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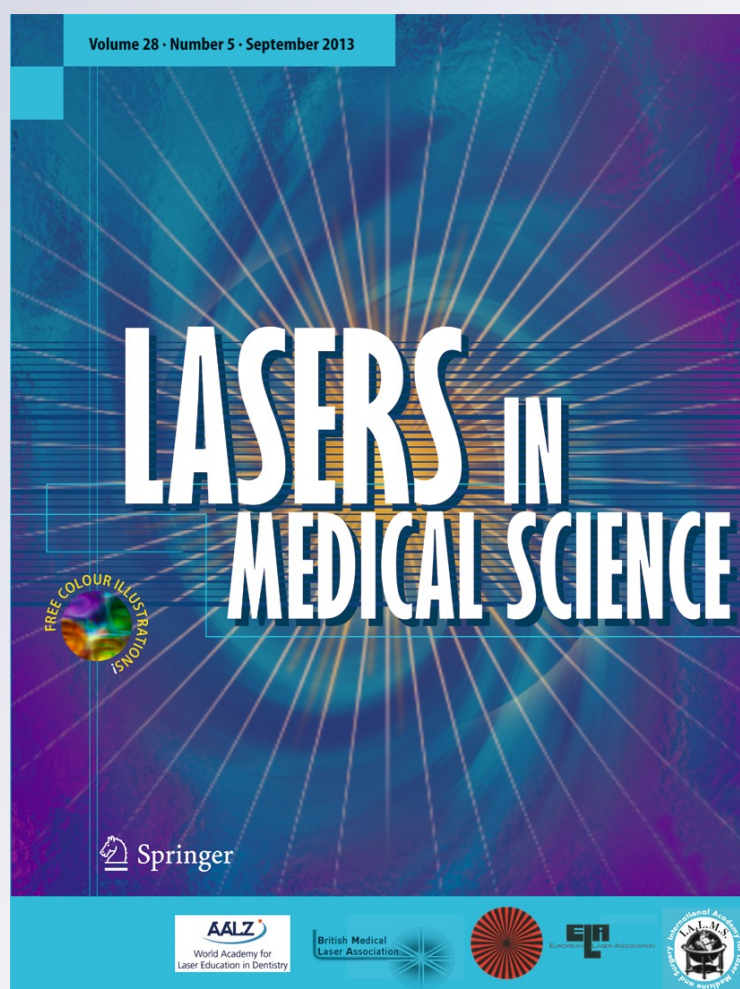
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Effects of different surface treatments on shear bond strength in two different ceramic systems

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Abstract The purpose of this study was to evaluate the influence of different surface treatments (sandblasting, acid etching, and laser irradiation) on the shear bond strength of lithium disilicate-based core (IPS Empress 2) and feldspathic ceramics (VITA VM 9). One hundred ceramic discs were divided into two groups of 50 discs each for two ceramic systems: IPS Empress 2 (group I) and VITA VM 9 (group II). Each of the two groups was further divided into five surface treatment groups (ten each) as follows: group SB, sandblasting with alumina particles (50 μm); group HF, 5 % hydrofluoric acid etching; group L, Er:YAG laser irradiation (distance, 1 mm; 500 mJ; 20 Hz; 10 W; manually, noncontact R14 handpiece); group SB–L, sandblasting + Er:YAG laser; and group HF–L, 5 % hydrofluoric acid + Er:YAG laser. Luting cement (Panavia 2.0) was bonded to the ceramic specimens using Teflon tubes. After 24 h of water storage, a shear bond strength test was performed using a universal testing machine at a crosshead speed of 0.5 mm/min. The data were analyzed with a two-way analysis of variance (ANOVA) and Tukey's honestly significant difference tests ($\alpha=0.05$). The two-way ANOVA indicated that the shear bond strength was significantly affected by the surface treatment methods ($p<0.05$), but there was no significant interaction between the ceramic systems. Group

SB–L had the highest mean values for each ceramic system. Sandblasting, followed by Er:YAG laser irradiation, enhanced the bond strength, indicating its potential use as an alternative method. The atomic force microscopic evaluation revealed that group SB had the most distinct sharp peaks among the groups.

