

Flexural and adhesive strength of 5Y-PSZ translucent zirconia after irradiation with Er:YAG laser

Resistência flexural e adesiva da zircônia translúcida 5Y-PSZ após irradiação com laser Er:YAG

Diana Ghiggi BASSO¹ , Patrícia Moreira de FREITAS² , Leonardo SARAIVA¹ , Paula BENETTI³ 

1 - Universidade de Passo Fundo, Faculdade de Odontologia, Programa de Pós-graduação em Odontologia. Passo Fundo, RS, Brazil.

2 - Universidade de São Paulo, Faculdade de Odontologia, Laboratório Especial de Laser em Odontologia. São Paulo, SP, Brazil.

3 - Universidade de Passo Fundo, Programa de Pós-graduação em Clínica Odontológica, Curso de Odontologia. Passo Fundo, RS, Brazil.

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ABSTRACT

Objective: To evaluate the effect of Er:YAG laser irradiation on the adhesive and fracture strength of 5 mol% yttria partially stabilized polycrystalline zirconia (5Y-PSZ). **Material and Methods:** Disc-shaped zirconia specimens (CPs) were divided into three groups (n=30): no treatment (Ctrl), airborne-particle abrasion with 50- μ m aluminum oxide (Abr), and Er:YAG laser irradiation (Las). Composite resin cylinders (2 mm diameter $\times$ 5 mm height) were adhesively cemented to the CPs (n = 10) and were tested for microshear bond strength (μ SBS). Fracture surfaces were examined by optical and scanning electron microscopy following fractographic principles, and failure modes were classified as adhesive, cohesive in resin, or mixed. Biaxial flexural strength (σ_f) was assessed using a piston-on-three-balls test (n=30), with the treated surface subjected to tensile stresses. The Weibull modulus (m) and characteristic strength (σ_0) were calculated. Data were analyzed by ANOVA and Tukey tests (5%) ($\alpha = 0.05$). **Results:** Laser-treated group showed μ SBS values similar to both airborne-particle-abraded and control groups ($p < 0.001$). Laser irradiation resulted in flexural strength values comparable to airborne-particle abrasion but significantly lower than the control group ($p < 0.001$). Fracture analysis revealed predominantly adhesive and mixed failures. **Conclusion:** High-power laser irradiation reduced the flexural strength of translucent zirconia similarly to airborne-particle abrasion, while reaching the adhesive performance comparable to airborne-particle abrasion.

KEYWORDS

Air abrasion; Ceramics; Flexural strength; Lasers; Shear strength.

RESUMO

Objetivo: Avaliar o efeito da irradiação com laser Er:YAG na resistência adesiva e à fratura da zircônia policristalina parcialmente estabilizada com 5 mol% de ítria (5Y-PSZ). **Material e Métodos:** Discos de zircônia (CPs) foram divididos em três grupos (n = 30): sem tratamento (Ctrl), jateamento com partículas de óxido de alumínio de 50 μ m (Abr) e irradiação com laser Er:YAG (Las). Cilindros de resina composta foram adesivamente cimentados na superfície dos CPs (n=10) e submetidos ao teste de resistência de união ao microcisalhamento (μ SBS). A resistência flexural biaxial (σ_f) foi avaliada (n=20) por meio do teste pistão sobre três esferas, com a superfície tratada submetida à tração. As superfícies de fratura foram examinadas por microscopia óptica e eletrônica de varredura, seguindo princípios fractográficos, e os modos de falha foram classificados como adesivo, coesivo em resina ou misto. O módulo de Weibull (m) e a resistência característica (σ_0) foram calculados. Os dados foram analisados por ANOVA e Tukey (5%) ($\alpha = 0,05$). **Resultados:** Las apresentou μ SBS semelhante a Abr e Ctrl ($p < 0,001$). A análise dos modos de falha revelou predominância de falhas adesivas e mistas. Las resultou em valores de σ_0 comparáveis ao Abr, porém significativamente inferiores ao Ctrl ($p < 0,001$). **Conclusão:** A irradiação com laser de alta potência reduziu a resistência da zircônia translúcida de forma semelhante ao jateamento, porém, alcançou adesão semelhante ao jateamento.

