Microtensile Bond Strength of a Resin Cement to Silica-Based and Y-TZP Ceramics Using Different Surface Treatments

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Keywords
Lithium disilicate ceramic; Nd:YAG laser; surface treatment; zirconia ceramic.

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Abstract

Purpose: To evaluate the effects of different surface treatments on the microtensile bond strength ($\mu$TBS) of bonding between resin cement and lithia or ziroria-based ceramics using an in vitro study.

Materials and Methods: Three zirconia ceramic blocks (IPS e.max ZirCAD) and three lithium disilicate ceramic blocks (IPS e.max CAD) were sintered and duplicated in resin composite. The zirconia specimens underwent various treatments ($n = 1$): (i) Sandblast + primer (ZiSa); (ii) sandblast + laser irradiation + primer (ZiSaLa); or (iii) laser irradiation + primer (ZiLa). The lithium disilicate specimens also underwent various treatments: (i) sandblast + HF + silane (LiSaE); (ii) sandblast + silane (LiSa); or (iii) sandblast + laser irradiation + silane (LiSaLa). The ceramic–composite blocks were cemented with resin cement and cut to produce bars with approx. 1 mm$^2$ bonding areas. The specimens were thermocycled, and bond strength tests were performed in a universal testing machine. The fracture type was determined by observing the fractured surface under a stereomicroscope. The mean bond strengths of the specimens were statistically analyzed using one-way ANOVA and Duncan’s tests ($\alpha = 0.05$).

Results: Mean comparison of the $\mu$TBS showed no significant difference between LiSaE and LiSa ($p > 0.05$), but significant differences between LiSaE and other groups ($p \leq 0.01$). No significant differences were found between the ZiSaLa and ZiSa groups ($p > 0.05$). The modes of failure in all groups were mostly adhesive (57% to 80%). The mean bond strengths in laser-irradiated ceramics were significantly lower than those from other surface treatments. All ZiLa specimens debonded before testing (pretest failure).

Conclusions: Lithium disilicate ceramic surface treated with a combination of sandblasting and silane application provided a bond strength comparable to that provided by sandblasting in combination with acid etching and applying silane. Groups treated with laser irradiation had significantly lower bond strengths than other groups.

Increasing esthetic demands have resulted in full ceramic restorations receiving considerable attention in contemporary cosmetic dentistry. The popularity of these restorations is increasing, due to their desirable esthetics and metal-free nature.\(^1\) Although full ceramic restorations are strong enough to resist occlusal loads on single crowns and short fixed partial dentures (FPDs), their application in long-span FPDs has been limited.\(^1\) The introduction of high-strength ceramics has led to an evolution in full ceramic restorations. Due to their high crystalline phase (aluminum oxide and zirconium oxide) contents, these ceramics have better mechanical properties than feldspathic, leucite-based, and lithium-disilicate-based ceramics,\(^2\) making them applicable to metal-free restorations in areas with higher occlusal loads.\(^2\)

Zirconia is a high-strength ceramic that has recently been introduced in full ceramic restorations.\(^3\) These full ceramic restorations, made of zirconium oxide, have several benefits, such as high flexural strength (more than 1000 MPa)\(^2\) and desirable optical characteristics, as well as compatibility with basic colors and reduced ceramic layer thickness to achieve desired colors.\(^1\) The success of ceramic restorations, among other factors, depends on achieving high retention and suitable marginal adaptation after luting.\(^4\) The high fracture resistance of zirconia ceramics means that crowns and FPDs made of
zirconium oxide could be cemented using conventional cements suggested by manufacturers; however, adhesive luting of these restorations has been recommended to improve their retention, marginal adaptation, and fracture resistance.\(^1\) It has been recommended that, where necessary, resin cements containing phosphate monomers are better materials for adhesive luting of zirconia ceramic restorations.\(^1,5\)

Some studies have suggested that the best long-term results are obtained when a sandblasted zirconia surface is luted with 10-methacryloxydecyl dihydrogen phosphate (10-MDP) monomer containing resin cements.\(^5\) The long-lasting bond between these cements and high-strength ceramics is due to direct bonding of the MDP ester group with the metal oxide.\(^2\) Common methods for ceramic surface treatment are grinding, abrasion with a diamond bur, sandblasting with aluminum oxide particles, acid etching, laser irradiation, silane application, or a combination of these methods.\(^5\) Although hydrofluoric acid etching followed by silane application is effective at creating successful bonding between glass ceramics and resin cement, both etching and silane have failed to bond well with some new ceramics.\(^3\) In particular, ceramics with high zirconia contents cannot be etched by hydrofluoric acid, because they do not contain a silicon dioxide (silica) phase.\(^9\) Furthermore, hydrofluoric acid poses a risk to dental staff and patients, and could damage the flexural strength of some ceramic materials.\(^10,11\)