Keywords Bond strength · Luting cements · Surface treatment · Ceramic · Er:YAG

Introduction

Ceramics are still the most aesthetically pleasing materials available for prosthetic dentistry because of their high resistance to abrasion, compression, chemical stability, biocompatibility, acceptable translucence, and fluorescence characteristics [1]. Feldspathic ceramics (silica-based ceramic) are the conventional ceramic materials for metal–ceramic restorations and consist of a mixture of feldspar and quartz [2]. They are frequently used to veneer metal frameworks and indirect restorations, such as inlays, onlays, and laminates. All-ceramic restorations have gained popularity among dentists and patients because of their favorable aesthetics and liability in conservative tooth preparations [3].

A lithium disilicate glass–ceramic core veneered with a sintered glass–ceramic provides higher strength and allows for the fabrication of short-span fixed dentures [4].

Some clinical studies have indicated that an insufficient luting performance of restorations may result in clinical failure [5, 6]. Obtaining desirable adhesion between cement and ceramic surfaces requires surface pretreatment to improve the retention, marginal adaptation, and fracture resistance of restorations [7, 8]. Surface pretreatment of porcelain increases surface area and creates microporosities on the surface, enhancing the potential for mechanical retention of the cement [9]. Different surface treatment

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methods (grinding, abrasion with diamond rotary instruments, airborne particle abrasion with Al_2O_3 , acid etching, sandblasting, and combinations of any of these methods) have been proposed to provide roughness and promote micromechanical retention [10].

Some authors declared that enhancing the roughness of a ceramic surface with hydrofluoric (HF) acid and then applying a silane coupling agent is the suggested surface treatment method for feldspathic ceramics [11, 12]. Conversely, for some conditions, such as when intraoral ceramic repair is needed, the use of acid etching is not recommended because of its harmful and irritating effects on oral tissues [13]. Other surface roughness methods can be assumed for this reason to induce similar or greater surface roughness on porcelains.

Another promising technique for the surface treatment of ceramic materials is laser irradiation [14]. Among the various laser types, the Er:YAG laser is highly recommended for use on the surfaces of dental materials, but there is no consensus in the literature regarding the best surface-conditioning method to produce optimal bond strength based on the cement and porcelain used [15].

Therefore, the purpose of this *in vitro* study was to investigate and compare the effects of Al_2O_3 sandblasting, HF acid etching, and their use in combination with ER:YAG laser irradiation on the surface roughness and shear bond strengths of lithium disilicate-based and feldspathic ceramics. The null hypothesis was that Er:YAG laser treatment would result in surface roughness and bond strength that is comparable with that of air abrasion and acid-etching surface treatment methods.

Materials and methods

Specimen preparation

One hundred ceramic discs were divided into two groups of 50 each for two ceramic systems: IPS Empress 2 (group I; Ivoclar Vivadent, Amherst, NY, USA) and VITA VM 9 (group II; VITA VM 9, VITA Zahnfabrik H Rauter GmbH & Co. KG, Bad Säckingen, Germany).

For group I, to obtain 50 lithium disilicate-based core ceramic discs (diameter, 10 mm; thickness, 1 mm), IPS Empress 2 wax patterns were prepared and invested in the IPS Empress 2 Speed investment (Ivoclar Vivadent, Amherst, NY, USA). The wax was eliminated in a burnout furnace (EP 600; Ivoclar Vivadent, Amherst, NY, USA) preheated to 850 °C with an alumina plunger for 90 min. The IPS Empress 2 ingots were softened at 920 °C and automatically pressed into the mold in a furnace.

After pressing and cooling to room temperature, the specimens were divested with 125- μm glass beads at a pressure of 4 bars, ultrasonically cleaned in a special liquid

(Invex liquid, Ivoclar-Vivadent) for 10 min, washed in running water, and dried. They were then subjected to airborne particle abrasion with 50- μm Al_2O_3 particles at a pressure of 2 bars (Korox, Bego, Bremen, Germany).

For group II, to obtain 50 feldspathic ceramic discs (diameter, 10 mm; thickness, 1 mm; VITA VM 9), metal molds with disc-shaped holes were used, and an impression of the metal mold was made with silicon putty (Virtual vinyl polysiloxane, Ivoclar, Schaan, Liechtenstein). The refractory die material (VITA Zahnfabrik H Rauter GmbH & Co. KG) was then poured into the silicon putty. Veneering porcelain powder (VITA VM 9 Powder, VITA Zahnfabrik H. Rauter GmbH & Co.) was mixed with the manufacturer-supplied condensing liquid and condensed using the vibration blotting technique. The obtained slurry was blotted with tissue to eliminate excess water and then condensed into the mold. The prepared discs were fired in a programmable vacuum porcelain furnace (VITA Vacumat 4000 Premium T; VITA Zahnfabrik H Rauter GmbH & Co. KG) in accordance with the firing programs provided by the manufacturer. No glaze was applied to the ceramic surface of the discs.