PALAVRAS-CHAVE

Abrasão dental por ar; Cerâmica; Resistência flexural; Lasers; Resistência de união.

INTRODUCTION

Dental zirconia exhibits high toughness and translucency, meeting aesthetic requirements and enhancing the strength of oral restorations. Zirconia is a polycrystalline ceramic that may exist in different crystallographic phases depending on temperature: monoclinic, stable at room temperature up to approximately 1,170 °C; tetragonal, stable between 1,170 °C and 2,370 °C; and cubic, present at higher temperatures until melting [1-3]. Among these phases, the tetragonal structure provides superior mechanical properties, whereas the monoclinic phase exhibits low flexural strength and hardness due to the high interatomic distance in the crystals.

To stabilize the tetragonal phase at room temperature, yttrium oxide is added, resulting in 3 mol% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP). This material exhibits high flexural strength (900-1,400 MPa) and fracture toughness (5 to 10 MPa^{1/2}) [4], primarily attributed to the transformation-toughening mechanism. Under localized stresses, such as those generated at the tip of a propagating crack, tetragonal crystals transform into the monoclinic phase, accompanied by a volumetric expansion from 3% to 5% that induces compressive stresses and counteracts crack propagation. This stress-induced phase transformation is the main mechanism responsible for the remarkable mechanical reliability of 3Y-TZP in dental applications [2,5].

Despite its mechanical superiority, 3Y-TZP presents limited translucency due to the intrinsic birefringence of tetragonal crystals and light scattering caused by grain boundaries, pores, and microstructural defects [3,6]. To meet the esthetic demands of monolithic restorations, modifications in zirconia composition and microstructure were introduced, including increased yttria content, reduced alumina concentration, higher sintering temperatures, and the promotion of a cubic phase [2,4,6]. These changes led to the development of more translucent zirconias [3,7], such as 4Y-PSZ and 5Y-PSZ; however, the increase in cubic phase content resulted in reduced strength and fracture toughness, since cubic zirconia does not undergo stress-induced phase transformation [1,2,8-11].

Regardless of composition, adhesive cementation of zirconia remains a significant clinical challenge. Unlike silica-based ceramics, zirconia lacks a glassy matrix and is therefore more resistant to acid etching, limiting conventional chemical conditioning strategies. Currently, airborne-particle abrasion with

aluminum oxide particles, with or without silica coating, is the most widely used surface treatment to enhance micromechanical retention, surface energy, and wettability of zirconia [12-15].

However, this mechanical surface treatment is not free of drawbacks. Airborne-particle abrasion may introduce surface defects, microcracks, and localized phase transformation as a result of particle impact [5,8,9,13,15-20]. While this protocol has been successfully applied to 3Y-TZP, concerns persist regarding its potential to compromise the structural integrity of translucent zirconia. Consequently, a central challenge in contemporary prosthodontics is to identify surface treatment protocols that promote reliable adhesion to translucent zirconia without damaging its mechanical strength.

From a chemical perspective, adhesive systems containing phosphate functional monomers, especially 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), have demonstrated effective bonding to zirconia. The phosphate groups of MDP chemically interact with zirconium dioxide, forming stable bonds that complement micromechanical interlocking [3,20-23]. Thus, combining mechanical surface modification with chemical adhesion promoters has been considered the most effective strategy for zirconia cementation.

To reduce the adverse effects associated with conventional airborne-particle abrasion, laser irradiation has been proposed as an alternative surface treatment [7,12,14,19,24-31]. Laser application induces primarily photothermal and thermomechanical effects, generating surface micro-irregularities through controlled ablation and potentially improving surface roughness and wettability [14,16,32,33]. Among available systems, the Er:YAG laser has received particular attention due to its ability to produce relatively homogeneous surface modifications.

Nevertheless, the evidence regarding laser treatment of zirconia remains inconclusive [18]. While some studies have reported bond strength values comparable to those achieved with airborne-particle abrasion in 3Y-TZP, others have demonstrated microcrack formation, phase transformation, and deterioration of mechanical properties, preventing laser irradiation from being considered a predictable substitute for conventional mechanical pretreatment. Moreover, most available data focus on 3Y-TZP, and the effects of laser irradiation on more translucent zirconias, particularly 5Y-PSZ, are still unclear.