To create a strong bond between a resin and ceramic, mechanical and chemical retention are needed. Roughening and cleaning the ceramic surface have been reported to achieve this.\(^5\) Accordingly, substitute methods have been recommended for the surface treatment of high-strength ceramics, including sandblasting, silica coating, laser irradiation, or a combination of these.\(^4,12\) Sandblasting is based on abrasion with particles of different sizes (between 20 and 250 \(\mu\)m).\(^13\) This abrasion process removes loose contaminated layers and creates a rough surface for mechanical retention by the luting agent. Additionally, surface roughening increases the surface area for efficient bonding and can lead to physicochemical changes, such as an increase in surface energy and wettability.\(^13\)

Applying silane coupling agents to prepared ceramic surfaces for chemical bonding has also been recommended.\(^5\) Silane is a bifunctional molecule that can bond with hydroxyl groups and silicon dioxide on the ceramic surface on one side, and with resin on the other side. Applying silane also increases the wettability of the ceramic surface.\(^5\)

As an alternative method, high intensity lasers have recently been studied for roughening ceramic surfaces and improving resin-ceramic bonding through micromechanical retention.\(^12\) Lasers have been suggested to modify material surfaces in a safe and easy manner.\(^14\) Laser applications in dentistry are constantly expanding. The effect of high intensity lasers on dental materials has been studied, with applications including bonding porcelain brackets and the surface treatment of leucite and alumina-based ceramics.\(^12\) It has been speculated that lasers can affect ceramic surface water and create microexplosions, which could lead to surface abrasion and, thus, increase surface roughness.\(^12\) Li et al reported that a neodymium-doped yttrium aluminum garnet (Nd:YAG) laser may improve the bond strength between a resin cement and ceramic by creating roughness on the ceramic surface.\(^15\) Roughening the ceramic surface with a Nd:YAG laser, after sandblasting, has also been reported to improve the bond strength between ceramic and resin cement.\(^16\)

Measuring the bond strength is one method to evaluate the effectiveness of an adhesive system and, consequently, to predict performance in vivo. Tensile and shear tests are mostly used for this purpose. Nonetheless, many studies have reported that the type of fracture occurring after a shear bond test is often cohesive (inside the resin cement) rather than adhesive (at the interface).\(^17\) Cohesive fracturing is rarely observed in clinical bonded restorations. In contrast, in vitro tensile bond testing causes more adhesive fractures, which is more useful for evaluating real bond strength.\(^18\) The results of these tests are severely affected by specimen geometry and nonuniform distribution of stress during force application.\(^19\) The microtensile bond strength test was introduced by Sano et al\(^20\) and concentrated the load on the adhesive interface more accurately. The specimen surface in this test is small (\(\approx 1 \text{ mm}^2\)), which leads to a more-uniform stress distribution during loading and results in a higher bond strength and fewer cohesive fractures.\(^19-21\) The microtensile test allows a more suitable settling of the specimens, more favorable stress distribution, and more sensible evaluation of bond strength.\(^22\)

The purpose of this study is to evaluate microtensile bond strength (\(\mu\)TBS) between a resin cement and silica-based or yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramics using a combination of different surface treatment methods, including hydrofluoric acid, sandblasting, and laser etching. The null hypothesis of this study is that there is no difference in long-term ceramic/cement bond strength using different surface treatment methods.

### Materials and methods

Blocks of lithium-disilicate ceramic (18.3 × 14.65 × 12.7 mm\(^3\)), IPS e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein) and zirconia ceramic (13.24 × 13.24 × 13.24 mm\(^3\)), IPS e.max ZirCAD (Ivoclar Vivadent) were purchased. Surfaces considered for bonding were all polished and cleaned using the following procedure: The surfaces were polished with 600-, 800-, and 1200-grit silicon carbide paper with water cooling, and cleaned by ultrasonication (Sterling Time Sonics; GVN Corp., Auburn Hills, MI) in 96% ethanol-water solution for 10 minutes. A two-piece brass mold was fabricated for each ceramic block, and each block was duplicated in composite resin (Clearfil AP-X; Kuraray, Kurashiki, Japan).