The bonding surfaces of the specimens in the two groups were polished using silicon carbide paper (grit 800) under water cooling and then polished with OpraFine Assortment (Ivoclar) for standardization. The surfaces were cleaned with ethanol and dried carefully in air before surface treatment. After the finishing procedures, the discs were subjected to ultrasonic treatment (Biosonic JR, Coltene Whaledent) in 99.5 % acetone to remove any surface residues and were dried. After these procedures, the specimens in both groups were further divided into five surface treatment groups of ten discs each.

Group SB (sandblasting)

For both groups, the ceramic surfaces were abraded with 50- μm Al_2O_3 particles at a pressure of 2.8 bars from a distance of 10 mm perpendicular to the treated surface for 20 s.

Group HF (acid etching)

For group I, the ceramic surfaces were etched with 5 % HF acid (IPS Ceramic Etching Gel, Ivoclar Vivadent) for 20 s; the gel was rinsed off with water for 20 s, and then the surfaces were dried under oil-free compressed air for 20 s. Similar procedures were performed for group II at 60 s for each procedure.

Group L (Er:YAG laser irradiation)

An Er:YAG laser (Fotona, At Fidelis, Ljubljana, Slovenia) was used to irradiate the ceramic surfaces in both groups. A contact handpiece (R14; 1.3 mm in diameter) with an

integrated spray nozzle was placed perpendicular to the ceramic surface at a 1-mm distance, and the entire ceramic area was manually scanned with water cooling for 20 s. The laser irradiation of all the specimens was performed by the same operator. The laser parameters were as follows: 500 mJ (pulse energy), 10 W (power), MSP mode (100- μ s pulse length), 20 Hz (pulses per second), and 37.68 J/cm² (energy density).

Group SB-L (sandblasting + Er:YAG laser)

For both groups, the ceramic surfaces were abraded using the same parameters described for group SB. After the sandblasting procedure, all the ceramic surfaces were irradiated with an Er:YAG laser using the same parameters described for group L.

Group HF-L (acid etching + Er:YAG laser)

For both groups, the ceramic surfaces were etched with 5 % HF acid using the same procedure described for group HF. The Er:YAG laser irradiation was performed using the same parameters described for group L.

After all the surface treatments, a silane-coupling agent (Clearfil Ceramic Primer, Kuraray, batch no. 00001A) was applied to the ceramic surface of all the specimens in both groups using a clean brush, and then the specimens were air dried using oil-free compressed air. To apply the resin cement, each treated ceramic was initially placed in its silicone mold. The resin cement (Panavia F 2.0, Kuraray Medical Inc., Osaka, Japan) was then mixed according to the manufacturer's instructions and injected into the Teflon tubes with an internal diameter and height of 4 and 3 mm, respectively. The bonding procedures were performed according to the manufacturer's recommendations. Curing light was applied to the top of the filled molds for 40 s (Bluephase, Ivoclar Vivadent). An oxygen-inhibiting gel (Oxyguard II, Kuraray) was applied to the exposed surfaces. After careful removal of the Teflon molds, every side of the cement cylinder was light cured for a total of 80 s. The bonded specimens were then stored in distilled water at room temperature for 24 h.

Evaluation of shear bond strength

The samples were placed into the test device using special acrylic molds. The knife-edge-shaped apparatus was placed between the joint of the ceramic disc and the resin material. The shear bond strength of the ceramic discs was evaluated using a universal testing machine (TSTM 02500, Elista Ltd Sti, Istanbul, Turkey) with a crosshead speed of 0.5 mm/min. The value at which the ceramic disc and the resin material ruptured was recorded (Fig. 1).

