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Repair Bond Strength and Surface Roughness of Zirconia Ceramics Treated via Carbon Dioxide Laser, Malachite Green, and Sandblasting. A Lab-Based Study

Eman M Alhamdan*

Department of Prosthetic Dental Sciences, College of Dentistry, King Saud University, P.O.Box 21069, Riyadh 11475, Saudi Arabia.

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Abstract

AIM Contemporary surface conditioners (carbon dioxide (CO_2) laser at different power outputs and malachite green (MG) activated by low-level laser therapy (LLLT) on the surface roughness (*Ra*) and repair bond strength (RBS) of zirconia bonded to composite MATERIALS AND METHODS 104 discs were prepared from pre-sintered zirconia ceramic. The allocation of the samples was based on the surface conditioner: Group 1 (SB), Group 2 (CO_2 laser-3 W), Group 3 (CO_2 laser-4 W), and Group 4 (MG-LLLT). *Ra* analysis and surface topography were assessed using a profilometer and SEM. A ceramic repair kit and composite were applied as restorative materials on 20 samples from each cohort, which were allocated to two subgroups: A (*without thermal aging)* and B (*with thermal aging).* RBS and failure mode were assessed using a universal testing machine and stereomicroscope. Intergroup comparisons were performed using ANOVA and Tukey's post hoc test *($p < 0.05$)*. RESULTS Group 3 (CO_2 laser-4 W) revealed the highest *Ra* and strongest RBS at baseline and after thermal aging, whereas Group 4 (LLLT (MG) demonstrated lower *Ra* and weakest bond at baseline and even after thermal aging. CONCLUSION A CO_2 laser at a power output of 4 can be used as a suitable alternative to sandblasting for repairing zirconia ceramics with composites.

Keywords: Carbon dioxide laser, low-level laser therapy, surface roughness, repair bond strength, sandblasting.

I. Introduction

Zirconia ceramic, a structured oxide ceramic, has found extensive applications in the construction of fixed dental prostheses [1,2]: However, ceramic restorations composed of tetragonal zirconia polycrystals stabilized with yttrium (Y-TZP) tend to chip [3]. Another issue with these restorations is that when the core is exposed, the adhesive securing the core to the porcelain veneer separates, a phenomenon known as delamination [4]. Furthermore, the high corrosion resistance and surface stability of zirconia ceramic complicate the process of achieving effective bonding [5]. In the past, a satisfactory bond between the repair material and the fracture surface was established by means of both chemical and mechanical conditioning regimes [6,7]. Sandblasting (SB) using aluminum oxide (Al_2O_3) particles is widely recognized as a standard method that enhances surface roughness (*Ra*) and improves the repair bond strength (RBS) of composites bonded to zirconia substrates [8]. However, it is essential to note that the SB of zirconia leads to deformation through phase transformation, potentially increasing its susceptibility to fracture.

To mitigate these side effects, alternative methods have been explored to improve the *Ra* and SBS of the ceramic composite while maintaining the mechanical properties. Lasers that utilize various parameters have garnered considerable attention [9–11].The CO_2 laser is frequently applied intraorally, particularly in both soft and hard tissue procedures [12,13].Previous studies have reported that it is highly effective for micromachining because this wavelength laser is nearly completely absorbed by zirconia ceramic and eventually improves the *Ra* and SBS [14–16]. However, it has also been reported that different power output settings of the CO_2 laser may result in different outcomes. Therefore, it is necessary to investigate which power output is suitable when a CO_2 laser is used in a conditioning regime to repair zirconia with composites.

Another technique that has gained recognition in the field of prosthodontics is photodynamic low-level laser therapy (LLLT) utilizing a photoactive dye [17]. This noninvasive method generates reactive oxygen species (ROS) by activating photoactive dyes with light of a certain wavelength [18,19]. Previous indexed literature has indicated that the use of LLLT as a surface pretreatment protocol positively affects the SBS of ceramic to cement [18,19]. Among the various photosensitizers utilized, malachite green (MG) has captured significant interest from scholars and researchers [20–22].This cationic dye has demonstrated excellent absorption in the red region of the visible spectrum [17]. However, it has not been utilized for the conditioning of zirconia surfaces and therefore requires further investigation.

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* Corresponding author: ealhamdan.ksu.edu.sa

Another crucial factor to consider is that an immediate RBS assessment does not evaluate the lifespan and sustainability of restoration. Artificial thermal aging is typically conducted in *in vitro* studies to simulate clinical oral conditions. Numerous studies have demonstrated that the durability of dental restorations decreases when these are subjected to thermocycling [8, 23] Moreover, the effects of various surface pretreatments on the RBS of zirconia bonded to the composite, both before and following thermal aging, remain undetermined.

