ORIGINAL RESEARCH

Comparative Analysis of Three Surface Treatments on the Bond Strength of Zirconia to Resin-luting Agents: An *In Vitro* Study

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Abstract

Aim: To study the impact of three different surface treatments namely sandblasting, silane-coupling agent, and laser on the retention of zirconia prosthesis and bond strength of zirconia to a resin-luting agent.

Materials and methods: Sixty zirconia crowns were fabricated and were divided into four groups of 15 samples each on the basis of surface treatments. A control group with no surface treatment (group A), laser-treated (group B), treatment with silane-coupling agent (group C), and sandblasting with aluminium oxide (Al_2O_3) particles (group D). Testing was then carried out using a universal testing machine (0.5 mm/min crosshead speed). At a point where the crown got separated from the tooth, the reading in kilogram force (kgF) was recorded. The data were collected and analyzed statistically.

Results: Group D produced the highest mean bond strength (17.5233 kgF) followed by group B (10.0067 kgF), group C (8.6907 kgF), and group A (3.3773 kgF). One-way ANOVA test showed a p-value more than 0.05, concluding no significant difference among the groups. Tukey's HSD *post hoc* test gave the p-value corresponding to the F-statistic of one-way ANOVA lower than 0.01 when intergroup comparison was done confirming a significant difference among the groups.

Conclusion: The bond strength significantly increased in the samples treated by sandblasting compared with those treated with laser and silane-coupling agents.

Clinical significance: The success of a zirconia prosthesis lies on its bonding with the tooth structure. Bond failure leads to loss of function and hence ends up in failure. Selection of the proper surface treatment will not only improve the bond strength but also amplify the retention of zirconia-based prosthesis, thereby reducing the failure of the final prosthesis. It also improves the longevity of the prosthesis and restores the lost function which is the basic clinical aim of a prosthodontic treatment.

Keywords: Bond strength, CO_2 laser, Sandblasting, Silane coupling, Zirconia.

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Introduction

Zirconia (ZrO_2) is a restorative material known for its esthetics and metal-free nature.[1] Other reasons for its increasing use in dentistry are its biologically inert nature, superior wear resistance, color stability, and poor thermal conductivity.[2] Being a chemically inert material, it shows no etching by conventional hydrofluoric acid. This reduces its bond strength between luting agents and tooth material. In order to achieve a reliable, long-lasting bond with zirconia whose physical and chemical properties differ from those of glass ceramic other alternative methods of improving bond strength were needed.[2] Hence pretreatment procedures play a vital role in enhancing the adhesion between the zirconia surface and the luting agent.[3]

The most widely practiced method to increase the bond strength is by roughening the zirconia surface at microscopic level by sandblasting. This allows the resin-luting agent to flow into the micro undercuts producing stronger mechanical lockings.[4]

The second popular method is a chemical bonding procedure. A silane-coupling agent is used to bond a ceramic material with the resin-luting agent. Water reacts with the silane molecule's alkoxy groups ($RO_3Si–$) and becomes a silanol group (SiOH). The final outcome is the siloxane (–Si–O–Si–O–) covalent bond that is formed by hydroxyl (OH) groups reacting with available Si and O on a ceramic surface. The methacrylate groups of resin-luting agents combine with the monomeric end of the silane molecules. As a result, a series of siloxane covalent bonds are generated between the methacrylate resin and the ceramic surface.[5]

The latest innovation in this field is the usage of lasers. The effect of lasers on zirconium oxide is documented by few studies. When the CO_2 laser is focused on ceramic surfaces, conchoidal tears

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appear due to induction of heat as an effect of surface warming. These are again surface irregularities that provide microundercuts and promote retention. But the sudden local temperature changes may lead to formation of internal stresses, which may be harmful.[3]

Though a lot of literature is available on these surface treatments, a comparative analysis among them based on their influence on the bond strength of zirconia to luting agents was not available. The aim of this *in vitro* study is to compare the influence of these three different surface treatments on the bond strength of a zirconia prosthesis to a resin-luting agent.

MATERIALS AND METHODS

This *in vitro* study was conducted in the Department of Prosthodontics and Crown Bridge and Implantology, Maharana Pratap College of Dentistry and Research Centre, Gwalior, over a time period of 9 months from 30th March 2018. The sample size of 60 was divided into four study groups with 15 samples in each.