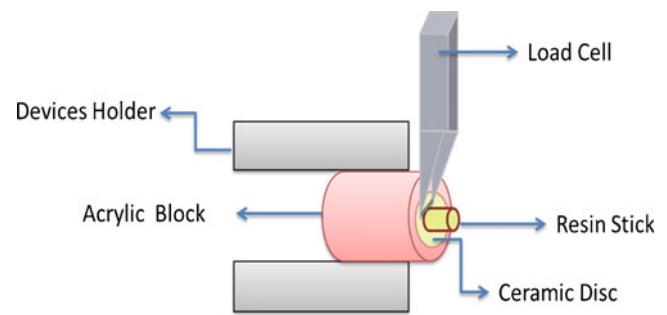


Fig. 1 Scheme of the test sample

After the shear bond strength test, ceramic bonding areas were observed with a stereomicroscope (Olympus CX41, $\times 40$, Japan) to identify the mode of fracture. The fractured surface was classified according to one of three types: type 1, adhesive failure between the resin cement and the ceramic; type 2, cohesive failure in the ceramic; and type 3, cohesive failure in the resin cement. The data were analyzed by two-way analysis of variance (ANOVA) and Tukey's honestly significant difference (HSD) tests (SPSS/PC version 17.0, SPSS, Chicago, IL, USA) for pairwise comparisons among the groups ($p < 0.05$).

Atomic force microscopic evaluation

One additional specimen from each group was evaluated by atomic force microscopy (AFM; NTEGRA Solaris, NTMDT, Russia). The digital images were obtained in air. A 0.01–0.025- Ω -cm gold-doped silicon tip (40 μ m) was used in the noncontact mode. The changes in the vertical position provided the height of the images, and these changes were registered as the bright and dark regions. A constant tip sample “tap” was maintained through a constant oscillation amplitude (set point amplitude). Five 25 \times 25- μ m digital images were acquired for each surface and recorded with a slow scan rate (1 Hz).

Results

Shear bond strength

The mean and SD bond strength values for each ceramic material in the five surface treatment groups are given in Table 1. As shown in Table 1, the two-way ANOVA indicated that shear bond strength was significantly affected by the surface treatment methods ($p < 0.05$) and that there was no significant interaction between the two ceramic materials ($p > 0.05$; Table 2). Tukey's HSD test results on shear bond strength of the lithium disilicate-based ceramic revealed significant differences among the groups ($p < 0.05$).

Table 1 Median (min–max) shear bond strength (MPa) values according to surface treatments

Group	Subgroup	M ± SD	Min	Max
Group I	SB	6.10±2.16 ^{ab}	2.43	10.30
	HF	6.40±1.72 ^{ab}	3.37	9.16
	L	4.47±2.48 ^b	0.70	7.63
	SB–L	7.99±3.48 ^a	3.01	13.1
	HF–L	4.20±1.61 ^b	1.91	7.12
Group II	SB	6.22±2.57 ^{ab}	2.58	11.85
	HF	5.61±1.28 ^{ab}	3.08	7.44
	L	4.86±1.36 ^{ab}	2.81	6.90
	SB–L	6.61±2.56 ^{ab}	3.76	12.32
	HF–L	3.96±1.53 ^b	1.68	6.95

Values with same lowercase letter are not significantly different at $p < 0.05$

SB sandblasting, HF etch, L laser, SB–L sandblasting + laser, HF–L etch + laser

Tukey's HSD test results revealed that group SB–L had significantly higher shear bond strengths than groups HF–L and L for group I ($p < 0.05$). There were no significant differences among the groups for group II ($p > 0.05$), but higher values were obtained from group SB–L.

Failure type analysis

The modes of failure for the specimens after the treatment methods are as follows: for the feldspathic ceramics, 31 specimens exhibited adhesive failure and 13 specimens exhibited cohesive failure; for the lithium disilicate-based ceramics, 44 specimens exhibited adhesive failure and no specimen exhibited cohesive failure. Six mixed failures were exhibited in both two ceramic systems.

AFM evaluation

The representative AFM images for the two ceramic groups treated with the surface treatment groups are presented in Figs. 2 and 3. The surface treatment groups exhibited similar topographies, excluding the surfaces treated with the Er:YAG laser

(group L). The group L surfaces exhibited moderate irregularity, with peaks and valleys, and less roughness than what was achieved with sandblasting and HF acid. Group SB had the most distinct sharp peaks among the groups (Figs. 2 and 3).

Discussion

The properties of a luting agent and the surface treatments for ceramic surfaces before cement application play a major role in the clinical success of many indirect ceramic restorations [16]. The intent of this in vitro study was to investigate the influence of different surface treatments of feldspathic and lithium disilicate-based ceramics on the bond strength of resin cement. Of all the surface treatments for feldspathic ceramic, HF acid etching achieved the highest bond strength. The finding of the present study that sandblasting and laser application resulted in a strong shear bond between the resin cement and the feldspathic or lithium disilicate-based ceramic was in agreement with the findings of other groups [14, 17–27].

