Effect of Surface Treatments on the Resin Bond to Zirconium-Based Ceramic

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> Purpose: This study tested the hypothesis that the tribochemical silica coating on ceramic surfaces increases the bond strength of resin cement to a glass-infiltrated zirconium-based ceramic. Materials and Methods: Fifteen blocks of In-Ceram Zirconia from CEREC InLab (5 per group) and 15 composite blocks (Z-250) 5 mm \times 5 mm \times 4 mm were made. The ceramic surfaces were polished, and the blocks were divided into three groups: (1) airborne abrasion with 110-µm aluminum oxide particles; (2) Rocatec system, tribochemical silica coating; and (3) CoJet system, tribochemical silica coating. The ceramic blocks were cemented to the composite blocks using Panavia F according to the manufacturer's specifications. All samples were stored in 37°C distilled water for 7 days and later sectioned in two axes using a diamond disk under cooling to obtain specimens with a cross-sectional area of approximately 1 mm² (n = 45). Each specimen was then attached with cyanoacrylate glue to an adapted device for the microtensile test, which was carried out on a universal testing machine. Results: The results were subjected to ANOVA and Tukey's test. Group 2 (23.0 ± 6.7 MPa) and group 3 (26.8 \pm 7.4 MPa) showed greater bond strength than group 1 (15.1 ± 5.3 MPa). There was no significant difference between groups 2 and 3. All failures were in the adhesive zone. Conclusion: The hypothesis was confirmed-the tribochemical systems increased the bond strength between Panavia F and In-Ceram Zirconia. Int J Prosthodont 2005; 18:60-65.

The bond between feldspar-based porcelains with high silica content and resin cements seems to be well-established, as the bond is provided by the etching with hydrofluoric acid and increased by the silane agent. The latter has the property of increasing the capacity of the cements to flow on the surface and into microretentions, improving the contact with the cements, as well as allowing a bond between the silica contained in the ceramic and the organic matrix of the resin cements through siloxane bridges.^{1–4}

However, hydrofluoric or sulfuric acids and silane coupling are not capable of providing a strong bond to aluminous ceramics with low silica content. This inefficiency has been proven by short- and long-term studies, which show the resistance to degradation of a compact ceramic surface with high crystal content.⁵⁻⁸ Glass-infiltrated zirconia-alumina ceramic (In-Ceram Zirconia, Vita) presents the same characteristics because of its high crystal content (aluminum oxide ± 67 wt%, zirconium oxide ± 13 wt%) and a limited vitreous phase (lanthanum aluminum silicate ± 20 wt%).⁹

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Although there are some studies on bond strength to the zirconium-based ceramics,¹⁰⁻¹² until now no study has investigated the bond strength using tribochemical systems (silica-coated and silanized zirconium ceramic surface) and monomer-phosphatebased resin cement (10-methacryloyloxydecyl-dihydrogen phosphate [MDP]). Moreover, the current study used a microtensile test, which is considered a more sensitive mechanical test, to evaluate the bond strength.¹³⁻¹⁸

The purpose of the present study was to investigate, by means of a microtensile test, the hypothesis that silica coating on ceramic surfaces increases the bond strength of resin cement to a glass-infiltrated zirconium-based ceramic.

Materials and Methods

Fifteen ceramic blocks of In-Ceram Zirconia for CEREC InLab (Vita) were made according to the manufacturer's specifications. The dimensions of these blocks were 5 mm \times 5 mm \times 4 mm.

The bonding surfaces were polished using a 600- to 1,200-grit metallographic paper (3M) in a polishing machine (Labpol 8-12, Extec). Each ceramic block was duplicated in resin composite (Z-250, 3M/ESPE; batch No. 9017B1) using a mold made of impression material (Express, 3M/ESPE; batch No. 7312). Composite layers were condensed (2 mm) and light cured for 40 seconds (XL 3000, 3M/ESPE; light intensity 500 mW/cm², distance 0) until completion of the mold. One resin composite block was obtained for each ceramic block.^{10-12,19,20}

Surface Treatment and Cementation of Ceramic Blocks

The bond surfaces of the ceramic blocks were treated (five blocks per treatment) as follows:

- Conditioning 1 (control): airborne abrasion (Micro-Etcher) with 110- μ m Al₂O₃ particles (blasting procedures: *a* = perpendicular to the surface; *b* = 10mm distance; *c* = 20-second time; *d* = 2.8-bar pressure).^{10-12,19,20}
- Conditioning 2 (Rocatec System, 3M/ESPE; batch No. 363), tribochemical silica coating: airborne abrasion with 110-µm Al₂O₃ particles (blasting procedures a, b, c, and d as above [Rocatec-Pre powder, 3M/ESPE]). Silica coating was then produced by blasting the surface with 110/µm silica-modified Al₂O₃ particles (Rocatec-Plus powder, 3M/ESPE) using the same procedures (a, b, c, and d). Finally, the silane agent was applied (Rocatec-Sil, 3M/ESPE) for 5 minutes.^{10-12,19,20}