Preparation of Specimens

Sixty noncarious, nondeformed, fully matured premolar teeth that were extracted for orthodontic treatment purpose were collected and cleaned with 9.5% normal saline. They were kept immersed in 8% hydrogen peroxide solution for 24 hours to clean debris and necrosed tissue. Three specimens were placed in autopolymerizing polymethylmethacrylate (PMMA) denture-base resin (DPI-RR, India) at an equal distance of 15 mm. They were kept parallel to each other at a level such that cemento-enamel junction was visible. The mounted specimens were retrieved after complete polymerization of PMMA. Twenty such acrylic blocks having three teeth specimens each were fabricated and numbered accordingly for groups A, B, C, and D with five blocks in each group.

Custom Tray Fabrication

A custom tray was fabricated for every acrylic block. A 1.5 mm-thick modeling wax (Tru Lon, India) was adapted over the specimen teeth as a spacer. Separating media (CMS) was applied and 5 mm-thick autopolymerizing PMMA resin in dough stage was adapted. The handle was attached in the center of the tray. The tray was then finished and polished.

Fabrication of Custom Jig

To standardize the tooth preparation, a custom jig was fabricated that secured the air rotor handpiece on a dental surveyor in the desired position. To fabricate the custom jig, a 10″ long stainless-steel rod with 9.5 mm diameter was attached in place of the surveying arm on the dental surveyor, which was movable only in the vertical direction and denoted as the vertical arm. At the bottom of that vertical arm, a handpiece-holding clamp with 17° angulation was secured with allen key and denoted as a handpiece-holding clamp. About 7.5″ long horizontal arm was welded at the top of the vertical arm. To this horizontal arm, a 12″ long and 4 mm in diameter paralleling rod was attached, which was movable and adjustable in horizontal as well as vertical direction.

Tooth Preparation to Receive Zirconia Crown

Tooth preparation for all ceramic crowns was done using an air rotor handpiece (NSK, Japan) mounted on the custom-made jig attached to the dental surveyor. The specimen tooth, which was to be prepared, was secured on the surveying table so that the long axes of the sample tooth, paralleling rod, and that of diamond bur on the air rotor handpiece were parallel to each other. This allowed for a standardization of tooth preparation.

Occlusal Preparation

Occlusal preparation was done by keeping the occlusal surface flat maintaining the 4 mm crown height from cementoenamel junction using a wheel-shaped diamond bur (WR 13C Mani Dia-burs; Mani Inc., Utsunomiya, Japan). Any sharp edges were rounded off.

Axio-proximal Preparation

The axio-proximal preparation was done by using No. 12 flat-end tapered diamond bur (Mani Dia-burs, Mani Inc., Utsunomiya, Japan). Preparation was finished so that all four axial walls (mesial, distal, buccal, and lingual) had 3° taper each and parallel to each other with shoulder finish line.

Impression Making and Fabrication of Dies

Two-step putty wash impressions by addition of silicone (Dentsply, Germany) in custom tray were made for each acrylic block. Each impression was beaded using Type-II dental plaster (Gyprock, India) and boxed using modeling wax, and then poured in Type-IV die stone (Kalabhai Ultrastone, India). After the final setting, the dies were retrieved.

Zirconia Crown Fabrication

Zirconia crowns were fabricated on each specimen's teeth using CAD-CAM. The crowns were fabricated in such a way that each crown had a loop measuring 5 mm in diameter on the occlusal surface for pull-out test.

Surface Treatment of Zirconia Crowns

Based on the surface treatment done, the zirconia crowns were then divided into four groups:

1. Group A: Control group of zirconia crowns with no surface treatment.
2. Group B: Zirconia crowns were treated with laser (4-W diode laser unit (Cheesell Den10B, GIGAA laser, Wuhan Giga Optronics Technology, UK). The samples in this group were treated with diode laser radiation. Laser energy in a noncontact mode (wavelength of 980 nm, 1500-Hz pulse-repetition rate, and power setting of 4W) is perpendicularly delivered to the surface 1 mm away using a 1000-μm-diameter straight probe. Surface irradiation was carried out for 5 seconds along with a fine water spray.
3. Group C: Silane-coupling agent treated zirconia crowns. Monobond S (Ivoclar, Switzerland) was selected as the test material. The samples were air-dried. The intaglio surface was painted with the silane-coupling agent. The surface was light-cured (Gnatus Equipamentos Medico, China) for 20 seconds.
4. Group D: Zirconia crowns were sandblasted inside a sandblasting machine (Twin-Pen, China) with alumina particles measuring 50 μm under 35-PSI pressure for 15 seconds, from a distance of 10 mm.