The six randomized treatment groups are outlined below. Surface treatment methods used in the study groups are summarized in Table 1. The same operator performed all the procedures.

Lithium disilicate ceramic blocks were randomly divided into three groups (one block per group) according to the type of surface treatment:

- **LiSa (lithium disilicate–sandblasted):** Ceramic surfaces in this group were sandblasted using 50-\(\mu\)m \(\text{Al}_2\text{O}_3\) particles under a pressure of 2.8 bar using a fine airborne-particle abrasion unit (Neosab3; TISSIDental, Milan,
Table 1 Surface treatments used in different study groups

<table>
<thead>
<tr>
<th>Study group</th>
<th>Ceramic</th>
<th>Surface treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 LiSa</td>
<td>Lithium disilicate</td>
<td>Sandblast–silane</td>
</tr>
<tr>
<td>2 LiSaE</td>
<td>Lithium disilicate</td>
<td>Sandblast–etch–silane</td>
</tr>
<tr>
<td>3 LiSaLa</td>
<td>Lithium disilicate</td>
<td>Sandblast–laser–silane</td>
</tr>
<tr>
<td>4 ZiSa</td>
<td>Zirconia</td>
<td>Sandblast–zirconia primer</td>
</tr>
<tr>
<td>5 ZiLa</td>
<td>Zirconia</td>
<td>Laser–zirconia primer</td>
</tr>
<tr>
<td>6 ZiSaLa</td>
<td>Zirconia</td>
<td>Sandblast–laser–zirconia primer</td>
</tr>
</tbody>
</table>

Zirconia ceramic blocks were also randomly divided into three groups (one block per group) according to the type of surface treatment:

(i) ZiSa (zirconia–sandblasted): The surface was initially sandblasted and cleaned using the same procedure as for the LiSa group. Zirconia primer (Monobond Plus; Ivoclar Vivadent) was then applied to the surface for 60 seconds and then dried under airflow.
(ii) ZiLa (zirconia–laser irradiated): After cleaning the surface, the Nd:YAG laser irradiated the surface using the same parameters outlined for the LiSaLa group. The zirconia primer was then applied for 60 seconds, and the surface was dried under airflow.
(iii) ZiSaLa (zirconia–sandblasted–laser irradiated): The surface underwent sandblasting, cleaning, Nd:YAG laser irradiation, zirconia primer application, and drying, all as outlined above.

After preparing the ceramic blocks, equal amounts of luting cement pastes A and B (Panavia F 2.0; Kuraray) were mixed thoroughly for 20 seconds, according to manufacturer instructions. Cement was applied to the surfaces of the ceramic and composite block, and the composite block was then placed onto the ceramic block. The blocks were placed into a loading unit (Dorsa, Tehran, Iran) under a 75 N load, according to ISO 4049–2009 instructions, and excess cement was removed. The cement was light-cured (Demi) for 40 seconds on each side of the specimen. The cement surface was then protected with an oxygen barrier using oxyguard gel (Oyiguard II; Kuraray) for 3 minutes. The oxyguard gel which was then removed by washing with distilled water. The block sets were stored in distilled water at 37°C for 24 hours. In the next step, the bonded blocks were embedded in acrylic resin. To obtain bar-shaped specimens with approximately 1-mm² cross-sectional areas, the blocks were sectioned using a low-speed cutting device (Isomet low speed saw; Buehler, Lake Bluff, IL). Each block was first cut longitudinally into a series of 1-mm thick slabs, then rotated 90°, and the cutting procedure was repeated. Bar-shaped specimens obtained from each block were considered for the study. The size of the specimens was 1.00 ± 0.02 mm². The specimens were placed in a thermocycler (Dorsa) and thermocycled between water baths at 5°C and 55°C, with a 20-second dwell time per bath and a 10-second transfer time between baths, for 3500 cycles.

