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The effect of using 40% HF treatment on the shear bond strength of the zirconia surface in comparison to conventional surface treatment methods

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Abstract

Introduction: The goal of this investigation was to examine how various surface preparation techniques affect the Shear Bond Strength (SBS) of resin cement to zirconia restorations.

Methods: 120 samples of zirconia ceramic disks in eight categories (10 x 3 mm) were randomly selected: 1. Control group (no surface treatments), 2. AL_2O_3 particle sandblasting, 3. 2.5 W Nd/YAG laser, 4. 3 W Nd/YAG laser, 5. Sandblasting plus 2.5 W Nd/YAG laser, 6. Sandblasting plus 3 W Nd/YAG laser, 7. 40% Hydrofluoric acid (HF), and 8. Sandblasting plus 40% HF. The analysis of one sample from each group was done using scanning. PANAVIA F2 dual-cure resin cement was then used to attach the samples to the composite resin. After thermocycling, the present researchers conducted the shear bond strength test utilizing the universal testing machine. The data were statistically evaluated using the Welch and Games-Howell tests, with a significance threshold of 0.05.

Results: A statistically significant difference ($P<0.001$) was seen between the SBS of the control and other groups. Comparison of SBS values with other groups revealed no statistically significant differences, except when compared to the control group ($P>0.05$). Sandblasting with HF acid etching had the greatest SBS, measuring 13.04 MPa. Out of all the groups, the SBS value was lowest for the control group (3.85 MPa). In terms of fracture type, the control group experienced more adhesive fractures. In the sandblasting and 3 W laser groups, the mixed and adhesive fractures were equal, and the mixed fracture was the most common type in the other groups.

Conclusion: The greatest SBS was obtained by sandblasting combined with etching. Nevertheless, this disparity was not statistically significant. In all groups but the control group, the SBS was not statistically significant.

Keywords: Shear bond strength, Surface treatment, Nd: YAG laser, Zirconia ceramic

Introduction

The advantages of zirconia restorations include biocompatibility, high aesthetics, high compressive strength, and a coefficient of thermal expansion similar to that of teeth.[1-2] Because zirconia ceramics and dentin do not bond well, various studies have been conducted to investigate the impact of surface treatment on the shear bond strength (SBS) of resin cement in zirconia ceramic-dentin bonding. Various techniques have been suggested for zirconia preparation that strengthen the bonding of the material with resin cement. These techniques consist of tribochemical silica coating,[3] laser therapy,[4,5] liner application,[6] selective etching,[7,8] and plasma spraying.[9] These sandblasting techniques increase the surface roughness of the zirconia structure, which improves SBS and micromechanical retention [10] .consist of Sandblasting creates acute fissures and defects in the structure that result in radial cracks.[11-13]

Additionally, abrasion and microscopic and macroscopic imperfections are created on the restoration surface by laser radiation, strengthening the binding.[14] Numerous research studies have prepared the zirconia surface using the Nd/YAG laser. Prior to bonding, this laser makes zirconia more wettable and rough on the surface.[14]

In zirconia restorations, there are different opinions on the use of 40% HF in Studies. In some Studies on zirconia restorations, there is no silica or glass phase, so HF acid and silane cannot be used for bonding, which causes poor zirconia-resin bonding.[15] Other Studies have suggested the use of hot acid etching or high-concentration acid for surface preparation.[16,17] It has also been stated that zirconia can be prepared with HF acid to increase bond strength and not to change the crystalline phase.[18] However, hot acid etching after sandblasting increases the surface roughness with the extensive distribution of deep and wide porosities in the zirconia network.[19] In this study, an attempt was made to examine and compare the groups that had the highest SBS. For this reason, according to the study by Ghoveizi et al,[20] the sandblasting and 2.5 and 3 W Nd: YAG laser groups, which had the highest SBS, were selected.

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Sandblasting and laser techniques were combined to examine the impact of these two groups. According to the study by YOU et al,[21] 10 minutes of immersion in 40% HF acid at room temperature is the optimal time and does not cause zirconia phase transformation. Finally, sandblasting with HF acid etching, which had the highest SBS, was selected according to Zhang et al.'s report.[19] These methods and their various combinations have not been compared yet. The goal of this investigation was to examine how various surface preparation techniques affected the SBS of resin cement to zirconia restorations. Various preparation methods and the control group would not differ in SBS, according to the study's null hypothesis.