The present research was based on the anticipation that there would be a significant difference in the *Ra* and RBS of zirconia ceramics bonded to composites when contemporary conditioners (CO_2 laser at different power outputs and MG activated by LLLT) were used compared to the control. Furthermore, it was also predicted that the RBS of the thermal aging group would be lower than that of the group exposed to baseline conditions (before thermocycling). Thus, this investigation aims to estimate the influence of different surface modifiers (CO_2 laser @ different power outputs and MG activated by LLLT) on the RBS and *Ra* of zirconia ceramics.

II. Materials and Methods

One hundred and four discs were prepared from a pre-sintered zirconia ceramic block (Nacera, Doceram, Germany) using ISOMET (Buehler, USA). The dimensional parameters consisted of a 10 mm diameter and 2 mm thickness. The discs were then heated at 1 450 °C for 12 h, followed by cooling at 23 °C. Surface polishing (Phoenix 4000, Buehler, USA) was performed under constant water irrigation with 600- and 1 200-grit silicon carbide paper (English Abrasives Ltd. England). The plates were then embedded in auto-polymerized acrylic resin (Takilon, Rodent srl, Italy) having a diameter and height of 4 cm respectively. The allocation of the specimens was based on the surface conditioner used (n = 26) [24]

Group 1 (SB) SB was conducted using 50-μm alumina particles (Korox; Bego, Bremen, Germany) by blasting at 2.5 bar pressure maintaining a distance of 25 mm from the abrasion tip for 15 s. An ultrasonic cleaner (W&H Dentalwerk Bürmoos GmbH, Bürmoos, Austria) filled with distilled water was used to clean the discs for 10 min [25].

Group 2 (CO_2 laser-3 W) Discs in this group were pretreated using a CO_2 laser (Smart us20D; Deka, Florence, Italy). 10.6 μm wavelength, 100 Hz pulse repetition, 160 ms pulse duration, with 3 W of output power were the set irradiation parameters. The laser irradiation time was 10 secs for each disc, with the laser tip being kept at a right angle to the horizontal axis of the discs at 2 mm distance [26, 27].

Group 3 (CO_2 laser-4 W) A CO_2 laser with the same parameters was used, except that the power output was increased to 4 W [27].

Group 4 (LLLT (MG) The surface of the zirconia samples was modified by applying 0.01 % w/v MG solution for 5 min followed by laser activation for 3 min at 5.4 J/cm^2 energy levels [23].

Ra analysis

A contact profilometer (Contour GT-K 3D Optical Microscope, Bruker, Tucson, AZ, USA) was used for *Ra* assessment on five discs from each group. A diamond stylus having a tip with a radius of 5 μm was traversed maintaining a constant load of 0.75 mN, at a rate of 0.5 mm/sec. Three traces were meticulously recorded at varying positions (parallel, perpendicular, and oblique), resulting in three readings for each sample. The average of three readings was taken as the mean [28].

Scanning electron microscopy (SEM)

To assess the surface characteristics, SEM was performed on one specimen from each group. For this technique, after conditioning, each zirconia plate from each group was gold-coated, followed by observation under SEM (LEO 1430, Carl Zeiss AG, Oberkochen, Germany) operated at 10 kV in combination with EDX for elemental analysis [29, 30].

Ceramic repair kit and composite application

Eighty ceramic plates, 20 from each group, were washed and dried after being coated with phosphoric acid etching gel (K-etchant gel), which was applied for 5 s. The Clearfil SE Bond Primer and Clearfil Porcelain Bond Activator were mixed in a 1:1 ratio and applied for a period of 5 s. The surface was then coated with Clearfil SE Bond, followed by light-curing using LED light (Valo Cordless, Ultradent, South Jordan, UT, USA) for 10 s. A plastic tube with an inner diameter of 3 mm and thickness of 2 mm was centrally positioned on each specimen. The resin composite (GrandioSo, Voco, Cuxhaven, Germany) was then introduced into the tube and subjected to light polymerization for 40 s using LED light. All ceramic specimens were then placed in an incubator maintained at 100 % humidity and 37 °C for 24 h [30].