Cementation of the Zirconia Crowns

A uniform layer of self-adhesive resin cement was dispensed using an automixing tip onto the intaglio surface of the crown and cemented on their respective specimen in their respective group. The resin cement was cured for few seconds. The excess flash material was removed. Then the final light curing was done thereafter for 20 seconds. To simulate the clinical condition, uniform finger pressure was applied during the cementation of crowns.

Storage

To simulate the oral environment, the test specimens were immersed in artificial salivary medium (Saleva by Global Dent Aids) at room temperature for 2 days before testing.

Testing

Universal testing machine (0.5 mm/min crosshead speed) was used. The test specimen was fixed on the lower jaw of the machine and the loop of the cemented crown was engaged by a stainless-steel wire fixed on the upper jaw of the machine. Testing was then carried out. At the point where the crown got separated from the tooth, the reading in kgF was noted for each specimen.

Statistical Analysis

Statistical analysis was carried out using Statistical Package for the Social Sciences (SPSS) also known as IBM SPSS statistics. One-way ANOVA and Tukey HSD *post hoc* tests were chosen for statistically analyzing the results obtained from the present study. One-way ANOVA test was chosen for statistically comparing the three test groups and the control group. Tukey's HSD *post hoc* test shows which groups among the sample in specific have significant differences.

RESULTS

The bond-strength values of all the groups were collected (Table 1). The group of zirconia crowns that were sandblasted (group D) came out to produce the highest bond strength with a mean strength of 17.5233 kgF, while the group with silane-coupling agent (group C) showed the lowest bond strength among the groups with surface treatments with a mean bond-strength value of 8.6907 kgF. The laser-treated group (group B) came out to have a bond strength of 10.0067 kgF. The control group (group A) in which no treatment was carried out showed the least of all other groups with a mean value of only 3.3773 kgF. The descriptive statistics obtained from the data of bond-strength testing like mean, sample variance, standard error, and standard deviation among different surface treatments groups were also recorded for statistical analysis (Table 2).

One-way ANOVA test was carried out to statistically compare the three test groups (Table 3). The *p*-value more than 0.05 concludes no significant difference among the groups corresponding to the one-way ANOVA test. To further determine the significant difference among the groups, Tukey's HSD *post hoc* test was carried out (Table 4). The *p*-value corresponding to the *F*-statistic of one-way ANOVA is lower than 0.01 when group A was compared with group B, group C, and group D; when group B was compared with group D and when group C was compared with group D. Hence, significant difference was found among these groups. When group B and group C were compared, the *p*-value was 0.53, which is higher than 0.01, suggesting that there is no significant difference. The mean bond strength in kgF and the standard deviation among the four groups were also presented graphically (Fig. 1).

DISCUSSION

Retention is an inherent fundamental requirement of a prosthesis. It can be defined as the property of a tooth preparation by which the prosthesis resists dislodgement in a vertical direction or along the path of placement.[6,7] While doing the tooth preparations for fixed prosthesis, certain principles must be followed, otherwise, there might be failure of the restoration due to distortion and dislodgment.[8] Therefore, these principles were followed while preparing the samples of the present study.

Table 1: Bond strength in kgF of the respective groups

Sample no.	*Group A (control)*	*Group B (laser treated)*	*Group C (silane-coupling agent)*	*Group D (sandblasting)*
1.	3.82	9.62	8.23	16.23
2.	3.17	9.71	8.38	16.39
3.	3.15	9.88	8.44	16.71
4.	2.99	9.81	8.47	17.25
5.	2.85	9.9	8.62	17.66
6.	3.71	9.65	9.1	17.77
7.	3.24	9.7	9.29	17.85
8.	3.87	10.41	9.21	17.91
9.	3.48	10.38	9.41	18.06
10.	3.86	10.46	9.06	18.16
11.	2.57	10.35	8.67	18.42
12.	3.79	10.04	8.92	18.26
13.	3.11	10.33	8.02	18.11
14.	3.81	10.11	8.13	18.01
15.	3.24	9.75	8.41	16.06
Mean	3.3773	10.0067	8.6907	17.5233