Materials and Methods

Following the manufacturer's instructions, 120 disk-shaped zirconia ceramic specimens (KATANA, Kuraray Noritake Dental Inc., Japan) measuring 10 mm × 2 mm were created using the CAD/CAM (imes-icore, Germany) technology. The silicon carbide abrasive paper (600-grit Matador 991A Soflex Starcke GmbH & co Melle Germany) was used to polish all of the specimens, following Ozlem Kara et al.[22]

Eight groups of 15 specimens each were randomly selected from the samples, and every group was subjected to various zirconia disk surface treatments. The control group did not receive any surface treatment. The surfaces of another group (group 2) were exposed to a 10-second, 2-bar pressure sandblasting of 50 µm Al2O3 particles at a distance of 10 mm. An Nd/YAG laser (Light Walker ATS, Fotona, Ljubljana, Slovenia) was used to treat two groups. It functioned at a frequency of 25 Hz with a wavelength of 1064 nm and 300µm fiber optic, for a total of 45 seconds.[20,23,24] The settings varied among these groups: one with 2.5 W power, 125 mJ energy density, and 355 microseconds pulse duration (group 3); another with 3 W power and 150mj energy density (group 4).[21] Additionally, two groups were treated with AL_2O_3 particles (the same as group 2) and then by a 2.5W Nd/ YAG laser (identical to group 3), and another group was treated with AL_2O_3 particles (the same as group 2) and then by a high-power Nd/YAG laser (the same as group 4). Finally, the two remaining groups were treated with 40% HF for 10 minutes and rinsed with water for one minute to de-ionize (group 7), and then another group was treated with AL_2O_3 particles (the same as group 2) and then treated with 40% HF (the same as group 7).

In order to maintain a stable and constant 1mm distance from the samples, a transparent plastic device was used to hold the 1mm tip perpendicular to the samples. An electron microscope (KYKY-EM3200-China and Hitachi-SU3500-Japan) with a × 5000 magnification was employed to randomly examine each group's single sample. The samples were then prepared in the following order in order to measure the SBS: Clear Tygon plastic tubes measuring 3 mm in inner diameter and 4 mm in height were filled with resin composite in shade A3.5 (Filtek Z250, 3M ESPE, St. Paul, MN, USA). A light-curing system (Helioulux DLX, Ivoclar Vivadent, Schaan, Liechtenstein) was then used to cure them for 40 seconds at a minimum intensity of 600 MW/cm². As directed by the manufacturer, zirconia disks were adhered to composites using dual cure resin cement (Panavia F2.0, Kuraray, Okayama, Japan). The surfaces of zirconia disks were prepared using ceramic primer (Z Prime Plus, BISCO INC., Schaumberg, USA) in compliance with the guidelines provided by the manufacturer. The zirconia disks and composite surface were coated with a mixture of dual cure resin cement. Every sample weighing 453.6 g was put under the Gilmore needle. The extra cement was removed after the initial curing period of 10 seconds. After three minutes of application of an oxygen-inhibiting gel (Oxyguard II), 40 seconds were spent on the final curing process in each of the four directions. Prior to undergoing 5000 heat cycles between 5°C and 55°C, each bath was exposed for 20 seconds, followed by a 10-second transfer period, and all samples were stored in a 37°C distilled water incubator for 24 hours. The samples were left at room temperature for an hour following thermocycling. After that, they were dried and subjected to an SBS test in a universal testing apparatus at a speed of 0.5 mm/s (Bongshin, DBBP-2t, Seongnam, Korea). Additionally, a stereomicroscope was used to inspect the fractured surfaces at 40 × magnification (Carl Zeiss, Germany) in order to identify the kind of fracture of the samples. As a result, two fracture groups were created from the samples. Fracturing the adhesive at the ceramic-resin contact or mixed fracture on the ceramic surface was accompanied by cement residues or cement residues and composite.[20] To analyze the data, SPSS version 24 was utilized. The Kolmogorov-Smirnov test was applied to confirm and validate the normal distribution of the data. The SBSs between the groups were analyzed using the Welch and Games-Howell methods. The chi-squared test was applied to compare the kinds of failures that occurred in each group. Every test was run with a significance threshold of 0.05.[20]

Results

The SBS values of surface preparation were determined by the Shapiro-Wilk test of normality. Histograms were drawn, and data distribution was normal ($P > 0.05$). Each study group's SBS mean ± standard deviation is shown in Table 1.

The study groups' SBSs were ranked from the highest to the lowest as follows: Sandblasting plus 40% HF acid etching, 40% HF acid etching, sandblasting, sandblasting plus 2.5 W laser, 2.5 W laser, sandblasting plus 3 W laser, 3 W laser, and control group.

