



Comparison of lithium disilicate–reinforced glass ceramic surface treatment with hydrofluoric acid, Nd:YAG, and CO₂ lasers on shear bond strength of metal brackets

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Abstract

Objectives To evaluate and compare the effects of different surface conditioning methods of lithium disilicate–reinforced ceramic on shear bond strength (SBS) of metallic brackets.

Materials and methods Thirty-six lithium disilicate ceramic blocks mounted in acrylic resin blocks were assigned to 3 groups ($n = 12$): 9.6% hydrofluoric acid (HF); neodymium-doped yttrium aluminium garnet (Nd:YAG) laser; and carbon dioxide (CO₂) laser. The glass ceramic surfaces were primed with a silane, and the brackets were bonded using a light-cured composite resin. SBS test was carried out in a universal testing machine at 0.5 mm/min crosshead speed until the brackets were debonded. The remaining adhesive was evaluated under a stereomicroscope in terms of the adhesive remnant index (ARI). The surface hardness was determined with a 100-gr force using a microhardness tester. Glass ceramic surface changes were evaluated using the scanning electron microscope. One-way ANOVA and post hoc Tamhane tests were used to compare microhardness values, and Kruskal-Wallis and Mann-Whitney U tests were used to analyze SBS values and ARI.

Results The median and interquartile range of SBS values in 3 groups were 6.48 (1.56–15.18), 1.26 (0.83–1.67), and 0.99 MPa (0.70–2.10), respectively. Microhardness analysis revealed significant differences between the CO₂ laser and intact porcelain groups ($P = 0.003$), without significant differences between the other groups. Group 1 exhibited the highest ARI.

Conclusion Neither CO₂ nor Nd:YAG lasers resulted in adequate surface changes for bonding of brackets on ceramics compared with the samples conditioned with HF. CO₂ laser decreased the microhardness of ceramics.

Clinical relevance Surface conditioning with HF resulted in clinically acceptable SBS values.

Keywords Hydrofluoric acid · Nd:YAG laser · CO₂ laser · Orthodontic brackets · Glass ceramics

Introduction

There is ever-increasing demand for adult orthodontic treatment [1]. Such treatment involves bonding of brackets to the surface of different types of dental restorative materials, including ceramics as one of the most commonly used esthetic materials for crowns and bridges [2, 3]. Lithium disilicate is a

relatively strong glass ceramic with a high crystalline content of up to 70%. It exhibits supernatural appearance, translucency, and strength and has clinically been advocated for the fabrication of inlays, onlays, laminate veneers, crowns, and three-unit fixed prostheses up to the premolar region [4–7]. In this context, IPS e.max CAD is a block of lithium disilicate ceramic introduced for the fabrication of esthetic restorations with the use of the computer-aided design and computer-aided manufacturing (CAD/CAM) system [4].

An important challenge in orthodontic treatment is to bond orthodontic brackets to porcelain surfaces because porcelain surfaces cannot be properly conditioned with conventional acid-etching techniques [8]. Optimal bond strength subsequent to surface treatment guarantees resistance against orthodontic forces and at the same time ensures ceramic integrity during debonding of brackets [3, 9]. Surface treatment might be implemented through chemical modification of the surface

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with acid etching and silane coating or using mechanical techniques such as air abrasion with alumina particles or tribochemical silica coating (CoJet) or a combination of these two techniques [5, 10].

Hydrofluoric acid (HF) appears to have the capacity to give rise to proper bond strength [11, 12]. In relation to glass ceramics, hydrofluoric acid etching, followed by the use of a silane coupling agent, has been advocated as a gold standard protocol for achieving a moistened rough surface for proper resin-to-ceramic bonding [13]. However, the technique has some disadvantages. HF is a strong acid and might be toxic to human tissues and irritate them; therefore, careful isolation is necessary in the region of interest, in addition to irrigation in association with a high-volume suction system, followed by immediate drying [14].