After thermocycling, the two ends of each specimen were attached with cyanocrylate adhesive (Mitreapel; Beta Kimya, Instanbul, Turkey) to the microtensile grip of a universal testing machine (Santam, Tehran, Iran). Microtensile bond strength testing was then performed. Specimens were put under tension at 0.5 mm/min speed and a 500 N maximum force until fracture. The interfacial area of the fractured specimen was then measured using a digital caliper (Mitutoyo Corp., Tokyo, Japan). The bond strength (σ, MPa) was then calculated according to the formula \( \sigma = \frac{L}{A} \), where \( L \) is the load (in N) required to fracture the specimen, and \( A \) is the interfacial area (mm²).

To determine the type of fracture in each specimen, the fractured surface was observed under a stereomicroscope (Olympus, Tokyo, Japan) at 10× magnification. The type of fracture was reported as either adhesive (a fracture at the interface between the ceramic and resin cement), cohesive (a fracture inside the resin cement itself), or mixed (a fracture with some resin cement remaining at the interface).

For each study group, one extra block was prepared for topographical analysis. After surface treatment, each block was dried, sputter-coated with gold, and observed using scanning electron microscopy (SEM). The surface morphology of each specimen was examined for roughness and porosity, with the roughness factor (Ra) and surface area (SA) measured using image analysis software (ImageJ, National Institutes of Health, Bethesda, MD). The SEM images were analyzed for surface topography and the underlying microstructure, which were compared among the different surface treatments.
Table 2 Statistical analysis of microtensile bond strength (MPa)

<table>
<thead>
<tr>
<th>Experimental groups</th>
<th>Group size</th>
<th>Minimum</th>
<th>Maximum</th>
<th>Mean</th>
<th>Standard error of mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>LiSaE</td>
<td>25</td>
<td>6.37</td>
<td>34.51</td>
<td>23.29a</td>
<td>1.53</td>
</tr>
<tr>
<td>LiSa</td>
<td>19</td>
<td>4.38</td>
<td>42.38</td>
<td>17.01ab</td>
<td>2.52</td>
</tr>
<tr>
<td>LiSaLa</td>
<td>10</td>
<td>1.95</td>
<td>10.15</td>
<td>6.05c</td>
<td>0.81</td>
</tr>
<tr>
<td>ZiSa</td>
<td>10</td>
<td>6.48</td>
<td>25.99</td>
<td>12.60bc</td>
<td>2.08</td>
</tr>
<tr>
<td>ZiSaLa</td>
<td>3</td>
<td>4.26</td>
<td>6.10</td>
<td>5.08c</td>
<td>0.54</td>
</tr>
<tr>
<td>ZiLa</td>
<td>0</td>
<td>——</td>
<td>——</td>
<td>——</td>
<td>——</td>
</tr>
</tbody>
</table>

Mean values and standard error of mean are from different independent replicates of groups, and different letters indicate a significant difference between the values of pairs of groups within columns at $p \leq 0.01$, according to Duncan’s multiple comparisons test.

electron microscopy (SEM; Cam Scan MV 1300, Oxford Instruments, Abingdon, UK) at 200× and 600× magnification.

Statistical analysis

In this study, the Shapiro-Wilk test was used to determine the normal distribution of the raw data. According to the normality test results, one-way ANOVA was used to compare the $\mu$TBS of each group. To compare two groups, Duncan’s multiple range test was used ($p \leq 0.01$). The first kind of error was considered to be $\alpha = 0.01$ in this study. The number of specimens, mean, standard error of mean, minimum, and maximum values of $\mu$TBS are presented in Table 2.

Results

All specimens in the ZiLa group debonded during cutting or thermocycling (pretest failure). As such, this group was excluded from the study.

The mean of $\mu$TBS was calculated for each group using SPSS software, based on the sum of the data and the number of samples obtained. ANOVA indicated that different ceramic treatments and resin cements significantly affected the $\mu$TBS ($p \leq 0.01$). Mean comparison of $\mu$TBS using Duncan’s multiple range test showed no significant differences between the LiSaE and LiSa groups ($p > 0.05$), but significant differences between the LiSaE group and all other groups ($p \leq 0.01$). No significant differences were found between the ZiSaLa and ZiSa groups ($p > 0.05$).

The highest $\mu$TBS obtained in this study was for LiSaE group (23.29 MPa), while the lowest was obtained for ZiSaLa (5.08 MPa). The modes of failure in all groups were mostly adhesive (57% to 80%).