Storage conditions

Twenty samples from each cohort were allocated to two subgroups, A (without thermal aging) and B (with thermal aging), based on the storage conditions they were exposed to (n =10)

1. **No exposure to artificial aging (A) (baseline)**
   Submerging discs in distilled water for 14 days
2. **Exposure to artificial aging (B)**

10 000 cycles of thermal exposure were performed in a thermocycler (Mechatronik, Feldkirchen, Westerham, Germany) by submerging the discs in two water tubs maintained at different temperature ranges of 5 – 55 °C for 30 s. The transfer time between the two submergences was 10 seconds [30].

RBS and failure mode assessment

A universal testing machine (UTM) (Instron Corp, USA) was used to obtain the maximum load required to fracture the bond between the composite and zirconia at a rate of 0.2 mm/min using a cylindrical piston. MegaPascals (MPa) were used as the unit of measurement. The specimen surfaces were then analyzed using a stereomicroscope (model SMZ 1500m, Nikon Instech, Kanagawa, Japan) at ×40 magnification to ascertain the type of failure (adhesive, cohesive, and admixed) [31, 32].

Data analysis

The Statistical Package of Social Sciences version 22 (IBM, Armonk, NY, USA) was used to analyze the data. The normality of data was assessed using the Kolmogorov-Smirnov test. Intergroup comparisons were performed using one-way analysis of variance (ANOVA) and Tukey's post hoc test *($p < 0.05$).*

III. Results

Surface topography via SEM

Fig. 1: A) Scanning electron microscopy visualization of zirconia ceramic subjected to sandblasting treatment. The micrograph exhibits minute porosities with a bubble-like morphology on the surface, attributed to the impact of Al_2O_3 nanoparticles during the blasting process. B) SEM micrograph of zirconia surface exposed to CO_2 laser irradiation at 4 W. The image demonstrates surface melting, enhanced roughness, and increased porosity, accompanied by matrix degradation. C) SEM representation of the surface modified with microgrooves activated by low-level laser therapy. The micrograph reveals preserved surface matrix integrity with discernible surface porosities.

Ra assessment

Table 1 presents the mean *Ra* scores of zirconia ceramics when conditioned with different conditioning agents. Group 3 (CO_2 laser-4 W) (1352.61 ± 0.025 µm) exhibited the highest *Ra* values. In contrast, Group 4 (LLLT (MG) (1000.37 ± 0.011 µm) demonstrated lower *Ra*. The intergroup comparison revealed that Group 1 and Group 3 showed no difference in their *Ra* ($p > 0.05$) Group 2 (CO_2 laser-3 W) (1 103.57 ± 0.043 µm) on the other hand exhibited significantly lower scores than Groups 1 (SB) (1 103.37 ± 0.011 µm) and 3 yet higher than Group 4 ($p < 0.05$).