Technological advances in the laser field have resulted in an increase in the use of different lasers in dentistry. In this context, different laser types, including neodymium-doped yttrium aluminium garnet (Nd:YAG), erbium-doped yttrium aluminium garnet laser (Er:YAG), and carbon dioxide (CO₂) lasers, have been applied in orthodontics to condition porcelain surfaces to prepare them for bonding brackets; however, contradictory results have been reported [15]. Poosti et al. reported that Nd:YAG lasers can be applied as an acceptable substitute for hydrofluoric acid [11]. This laser has a solid active medium, delivering beams at a wavelength of 1064 nm, properly absorbed by water and pigmented tissues [16].

Ahrari et al. reported that CO₂ laser at 10- and 15-W power resulted in stronger bond strength in feldspathic porcelains compared with HF [8]. The active component of this laser consists of a gas, and its beam wavelength is 10,600 nm with proper absorption by water and hydroxyapatite. In addition, this wavelength is properly absorbed by porcelain and is capable of creating porosities using superficial heat; such micro-porosities improve the mechanical bond between the resins and ceramic surfaces [16].

Silane primers can increase the surface wettability of porcelains; therefore, they can improve the bond strength between composite resin and ceramic surfaces [14]. Studies have shown that silane coupling agents can increase the bond strength of brackets to porcelain; however, there is the risk of cohesive failure during the debonding procedure [1].

Considering the controversies over the use of lasers to prepare porcelain surfaces for bonding procedures as a result of the use of different approaches and techniques, this study was undertaken to evaluate and compare the effects of three different surface treatment techniques (HF + silane, Nd:YAG laser + silane, and CO₂ laser + silane) on the shear bond strength (SBS) of metallic brackets bonded to lithium disilicate-based ceramics and to determine the mode of failure after debonding.

Materials and methods

A total of 36 lithium disilicate-based all-ceramic blocks (IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) were included in this experimental in vitro study; the blocks, measuring 6 mm in diameter and 3 mm in thickness, were fabricated according to the manufacturer's instructions and mounted in self-curing acrylic resin (Acropars, Marlic, Tehran, Iran).

Then the samples were randomly assigned to 3 groups ($n = 12$) in terms of surface conditioning applied. In group 1, the ceramic block surfaces were etched with 9.6% HF acid (porcelain etch gel, Pulpdent Corp., Watertown, USA) for 2 min. The gel was removed with a cotton roll, followed by rinsing for 2 min and drying with oil-free air for 15 s.

In group 2, Nd:YAG laser beams (Lucid Q-PTP, Seoul, Republic of Korea) were used for surface conditioning under a glass shield. The laser beams consisted of photons at a wavelength of 1064 nm that were pulsed, with duration of 100 μ s and a repetition rate of 20 Hz. The laser beams were delivered perpendicular to the target area at a 1-mm distance in 10 s, with a beam spot size of 3 mm² and an energy density of 2 J/cm², using a sweeping motion. Subsequently, the samples were rinsed for 15 s and dried for 15 s.

In group 3, CO₂ laser beams (Deka, Calenzano, Italy) were used for surface conditioning under a glass shield. The laser beams consisted of photons at a wavelength of 10,600 nm that were pulsed with a repetition rate of 200 Hz and power of 5 W. The beams were delivered perpendicular to the target area at a 2-mm distance for 10 s, using a sweeping motion. Subsequently, the samples were rinsed for 15 s and dried for 15 s.

Subsequent to glass ceramic surface conditioning, a silane coupling agent (Bond Enhancer, Pulpdent Corp., Watertown, USA) was applied to ceramic surfaces with the use of a brush and allowed to dry for 60 s, followed by air drying for 30 s.

A total of 36 stainless steel standard edgewise maxillary central incisor brackets with a slot size of 0.018 in (Ortho Organizers, Carlsbad, CA, USA) and a base surface area of 13.11 mm² were used in this study. The brackets were bonded to glass ceramic surfaces with the use of a light-cured composite resin (Transbond XT, 3M Unitek, CA, USA). Excess composite resin was removed with a dental explorer, and the samples were light-cured for 20 s using a light-curing unit (Ortholux LED Curing Light, 3M Unitek, CA, USA) with a light power of 450 mW/cm² and 1-mm distance of light cure source to the brackets during curing.

In the next stage, all the samples were incubated in distilled water at 37 °C for 24 h, followed by 5000 rounds of thermocycling in water baths (Delta Tpo2, Nemo, Iran) at 5 °C/55 °C. The samples were coded and blinded to the operator.