 Conditioning 3 (CoJet System, 3M/ESPE; batch No. 68421), tribochemical silica coating: Initially, the surface was treated as in conditioning 1 and then subjected to airborne abrasion with 30-μm Al₂O₃ particles modified with salicylic acid (procedures a, b, c, and d [CoJet-Sand, 3M/ESPE]). Silane agent was then applied (ESPE-Sil, 3M/ESPE).²⁰⁻²²

Other ceramic samples were treated and analyzed under scanning electron microscope (SEM) at 2,000 \times magnification (Jeol JSM-T330A) to observe the topographic patterns obtained with the surface treatments.

Panavia F dual-cure resin cement (Kuraray; batch No. 51133; composition: 78% filler, MDP, dimethacrylates, chemical and photoinitiators), manipulated according to the manufacturer's specifications, was used to cement each ceramic block to its corresponding resin composite block using a Centrix syringe. The ceramic-cement-resin set was placed into a press with the interface perpendicular to a vertical load (750 g/10 min). Any excesses were removed. The cement was light cured for 40 seconds at each margin (XL 3000; light intensity 500 mW/cm², distance 0),²³ and Oxyguard (Kuraray) was applied to the external edge of the interfaces. The set was then removed from the press, rinsed with air-water spray, and stored in distilled water at 37°C for 7 days.^{10–12,19,20}

Production of Specimens for Microtensile Test

Initially, the cemented blocks were bonded with cyanoacrylate glue (Fig 1) to a cylindric metallic base and coupled to an adapted cutting machine. The cuts were made with a slow-speed diamond wheel saw under cooling. The first cut (\pm 0.5 mm) was disregarded because the results could be influenced by excess or absence of resin cement at the interface.^{14,24} Afterward, three cuts approximately 1 mm thick were made (Fig 1). Each slice was then rotated 90 degrees and bonded to other metallic bases (Fig 2). A \pm 0.5-mm portion was disregarded for the same reason described above. Three additional cuts approximately 1 mm thick were made. This procedure was repeated with the other two slices. A total of nine specimens were obtained for each cemented set, 45 specimens per experimental group (Fig 2). The specimens had the following characteristics: untrimmed rectangular (bar specimens); nearly symmetric square cross-sectional area of 1.0 \pm 0.1 mm²; and length of about 10 mm.^{3,20,25-28} Only the internal specimens were used (B2 in Fig 2). Three groups were therefore established:

 Group 1: 45 zirconium ceramic specimens conditioned with Al₂O₃ (control) **Fig 1** (*right*) Cutting of cemented blocks (*left*); three slices are obtained (*right*).

Fig 2 (*below*) Cutting of slices (*A*); specimens obtained from a block (*B1*); specimens used in the study (*B2*); form of specimens (*C*); and adhesive zone (*D*).





- Group 2: 45 zirconium ceramic specimens conditioned with SiO_v (Rocatec)
- Group 3: 45 zirconium ceramic specimens conditioned with SiO_v (CoJet)

Microtensile Testing

Before the test, the adhesive area of each sample was measured using a digital caliper (Mitutoyo). Each specimen was bonded with cyanoacrylate adhesive to an adapted device perpendicular to the force applied, therefore minimizing the bending forces in the adhesive zone during microtensile testing. Only the extremities of the specimens were bonded. This set (device and specimen) was adapted to a universal testing machine (EMIC DL-1000) and subjected to tensile load (cross-head speed of 1 mm/min) until fracture.

Tensile bond strength calculations were made using the formula:

L(K/A)

where L = test load (kgf); A = adhesive area (mm²); and K = constant (acceleration because of gravity, g = 9.8 m/s²). The data obtained were subjected to statistical analysis by analysis of variance (ANOVA) and Tukey's test (α = .05).

The specimens tested were analyzed under optical microscope (Zeiss MC 80 DX) with a magnification of

 $50\times$ to evaluate the failure mode (adhesive, cohesive, or mixed).

Results

The statistical analyses showed that the mean bond strengths of group 2 (23.0 \pm 6.7 MPa) and group 3 (26.8 \pm 7.4 MPa) were statistically significantly superior to that of group 1 (15.1 \pm 5.3 MPa) (*P* < .05; Tukey value = 3.5). There was a significant increase in the microtensile bond strength when the ceramic surface was subjected to the tribochemical treatment (groups 2 and 3). Notwithstanding the groups investigated, all tested samples (100%) presented mixed fractures, namely adhesive failures between resin cement and ceramic, and cohesive failures of the cement.