SEM analysis of the lithium disilicate ceramic surface after sandblasting and laser irradiation revealed an obvious surface roughness (Fig 1), whereas the specimen surfaces of lithium disilicate ceramic after sandblasting and acid etch exhibited a moderately rough surface with some grooves (Fig 2). Laser-irradiated zirconia surfaces with or without sandblasting revealed large deep pits with surface cracks (Figs 3 and 4); however, the texture of sandblasted zirconia surface was less rough than that of other zirconia groups without surface cracks (Fig 5).

Discussion

This study evaluated the influences of several combined surface treatment methods on the strength of bonding between zirconia- and lithium disilicate-based ceramics and a resin cement. The results of this study reject the null hypothesis, because various
surface treatments affected the $\mu$TBS between the ceramic and resin cement.

Given that most clinical fractures of full ceramic restorations begin in the cement or internal surface of these restorations, the continuity of adhesive cement with the ceramic surface has a significant role in the durability of these restorations.\textsuperscript{13} Furthermore, bonding these restorations with resin cement is recommended for improving marginal integrity and retention. Therefore, to form a strong bond between ceramic and resin cement, ceramic surface treatment is obligatory.\textsuperscript{1} Etching the internal surface of full ceramic restorations made from glass ceramics (including lithium disilicate-based ceramics) with hydrofluoric acid, followed by silane application, has been proven to be an effective method for bonding with resin cements,\textsuperscript{24-26} and is considered the gold standard in evaluating bond strength; however, some studies have recommended eliminating HF etching, only applying silane using a special technique.\textsuperscript{27}

In some new high-strength ceramics, such as zirconia ceramics, the glassy phase does not exist. This has led to standard surface treatments not effectively developing durable bonds between ceramics and resin cement.\textsuperscript{8,9} Therefore, it is necessary to find a suitable surface treatment method to produce a long-lasting bond between zirconia ceramic and resin cement.

In this study, ceramic specimens were bonded to composite blocks instead of dental tissues. The structure of the composite blocks was homogeneous, thus preventing errors in interpreting the bond strength that can be caused by the heterogeneous microstructure of dentin.\textsuperscript{28}

In this study, the $\mu$TBS was used to evaluate bond strength. This test is more desirable than the shear bond test, which mostly results in cohesive fractures far from the resin/ceramic interface and errors in bond strength evaluation.\textsuperscript{29,30} Sano et al demonstrated that tensile bond strength has an inverse relationship with the cross-sectional area of the bonded surface, and recommended using the microtensile bond test.\textsuperscript{20}

Comparing the $\mu$TBS of studied groups, the LiSaE group showed the highest bond strength, although there was no significant difference between the LiSaE and LiSa groups ($p > 0.05$). These findings showed that etching the lithium
disilicate ceramic surface and applying silane created an acceptable bond with resin cement. IPS e.max CAD ceramic is a glass ceramic based on lithium disilicate that, after sintering and crystallization, contains lithium disilicate crystals buried in glassy phase. Because of the silica phase present in this ceramic, etching with 9% hydrofluoric acid can partially dissolve the glassy matrix and increase the amount of silica phase available for bonding. Therefore, the chemical bonds between the resin cement and ceramic surface through silane molecules are enhanced.

Using silane to increase the bond strength between the resin cement and ceramic surface has been studied previously. Silane is a bifunctional molecule that hydrolyzes when reacted with water to form silanol (-SiOH) groups. These groups can react with silica available on the surface of glass ceramics to form siloxane (-Si–O–Si–O-) networks. Meanwhile, the methacrylate end of the silane molecule undergoes free-radical polymerization, reacting with methacrylate groups in the resin cements to form chemical bonds. Ozcan and Vallittu evaluated the influence of acid etching and sandblasting on some ceramics, reporting that hydrofluoric acid etching followed by silane application was the most effective surface treatment method for glass ceramics. This was in agreement with the findings of this study.

Sandblasting with aluminum oxide particles has been recommended as a method for roughening the ceramic surface and improving surface characteristics, including surface energy and wettability. Kiyani et al also found that etching with hydrofluoric acid after sandblasting, followed by silane application, was an effective method for preparing a long-lasting bond between lithium disilicate ceramic and resin cement.