Table 2 shows the Games-Howell test pairwise comparison of groups. Welch's test, which compares groups pairwise, revealed a significant difference in the mean bond strength between them ($P < 001$). The control, sandblasting, 2.5 W laser, 3 W laser, sandblasting plus 2.5 W laser, sandblasting plus 3 W laser, 40% HF acid etching, and sandblasting plus 40% HF acid etching groups showed statistically significant differences in SBS

Table 1. The Shear Bond Strength (Mean ± Standard Deviation) of Groups With Different Surface Treatments

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
Control	14	3.8571	.82285	.21992	3.3820	4.3322	2.92	5.51
Sandblast	14	11.2336	3.20918	.85769	9.3806	13.0865	3.89	16.21
2.5-W laser	14	10.3543	2.30198	.61523	9.0252	11.6834	7.22	14.18
3-W laser	14	9.5586	1.80678	.48288	8.5154	10.6018	7.47	14.09
Sandblast + 2.5-W laser	14	10.4330	2.65101	.70851	8.9024	11.9636	6.34	14.77
Sandblast + 3-W laser	14	9.7686	3.86172	1.03209	7.5389	11.9983	5.07	16.91
HF40%	14	11.6236	3.95456	1.05690	9.3403	13.9069	7.06	20.47
Sandblast + HF 40%	14	13.0414	4.18085	1.11738	10.6275	15.4554	7.04	21.38

Table 2. Pairwise Comparison of Different Surface Treatment Methods

	(I) group	(J) group	Sig/ *P*-value
Games-Howel	Control	Sandblast	.000
		2.5-W laser	.000
		3-W laser	.000
		Sandblast + 2.5-W laser	.000
		Sandblast + 3-W laser	.001
		HF40%	.000
		Sandblast + HF 40%	.000
	Sandblast	2.5-W laser	.989
		3-W laser	.686
		Sandblast + 2.5-W laser	.996
		Sandblast + 3-W laser	.953
		HF40%	1.000
		Sandblast + HF 40%	.896
	2.5-W laser	3-W laser	.967
		Sandblast + 2.5-W laser	1.000
		Sandblast + 3-W laser	1.000
		HF40%	.963
		Sandblast + HF 40%	.443
	3-W laser	Sandblast + 2.5-W laser	.966
		Sandblast + 3-W laser	1.000
		HF40%	.642
		Sandblast + HF 40%	.141
	Sandblast + 2.5-W laser	Sandblast + 3-W laser	.999
		HF40%	.979
		Sandblast + HF 40%	.521
	Sandblast + 3-W laser	HF40%	.907
		Sandblast + HF 40%	.411
	HF40%	Sandblast + HF 40%	.981

($P < 001$). The comparison of the results of different groups (sandblasting, 2.5 W laser, 3 W laser, sandblasting plus 2.5 W laser, sandblasting plus 3 W laser, 40% HF acid etching, and sandblasting plus 40% HF acid etching) revealed no variation in SBS that was significant in terms of statistics ($P > 0.05$). The stereomicroscopic results evaluation of the fracture surface of the samples showed that most of the fractures were of an adhesive type in the control group. In the sandblasting and 3 W laser groups, the adhesive fracture was similar to the mixed fracture type. In the other groups (2.5 W laser, sandblast plus 2.5 and 3 W lasers, HF acid etching, and sandblasting plus HF acid etching), however, the mixed fracture was more evident.

The pictures from the electron microscope showed that the structure of the zirconia disks in all 7 groups underwent superficial morphological changes, indicating an improvement in the SBS in all groups. However, the control group showed no change in structure (Figures 1 and 2).

Discussion

The impact of various surface preparation techniques on the SBS of resin cement to Katana zirconia ceramic upon thermocycling was examined in this work. Less than 1 W lasers do not significantly increase roughness in pilot testing. However, even in short pulse mode, lasers with 1–2 W of power destroy, fuse, and remove zirconia's surface. The reduction in roughness (micromechanical bond) caused by a laser compared to sandblasting reduces the bond strength.[20]

The findings demonstrated that the surface characteristics of zirconia ceramics were altered by sandblasting, ND-YAG lasers of varying strengths, and 40% HF acid etching. Comparing the various groups, we found that sandblasting plus HF acid etching revealed the maximum surface roughness in this respect, which is consistent with the results of previous research.