Table 2: Descriptive statistics of all the groups

Surface treatments	*N*	*Mean*	*Sample variance*	*Std. error*	*Std. deviation*	*95% Confidence interval for mean*	
						Upper bound	*Lower bound*
Group A	15	3.37	0.1739	0.107	0.4170	3.60	3.14
Group B	15	10.06	0.0952	0.079	0.3085	10.16	9.85
Group C	15	8.69	0.1980	0.11	0.4449	8.93	8.44
Group D	15	17.52	0.6280	0.204	0.7924	17.92	17.12
Total	60	9.89	26.2262	0.661	5.1211	11.19	8.60

Table 3: ANOVA table for laser, silane-coupling agent, and sandblasting groups

Source	*N*	*Mean*	*Sample variance*	*Std. error*	*Std. deviation*
Between groups	1532.0138	3	510.6713	1865.5	1.11
Within groups	15.3297	56	0.2737		
Total	1547.3435	59			

The mean difference is significant at the 0.05 level

Table 4: Tukey HSD *post hoc* test for laser treatment, silane-coupling agent, and sandblasting

Surface treatments	*N*	*Mean*	*Sample variance*
A vs B	39.33	0.0010053	$^*p<0.01$
A vs C	49.07	0.0010053	$^*p<0.01$
A vs D	104.71	0.0010053	$^*p<0.01$
B vs C	9.74	0.533245	$^*p<0.01$
B vs D	65.38	0.0010053	$^*p<0.01$
C vs D	55.64	0.0010053	$^*p<0.01$

*The mean difference is significant at the 0.01 level

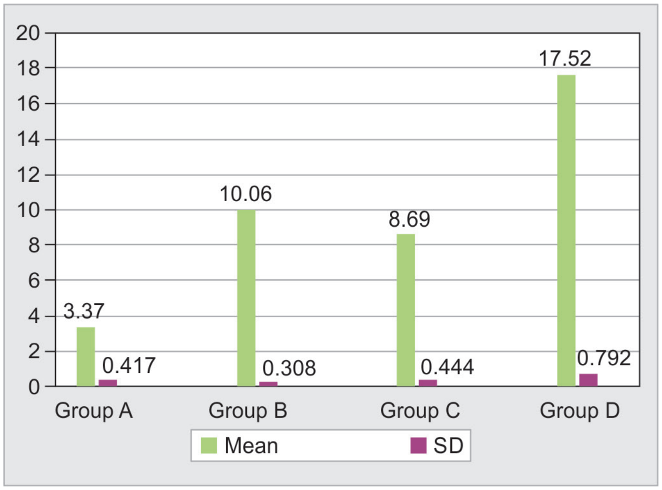

Fig. 1: Graphical representation of mean bond strength in kilogram-force (kgF) and standard deviation among the four groups

Factors that are taken into consideration for increasing the retention of a fixed restoration are:

- Magnitude of forces
- Geometry of tooth preparation
- Intaglio surface roughness of the restoration
- Adhesive properties of the luting material

The magnitude of forces that tend to dislodge a restoration along its path of withdrawal is small compared with those of seating or tilting forces. The magnitude depends on the stickiness of the various foodstuff, texture, and the surface area of the prosthesis being dislodged.[8]

Theoretically, a tooth preparation with parallel opposing axial walls provides maximum retention. A small taper or angle of convergence is provided as a convenience form during tooth preparation. As long as this taper is limited, the luted restoration will be retained by the preparation because of reduced path of withdrawals. With the increase of taper, the restoration will have more freedom of movement and thus there will be reduction in the retention. For the first time, Jorgensen experimentally demonstrated the relationship between the degree of axial wall taper and the magnitude of retention for the first time. If the paths of withdrawal are limited, the retention depends on the total length of the path and the total surface area in sliding contact. Hence, preparations with smaller axial walls are less retentive than the ones with long axial walls.[8] In the present study, the axial wall length was kept 4 mm, which is long enough to provide retention with a minimum taper of 3° using a custom-made jig.

It has been observed that retentive failure occurs at the cement–restoration interface for restorations with smooth intaglio surfaces. This can be solved by etching the tooth surface, roughening, or grooving the inner surface of the restoration.