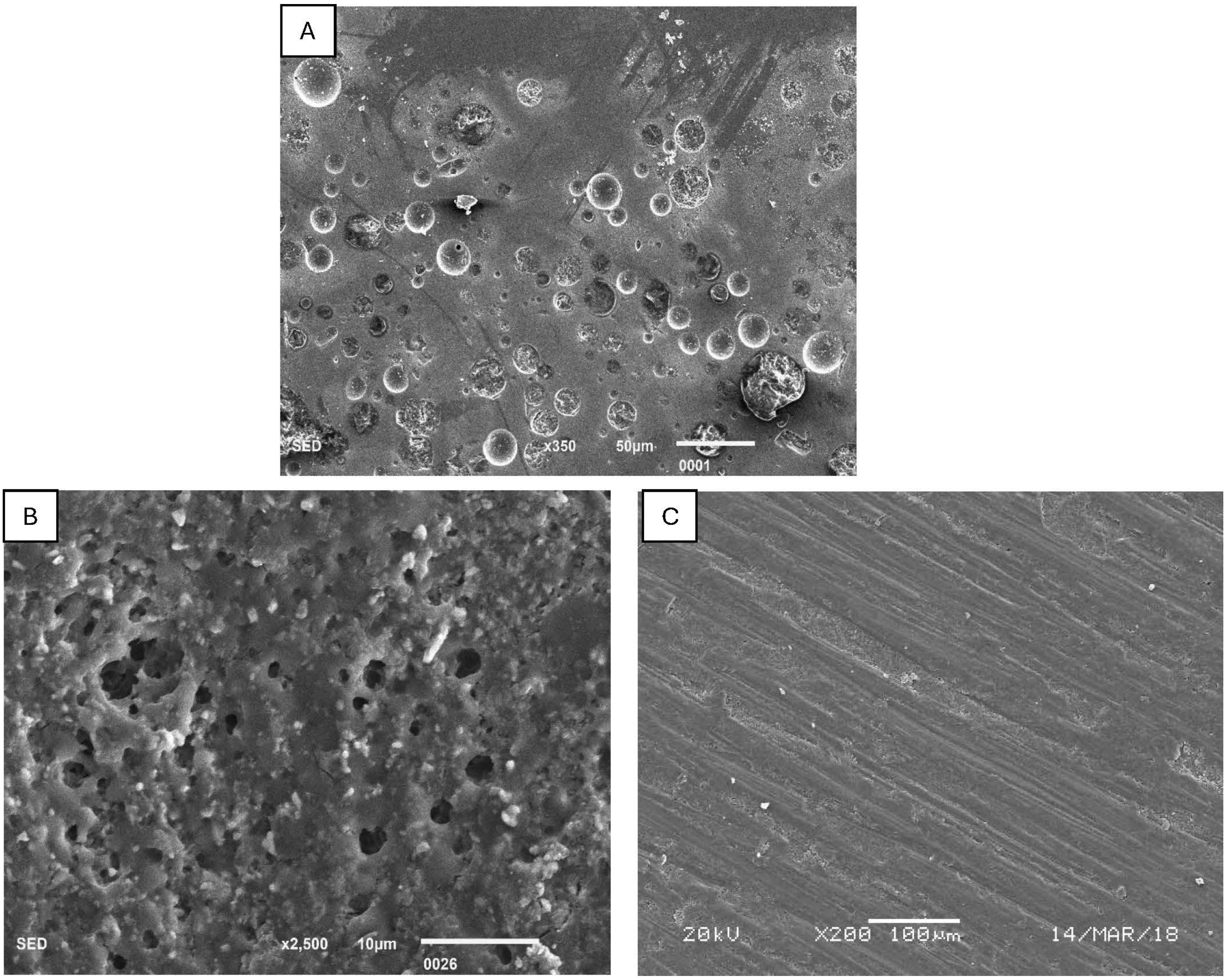

Fig. 1: A) SEM image when zirconia ceramic treated by SB. SEM image shows small pores with bubble appearance on the surface due to the blasting effect of Al_2O_3 nanoparticles. B) SEM image when zirconia surface is treated with CO_2 laser-4 W. Melting and increase in surface roughness and pores are visible with loss of matrix. C) SEM image showing the surface treated with MG activated by LLLT. No loss of surface matrix with visible surface porosities.

Table 1: Surface roughness (*Ra*) on zirconia ceramics after the application of different conditioners.

Investigated groups	*Mean ± SD (μm)*	*p- value!*
Group 1: SB	1 334.15 ± 0.062^{a}	*<0.05*
Group 2: CO$_2$ laser-3 W	1 103.57 ± 0.043^{b}	
Group 3: CO$_2$ laser-4 W	1 352.61 ± 0.025^{a}	
Group 4: LLLT (MG)	1 000.37 ± 0.011^{c}	

Sandblasting (SB), Carbon dioxide laser (CO$_2$), Low-level laser therapy (LLLT), Malachite green (MG)
Different superscript characters denote statistically significant differences! (Post Hoc Tukey Multiple Comparison Test)

Table 2: RBS of zirconia ceramics bonded to composite after the application of different surface modifiers.

Investigated groups	*Before thermo-cycling Mean ± SD (MPa)*	*After thermo-cycling Mean ± SD (MPa)*	*p- value!*
Group 1: SB	14.99 ± 0.12 $^{a, A}$	13.11 ± 0.11^{a,B}	*<0.05*
Group 2: CO$_2$ laser-3 W	13.86 ± 0.07 $^{b, A}$	11.16 ± 0.09$^{b, B}$	
Group 3: CO$_2$ laser-4 W	15.19 ± 0.21$^{a, A}$	13.22 ± 0.32^{a,B}	
Group 4: LLLT (MG)	11.24 ± 0.21$^{c, A}$	9.64 ± 0.82^{c,B}	

Sandblasting (SB), Carbon dioxide laser (CO$_2$), Low-level laser therapy (LLLT), Malachite green (MG)
! ANOVA
Different superscript lowercase alphabets denote statistically significant differences in each column.
Different superscript upper case alphabets denote statistically significant differences in each row(Post Hoc Tukey Multiple Comparison Test)