Identifying surface treatment strategies capable of balancing adhesive effectiveness and preservation of zirconia structural integrity remains of utmost importance. Therefore, the present study aimed to evaluate the effect of high-power Er:YAG laser irradiation on the flexural and adhesive bond strength of 5 mol% yttria-partially stabilized polycrystalline zirconia (5Y-PSZ). The hypotheses were that Er:YAG laser irradiation improves the adhesive bond strength of translucent zirconia while preserving its flexural strength.

MATERIALS AND METHODS

Obtaining specimens

The materials used in this study are described in Table I. A block of translucent zirconia (5Y-PSZ) was sectioned with a diamond saw on a cutting machine (Metallographic Cutter Machine - LCD 1,200 RPM - Biopdi) in the green phase into small blocks. The blocks were machined into a cylindrical shape and sliced to obtain disc-shaped specimens ($N = 90$) with dimensions of 0.86 mm thickness and 14.8 mm diameter measured with a digital caliper (ABS Absolute Digimatic, Mitutoyo). The specimens were smoothed with 1,000 grit sandpaper before sintering according to the manufacturer's recommendations. The final dimensions (0.7 mm thickness x 12 mm diameter) were verified with a digital caliper. The specimens were divided into three experimental groups ($n = 30$) according to the surface treatment: no treatment (Ctrl), airborne-particle abrasion using Al_2O_3 (Abr), and irradiation with high-power Er:YAG laser (Las).

Airborne-particle abrasion

To minimize operator-related variability during airborne-particle abrasion and laser irradiation, all procedures were performed by a single previously trained operator under

standardized conditions. One surface of each specimen was initially coated with a permanent marker to serve as a visual indicator for surface treatment. Subsequently, airborne-particle abrasion was performed using 50- μ m aluminum oxide (Al_2O_3) particles, applied perpendicularly to the surface at a working distance of 10 mm and an approximate pressure of 2 bar, until complete removal of the marker layer was achieved.

Er:YAG Laser Irradiation Protocol

Irradiation was performed by a single trained operator using an Er:YAG laser (LiteTouch™, Light Instruments, Israel) (wavelength = 2,940 nm) equipped with an R02-C handpiece and a cylindrical sapphire optical fiber (AS7077X, 0.8 mm diameter x 8 mm length; beam area of 0.005 cm²) under water irrigation (20 mL/min). The irradiation protocol was carefully controlled regarding application time (30 s per specimen), working distance of 1–2 mm from the material surface, scanning pattern, incidence angle, and water cooling. In addition, the handpiece movement was performed using continuous and standardized horizontal and vertical motions guided by a red aiming beam projected onto the target area of the zirconia surface. The opposite (non-irradiated) surface was previously identified and marked to prevent unintentional exposure.

Different Er laser irradiation protocols were evaluated in a pilot study to identify parameters capable of promoting surface modification without inducing evident surface damage to the zirconia ($n=2$). The pilot analysis was based on qualitative optical and scanning electron microscopy (SEM) observations of the irradiated surfaces, focusing on the presence of irregularities, melting areas, and crack formation. Four irradiation protocols based on Moretto et al. [28] were tested by varying pulse energy (200 or 250 mJ), repetition rate (10 or 15 Hz), power output (2.5 or 3.0 W), and irradiation angle (45° or 90°), as shown in Table II.

Table I - Description of materials used in the study

Material	Description	Chemical composition*
Ceramill Zolid FX – White (5Y-PSZ)	Fourth-generation ultra-translucent zirconia	ZrO_2 , HfO_2 , Y_2O_3 , Y_2O_3 $Y_2O_3 \geq 9.15 - 9.00\%$ $HfO_2: \leq 5$ $Al_2O_3: \leq 0.5$
Aluminum oxide (Al_2O_3)	Abrasive agent	Aluminum oxide with a mean particle size of 50 μ m

*Provided by the manufacturers.

