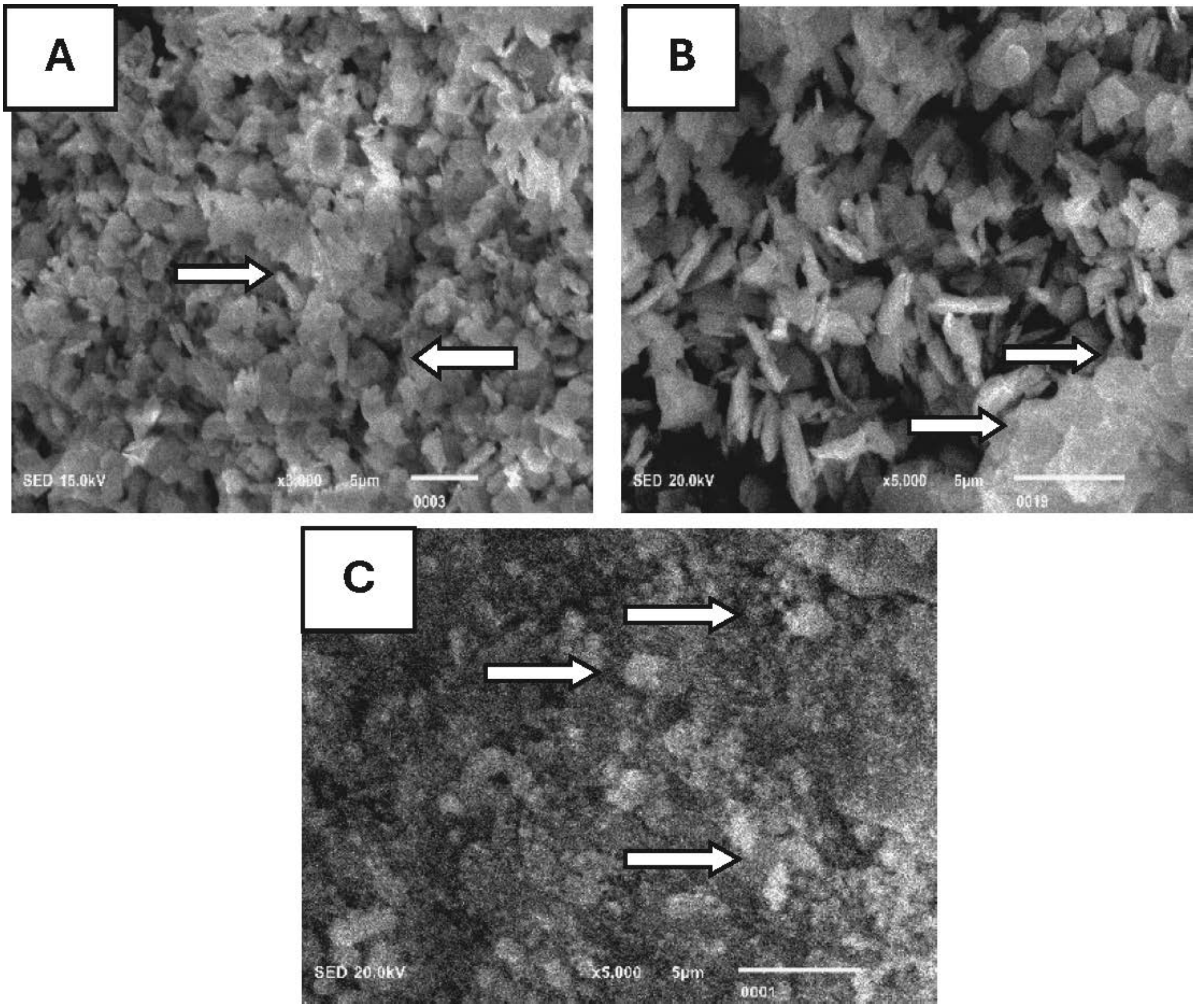

Fig. 1: (A) SEM analysis shows Y-TZP coated with Nano-HA displaying a rose petal-like pattern (Arrows) (B) The topographic analysis revealed well-structured, regular, and clearly defined horizontal and cross-surface patterns, with no visible microcrack melting and fusion of crystals being observed (Arrow) (C) SEM image of Y-TZP coated with tribo-chemical silica coating melting and subsequently bonding of silica is observed on the zirconia surface (Arrow).

Notably, no evidence of microcrack formation or crystal melting is observed (arrow). (C) SEM image of Y-TZP coated with a tribochemical silica coating. In contrast to the nano-HA coating, evidence of silica melting and subsequent bonding to the zirconia surface is apparent (arrow).

(2) *Ra* analysis

Table 1 presents the *Ra* of Y-TZP after the application of different pretreatment regimes. The maximum roughness was exhibited by Group 4 (Nano-HA) (1 421.26 ± 0.062 μm) specimens. However, Group 2 (TBC) (1 000.57 ± 0.043 μm) conditioned discs demonstrated minimum scores of roughness. Comparison analysis among different groups indicated that Group 3 (FS laser) (1 410.11 ± 0.053) and Group 4 showed comparable scores for Ra. *($p > 0.05$)* Group 1 (APA) (1 283.61 ± 0.021 μm) conditioned Y-TZP discs revealed higher bond strength scores than Group 1 yet lower than Group 3 and 4 *($p < 0.05$).*

Table 1: *Ra* on yttria-stabilized tetragonal zirconia polycrystals after the application of different pretreatment regimes.