RBS evaluation

No exposure to artificial aging

Table 2 shows the RBS of zirconia ceramics bonded to composite resin when conditioned with different pretreating agents. Group 3 (CO$_2$ laser-4 W) (15.19 ± 0.21 MPa) revealed the strongest repair bond value. Group 4 (LLLT (MG) (11.24 ± 0.21 MPa) demonstrated the weakest bond. Comparison among different groups revealed that Group 1 (SB) (14.99 ± 0.12 MPa) and Group 3 showed comparable RBS. ($p > 0.05$) Group 2 (CO$_2$ laser-3 W) (13.86 ± 0.07 MPa) on the other hand exhibited significantly lower scores than Groups 1 (SB) (14.99 ± 0.12 MPa) and 3 yet higher than Group 4 ($p < 0.05$)

Exposure to artificial aging

Group 3 (CO$_2$ laser-4 W) (13.22 ± 0.32 MPa) exhibited the strongest repair bond value. Group 4 (LLLT (MG) (9.64 ±0.82 MPa) demonstrated the weakest bond. Comparison among different groups revealed that Group 1 (SB) (13.11 ± 0.11 MPa) and Group 3 showed comparable RBC. ($p > 0.05$) Group 2 (CO$_2$ laser-3 W) (11.16 ± 0.09 MPa) on the other hand exhibited significantly lower scores than Groups 1 (SB) (14.99 ± 0.12 MPa) and 3 yet higher than Group 4 ($p < 0.05$)

Failure mode assessment

Cohesive failure was observed predominantly in the groups treated with SB and CO$_2$ laser at 4 W. In contrast, the other groups exhibited all types of failure (Fig. 2).

IV. Discussion

For the study, it was assumed that modern conditioning methods (CO$_2$ laser with varying power outputs and MG activated by LLLT) would yield a notable difference in *Ra* and RBS of zirconia ceramics compared to composite, relative to the control. Additionally, it was anticipated that specimens subjected to thermal aging would demonstrate a reduced RBS compared with those under baseline conditions (before thermocycling). The selection of CO$_2$ laser energy parameters was informed by previous research findings, and their impact on zirconia‘s mechanical properties was evaluated to determine their effectiveness [33–35].

The CO$_2$ laser was used with different power outputs as it has been observed in the previous available literature that the CO$_2$ laser results in significantly different outcomes when set at different power outputs [36–38] In the present analysis, it was indicated that CO$_2$ laser at a power of 4 W enhances the *Ra* and SBS of zirconia ceramics bonded to a composite compared to control specimens to a significantly higher extent than CO$_2$ laser at a power of 3 W. This finding can be supported by the observation that zirconia ceramic exhibits a higher capacity for absorbing CO$_2$ laser at 4 W. The increase in temperature results in the destruction by generating porosity. This approach enhances the micromechanical interlocking of the resin composite and ultimately strengthens the bond integrity [39,40]. This is in agreement with the outcomes of *in vitro* analysis by Kunt and coauthors [41].

The difference in outcomes at the power of 4 W and 3 W can be explained by the fact that the conversion of light