In this study, a special method was used to apply silane to the ceramic surface. After sandblasting and applying silane to the ceramic surface, it was dried with hot airflow, rinsed with boiling water, and then dried again with hot airflow. When silane is applied to the ceramic surface, it forms three layers, in which the two superior layers are physisorbed. The adhesion between these layers and the lower layer is weak, while only the lower layer is strongly chemically bonded to the surface. Rinsing with boiling water removes the two upper layers, leaving just the lower bonded layer intact. Meanwhile, drying silane with hot airflow has been shown to form siloxane bonds via condensation reactions, which helps to increase bond strength. The result of this study showed that applying silane to the sandblasted glass ceramic surface using the method mentioned above obtained a bond between the lithium disilicate ceramic and resin cement similar in strength to that achieved by hydrofluoric acid etching. Therefore, the need to etch with hydrofluoric acid is eliminated, which is advantageous because of the hazards HF poses to patients and dental staff, and its destructive effect on glass ceramic flexural strength. These results are in agreement with the findings of Hooshmand et al, who recommended the same silane application method.

Comparisons between the μTBS in the LiSaE and LiSaLa groups showed that values obtained for the LiSaE group were significantly higher (p < 0.01). This demonstrated that using a Nd:YAG laser with the parameters applied in this study might not be effective for improving the μTBS between lithium disilicate ceramic and resin cement. Laser energy is thought to affect ceramic surface water, achieving surface roughness by creating microexplosions, and, thus, improving resin/cement bonding. The parameters employed in laser irradiation are important for determining its effect on the ceramic surface. The parameters applied to surface treatment of the lithium disilicate ceramic in our study were determined based on Kara et al’s findings of no significant differences between surface roughness resulting from Nd:YAG laser irradiation and hydrofluoric acid etching. However, in this study, SEM analysis (Fig 1) showed that laser irradiation under the applied parameters resulted in destruction of the ceramic surface structure in the LiSaLa group in comparison with that of the LiSaE group (Fig 2). This destruction was speculated to cause the significant decrease in observed bond strength. Furthermore, considering that the bond strength in the LiSaLa group was significantly lower than that of the LiSa group (p < 0.01), we can conclude that Nd:YAG laser irradiation, using the parameters applied in this study, resulted in a decreased bond strength between the ceramic and resin cement, and, therefore, should not be used in practice.

Comparing the μTBS of the LiSaE group (considered as gold standard in this study) and the ZiSa and ZiSaLa groups showed that the ZiSa and ZiSaLa bond strength were significantly lower (p ≤ 0.01). Also, as mentioned earlier, all ZiLa specimens were debonded during preparation or thermocycling. It can be speculated that using the Nd:YAG laser alone (with the applied parameters) or after sandblasting would result in lower bond strength between the zirconia ceramic and resin cement compared to the gold standard group.

High-magnification SEM analysis of laser-treated zirconia specimens, with or without sandblasting, revealed microcracks, along with some areas of large deep pits (Figs 3 and 4). This indicated that destruction of the ceramic surface resulted from Nd:YAG laser irradiation under the parameters applied in this study. Choosing an inappropriate Nd:YAG laser output power has been shown to cause thermal damage in the superficial layer of the zirconia ceramic. This layer easily separates from underlying layers due to weak attachment, causing specimen debonding. It can be assumed that the output power selected in this study was inappropriate, thus causing debonding in all ZiLa specimens and most specimens in the ZiSaLa group. These results were in accordance with those of Akyil et al, who reported that Nd:YAG laser irradiation on the zirconia surface resulted in reduced bond strength between the ceramic and resin cement compared to the untreated group. The results of Liu et al’s study, in which they suggest that different Nd:YAG laser irradiation parameters do not improve the bond strength between zirconia ceramic and resin cement compared to a control group, were also in agreement with those of this study. However, Paranhos et al reported that Nd:YAG laser, either with or without sandblasting, could create higher bond strength between zirconia ceramic and resin cement compared to sandblasting alone. The difference could be because they evaluated bond strength using the shear bond test. Furthermore, thermocycling was not used to simulate mouth conditions.

The bond strength values obtained from different bond tests can be a valuable indicator for evaluating resin/ceramic bonds, but only when the fracture occurs in the adhesive area. In this
study, the most common type of fracture in all groups was ad-
hesive (occurring in the resin/ceramic interface), showing that
stress had been transferred to the bonded area in a homogeneous
manner.

The mechanical properties and phase changes of zirconia
were not evaluated after the proposed treatments in this study.
This might be a limitation, as mechanical stress can result
from tetragonal-to-monoclinic phase changes in zirconia-based
ceramics.\(^1\) Another limitation was that the initial bond strength
was not evaluated, and should be assessed in future studies.
Considering that bond strength tests are only one parameter
for predicting clinical outcomes, long-term clinical trials
are suggested for evaluating the methods proposed in this
research.

