Ceramic Repair: Influence of Chemical and Mechanical Surface Conditioning on Adhesion to Zirconia

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Abstract

Aims: To evaluate the influence of mechanical surface treatment and chemical primer application on the composite shear bond strength to zirconia.

Methods: Eighty blocks of Lava Frame Zirconia were randomly assigned to the experimental groups, according to the several possible combinations between mechanical (untreated/aluminum oxide sandblasting) and chemical treatment (no primer/Monobond Plus/Experimental Zirkon-Primer/Z-Prime Plus) (n=10). Adhesive system and composite resin were then applied, and samples were thermal cycled (5ºC-55ºC, 500 cycles). Shear bond strength tests and failure mode analysis were performed. SBS data were analyzed by two-way ANOVA, followed by Student-Newman-Keuls post-hoc tests, and failure mode by Kruskal-Wallis and Mann-Whitney tests (p<0.05).

Results: Shear bond strength mean values ranged from 6.9 to 23.2 MPa. Surface treatment with aluminum oxide sandblasting allowed a statistically significant increase (p<0.05) in bond strength values. The group treated with Z-Prime Plus achieved (p<0.05) higher bond strength results than the other chemical treatments.

Conclusions: The surface mechanical treatment almost doubled the bond strength values. Z-Primer Plus have enhanced bond strength values of composite to zirconia.

Key Words: Zirconia, Shear Strength, Dental restoration repair, Primer, Air abrasion

Introduction

Zirconia has been increasingly used in dentistry due to its exceptional bio-compatibility, mechanical properties, esthetic and the high demand for metal-free [1-3]. Although, it can be used as single restorative material, it is mainly used as a core material, veneered by a stratified feldspathic ceramic [4,5]. Zirconia core rarely fractures [6,7], but the veneering feldspathic ceramic has shown a high incidence of chipping and delamination exposing the underlying zirconia [8], when compared to conventional metal-ceramic restorations [9].

The location and extent of the fracture will determine either the restoration’s repair or replacement. The lower cost involved and the possibility of being performed in just one appointment, are some of the advantages of repair [10]. To perform ceramic restorations repair, some systems, using composite resins, were developed [11].

With minor differences in the literature, the protocol for bonding composite resins to feldspathic ceramic advocates the use of sandblasting, with aluminum oxide, or hydrofluoric acid conditioning to create micro-retentions, followed by silane application, to chemically interact with silica [11-13].

Since zirconia cannot be effectively conditioned with hydrofluoric acid and has no silica in its structure, the feldspathic ceramic adhesion protocol is not efficient [14]. New methods of surface conditioning have been proposed to increase bond strength between composite resins and zirconia, such as silica coating techniques and zirconia primers [14-21]. These new developed primers usually contain phosphate ester, carboxylic acid and other organic acidic monomers that may react with hydroxyl groups presented on zirconia surface, allowing the contact of the composite resin with zirconia [16,22-24].

Aims

The aims of this in-vitro study were to assess the effect of a mechanical surface treatment technique and three different priming agents on the shear bond strength (SBS) of a composite resin to zirconia. The null hypotheses to test were that 1) mechanical treatment with 50 μm Al,O₃ does not increase the SBS of resin composite to zirconia, and 2) chemical treatment with zirconia primers does not increase the SBS of resin composite to zirconia.

Methods

The sample size (n=10) was estimated with a power analysis to provide statistical significance alpha=0.05 at an 80% power.

Eighty blocks of pre-sintered high-purity zirconium-oxide ceramic (Lava Frame Zirconia, 3 M ESPE, Seefeld, Germany) sized 12x12x6 mm were prepared with an Isomet 1000 precision saw (Buehler Ltd, Lake Bluff, Illinois, USA), and polished with silicon carbide paper strips grit-220, 400 and 600, under running water for 5 seconds.

After sintered, the zirconia blocks were randomly assigned to two surface treatment groups. One group was submitted to sandblasted with 50 μm aluminum oxide particles for 5 seconds at a pressure of 40 psi and a distance of 10 mm, and the other had no surface treatment. Specimens were then immersed and ultrasonically washed for 1 minute in distilled water.