In each group, SBS was evaluated using a universal testing machine (K-21046, Walter + bia, Lohningen, Switzerland) at a crosshead speed of 0.5 mm/min. The shear force was applied with the use of a beveled flat-end shearing blade with the force delivered parallel to the glass ceramic–bracket interface and until debonding occurred. The maximum shear bond strength of each sample was measured in N (newton) and then divided by the cross-sectional area of the bracket (11.13 mm²) to obtain the shear bond strength in MPa.

The bond failure site for each bracket was evaluated under a stereomicroscope (SM P200, HP, USA) at $\times 10$ magnification and classified according to adhesive remnant index (ARI) (Artun and Bergland) [17]:

- Grade 0: no adhesive remnants on the ceramic surface
- Grade 1: adhesive remnants covering $\leq 50\%$ of the ceramic surface
- Grade 2: adhesive remnants covering $> 50\%$ of the ceramic surface
- Grade 3: all the adhesive left behind on the ceramic surface

Microhardness test was carried out using a digital microvickers hardness tester (Microet, Buehler, Tokyo, Japan) at 100-gr force for 20 s. A pyramidal point was applied, and a diagonal length of indentation was measured [18] Five samples from each group were randomly selected for 3 VH measurements per ceramic sample. Microhardness of 5 samples that did not undergo any surface treatment was measured as controls. The mean value of each specimen was compared with the mean value of the control group.

Scanning electron microscope (SEM) analysis was used to compare surface microstructures after bracket debonding between the three groups. A random sample from each group was sputter coated with gold and examined under a scanning electron microscope (INCAx-sight, England) at $\times 2000$ magnification.

We should check model assumptions before performing parametric tests. For this purpose, we used Kolmogorov-Smirnov and Shapiro-Wilk test in order to find whether our data have normal distribution. We also performed these tests on residuals. Both tests showed that ARI scores and bond strength did not have normal distribution. Also their residuals were not normal too (P value < 0.001). However, microhardness and its residuals had normal distribution according to Kolmogorov-Smirnov test (P value = 0.060) and Shapiro-Wilk test (P value = 0.106). We also used Levene's test to check homogeneity of variances. We realized that ARI score and bond strength do not have homogeneous variances (P value < 0.001). However, we had homogeneous variances in microhardness among groups (P value = 0.051).

Kruskal-Wallis and Mann-Whitney U tests were used to analyze bond strength values and ARI. One-way ANOVA

and post hoc Tamhane tests were used to compare surface hardness values. All the statistical analyses were performed with SPSS 22. Statistical significance was set at $P < 0.05$.

Results

According to Table 1 and Fig. 1, the median and interquartile range of shear bond strength in the HF, Nd:YAG laser, and CO₂ laser groups were 6.48 (1.56–15.18), 1.26 (0.83–1.67), and 0.99 (0.70–2.10) MPa, respectively. Kruskal-Wallis test showed significant differences between the three groups ($P = 0.009$). In addition, Mann-Whitney U test showed a significant difference between the HF and Nd:YAG laser groups ($P = 0.009$) and between the HF and CO₂ laser groups ($P = 0.008$). There was no significant difference between the laser groups ($P = 0.817$).

Table 2 shows ARI scores of the groups. Kruskal-Wallis test revealed significant differences between the three groups ($P < 0.001$). Furthermore, Mann-Whitney U test showed a significant difference between the HF and Nd:YAG laser groups ($P < 0.001$) and between the HF and CO₂ laser groups ($P < 0.001$). There was no significant difference between the laser groups ($P = 1.00$).

Twelve randomly selected samples were reevaluated by the same observer after 1 week and the Kappa test was used to examine intra-examiner reliability (Kappa value = 0.96). Inter-examiner reliability of ARI was evaluated by Cohen's Kappa test (Kappa value = 0.92).

Table 3 and Fig. 2 show the mean \pm SD of microhardness values in the three study groups. The control and CO₂ laser group exhibited the highest and lowest mean microhardness values, respectively. Tamhane test revealed a significant difference between the CO₂ laser and control groups ($P = 0.003$). There were no significant differences between the other paired groups.