**Conclusions**

Allowing for the limitations of this study, it was concluded
that:

1. Treating a lithium disilicate ceramic surface with sand-
blasting and acid etching produced the highest \(\mu\)TBS.
2. When silane was applied in the proposed manner to a
sandblasted lithium disilicate surface, the bond strengths
were similar to those obtained by sandblasted and etched
surfaces.
3. Applying a Nd:YAG laser with or without other treatment
methods to the surface of both ceramics tested reduced
the bond strength between the ceramic and resin.
4. The most frequent mode of failure in all groups was
adhesive, occurring at the interface between the ceramic
and resin cement.

**References**

zirconium oxide ceramic surface treatments on the bond strength
methods on the microtensile bond strength of phosphate
monomer-based cement on zirconia ceramic in dry and aged
3. Blatz MB: Long-term clinical success of all-ceramic posterior
aging on microtensile bond strength of dual-cured resin cements
to pre-treated sintered zirconium-oxide ceramics. Dent Mater J
2009;25:392-399
5. Blatz MB, Sadan A, Kern M: Resin-ceramic bonding: a review of
methods and durability. Dent Mater J 1998;14:64-71
7. Calamia JR: Etched porcelain veneers: the current state of the art.
Quintessence Int 1985;1:5-12
9. Ozcan M, Vallittu PK: Effect of surface conditioning methods on
the bond strength of luting cement to ceramics. Dent Mater J
2003;19:725-731
hydrofluoric acid etching duration on the roughness and flexural
strength of a lithium disilicate-based glass ceramic. Braz Dent J
2011;22:45-50
11. Hooshmand T, Parvizi Z, Keshvad A: Effect of surface acid
etching on the biaxial flexural strength of two hot-pressed glass
bond strength of composite resin to glass-infiltrated alumina
2010;28:341-346
strength of a resin cement to glass infiltrated zirconia-reinforced
ceramic: the effect of surface conditioning. J Prosthodont J
2006;22:283-290
microshear bond strength to zirconia ceramic using Er:YAG and
tribochemical silica coating as surface conditioning. Lasers Med
Sci 2013;30:787-795
15. Li R, Ren Y, Han J: Effects of pulsed Nd:YAG Laser irradiation
on shear bond strength of composite resin bonded to porcelain. O
Hux Xi Kou Quan Yi Xue 2000;18:377-379
bond strength between a resin cement and an aluminous
ceramic treated with Nd:YAG laser, Rocatec system, or
aluminum oxide sandblasting. Photomed Laser Surg
2005;23:543-548
17. Della Bona A, Northeast SE: Shear bond strength of resin bonded
18. Chadwick RG, Mason AG, Sharp W: Attempted evaluation of
three porcelain repair systems—what are we really testing?. J
Oral Rehabil 1988;25:610-165
bond strength testing of luting cements to prefabricated
CAD/CAM ceramic and composite blocks. Dent Mater J
2003;19:575-583
mineralized and demineralized human and bovine dentin. J Dent
Res 1994;73:1205-1211
21. Della Bona A, Van Noort R: Shear vs. tensile bond strength of
methods on the bond strength of resin to zirconia-alumina
ceramic: microtensile versus shear test. Dent Mater J
2008;27:849-855
23. Hooshmand T, Van Noort R, Keshvad A: Bond durability of the
resin-bonded and silane treated ceramic surface. Dent Mater
2002;18:179-188
24. Tylka DF, Stewart G: Comparison of acidulated phosphate
fluoride gel and hydrofluoric acid etchants for porcelain-
25. Aida M, Hayakawa T, Mizukawa K: Adhesion of composite to
porcelain with various surface conditions. J Prosthet Dent
1995;73:464-470
Prosthet Dent 1998;60:443-447
27. Hooshmand T, Van Noort R, Keshvad A: Bond durability of the
resin-bonded and silane treated ceramic surface. Dent Mater
2002;18:179-188
treatment on the resin bond to zirconium-based ceramic. Int J
Prosthodont 2005;18:60-65
29. Escribano NI, Del-Nero MO, Macorra JC: Inverse relationship
between tensile bond strength and dimensions of bonded area. J