Surface roughness is generally regarded as a basic technique to enhance the quality of resin cement to ceramic bonding using a micromechanical mechanism.[25] Both methods of grinding and sandblasting can remove contamination, increase the surface area, and increase wettability.[20,26] Several studies have shown that sandblasting with AL2O3 particles is an important step in achieving stable bond strength in ceramics. Generally, different particle sizes ranging from 50 to 110 microns have been used in sandblasting, although differences in particle size and preparation time can cause different results.[27,28] High pressure applied during sandblasting may result in transformation toughening and hasten the development of microcracks, which would ultimately diminish zirconia's mechanical qualities.[29]

Because of increased surface roughness caused by the

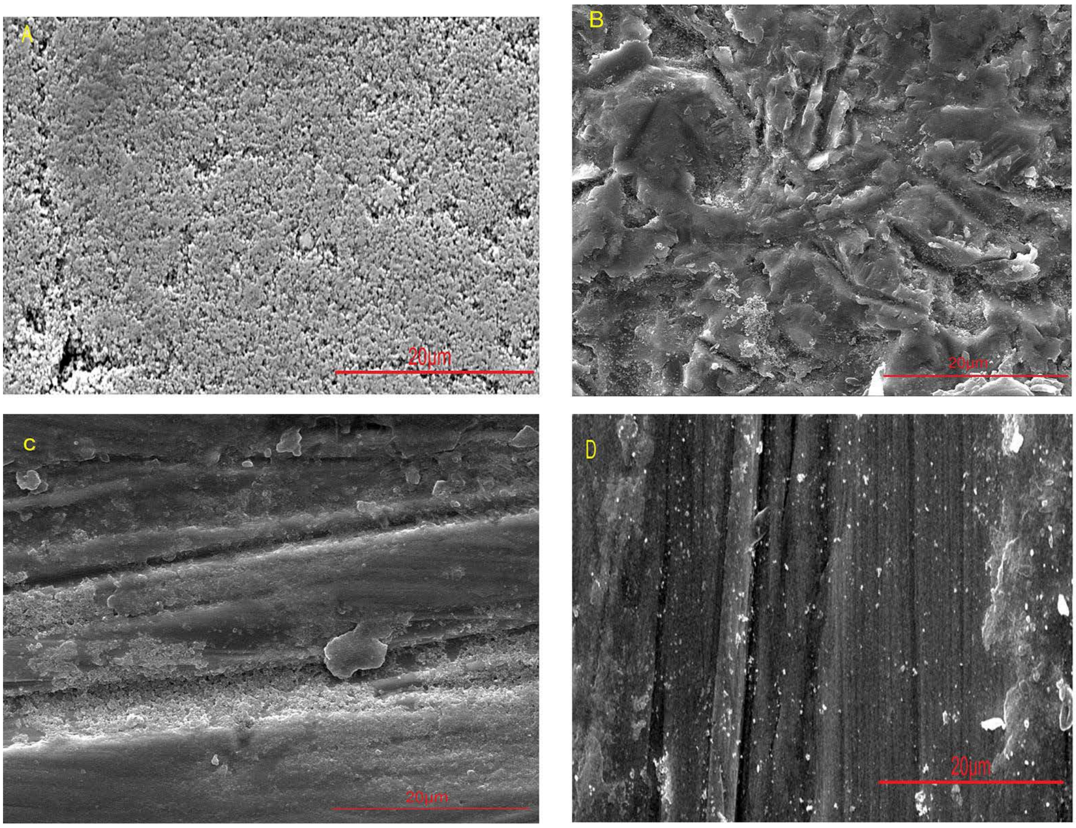

Figure 1. SEM microscopic images with 5000 × magnification. A: Control group (flat surface), B: Sandblast group (Irregular furrows), C: 2.5 power laser group (bubbles and blisters), D: 3 power laser group (bubbles and blisters)