Each group was further divided into four subgroups, according to the primer application: A) control group (no primer), B) Monobond Plus (Ivoclar Vivadent AG, Schaan, Liechtenstein), C) Z-Prime Plus (Bisco, Schaumburg, Illinois, USA), D) Experimental Zirkon-Primer #1043073 (Voco, Cuxhaven, Germany).

One layer of the selected primer was applied to a
standardized 3 mm diameter area, left undisturbed for 60 seconds and air dried for 5 seconds.

The restorative procedure was the same in every specimen. First, the fluid resin from Adpter Scotchbond Multi-Purpose system (3M ESPE Dental Products, St. Paul, Minnesota, USA) was applied and light cured for 10 seconds. Then, 2 mm increments of restorative composite resin Tetric EvoCeram (Ivoclar Vivadent AG, Schaan, Liechtenstein) were applied and light cured for 20 seconds each. Materials were light cured using an Ortholux LED Curing Light (3M Unitek, Puchheim, Germany), with 800 mW/cm².

After 24 hours storage period in an incubator at 37°C and 100% humidity, all specimens were thermal cycled (500 cycles at 5°C and 55°C), and included in a single plane lap shear bond strength device [25]. Tests were performed in a universal machine (Instron 4502, Instron Ltd., Bucks, England) with a crosshead speed of 0.5 mm/min and a load cell of 1 kN.

Failure mode was determined using a stereomicroscope (EMZ-8TR, Meiji Techno Co., Saitama, Japan) with a 20x magnification and classified, by two independent observers as: adhesive, when the failure occurred at the adhesive interface, or mixed, when the combination of cohesive in composite and adhesive failure occurred [17,26,27].

The results were statistically analyzed using IBM SPSS Statistics 20 (SPSS Inc., Chicago, Illinois, USA). After assessing normality and homoscedasticity with Kolmogorov-Smirnov and Levene’s tests, shear bond strength data was analyzed using two-way ANOVA, followed by Student-Newman-Keuls post-hoc tests. Kruskal-Wallis and Mann-Whitney tests were used to analyze failure mode data. A statistical significance of 0.05 was set for both statistical analyses.

Results

Shear bond strength (SBS) values and failure mode distribution are presented in Table 1. The bond strength mean values ranged from 6.9 MPa, in the Experimental Zirkon-Primer group with no surface treatment, to 23.2 MPa, in the Z-Prime Plus sandblasted group.

Two-way ANOVA showed that both mechanical (p<0.001) and chemical surface treatment (p=0.001) significantly influenced SBS values (Table 2). No interaction was detected between these two factors (p=0.515). Aluminum oxide sandblasting achieved higher SBS values than no mechanical surface treatment (Figure 1). Regarding the chemical treatments Z-Prime Plus achieved higher (p<0.05) SBS values than all the other groups (Figure 2). No statistical differences were found between the control, Monobond Plus and Experimental Zirkon-Primer #1043073.

Mann-Whitney test revealed a statistically significant (p=0.002) influence of the surface treatment on the failure mode. Sandblasting predominantly led to mixed mode, while no mechanical surface treatment resulted in mainly adhesive failures. Kruskal-Wallis test showed no statistical differences among the zirconia primers (p=0.141).

Discussion

The direct extrapolation of in vitro studies results to the clinical performance of restorative materials should be carefully made. However, achieving high bond strength values is important for the clinical success of any restoration [23,28].

As expected, and in agreement with previous reports, ceramic surface pre-treatment with aluminum oxide sandblasting yielded higher SBS values than those obtained in the non-sandblasted group [29-31]. Ceramic sandblasting increases surface roughness, area and energy, improving the adhesive forces achieved [15,29,30,32]. This improvement has also been justified by the cleaning effect and chemical activation of the surface [27]. Despite concerns that the impact of aluminum oxide particles on the ceramic surface may generate fracture lines that lead to a decrease in cohesive strength, some researchers report that a transformation from tetragonal to monoclinic phase on Yttrium stabilized tetragonal zirconia polycrystals due to sandblasting counteracts the effect of these flaws on the material strength [14,33].