The most commonly used luting materials are self-adhesive resin cements and resin-modified glass ionomer. They provide adhesion to both the teeth and the ceramic material. They are quick and easy to apply. These require no additional priming, and thus are less technique-sensitive.[9] They improve retention, marginal adaptation, and fracture resistance of the restoration.[10] Self-adhesive resin cements are "tack cured" for few seconds. This partially cures the cement and facilitates the removal of excess flash material when it is soft. After removing the excess cement, it is finally cured.[9]

Brittle materials like ceramics do not undergo plastic deformation like metal alloys. Thus, their modulus of elasticity and behavior under functional stress is different from metals. Crack propagation is allowed by crystalline materials when the surface is damaged by external forces. Therefore, choosing the correct type of luting agent and the technique of application for permanent cementation plays a major role in the clinical success of all metal-free restorations.[9]

In the present study, the best means of increasing the resin bond of zirconia to the tooth structure is by air abrasion. Awliya et al. conducted a study to compare the shear bond strength (MPa) of resin cement to ceramic after different surface treatments where similar results were seen. They found sandblasting to produce the highest shear bond strength of 11.99 (MPa), followed by diamond abrasion + phosphoric acid (9.13 MPa) and hydrofluoric acid treatment (5.38 MPa).[11] Kern et al. concluded in their study that the bond strength of zirconia to luting agents decreased over time if the zirconia is not sandblasted using alumina dust.[12] Thus, sandblasting proved to have a good impact on the bond strength similar to the present study. Wolfart et al. observed the increase of bond strength after sandblasting.[13] Cavalcanti et al. concluded in their study that sandblasting with Al_2O_3 provided better shear strength than Er:YAG laser treatment.[14] Moradabadi et al. in their study said that air particle abrasion provides higher shear strength than acid etching.[15] Liu et al. in their study found that the specimens that underwent sandblasting had the best shear bond strength (25.1 ± 2.7 MPa) as compared with other surface treatments like acid etching (21.5–22.5 MPa).[16] In the present study, it was observed that sandblasting provided the highest bond strength with 17.5233 kgF.

For the purpose of sandblasting, various sizes of aluminum oxide particles, between 50 and 110 μm, are used.[17] Sciasci et al. concluded that particle size of the alumina dust never affected the bond strength between zirconia and the resin cements.[18] Miragaya et al. and Akbarzadeh et al. have mentioned in their studies about air abrasion affecting a tetragonal-to-monoclinic phase transformation on the surface of the zirconia ceramic, thereby increasing the bond strength of the adhesive resin used in their study.[19,20] The bombardment of aluminum oxide particles under high pressure during blasting induces microcracks, thereby reducing the mechanical properties of zirconia.[4] In the present study, 50-μm Al_2O_3 particles were used, which showed the highest bond-strength values among all three.

Silane-coupling agents as described by Blatz et al. contain a silane coupler and a weak acid, which encourages the formation of siloxane bonds.[21] A silane-coupling agent helps in the bonding between a ceramic surface and a resin-luting agent. When an alkoxy group (RO_3Si–) from the silane molecule comes into contact with water molecules, they form silanol groups (SiOH). The hydroxyl (OH) groups of silanol groups further react with available Si and O on the ceramic to form siloxane (–Si–O–Si–O–) covalent bonds. The methacrylate groups of resin-luting agents react with the monomeric end of the silane molecules. This results in the formation of a strong network of siloxane covalent bonds in-between the ceramic surface and methacrylate resin.[5]

Atsu et al. evaluated 6 different ceramic surfaces treated with different coupling agents and concluded that the untreated control had the least bond strength (15.7 ± 2.9 MPa), followed by the group silanized with a silane-coupling agent (16.5 ± 3.4 MPa), adhesive 10-methacryloyloxydecyl dihydrogen phosphate monomer (MDP) containing bonding/silane-coupling agent mixture (18.8 ± 2.8 MPa), silica coating using 30-μm Al_2O_3 particles modified by silica (21.6 ± 3.6 MPa), silica coating and silanization (21.9 ± 3.9 MPa), and the highest was from silica coating and application of an MDP containing bonding/silane-coupling agent mixture (22.9 ± 3.1 MPa).[1] The results were similar, where using silane alone proved to improve the bond strength. In the study conducted by de Souza et al., the bond strength after chemical surface treatment with MDP and 6-(N-(4-vinylbenzyl) propylamino)-1,3,5-triazine-2,4-dithione (VBATDT) were 12.78 MPa and 22.77 MPa, respectively, which was higher than without surface treatments (9.17 MPa).[22] Qeblawi et al. in their study concluded that a combination of mechanical and chemical conditioning of the zirconia surface helps to develop a durable resin bond to zirconia.[23] Chai et al. observed that the shear bond strengths of zirconia with silane coupling increased (11.4 ± 5.4 MPa) than that of non-silane-treated zirconia (5.7 ± 4.3 MPa).[5] In all the above-mentioned scientific researches, it was seen that using a silane-coupling agent used for salinization facilitates the bond strength by forming a chemical bond to resinous material via cross-linkages with methacrylate groups. It also improves the substrate surface energy and surface wettability of the resin.[23] In this study, the property of silane-coupling agent was put to test with other popular methods in order to compare the bond strength.