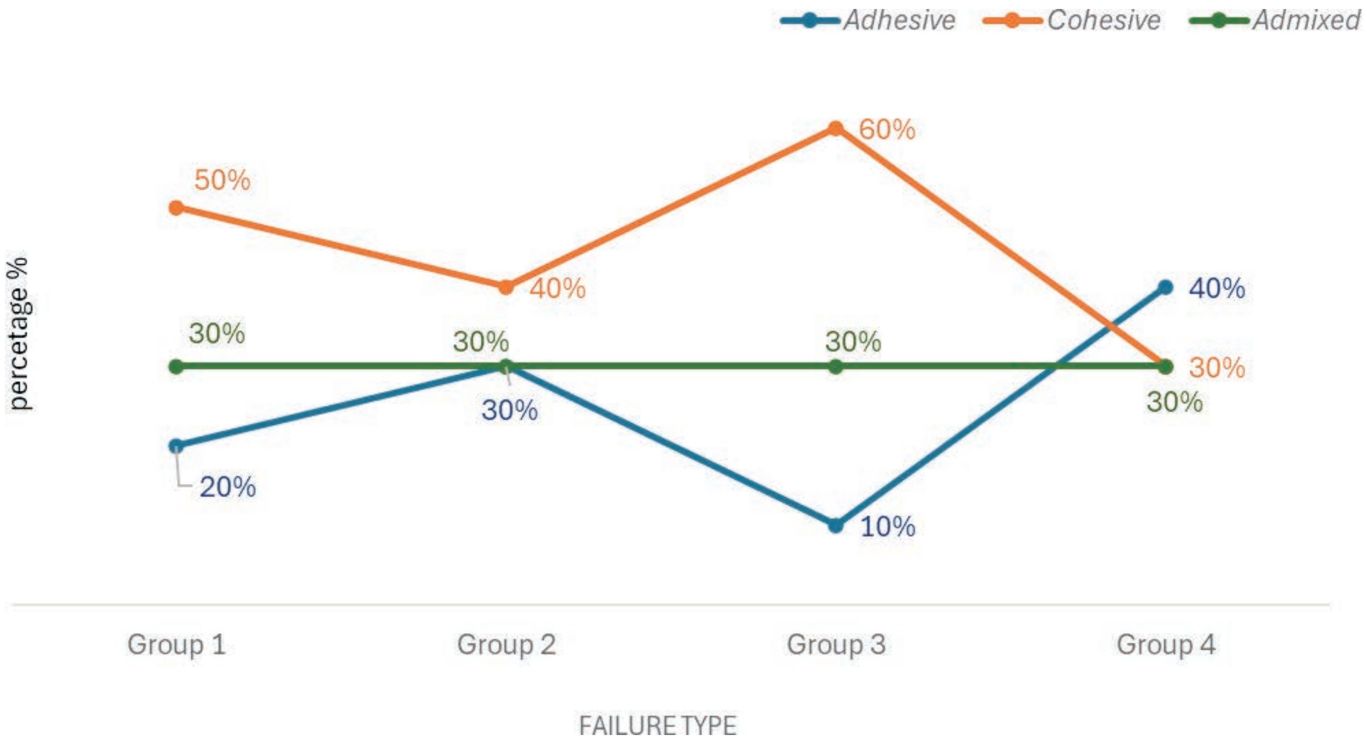

Fig. 2: Modes of failure distribution in different experimental groups.

into heat is the primary effect of laser energy, and the absorption of this energy by the material‘s surface is the most significant interaction between the two. Therefore, the author of the present study has emphasized the need to select the appropriate laser parameters, as this would significantly affect the outcomes [12, 34, 42] The comparable *Ra* and bond scores after SB can be explained by the fact that conditioning with SB enhances *Ra* scores as well as increases the bonding surface area when used for repairing zirconia. Demir and coauthors asserted that micromechanical retention before bonding with the resin cement can be achieved using SB [43]. This also leads to phase transformations from the metastable tetragonal phase to the monoclinic of zirconia, which increases the compressive stress in the outer surface [44]. This stress serves to counterbalance tensile stress that may arise under load, thus enhancing the strength of the material in the layers near the surface [12].

The lowest *Ra* and RBS values were observed in specimens pretreated with MG activated by LLLT. However, this study represents the first investigation to elucidate the effect of MG-activated LLLT on the *Ra* and RBS of zirconia with composites. The researcher formulated a hypothesis based on previous work by Alkhudhairy and Alfawaz [17]. They suggested that water absorption occurs on the hybrid ceramic surface when MG is applied as a surface pretreatment protocol. This water adversely affects the SBS. Moreover, the diminished bond strength may be attributed to the cationic nature of the dye [17, 45].Nevertheless, further research is necessary to corroborate the findings of this study.

Previous studies advocated that artificial aging, which is performed to simulate the oral environment, affects the durability of restorations. Passia *et al.* and Cinar *et al.* proclaimed in their lab-based analysis that bond strength decreases significantly after aging when compared to baseline conditions without aging [46, 47]. It was also reported that post-repair, thermocycling unfavorably impacts adhesion due to mechanical stresses at the bonding interface, stemming from differences in the coefficient of thermal expansion of the materials, alongside hydrolytic degradation of hydrophilic components in adhesives [48].

The failure mode analysis findings revealed that cohesive failure was evident when SB and the CO_2 laser at 4 W were used. This occurrence might be explained by improved surface interaction between the zirconia and composite materials. However, the remaining groups demonstrated mixed failure types. The current study has several constraints. The primary limitation is its laboratory-based nature, which limits its applicability to real-world clinical settings. Varying concentrations of MG and different laser parameters could have produced alternative results. The samples used did not accurately replicate the shape and size of oral structures. Additionally, incorporating both mechanical and thermal aging processes would have more accurately simulated the oral environment. Consequently, further *in vitro* research and clinical trials are necessary to validate these findings.

V. Conclusions

A CO_2 laser with 4 power output can effectively replace sandblasting methods when fixing zirconia ceramics using composites.

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