Even though ultrasonic cleaning is not reproducible in a

<p>| Table 1. Descriptive statistics of shear bond strength (SBS) and failure mode ( (n=10) ). |</p>
<table>
<thead>
<tr>
<th>Mechanical treatment</th>
<th>Chemical treatment</th>
<th>SBS (MPa)</th>
<th>Failure mode [n (%)]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean ± SD</td>
<td>Adhesive</td>
<td>Mix</td>
<td></td>
</tr>
<tr>
<td>No treatment</td>
<td>No primer</td>
<td>7.2 ± 3.5</td>
<td>7 (70%)</td>
</tr>
<tr>
<td></td>
<td>Monobond® Plus</td>
<td>7.7 ± 3.6</td>
<td>7 (70%)</td>
</tr>
<tr>
<td></td>
<td>Z-Prime™ Plus</td>
<td>11.6 ± 6.7</td>
<td>4 (40%)</td>
</tr>
<tr>
<td></td>
<td>Experimental Zirkon-Primer</td>
<td>6.9 ± 2.8</td>
<td>6 (60%)</td>
</tr>
<tr>
<td>50 µm Al₂O₃ Sandblasting</td>
<td>No primer</td>
<td>13.7 ± 4.2</td>
<td>4 (40%)</td>
</tr>
<tr>
<td></td>
<td>Monobond® Plus</td>
<td>15.3 ± 6.4</td>
<td>4 (40%)</td>
</tr>
<tr>
<td></td>
<td>Z-Prime™ Plus</td>
<td>23.2 ± 4.1</td>
<td>1 (10%)</td>
</tr>
<tr>
<td></td>
<td>Experimental Zirkon-Primer</td>
<td>16.3 ± 10.7</td>
<td>1 (10%)</td>
</tr>
</tbody>
</table>

<p>| Table 2. Two-way ANOVA. |</p>
<table>
<thead>
<tr>
<th>Source</th>
<th>df</th>
<th>Sum of squares</th>
<th>Mean square</th>
<th>F</th>
<th>p*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mechanical treatment</td>
<td>1</td>
<td>1539.8</td>
<td>1539.8</td>
<td>45.9</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>Chemical treatment</td>
<td>3</td>
<td>595.4</td>
<td>198.4</td>
<td>5.9</td>
<td>0.001</td>
</tr>
<tr>
<td>Mechanical * chemical treatment</td>
<td>3</td>
<td>77.5</td>
<td>25.8</td>
<td>0.8</td>
<td>0.515</td>
</tr>
<tr>
<td>Error</td>
<td>72</td>
<td>2416.9</td>
<td>33.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>80</td>
<td>17589.8</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*p<0.05 indicates statistically significant differences.
clinical scenario, the method was used in this study, in order to guarantee a clean surface of the sample.

Thermal cycling was performed, according to the ISO 11405 specifications for testing adhesion to tooth structure. This procedure could be controversial and although it has been stated that 500 cycles do not stand for a sufficient amount of aging [34,35], all specimens were treated in the same way.

The use of zirconia primers has also been described in literature as an effective method to increase bond strength between zirconia and composite resins [16,36,37]. However, in the present study only Z-Prime Plus application resulted in higher SBS than the group without primer. Z-Prime Plus main components are organophosphate and carboxylic acid monomers. Organophosphates monomers have two functional groups: a phosphoric acid group that can bond to zirconia surface oxides and, usually, a methacrylate group to copolymerize with organic monomers of the composite.

Regarding failure mode statistical differences were found only between the two mechanical surface treatments tested with higher number of mixed failures in the sandblasted group. This result matches with the higher mean SBS values observed in this group. As in other studies higher SBS values seem to be related to a predominance of mixed failure modes [20,39]. Techniques and materials used in this study should be tested in more demanding laboratory conditions to closer simulate clinical environment.

Conclusions

Within the limitations of the present study, both null hypotheses have to be rejected, as 1) sandblasting the zirconia surface with 50 μm ALO3 particles, and 2) the chemical treatment with Z-Prime Plus increased the shear bond strength of the resin composite to zirconia.

Acknowledgements

The authors thank VOCO, Ivoclar Vivadent and 3M/ESPE for providing the materials for this study and GITO Laboratory for sintering the zirconia. This work was performed at UICOB, R&D unit nº 4062 of FCT.

References