The evaluated protocols produced only subtle surface alterations without visible crack formation (Figure 1). Based on the SEM findings and previous studies demonstrating that higher pulse energies may induce excessive surface deterioration and strength reduction in zirconia ceramics, the Protocol 3 using 250 mJ, 10 Hz, and 2.5 W was

selected for the subsequent analyses. This protocol promoted homogeneous surface modification while avoiding evident morphological defects. In addition, lower repetition rates are associated with reduced thermal accumulation during irradiation, which may decrease the risk of thermally induced damage to zirconia surfaces.

Table II - Irradiation protocols tested in the pilot study

Protocol	Pulse energy (mJ)	Repetition rate (Hz)	Power (W)	Irradiation angle (°)
1	200	15	3.0	90
2	200	15	3.0	45
3	250	10	2.5	90
4	250	10	2.5	45

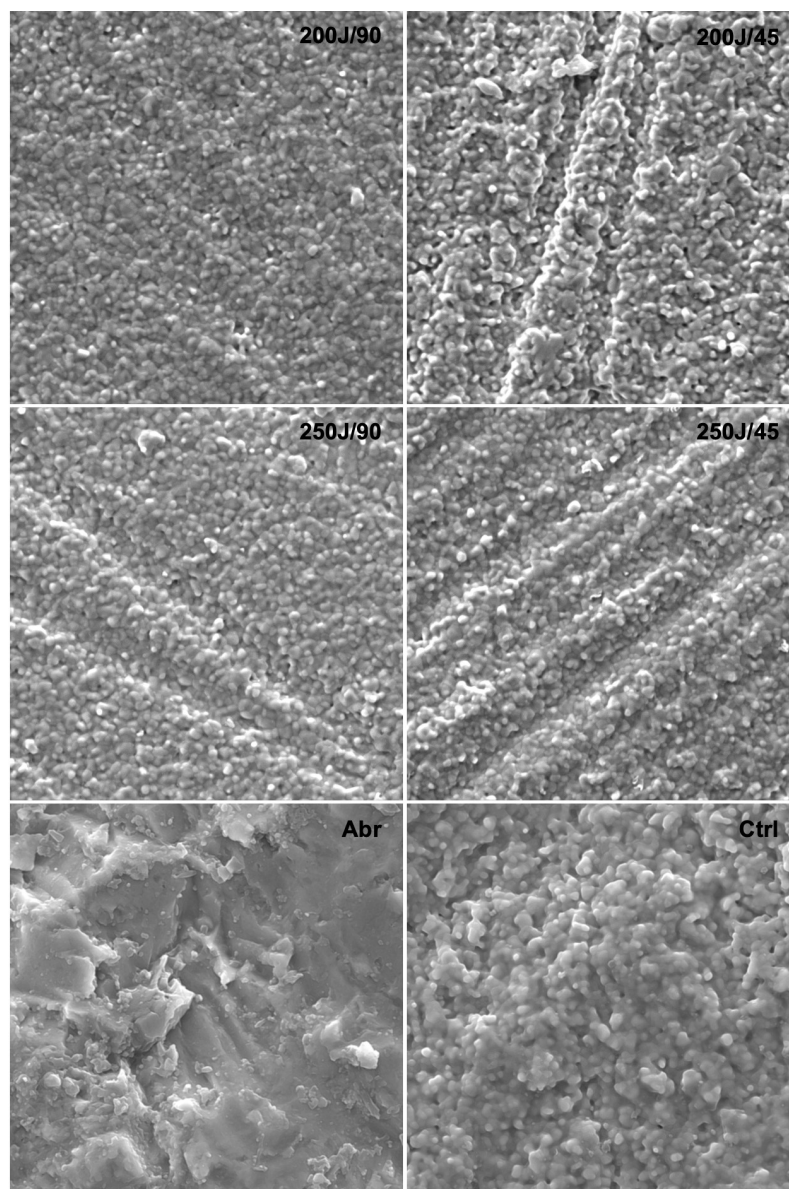


Figure 1 - Representative SEM images (20,000× magnification) of zirconia surfaces after different Er laser irradiation protocols, airborne-particle abrasion with alumina (Abr), and untreated control surface (Ctrl). Laser irradiation produced subtle and homogeneous surface alterations without evident crack formation, whereas airborne-particle abrasion resulted in more irregular topography.