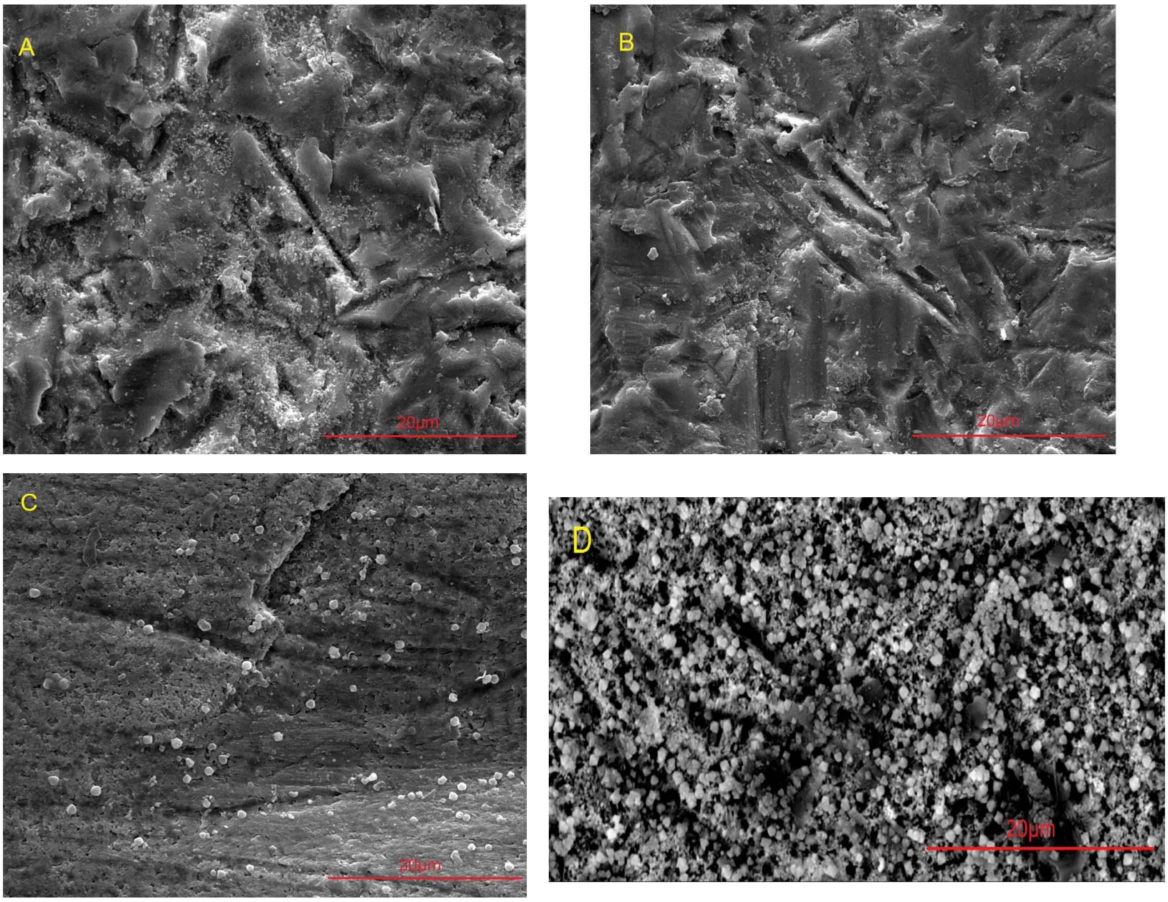

Figure 2. SEM microscopic images with 5000 × magnification. A: Sandblasting with laser power 2.5 (Irregular furrows with bubbles and blisters), B: Sandblasting with laser power 3 (Irregular furrows with bubbles and blisters), C: HF%40 (octahedrons), D: Sandblasting with HF%40 (sponge-like)

Nd: YAG laser, the SBS rises.[23,24] It should be mentioned, though, that the ceramic surface may melt as a result of the elevated temperature during irradiation; expansion happens during melting, and contraction happens right away following solidification. Surface cracks may result from the stress brought on by temperature fluctuations.[30] Microcracks were formed on the ceramic surface of the samples exposed to the 2 W power laser, according to the same Nd:YAG laser examination. Blister-like spheres with bubbles encased in a porous layer with apertures of varying diameters were produced by raising the power from 2 to 2.5 W. However, when the power was raised to 3 W, the blister-like spheres decreased and only more microcracks appeared.

Zirconia's surface roughness keeps getting rougher after being hot-etched for 10, 30, and 60 minutes at 100 ºC.[31] The advantages of HF acid are low price, ease of application, and no requirement for equipment, and its disadvantage is that it can be toxic and dangerous during clinical work.[32] Therefore, using lasers can be safe for the patient.[33] In SEM and Atomic Force Microscopy (AFM) analysis, in contrast to sandblasting, hot acid etching is rougher, and the porous structure is what gives the bond strength a greater value.[34]

The SBS of samples prepared by sandblasting was higher than that of the 2.5 and 3 W laser and sandblasting plus 2.5 and 3 W laser. Because the zirconia surface is roughened by the particles of aluminum oxide and promotes micromechanical adhesion, sandblasting produces stronger bonds.[20] However, the surface roughness created by a laser is probably less than that of sandblasting and sandblasting plus laser 2.5 and 3 W. The 2.5 and 3 W laser irradiation probably reduces the surface roughness compared to sandblasting.