Figures 3, 4, and 5 demonstrate SEM photographs of glass ceramic surfaces conditioned with 9.6% HF, Nd:YAG laser, and CO₂ laser, respectively. Glass ceramic samples treated with HF exhibited a homogeneously rough pattern and exposed ceramic crystals. Surface treatment with Nd:YAG laser resulted in small homogeneous depressions and mesh-like surfaces. The CO₂ laser-irradiated glass ceramic surfaces exhibited pores, fissures, and microcracks.

Table 1 The shear bond strength values (MPa) of metal brackets bonded to lithium disilicate glass ceramic surfaces in three different treatment groups

Groups	<i>N</i>	Median	Interquartile range	Minimum	Maximum
HF	12	6.48	13.62	0.28	16.22
Nd:YAG	12	1.26	0.84	0.00	6.69
CO ₂	12	0.99	1.40	0.60	3.66

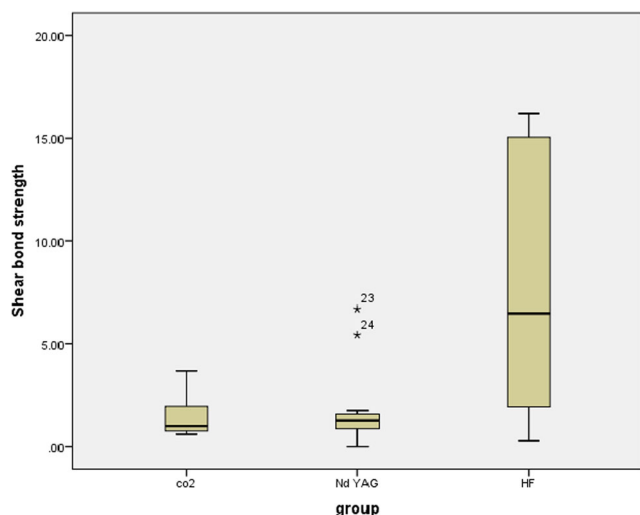


Fig. 1 Box plot of shear bond strength values of brackets bonded to lithium disilicate-based ceramic in the three treatment groups

Discussion

Patients’ increasing demands for esthetic procedures has resulted in the emergence of lithium disilicate crowns as a solution for esthetic and functional problems in prosthodontics [15]. This dental material consists of lithium silicate with micron-size lithium disilicate crystals in between, composed of submicron lithium orthophosphate crystals, giving rise to a highly filled glass matrix. Such a modification in the shape and volume has led to an increase in the material’s flexural strength up to approximately 360 MPa [6, 19].

Various mechanical (roughening the surface with a diamond bur or microetching with aluminum oxide particles) and chemical (etching with hydrofluoric acid or phosphoric acid, tribochemical silica coating, and use of silane coupling agents) techniques have been introduced for surface conditioning so that an effective bond can be achieved between stainless steel orthodontic brackets and porcelain surfaces [20].

Some studies found that enamel air abrasion followed by acid etching resulted in significantly higher SBS compared with acid etching or air abrasion alone [21, 22]. It has been reported that mechanical roughening of porcelain by air

Table 2 Descriptive statistics of ARI scores of lithium disilicate glass ceramic surfaces in the 9.6% HF, Nd:YAG laser, and CO₂ laser groups

Group	Adhesive remnant index (ARI) scores				
	0	1	2	3	Total
HF	3	2	1	3	12
Nd:YAG	12	0	0	0	0
CO ₂	12	0	0	0	0

Table 3 Vickers microhardness values of lithium disilicate glass ceramic in the 9.6% HF, Nd:YAG laser and CO₂ laser groups, and the control group

Groups	N	Mean	SD	Minimum	Maximum
HF	5	622.79	97.21	474.56	737.96
Nd:YAG	5	606.00	138.79	462.30	767.56
CO ₂	5	480.43	43.21	418.00	538.10
Control	5	624.23	33.08	588.33	666.36

abrasion or by diamond stone increases SBS significantly, but it also results in a higher risk of crack initiation and propagation within the porcelain [23, 24]

In the present in vitro study, the effect of three surface conditioning techniques was evaluated on shear bond strength of metallic orthodontic brackets to lithium disilicate-based ceramic surfaces.