With the introduction of newer wavelengths and improvement of existing ones, application of lasers has extended their usage to a wide range of procedures. In this study, a diode laser was used for irradiating the zirconia surfaces. A laser causes local temperature changes due to heating and cooling phases. This creates internal tensions that can weaken a material. The mechanical properties are reduced by these temperature changes, especially due to phase transformation.[14] Thus, in the current study, a lower power setting was chosen and the surfaces that were irradiated were kept cool by running water.[24]

The very important interaction between the laser and the material (thermomechanical effect) is the absorption of the laser beam energy by the material surface. The output energy and pulse rate of the laser beam showed to produce directly proportional effects on the energy density and thermal effects on the material surface.[10]

With the absorption of the energy from the laser beam, a phenomenon called heat induction produces "shell-like ruptures" on the surface of the ceramic, leading to the formation of micromechanical roughness. The resin cementing material penetrates into these cracks, sets, and forms resin tags. As a result, a stronger bond is formed between the ceramic surface and the resin material.[3] Micromechanical retention and shear bond strength of resin cement to the zirconia surface is increased by roughening on the zirconia surface.[10]

Unlike the results of the present study, Ural et al. compared sandblasting, hydrofluoric acid etch, and CO_2 laser treatments on zirconia surface and found the laser had the highest mean shear bond strength (20.9 MPa), followed by sandblasting (16.4 MPa) and hydrofluoric acid (14.1 MPa).[3] Paranhos et al. concluded that when zirconia was treated with Nd:YAG laser, the shear bond strength was higher (14.09–16.20 MPa) than CO_2 laser (6.24–10.51 MPa) and non-laser zirconia (4.65–8.79 MPa). Both the bond strength and surface roughness were high for the Nd:YAG laser than the CO_2 laser and other abrasion treatments.[25] In a study conducted by Kasraei et al., it was observed that there was a significant increase in the shear bond strength values of the CO_2 laser-treated zirconia (12.12 ± 3.02 MPa) than that of the untreated control samples (5.97 ± 1.14 MPa).[26] Kasraei et al. evaluated the effects of CO_2 and Er:YAG laser treatment on the bond strength of resin cement to zirconia ceramics and concluded that CO_2 lasers provided higher bond strength values (12.12 ± 3.02 MPa) than the Er:YAG laser treatment (8.65 ± 1.75MPa).[27] The study that was done by Akhavan Zanjani et al. concluded that sandblasting using alumina particles has a larger impact and a better mean micro-shear bond strength (37.3066 MPa) than laser treatments using CO_2 (29.0802 MPa) and Er,Cr:YSGG lasers (27.5204–21.9773 MPa).[28] In their study also, Cavalcanti et al. concluded that Al_2O_3 sandblasting provides better shear strength than Er:YAG laser treatment.[14] As we see that there were studies where the results contradicted each other, a detailed comparative analysis was required to evaluate the effect of lasers on zirconia and its effectiveness over sandblasting and silane-coupling agents.

There are few limitations in this study, such as the surface analysis tests were not conducted. To evaluate the hydrolytic degradation, stability and durability of the resin-zirconia bond artificial aging procedures, such as fatigue, thermocycling, and long-term water storage, were not performed. Thus, further research is required to address the limitations of the present study.

CONCLUSION

Within the limitations of the present study, it can be concluded that all the three surface treatments (sandblasting with alumina particles, surface treatment using silane-coupling agent, and laser treatment) increased the bond strength adequately. The bond strength significantly increased in the samples treated by sandblasting compared with the samples treated with silane-coupling agents and laser. It was also seen that statistically, the laser-treated samples and the samples treated with silane-coupling agent showed no significant difference.

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