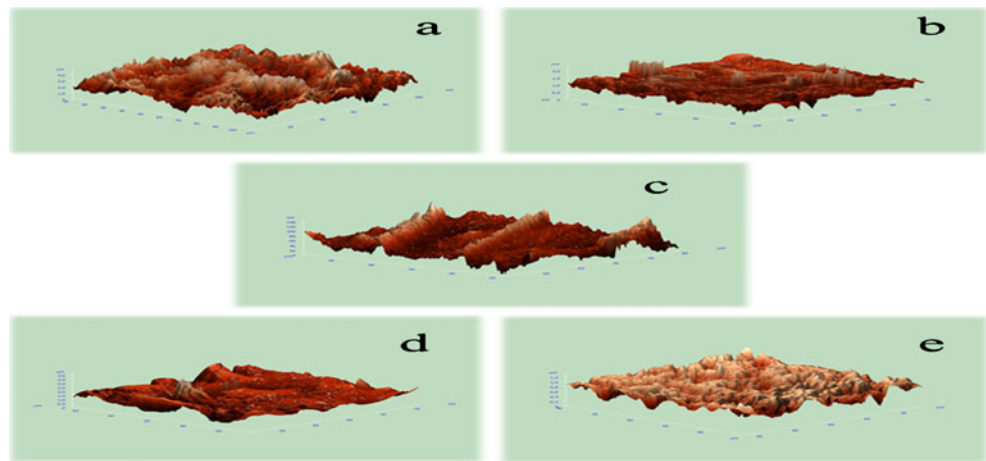
Airborne particle abrasion with Al_2O_3 particles is effective and practical for creating an activated and roughened surface on aluminum oxide ceramics [8, 28]. Changes in surface topography occurred after sandblasting procedures [29–31]. This technique was included in the present study, as it is a commonly used procedure in prosthodontic laboratories, and dental offices have miniaturized devices that facilitate its use [28]. Sandblasting produces a rough irregular surface with an increased surface area and enhances the wettability of the ceramic and composite resin. However, excessive sandblasting induces chipping or a significant loss of porcelain material, and it is not recommended for cementation of silica-based and feldspathic ceramic restorations [32–34].

Particle size, procedure duration, and pressure and distance used in the procedure are important factors in the performance of a cement bond [26, 28, 35, 36]. It was indicated that a sandblasting procedure with very large particles or with a very high pressure does not increase the bond strength; nevertheless, it causes maladaptiveness in the restoration as a result of wear [35–37]. Kern and Thompson [32] reported that at the end of sandblasting procedures, material loss in the IPS Empress ceramics was 36-folds higher than that in the In-Ceram. Spohr et al. [37] used an electron microscope to analyze the IPS Empress 2 ceramics

Table 2 Results of two-way ANOVA

Source	SS	df	MS	F	p
Ceramics	182.493	1	182.493	0.766	0.384
Surface treatments	6,538.319	4	1,634.580	6.863	0.000
Intercept	159,106.052	1	159,106.052	668.032	0.000
Ceramics* surface treatments	506.226	4	126.556	0.531	0.713

Fig. 2 AFM images of Group I. **a** SB. **b** HF. **c** L. **d** SB–L. **e** HF–L



to which 50- and 100- μm Al_2O_3 particles were applied. They performed a shear bond test and indicated that surfaces on which 100- μm Al_2O_3 particles were applied did not possess adequate retentive properties, whereas surfaces prepared using 50- μm Al_2O_3 particles were more highly etched. They also found at the end of the test that there was a significant increase in the strength of the cement bond. Other researchers reported that chemical and mechanical etching methods can be used together [28, 32, 38, 39]. In the present study, a statistical difference was found between the bonding resistance of the group I and II samples conditioned with 50- μm Al_2O_3 particles and the samples to which 50- μm Al_2O_3 particles were applied.

Among the chemical treatments of ceramic surfaces, etching with HF acid is one of the most commonly used methods [9, 17, 40–42]. In the procedure, the glassy matrix is selectively removed, and crystalline structures are exposed. Crystals influence the formation of microporosities [43]. Microporosity increases the surface area and makes micromechanical interlocking of resin possible [16]. Previous studies [18, 25, 44] demonstrated that HF + silane was consistently the most effective ceramic surface treatment, although it was not always significantly better than the other