According to previous studies, usage of hydrofluoric acid results in a significant increase in the bond strength. The mechanism of action of HF on lithium disilicate glass ceramic depends on the ability of the acid to react with the silica phase to achieve micromechanical retention through microporosities. As a result, the glassy matrix dissolves partially, exposing lithium disilicate crystals, which increases the formation of retentive channels. This gives rise to an increase in surface area, improving the quality of the bond [7, 20]. However, it has been reported that HF is highly toxic, reactive, and corrosive [25].

Application of different laser types, including Nd:YAG, Er:YAG, and CO₂ with different settings, has been suggested in order to circumvent such limitations [16]. Several studies evaluated the effects of laser pretreatment of enamel surfaces on SBS of brackets [21]. Some studies concluded that there is

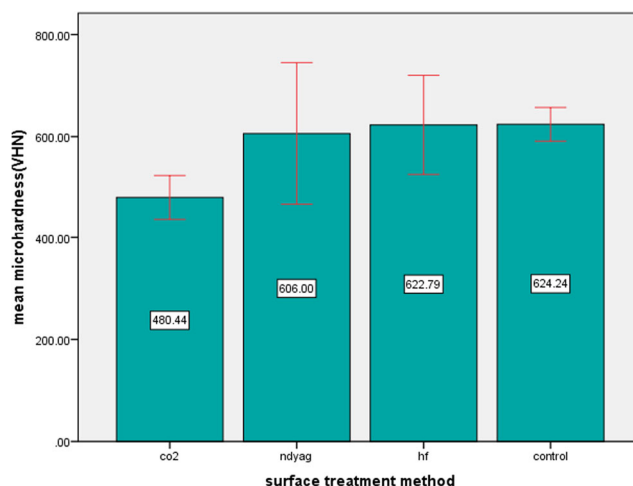


Fig. 2 Means and standard deviations of microhardness test values on lithium disilicate-based ceramic in the three treatment groups and the control group

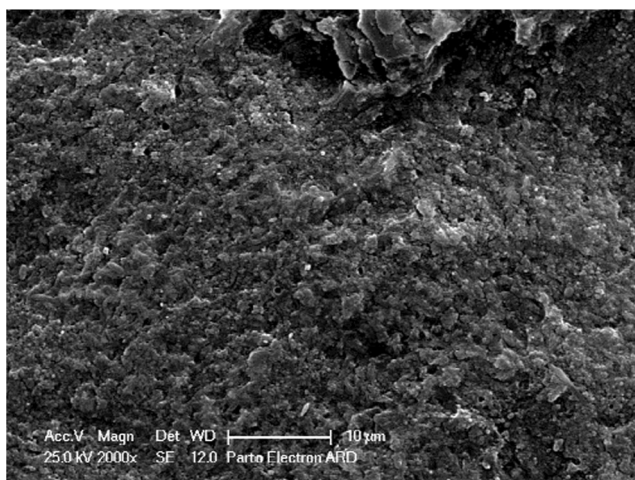


Fig. 3 SEM images of glass ceramic surfaces conditioned by 9.6% HF

no significant differences in the mean SBS of the Er:YAG laser group and acid-etched group [26]. Some studies have demonstrated that Nd:YAG laser with appropriate power settings can be used as an alternative method for etching the porcelain surface [6, 11]. Nd:YAG laser beams are absorbed by hard tissues, modifying the surface characteristics. This laser creates surface roughness by melting and random recrystallization, improving the resin–ceramic bond strength [16]. It has been reported that the CO₂ laser is a proper choice for modifying porcelain surfaces because its beams are almost completely absorbed by the porcelain. During induction of heat on the porcelain surfaces with focused CO₂ laser beams, conchoidal tears (typically resulting from surface heating) appear on the surface. The tears are thought to provide mechanical retention between the composite resin and the porcelain surface [12].

In the present study, a silane was used to prime the glass ceramic surfaces subsequent to surface conditioning.