Investigated groups	*Mean ± SD (μm)*
Group 1: APA	1 283.61 ± 0.021^{b}
Group 2: TBC	1 000.57 ± 0.043^{c}
Group 3: FS laser	1 410.11 ± 0.053^{a}
Group 4: Nano—HA coating	1 421.26 ± 0.062^{a}

ANOVA

Air-particle abrasion (APA), tribo-chemical silica coating (TBC), Femtosecond laser (FS), Nano-Hydroxyapatite (HA)

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

(3) Bond strength analysis

Table 2 presents the SBS of Y-TZP bonded to resin luting cement after the application of different pretreatment

Table 2: SBS of yttria-stabilized tetragonal zirconia polycrystals bonded to resin luting cement after the application of different pretreatment regimes.

Experimental groups	*Mean ± SD (MPa)*	*p- value!*
Group 1: APA	15.02 ± 0.06^{b}	
Group 2: TBC	14.54 ± 0.09^{c}	*< 0.05*
Group 3: FS laser	17.99 ± 0.12^{a}	
Group 4: Nano-HA coating	18.34 ± 0.16^{a}	

! ANOVA

Air-particle abrasion (APA), tribo-chemical silica coating (TBC), Femtosecond laser (FS), Hydroxyapatite (HA)

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

regimes. The maximum bond integrity scores were exhibited by Group 4 (Nano-HA) (18.34 ± 0.16 MPa) discs. However, the minimum SBS was indicated by Group 2 (TBC) (14.54 ± 0.09 MPa) conditioned Y-TZP plates. Intergroup comparison analysis revealed that for Group 3 (FS laser) (17.99 ± 0.12 MPa) and Group 4 no significant difference in the bond strength could be determined *($p > 0.05$).* Group 1 (APA) (15.02 ± 0.06 MPa) conditioned Y-TZP discs exhibited higher bond strength scores than Group 1 yet lower than Groups 3 and 4 *($p < 0.05$).*

(4) Bond failure assessment

Fig. 2 presents the modes of failure distribution in different experimental groups. Groups 3 and 4 mostly exhibited a cohesive mode of failure. However, Groups 1 and 2 presented all types of failures.

IV. Discussion

This current research was based on the hypothesis that when contemporary conditioning techniques (TBC, FS laser, and Nano-HA coating) are applied, the *Ra* of Y-TZP would be comparable to that obtained with APA. It was also anticipated that SBS outcomes of zirconia modified using the latest regimes would be the same as that of the control specimens. The outcomes revealed that both hypotheses could be completely rejected as all the groups presented significantly different *Ra* and SBS to that obtained with APA. The SBS test was selected, acknowledging its limitations, as alternative specimen preparation could lead to interfacial defects. During tensile loading, crack propagation may occur, leading to potential premature failure of the interface at a lower stress level [14].

Attia and colleagues proposed that the minimum acceptable range for clinical bonding in restorative dentistry is between 10 and 13 MPa. The Nano-HA coating and FS laser-treated group displayed significantly higher scores for *Ra* and bond strength [15]. Diverse coating approaches are available for utilizing the advantages of HA. The method of thermal coating possesses the capacity to modify the zirconia ceramics as per