Discussion

Some studies¹⁰⁻¹² have shown that blasting with AI_2O_3 particles combined with a monomer-phosphate-based resin cement allows a significant bond strength to the yttrium oxide-partially stabilized zirconia ceramic. Our study showed that the groups treated with the Rocatec and CoJet systems (silica coating + silanization) combined with the monomer-phosphate-based resin cement presented higher bond strength compared to the samples blasted with AI_2O_3 particles. Therefore, it is safe to suggest that using a tribochemical system



Fig 3a SEM view of ceramic surface treatment: In-Ceram Zirconia blasted with Al_2O_3 particles.



Fig 3b SEM view of ceramic surface treatment: In-Ceram Zirconia treated by Rocatec system. Silica particles bonded to ceramic surface (*arrow*) can be observed, different from Fig 3a.



Fig 3c SEM view of ceramic surface treatment: In-Ceram Zirconia treated by CoJet system. The same topographic pattern as with Rocatec system is verified (arrow).

combined with monomer-phosphate-based resin cements is the best alternative for the cementation of zirconium ceramic.

The resistance to etching by hydrofluoric acid is well-established because of the inability of this acid to degrade a compact ceramic surface with a high alumina-zirconia content⁵⁻⁸; when this treatment is executed in feldspar-, leucite-, and lithium disilicate-based ceramics, it promotes the dissolution of the glass matrix.²⁻⁴

The bond to the ceramic substrates seems to be related to the presence of silica in the material, which is compatible with the silane agent.¹ The silane coupling agents present a bond function between the silica contained in the ceramic and the organic matrix of the resin cements (siloxane bonds). They also increase the capacity of the cements to flow on the surface and into microretentions, optimizing the microscopic interaction between the ceramics and resin cements.¹⁻⁴ This relationship—silica-silane and bond capacity—is well-established in the feldspar-, leucite-, and lithium disilicate–based ceramics.

In-Ceram Alumina (Vita) presents 80 wt% to 82 wt% alumina and less than 5 wt% silica in its composition.²⁹ These microstructural characteristics limit a reliable bond to resin cements when treated by traditional methods, such as hydrofluoric acid or blasting with Al_2O_3 particles.

One study showed a significant increase of silica in the surface of In-Ceram ceramic (15.8 wt% to 19.7 wt%) after blasting with Rocatec-Plus (silica coating) compared to samples blasted only with Rocatec-Pre (Al_2O_3 particles).²⁹ The study suggested that the silicacoated surface (Rocatec system) could develop a better bond strength between the In-Ceram ceramic and resin cements because of the increase of silica content and the interaction with the silane agent (Rocatec-Sil) and, later, with resinous materials. This increase in bond capacity has been proven in several studies.⁵⁻⁸ It was noted that the silane agent Rocatec-Sil (ymethacryloxypropyltrimethoxysilane [MPS]) presents a chemical bond to the coated silica (Rocatec-Plus) and no bond to the alumina of In-Ceram ceramic.³⁰ Those authors agreed that the silica coating and silanization are crucial to allow a high and stable bond strength to In-Ceram.

By means of SEM (Fig 3), we noticed that the airborne particle abrasion with SiO_x using the Rocatec (group 2) and CoJet (group 3) systems increased the silica content on the ceramic surface compared to the conventional treatment (group 1). The increase of silica content on the ceramic surface has been shown in some studies.^{6–8,19,20,28} This silica coating suggests a better bond strength, which was confirmed by our study.

Procera AllCeram ceramic (Nobel Biocare) presents similar difficulty in surface conditioning as In-Ceram ceramic because of its high alumina content (In-Ceram Alumina \pm 80 wt%, Procera AllCeram \pm 99.9 wt%) and because of its low silica content.⁶ Silica coating and silanization using the Rocatec system in densely sintered alumina ceramics also promotes a significant increase in the bond strength to bis-GMA-based resin cement.³¹