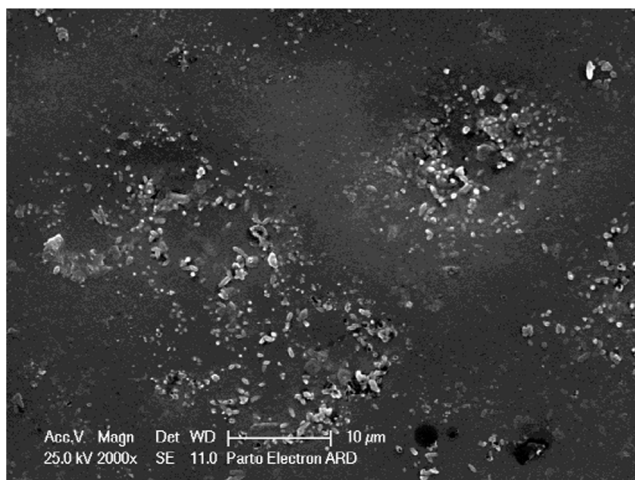


Fig. 4 SEM images of glass ceramic surfaces conditioned by Nd:YAG laser

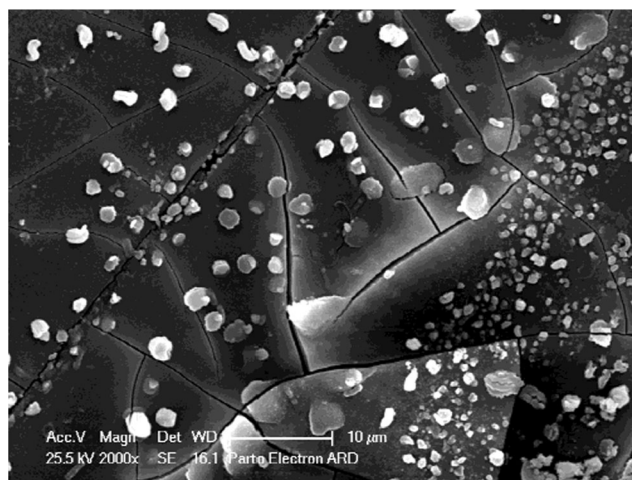


Fig. 5 SEM images of glass ceramic surfaces conditioned by CO₂ laser

Application of silanes can improve the bond strength at bracket–ceramic surface interface because silanes can form chemical bonds with inorganic and organic surfaces [27]. A silane coupling agent fuses the silica unit in the ceramic with the methacrylate monomers in the adhesive [9].

Orthodontic brackets bonded to ceramic surfaces might exhibit failure at the ceramic–bonding agent interface in the oral cavity as a result of thermal changes and heavy orthodontic forces applied by the archwire during orthodontic movements. Thermocycling regimens between 500 and 7000 rounds have been applied to verify whether temperature fluctuations are able to increase stresses in the light-cured materials in the simulated oral conditions before mechanical tests. Some studies have shown that thermocycling might exert negative effects on shear bond strength [14, 27]. In the present study, samples underwent 5000 thermal cycles in a thermal cycler at 5 °C/55 °C, which might have played a role in decreasing the shear bond strength.

SBS is the principal factor used to evaluate bonding agents [28]. The ability of adhesive resins to bond to tooth structure or to a second restorative material is measured by SBS. This almost simple procedure employs a chisel-shaped tool mounted in a universal testing machine to fracture a disc of bonded material from the bonding substrate by force [29]. It has been reported that a minimum bond strength of 6–8 MPa is necessary for efficient clinical orthodontic bonding [30], whereas 13 MPa has been reported to be the maximum permissible bond strength between porcelain and the adhesive to avoid cohesive porcelain failure [31]. Therefore, in the present study, only the HF group exhibited proper orthodontic bonding to lithium disilicate–based porcelain. Cevik reported that in the feldspathic and lithium disilicate ceramic systems, surface conditioning with Nd:YAG laser, followed by a 2500-round thermocycling procedure, resulted in clinically unfavorable shear bond strength, consistent with our results. In our study, the minimum values of SBS were far below of 6–8 MPa

in all 3 groups; therefore, even in HF group, some samples did not show sufficient SBS.