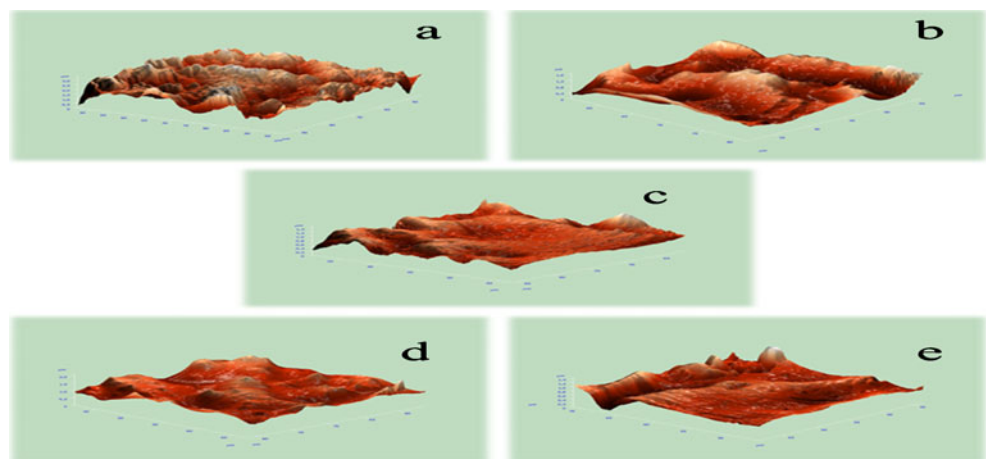
treatments that involved roughening and silane application [17]. In the present study, there were significant differences between the HF acid etching and the other techniques. HF acid is well recognized to have hazardous effects *in vivo*; it is harmful and irritating to soft tissues, [15] and this method cannot be considered for practical use in dentistry, particularly for intraoral ceramic repairs [17].

When Er:YAG laser irradiation is applied to a ceramic surface, it is expected that the laser irradiation will create a rough surface by removing the glass phase of the ceramic [27, 45] and increase the micromechanical retention of resin. However, in this study, the Er:YAG laser irradiation actually produced the lowest shear bond strength [27]. Erosion and melting were observed, but no fissures or cracks were observed on the surface after Er:YAG laser irradiation using AFM [27].

Similarly, Shiu et al. [17] observed that Er:YAG laser irradiation of a feldspathic ceramic surface at an energy setting of 500 mJ/pulse and 4 Hz for 2 min was insufficient to cause adequate surface roughening. They also found that the surface treatment proposed with Er:YAG laser resulted in low bond strength [27].

In the present study, the methodology for Er:YAG laser application on ceramic surfaces (10 W, 20 Hz, 500 mJ, and

Fig. 3 AFM images of Group II. **a** SB. **b** HF. **c** L. **d** SB–L. **e** HF–L



37.68 J/cm²) was based on a study performed by Silveira et al. [16], who defined this protocol after a series of pilot studies on In-Ceram alumina and observed that the Er:YAG laser was the most effective surface treatment, followed by Al₂O₃ sandblasting.

The combination of Al₂O₃ and Er:YAG laser (SB–L) treatment resulted in a better performance than Er:YAG laser irradiation alone. The use of Er:YAG laser irradiation and acid etching (HF–L) for ceramic surface treatment resulted in lower bond strengths than those observed in other groups [17]. Laser irradiation after HF acid etching may remove crystalline components rather than glass components and may decrease the bonding to the porcelain surface. Conversely, laser irradiation after sandblasting may remove aluminum particles from the porcelain surfaces, thus enhancing the bonding. These mechanic alterations may affect the bond strength between resin cement and porcelain surfaces.

This study had limitations in its ability to simulate clinical loading forces on restorations and oral environmental changes. In the shear bond test, the loading was monotonic instead of representative of cyclic fatigue, and the temperature and moisture of the oral cavity were not simulated; these factors should be included in investigations as many factors affect the bond strengths of the resin luting cements applied to the ceramics [28]. Future studies with a model that more closely resembles the oral environment and simulates clinical loading conditions are required [17].

Conclusion

The present study demonstrated that the shear bond strengths of the resin cements tested on ceramics after surface treatments varied in accordance with the type of ceramic. For lithium disilicate-based ceramics, the shear bond strength was significantly different between the SB–L group and the HF–L and L groups. However, no significant difference was observed for feldspathic ceramic.

Low bond strength was observed in the HF–L and L groups, indicating that these treatments are inadequate for clinical use within the parameters tested in this study. Laser irradiation used alone or combined with HF acid and SB within the parameters is not an alternative method for improving the bonding of resin cement to ceramic surface.

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