Preparation of test specimens for the bond strength test

Composite resin cylinders (n=30) were fabricated by condensing the material (Fill Magic Resin; Vigodent) into a two-part metallic mold with internal dimensions of 2 mm in diameter and 5 mm in height. The resin cylinders were bonded to the center of the zirconia treated surfaces (n=10) using an MDP-containing resin cement (Panavia F 2.0; Kuraray Noritake Dental, Tokyo, Japan). A static load of 750g was applied to standardize the cement layer thickness. Excess cement was removed with a microbrush to control the adhesive interface area. The cement was photoactivated for 60 seconds using a LED curing unit (Emitter Now, Schuster, 1,250 mw/cm²).

Micro-shear bond strength test

The specimens were subjected to a shear strength test (SBS) using a universal testing machine (EMIC DL 2000, São José dos Pinhais, PR, Brazil), with a 200 N load cell, at a crosshead speed of 0.5 mm/min. The shear force was applied parallel to the cement-zirconia interface until failure. The maximum fracture load (N) was recorded, and the bond strength values (σs, MPa) were calculated by dividing the fracture load by the adhesive interface area (mm²), according to the ISO 6872 [34]. Fracture surface analysis was performed using a stereomicroscope (10X) to determine the failure mode. Failures were classified as adhesive (at the cement-ceramic interface), cohesive (within the resin cement or composite resin), and mixed (a combination of adhesive and cohesive failures).

Biaxial bending strength test

Specimens from each experimental group were placed in a biaxial flexural test device (piston on three-balls, ISO 6872 [34]) with the treated surface facing downward and subjected to tensile stress. The test was performed in a universal testing machine (EMIC DL 2000; São José dos Pinhais, PR, Brazil) equipped with a 2,000 N load cell. A compressive load was applied by a piston at a constant crosshead speed of 0.5 mm/min to the center of the untreated surface until fracture, and the maximum load (N) was recorded. The biaxial flexural strength (σf) was calculated using Equation 1, which accounts for the fracture load, specimen thickness

and diameter, the diameter of the support circle formed by the three spheres, and the diameter of the loading piston [34].

$$\sigma_f = 3P \frac{(1+\nu)}{4\pi t^2} \left[1 + 2 \ln \left(\frac{a}{b} \right) + \left(\frac{1-\nu}{1+\nu} \right) \left(1 - \frac{b^2}{2a^2} \right) \left(\frac{a^2}{R^2} \right) \right] \quad (1)$$

Where,

σf = bending strength (MPa)

P = maximum fracture load (N)

t = sample thickness (mm)

R = radius of the sample (mm)

a = radius of the support circle (mm)

b = radius of the load piston (mm)

ν = Poisson's ratio

Analysis of statistical data

The biaxial flexural strength (σf) and shear bond strength (σs) data passed the normality and homogeneity of variance tests (Shapiro-Wilk and Levene, respectively) (α = 0.05). Therefore, data were analyzed using one-way analysis of variance (ANOVA), followed by Tukey's post hoc test, at a significance level of 5%. Weibull analysis was also performed to determine the Weibull modulus (m) and characteristic strength (σ₀), which were calculated and compared among the experimental groups.

RESULTS

Significant differences in bond strength were observed among the groups (p < 0.001) (Table III). Airborne-particle abrasion significantly increased bond strength compared to the control. High power laser irradiation (Las) promoted intermediate bond strength values, with no statistically significant differences compared to Abr and Ctrl groups (p < 0.001). The predominant modes of failure were adhesive and mixed failures across all groups.

Las showed characteristic strength similar to Abr, however, lower than the control group (p < 0.001) (Table IV). No differences between the groups were found in the Weibull modulus. The ceramic surface in the region opposite to the contact with the load-applying piston, subjected to tension, was identified as the origin of the fractures.

Table III - Results of the comparative analysis of bond strength data for the experimental groups

Group	N	σ_s (MPa)*	Standard Deviation	Failure mode (%)		
				Adhesive	Cohesive	Mixed
Ctrl	10	6.8 B	3.8	50	10	40
Abr	10	12.8 A	5.5	40	10	50
Las	10	8 AB	4.5	40	10	50

* Different letters indicate statistically significant differences among the experimental group means ($p < 0.001$). Statistical power of 74%.