The glass-infiltrated alumina-zirconia ceramic assessed in this study also presents difficulty in surface conditioning because of its high crystal content (aluminum oxide \pm 67 wt%, zirconium oxide \pm 13 wt%, vitreous phases, lanthanum aluminum silicate \pm 20 wt%)⁹ compared to Procera AllCeram (99.9% alumina). Nevertheless, because of the presence of glass on the ceramic surface, it is safe to suggest that the In-Ceram Zirconia can present greater facility for silica coating than ceramics without vitreous phases, such as densely sintered alumina or yttrium oxide-partially stabilized zirconia ceramics. This phenomenon may occur by a better fixation of silica particles in the vitreous phases of the glass-infiltrated ceramics than in the compact surface of densely sintered alumina or yttrium oxide-partially stabilized zirconia ceramics. When comparing the results of another study with the same methodology²⁸ to our results, the densely sintered alumina ceramic seems to present a lower bond strength (AI_2O_3 12.7 MPa; Rocatec 17.1 MPa; CoJet 18.5 MPa)²⁸ than the glass-infiltrated zirconium ceramic we evaluated (AI_2O_3 15.1 MPa; Rocatec 23.0 MPa; CoJet 26.8 MPa). Further studies are necessary to confirm this hypothesis.

Some studies observe that the use of monomerphosphate-based resin cement allows a high and stable bond strength to zirconia ceramic blasted with Al_2O_3 particles.¹⁰⁻¹² Comparing the results of these studies to ours, it can be suggested that the association of monomer-phosphate-based resin cement to tribochemical systems may allow a better bond strength to zirconium ceramics than the same ceramics blasted with Al_2O_3 particles and cemented with the same resin cement. Therefore, there could be an additional effect of the silica coating and chemical bond of the monomer-phosphate to the zirconium-aluminum oxide. Further studies are necessary to confirm this hypothesis.

The CoJet system is similar to the Rocatec treatment method: (1) airborne particle abrasion with AI_2O_3 ; (2) silica coating; and (3) silanization. This system was optimized for intraoral silica coating, allowing direct resin composite repairs of fractured metal-ceramic or metal-free crowns.^{21,22} In the present study, a significant increase in the bond strength of the ceramics treated by the CoJet system (group 3) was observed compared to the ceramics treated conventionally (group 1). The mechanisms to promote bonding for this system are supported by the same arguments as for the Rocatec system.

Although there are some studies^{10–12} on the bond strength (conventional tensile test) of the zirconiumbased ceramics and monomer-phosphate-based resin cements, various investigations^{13–18,24,25} consider this mechanical test limited to evaluate the real bond strength between two substrates. When the aim of a study is to evaluate the adhesive properties of two substrates, one of the fundamental aspects is to use mechanical tests that can record the real bond strength.

The aim of mechanical bond strength tests (eg, tensile, microtensile, shear) is to apply a load on the samples to produce a homogeneous stress at the adhesive interface until rupture of the sample. Untrimmed samples with a nearly square cross-section and cross-sectional area of $1.0 \pm 0.1 \text{ mm}^2$ (microtensile test) exhibit homogeneous stress distribution at the interface, increasing the level of adhesive failure between the ceramic and resin cement.^{26,27} Specimens that exhibit a lower maximum stress have a greater chance of surviving longer.^{25–27} This is thought to provide more accurate results for validating the "adhesive potential" of a bond between two materials.^{3,32}

Thus, for a test to reproduce the real bond strength between an adhesive (resin cement) and a substrate (dental, metallic, ceramic, or polymeric substrate), it is essential that the interfacial zone be the most stressed zone, notwithstanding the mechanical test used.^{3,13,14,32} According to some stress distribution analyses, 15-17,24 some mechanical tests do not actually stress the interfacial zone. The shear test, for example, is criticized because the stress is nonhomogeneously distributed at the adhesive interface, requiring more substrate. Thus, the stress concentrates more in a restricted zone away from the adhesion zone; then, most of the fractures occur in the substrate. This phenomenon prevents the measurement of true interfacial bond strength and limits further improvements in the adhesive systems (underestimated and misinterpreted results). Analyses of the failure mode and fractography reduce the risk of data misinterpretation such as, "The bond strength was higher than the cohesive strength of the substrate."3,32,33

The present study used a method that is considered reliable according to studies carried out to compare different mechanical tests and investigate the bond strength among different materials. Therefore, some important factors of our study should be discussed:

- The cementation of the ceramic blocks to the composite, not to the dentin, is justified by the microstructural variations of the dentin substrate, which could yield false interpretations of the results. The composite blocks, on the other hand, were obtained under similar experimental conditions. Furthermore, the purpose of our study was to evaluate the bond strength of resin cement to a ceramic, varying the ceramic surface treatment, regardless of the dental structure.
- All failures occurred in the adhesive zone (no failure between the resin cement and composite), justifying the cementation between ceramic and composite blocks.
- The untrimmed rectangular specimens (bar specimens) with a cross-sectional area of approximately 1 mm² can be used to evaluate the bond strength between glass-infiltrated zirconium ceramic and resin cement.

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