The highest ARI score was detected in the HF group (50% of samples had score 3), which also exhibited the maximum SBS. In both laser groups, ARI values indicated the predominance of debonding failures with a score of 0, which means no bonding resin was observed on the ceramic surface. It can be concluded that HF gave rise to better wettability of the glass ceramic surface compared with the laser groups.

In the current study, the effect of three surface conditioning techniques on the microhardness values of lithium disilicate-based ceramics was compared with a control group. The base of Vickers microhardness test is to use the indenter under a certain force to the pyramidal contact area of the indentation [32]. The thickness of the ceramics used in this study was designed to be as close to that in the clinical practice as possible. According to our findings, 5-W CO₂ laser beams decreased the microhardness of lithium disilicate-based ceramic compared with the control group, while HF and Nd:YAG laser groups exhibited no significant difference from the control group. It seems that microcracks on the glass ceramic surfaces conditioned with CO₂ laser may have resulted in a decrease in hardness values. Gamal reported that 5-W CO₂ laser beams increased the microhardness of lithium disilicate ceramics, whereas CO₂ laser beams at 10 W or Nd:YAG laser beams did not change microhardness [18].

SEM images showed that HF interacted with lithium disilicate ceramic surface and dissolved surface substances, producing surface porosities, which resulted in clinically acceptable shear bond strength values.

Surface conditioning with 5-W CO₂ laser beams resulted in non-retentive pores and cracks, which might have contributed to a decrease in microhardness values of lithium disilicate-based ceramics compared with other groups; 2-W Nd:YAG laser beams gave rise to shallow surface porosities with a minor role in mechanical retention.

Josko et al. reported that Er:YAG and Nd:YAG laser beams did not result in adequate surface modifications for bonding of orthodontic brackets to glazed lithium disilicate ceramic surfaces compared with the control group in which 9.5% HF was used, consistent with our results [15]. Rocca reported that CO₂ and Nd:YAG laser beams induced chemical and physical surface modifications in ceramics, indicating the possibility of an improvement in the bonding of the ceramics evaluated, which is different from our results [6]. However, these two studies used SEM analyses and did not evaluate the shear bond strength.

In this study, the samples were etched with HF for 2 min. Some studies reported that there were no significant differences in SBS values according to conditioning time with HF from 20 to 120 s for feldspathic porcelains and glass ceramics [33, 34]. Ahrari et al. found that surface conditioning with 9.6% HF for 2 min resulted in acceptable SBS of resin cement to a lithium disilicate glass ceramic [4]. Straface et al.

concluded that the highest SBS values of CAD/CAM materials bonded to composite resin cements were achieved with 15 to 60 s etching time with 5 and 9% HF [35]. HF is a hazardous acid and prolong etching time might increase the risk of intraoral exposure. This factor should be taking into consideration in future studies.

In the present study, SBS values were not tested 24 h after bonding. Therefore, these values were not available as a baseline to compare SBS before and after thermocycling. The complexity of the oral cavity and variables such as temperature, stress, humidity, acidity, and plaque might interfere with determination of proper orthodontic bonding in vitro [36]. Therefore, it is necessary to evaluate the bond strength of stainless steel orthodontic brackets to ceramics in situations as similar to the oral cavity as possible. Another limitation of this study is the small sample size. Future studies with larger sample size, different types of lasers with different parameters, and on different dental porcelains are suggested.

Conclusion

Under the limitations of the present in vitro study, it can be concluded that:

1. Application of 9.6% HF on the lithium disilicate ceramic surface resulted in clinically acceptable shear bond strength values.
2. Neither CO₂ nor Nd:YAG laser beams provided adequate surface treatment for bonding of orthodontic brackets to lithium disilicate ceramics compared with samples conditioned with 9.6% HF, and application of these two lasers with the mentioned physical parameters for metallic bracket bonding does not seem to be rational.
3. Bond failure in both laser groups occurred at resin-porcelain interface, while failure in the HF group occurred at bracket-adhesive interface in most samples, indicating a lack of adequate bond strength between the porcelain and adhesive in both laser groups.
4. CO₂ laser with the mentioned parameters decreased the microhardness of lithium disilicate ceramic surface.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent For this type of study, formal consent is not required.

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