Table IV - Results for biaxial flexural strength (σ_f), Characteristic strength (σ_0), and Weibull modulus (m) of the experimental groups*

Group	n	σ_f (SD) (MPa)	σ_0 (MPa)	m
Ctrl	30	265.8 (112.1) A	300.6 A	2.7 A
Abr	30	172.7 (73.2) B	194.8 B	2.6 A
Las	30	181.3 (54.2) B	200.8 B	3.6 A

* Distinct letters show the differences between the means of experimental groups ($p < 0.001$). Statistical power of 94%.

DISCUSSION

The present study evaluated the effect of high-power Er:YAG laser irradiation on the flexural and adhesive strength of translucent zirconia (5Y-PSZ), compared with conventional airborne-particle abrasion. Based on the results, the null hypothesis was partially rejected, as Er:YAG laser irradiation did not preserve flexural strength of 5Y-PSZ, although it maintained adhesive performance comparable to airborne-particle abrasion.

Er:YAG laser irradiation resulted in bond strength values comparable to both airborne-particle abrasion and untreated zirconia. These findings indicate that, within the tested parameters, laser irradiation did not improve resin–zirconia bonding beyond conventional methods, but also did not weaken it. The predominance of adhesive and mixed failure modes across all groups suggests that the integrity of the resin–zirconia interface remains the main limitation, regardless of surface treatment [14,22,30].

Biaxial flexural strength results demonstrated that both Er:YAG laser irradiation and airborne-particle abrasion significantly reduced the strength of 5Y-PSZ compared with untreated specimens. These findings corroborate previous reports indicating that surface modification strategies involving localized energy transfer—either through particle impact or laser irradiation—may introduce surface and subsurface damage that negatively affects the mechanical performance of translucent zirconia materials [5,9,16]. Similar reductions in flexural strength following laser

treatment were reported by da Silva et al. [29] and Çağlar and Yanıkoğlu [16], reinforcing concerns regarding the structural sensitivity of translucent zirconia to aggressive surface treatments.

Laser–material interaction is primarily governed by photothermal and thermomechanical mechanisms. However, zirconia exhibits limited intrinsic absorption of Er:YAG laser energy due to its white color and absence of water content [1,16,27,28]. Consequently, surface modification depends on the use of high-energy parameters capable of inducing nonlinear optical phenomena and multiphoton absorption, which may result in localized heating, phase transformation, and microstructural damage [25,35]. Previous studies demonstrated that increasing pulse energy and irradiation time enhances surface roughness, but at the cost of severe surface deterioration and strength reduction, particularly in translucent zirconias [24,31].

In the present study, laser parameters were selected based on a pilot investigation to minimize surface damage while still inducing surface irregularities. Irradiation with pulse energies of 200 to 250 mJ produced subtle surface alterations without visible cracking under optical and scanning electron microscopy. Nevertheless, despite the absence of macroscopically detectable defects, flexural strength values were significantly reduced, suggesting that microstructural changes below the surface or residual stresses induced by irradiation may have contributed to the observed mechanical degradation.

This effect is especially relevant for 5Y-PSZ, which contains a higher proportion of the cubic phase and lacks the stress-induced transformation toughening mechanism characteristic of 3Y-TZP [1,2,8,9].

A study demonstrated the effects of Er:YAG laser irradiation and alumina air abrasion on the surface roughness and morphological characteristics of 3Y-TZP, showing that greater surface roughness occurred when the laser output power and irradiation time were increased. However, increasing the laser irradiation parameters is unsuitable for surface treatment due to severe damage and phase transformation of the zirconia [28-31]. A recent study showed that the use of pulse energy values between 400 mJ and 600 mJ excessively deteriorated the material and was not indicated for surface treatments [28]. Irradiation with a pulse energy of 200 mJ caused slight alterations, with characteristics intermediate between the effects of air abrasion of particles and a higher laser intensity [28]. Therefore, lower energy options (200-250 mJ) should be applied for ceramic surface treatment. The laser irradiation protocols used in the present study, with pulse energies of 200 mJ and 250 mJ, produced only small alterations in the topography of the zirconia surfaces. The irradiation beam angle of 90° or 45° did not significantly alter the zirconia topography. Thus, a surface irradiation energy of 250 mJ was selected for the flexural strength analysis.