The conclusions of Ghoveizi et al,[20] Hatami et al,[35] and Amiri et al[36] were in line with the current study's findings. Yet, Akar et al[37] discovered that, in contrast to the current investigation which utilized 50-micron particles, the laser's SBS was higher than that of sandblasting. This is most likely because the 120-micron particle size causes the tetragonal to monoclinic phase to change. Also, their study used Kuraray Noritake Dental disks and Bifix SE cement, while the current study used Katana disc and Panavia F2 cement. In our study, sandblasting plus HF acid etching and HF acid etching alone were more effective than the sandblasting and control methods, which is because the use of etching causes the formation of octahedrons, thereby increasing the porosity and micromechanical retention.[19] Sandblasting with etching increased this porosity and micromechanical retention and had a higher degree of roughness than the control, sandblasting, and laser groups. Zhang et al,[19] Kim et al,[38] Pin LV et al,[39] and Savaş et al[40] reported results consistent with our study. However, URAL et al[40] showed sandblasting had a higher SBS than HF acid etching, which is probably due to the use of 9.6% HF acid, compared to the 40% HF acid used in the present study.[41] Further, unlike our study, according to Marco Colombo, sandblasting has a stronger connection than acid etching, which is probably due to the different types of acid used, hydrochloric acid and ferric chloride being used in this study versus 40% HF used in the current study.[42]

The sandblasting plus etching and HF acid etching groups in our study had stronger bonds than the 2.5 and 3 W laser groups. This runs counter to the findings of Benli et al.'s research[35] wherein the laser's bond strength was greater than the etching group's. This is most likely because there was not any thermocycling in this study, and 4.9% HF acid was used in this study, unlike the present study that used 40% HF acid, which could not find a suitable micromechanical bond with zirconia at room temperature. Furthermore, unlike our study, Huang et al[43] showed that the laser's bond strength was greater than the etching group's, most likely as a result of the low HF (4.9% HF was used in this study, unlike 40% HF used in our study) and also the short application time (60 seconds in this study compared to 10 minutes in our study).

SEM images showed that various preparation methods influenced the zirconia surface differently. The control group had a smooth, non-porous surface, which resulted in lower micromechanical retention and bond strength than the other groups. Sandblasting, on the other hand, strengthened the connection by creating porosity on the zirconia surface. Considering the research of Amiri et al,[36]even brief bursts of laser radiation destroy, fuse, carbonize, and remove zirconia's surface, increasing porosity and bond strength in comparison to the control group. They demonstrated that the porosity was decreased and the bond strength was adversely affected by sandblasting plus 2.5 and 3 W laser, and in this study, sandblasting had a higher strength than sandblasting plus laser. When HF acid is combined with zirconia for 10 minutes, octahedrons are formed, which increases the porosity and micromechanical retention.[19] The strongest bond in the groups is produced by these octahedrons, and sandblasting combined with acid etching increases the effectiveness of just etching or sandblasting alone. The present study was conducted in an in vitro environment, and its results were recorded in laboratory conditions. Naturally, the study needs to be validated in an in vivo environment. To be closer to clinical conditions, the samples should be stored in distilled water for a longer period.

Conclusion

Based on the study's findings, the following outcomes were attained:

1. Among the study groups, sandblasting combined with etching had the greatest SBS value.
2. Of the groups, the control group's SBS value was the lowest.
3. SBS among the groups under study, ranked from the highest to the lowest, was found for sandblasting plus HF etching, HF etching, sandblasting, sandblasting plus 2.5 W laser, 2.5 W laser, sandblasting plus 3 W laser, 3 W laser, and control group.

4. Surface preparation of zirconia (sandblasting, laser, and 40% HF etching) significantly increases the SBS to resin cement compared to no preparation.
5. Preparation by sandblasting, laser, and 40% HF acid etching changes the surface morphological characteristics.
6. Preparation by sandblasting, 2.5 W laser, 3 W laser, sandblasting plus 2.5 W laser, sandblasting plus 3 W laser, 40% HF acid etching, and sandblasting plus 40% HF acid etching was significantly more effective than the control group.

Authors' Contribution

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Writing–original draft: Aynaz Samadnezhad.
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Competing Interests

The authors declare no conflict of interest.

Ethical Approval

This research is an in vitro study, so there are no ethical issues to be stated.

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