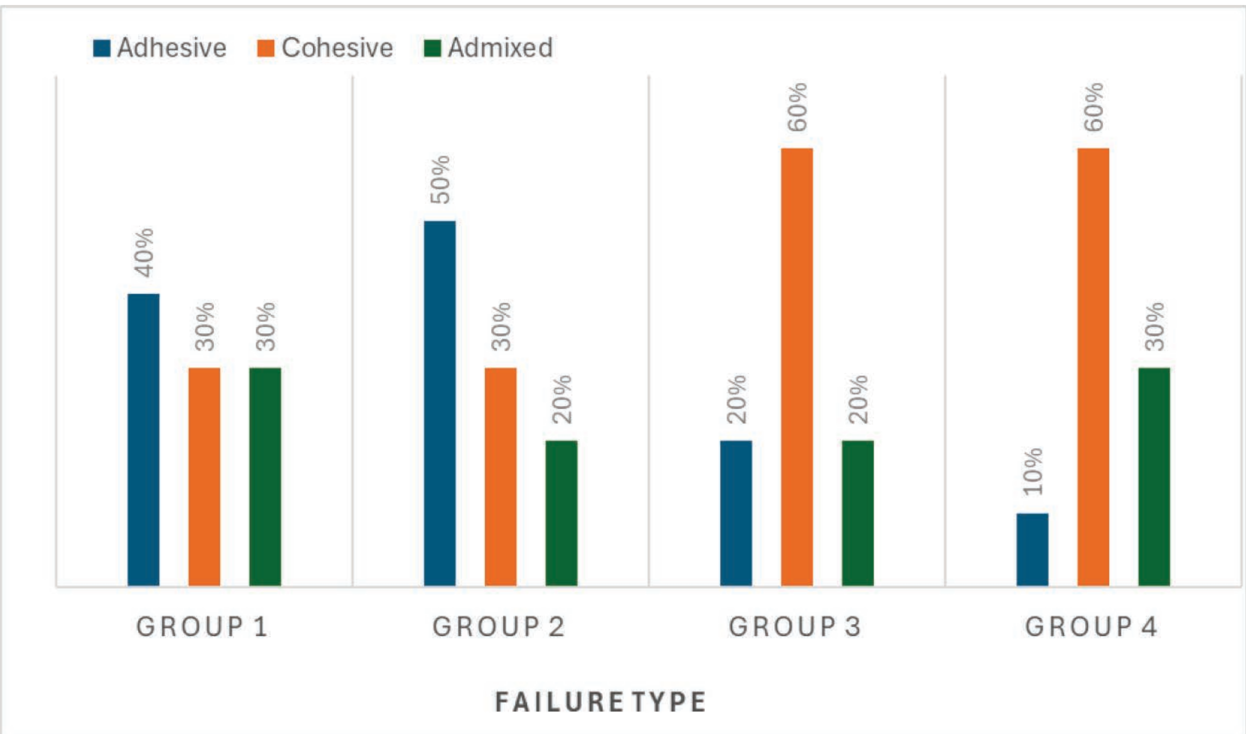

Fig 2: Modes of failure distribution in different experimental groups.

the research analysis conducted by Azari *et al.*[16]. They revealed that Nano-HA coating improves SBS by forming a thin and uniform coating of adequate strength. This coating also increases the *Ra* when used for implant coating, which is in agreement with the outcomes of existing analysis [16]. The augmented *Ra* scores can also be corroborated by the SEM analysis, which displayed a rose petal-like pattern on Y-TZP discs. Whereas, Moezzizadeh and coauthors displayed contradictory outcomes in their study by proclaiming that Nano-HA coating resulted in weaker bonds as compared to sandblasting for zirconium samples [17]. Likewise, the higher *Ra* and bond integrity of FS laser may be clarified by considering that laser engraves a cross-stripe pattern on the surface of Y-TZP which becomes responsible for the resin luting micro retention, thus eventually enhancing the SBS [10]. FS laser machining has demonstrated significant efficacy in the microprocessing of ceramic materials due to its ultrashort pulse duration and high peak power. This technique facilitates local melting, vaporization, or ablation of samples within an exceptionally brief laser-material interaction period.[18] This finding can also be supported by the SEM analysis of FS-conditioned zirconia ceramics discs. The topographic analysis revealed well-structured, regular, and clearly defined horizontal and cross-surface patterns, with no visible microcrack. Prieto *et al.*, Yeğin *et al.* and Akpinar *et al.* in their lab-based analysis reported the same outcomes.[19–21]

Based on the available literature, it can be witnessed that APA roughens the zirconia surface to enable enhanced bonding, yet it compromises the material mechanical properties. However, Kern *et al.* indicated that the application of APA with 50-µm alumina particles at a pressure of 2.5 bar or lower effectively reduces damage while generating adequate microporosity, and wettability and thus augments the bond strength [22]. Considering this evidence, the current study involved air-abrasion of the specimens with 50-µm Al_2O_3 particles at a pressure of 2.5 bar. The increased *Ra* scores observed can further be confirmed with the SEM. In contrast, TBC pretreated discs displayed significantly lower scores of *Ra* and SBS than the control specimens. Past research has reported that silica coating helps absorb the kinetic energy during impact, providing a gentler modification with lower *Ra* scores [23]. Nagaoka and colleagues did not elucidate the exact mechanism by which this works but they stated that upon impact with the zirconia surface, silica-coated alumina particles experience a conversion of a portion of their kinetic energy into thermal energy, resulting in a localized rise in temperature [24]. Consequently, a portion of the silica particles undergoes melting and subsequently bonds to the zirconia surface. At the same time, certain alumina particles undergo fracturing and silica particles do not undergo melting followed by their deposition on the surface. These unfused silica particles remaining on the zirconia surface negatively impact the bond strength of luting cement [24, 25]. However, it was evidenced that when TBC is used with silane, it increases the surface receptiveness of zirconia for chemical interaction.

The present study has several limitations. Its laboratory-based nature restricts the generalizability of findings to clinical practice. Furthermore, the investigation focused on individual femtosecond laser parameters and their influence on surface roughness and bond strength, and only one resin cement was tested. Consequently, future research should investigate the combined effects of hydroxyapatite coating and FS laser treatment on the *Ra* and shear bond strength of zirconia in a clinical setting.

V. Conclusions

Nano-hydroxyapatite coating and the femtosecond laser can be used as a suitable alternative to air particle abrasion without affecting the physical and mechanical properties of zirconia ceramic.

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Ethical Statement: *The research experiments conducted in this article were approved by the Ethical Committee and responsible authorities of our research organization(s) following all guidelines and regulations.*

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