Pulse width is directly associated with the interaction of light and thermal effects on the substrate. The smaller the pulse, the faster it will modify the irradiated surface, resulting in more efficient ablation and less thermal damage to the ceramic surface [29]. Similarly, the number of pulses per second (in Hz) is also related to the thermal effects on the irradiated tissue. The higher the repetition rate, the greater the risk of increased temperature. Therefore, the investigations in this study were conducted using a short pulse (100 - 200 μ s), selecting the protocol with a repetition rate of 10 Hz.

A previous study demonstrated that the alumina air abrasion method provided rougher surfaces than Er laser irradiation.YAG [32], corroborating the findings of the present study, which showed more irregularities in zirconia subjected to air abrasion, groups irradiated with Er:YAG lasers of different parameters produced similar, slightly scratched surface areas. Although laser irradiation and abrasion with airborne

particles produced different topographies, they may have caused similar microstructural damage, stress-induced phase transformations, or alterations in crystal arrangements, which could have contributed to the decrease in strength observed in the treated zirconia compared to the control group.

From a clinical perspective, the reduction in flexural strength associated with both laser irradiation and airborne-particle abrasion raises concerns regarding the use of aggressive surface conditioning protocols on translucent zirconia, particularly in thin restorations or minimally invasive designs. Although adhesive cementation may partially offset strength loss by improving stress distribution and structural support, surface treatments must be carefully balanced to avoid compromising the intrinsic mechanical integrity of 5Y-PSZ [5,9]. In the present study, laser irradiation and air abrasion with alumina particles decreased the mechanical strength of translucent zirconia, which could potentially be recovered with adhesive cementation to dental substrates or other materials. Future evaluations of the longevity of zirconia cemented with adhesive could be performed by simulating the intraoral environment with cyclic thermomechanical fatigue.

A limitation of this study is the manual application of the laser and airborne-particle abrasion, which may introduce operator-dependent variability. However, all irradiations were performed by a single trained operator under standardized parameters and application procedures to improve reproducibility.

Er:YAG laser irradiation did not provide a mechanical advantage over airborne-particle abrasion for 5Y-PSZ. Although adhesive performance was maintained, the adverse effect on flexural strength indicates that high-power laser irradiation cannot be considered a risk-free alternative to conventional mechanical pretreatment. Further studies incorporating thermomechanical fatigue and aging protocols are needed to better predict the long-term clinical performance of laser-treated translucent zirconia restorations.

CONCLUSION

Within the limitations of this in vitro study, high-power Er:YAG laser irradiation altered the surface of 5Y-PSZ translucent zirconia but resulted in a reduction in biaxial flexural strength similar to that observed with airborne-particle abrasion.

Adhesive bond strength to resin cement was maintained and comparable among laser-treated, air-abraded, and untreated surfaces. Therefore, although Er:YAG laser irradiation did not improve adhesion relative to conventional abrasion, it also did not impair bonding performance; however, its negative impact on flexural strength indicates that it should be used with caution when conditioning translucent zirconia restorations.

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Data availability

The raw data supporting the conclusions of this article will be made available by the authors on reasonable request.

Author's Contributions

DGB, PB, Conceptualization. DGB, PMF, LS, PB: Methodology. DGB, PMF: Validation. DGB, PB: Formal Analysis and Data Curation. DGB, PB: Writing – Original Draft Preparation. PMF, LS: Writing – Review & Editing. PMF, PB: Supervision.

Conflict of Interest

The authors declare no conflicts of interest.

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Regulatory Statement

As this was an in vitro study, approval by an ethics committee was not required.

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Paula Benetti**(Corresponding address)**

Universidade de Passo Fundo, Programa de Pós-graduação em Clínica Odontológica, Curso de Odontologia, Passo Fundo, RS, Brasil.

E-mail: paulabenetti@upf.br

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