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Pilot Evaluation of Four Experimental Conditioning Treatments to Improve the Bond Strength between Resin Cement and Y-TZP Ceramic

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Abstract

Purpose: This study evaluated the bond strength between resin cement and Y-TZP ceramic (Yttrium-stabilized Tetragonal Zirconia Polycrystalline) submitted to different surface conditionings.
Materials and Methods: Fifty Y-TZP ceramic discs (Ø = 10 mm) were allocated into five groups: Gr1 (control)—no conditioning; Gr2—tribochemical silica coating (30-μm SiO_2) before sintering; Gr3—air abrasion with 50-μm Al_2O_3 before sintering; Gr4—air abrasion with 110-μ Al_2O_3 before sintering; Gr5 – air abrasion with 50-μm Al_2O_3 after sintering. After specimen preparation, cylinders of composite resin were prepared and immediately cemented onto the ceramic. A shear test was performed.
Results: One-way ANOVA indicated a statistically significant difference among the groups ($p = 0.0019$). The mean shear bond strengths (MPa) were: Gr1 = 4.7 ± 0.8,[b] Gr2 = 4.6 ± 0.9,[b] Gr3 = 6.4 ± 1.0,[a] Gr4 = 6.5 ± 1.8,[a] Gr5 = 6 ± 1.3[ab] (same superscript letter indicates statistical similarity). Adhesive fracture between the ceramic and resin cement was the most common failure. No complete cohesive fracture at the ceramic or composite cylinders was noted.
Conclusion: Within the limitations of this study, additional surface treatment with air abrasion before and after sintering provided a significant increase in bond strength. Tribochemical silica coating before sintering was not effective as a surface treatment.

Clinical trials have reported favorable clinical findings with high-strength ceramics (glass-infiltrated alumina or alumina/zirconia ceramics, yttrium oxide-stabilized tetragonal zirconia ceramics [Y-TZP], and densely sintered alumina ceramics) veneered with feldspathic ceramic.[1] Dental ceramics with an increased crystalline content present greater mechanical properties, with an indication of placement in areas where high resistance is required.[2]

On the other hand, the decrease in the glass phase led to acid-resistant ceramics for the bonding process. Acid etching does not produce significant topographic changes of these high-ceramic substrata, reducing the ability to achieve proper micromechanical bonding of resin materials.[3,4] Due to a microstructure with high-crystalline content (aluminum and zirconia oxides), these ceramics could not be etched by hydrofluoric acid gel, since this agent does not etch the high-strength crystalline phase but the glass phase.[3-9]

Y-TZP ceramic presents a high-initial strength (2000 N) and fracture toughness (9–10 MPa/m$^1/_2$).[10] Yttrium oxide is a stabilizing oxide added to pure zirconia to stabilize it and generate a multiphase material known as "partially stabilized zirconia." Tensile stresses acting at the crack tip induce a transformation of the metastable tetragonal zirconium oxide form into the monoclinic form. This transformation is associated with a localized volumetric increase of 3% to 5%. This increased volume creates compressive stresses around the tip of the crack, which counteracts the external tensile stresses acting on the fracture tip. This physical property is known as transformation toughening.[10] Y-TZP materials are composed of a glass-free polycrystalline microstructure, with approximately 95% zirconium oxide and 5% yttrium oxide (dense high-crystalline microstructure),[10] characterizing Y-TZP as an acid-resistant or nonetchable material[2-5] that is relatively resistant to oxide deposition by air abrasion.

Current studies have indicated that laboratory or chairside tribochemical silica coating by air abrasion with 110-μm and 30-μm silica-coated aluminum particles leads to silica embedding onto the ceramic surface. These surfaces, artificially modified by silica oxide, chemically react to the silane coupling agents, enhancing the bond to resins.[3,5,8,11–18]

Increasing the adhesion between Y-TZP and luting cements is important for all-ceramic inlay-retained fixed partial dentures (FPDs) and resin-bonded FPDs.[19] Although the adhesion to Y-TZP ceramic using resin-cement-containing methacryloxydecyl phosphoric acid monomers (MDP) appears to be stable,[13,14] there is no consensus in the literature, and the findings are contradictory regarding the use of Y-TZP surface conditioning and resin cement.[15,16] Airborne-particle abrasion, tribochemical silica coating, acid etching, plasma spraying, low-fusing porcelain layers, silane application, and heat-induced maturation-selective etching techniques have been evaluated as Y-TZP surface-conditioning methods.[13–16,20–23] Moreover, the damage derived from air abrasion with 110-μm Al_2O_3 particles could induce negative changes in the structure, as a transformation from a tetragonal to a monoclinic phase has been shown to reduce the fatigue strength of zirconia-based ceramic materials.[24,25]

Recently, a simplified approach for cementing inlays, onlays, crowns, and FPDs has been marketed. No dentin-enamel pretreatment is indicated in this one-step technique. The organic matrix consists of multifunctional phosphoric acidic methacrylates, which contribute to the adhesion to tooth tissue. The content of inorganic fillers is about 72wt%. The fillers are of basic nature and are able to undergo a cement reaction with the acidic groups of the functional monomers. Due to the cement reactions, the material's pH value increases from one to six during the setting reaction.[24] The dominant setting reaction starts with free-radical polymerization, which can be initiated both by light or by a redox system, such as, with the reactions of dual-curing composite materials. Additionally, phosphoric acidic methacrylates in the monomer mixture can react with the basic fillers and the hydroxyapatite of the tooth hard tissue. Water is released in this reaction, accelerating the neutralization reaction.[26–28]

Airborne-particle abrasion of Y-TZP surfaces before sintering may improve the resin bond strength to this ceramic and prevent damage to the Y-TZP microstructure. To the current authors' knowledge, as of this writing, no studies have assessed this alternative conditioning method of the Y-TZP surface. The objective of this study was to evaluate the shear bond strength of a resin cement to a Y-TZP ceramic submitted to one of four surface conditioning methods when compared to the no-treatment condition.

Materials and methods

Fifty disc-shaped Y-TZP ceramic specimens (LAVA Frame, lot n. 231719, 3M ESPE, Seefeld, Germany) (Ø = 10 mm) were produced (B2 shade, sintered following manufacturers' recommendations) and embedded in polymethylmethacrylate (Technovit 4071, Heraeus Kulzer, Hanau, Germany) using polyethylene molds, with one side of the disc exposed for study. All specimens were ultrasonically cleaned in 95% ethylic alcohol for 10 minutes (Ultrasonic machine, Vitasonic, Vita, Bad Sackingen, Germany), and allocated into five groups, as described below:

Gr1: no conditioning (control).
Gr2: chairside air abrasion with 30-μm SiO_x (CoJet-Sand, 3M ESPE) before sintering. For air abrasion, a device (CoJet-Prep, 3M ESPE) was used following these parameters: perpendicular to the surface; distance = 10 mm for 15 seconds; pressure = 2 bar.
Gr3: air abrasion with 50-μm Al_2O_3 before sintering (parameters for air abrasion: perpendicular to the surface; distance = 15 mm for 15 seconds; pressure = 4 bar).
Gr4 air abrasion with 110-μm Al_2O_3 before sintering (parameters for air abrasion: perpendicular to the surface; distance = 20 mm for 15 seconds; pressure = 4 bar).
Gr5 air abrasion with 50-μm Al_2O_3 after sintering (parameters for air abrasion: perpendicular to the surface; distance = 10 mm for 20 seconds; pressure = 4 bar).

After surface conditioning, the MPS-based silane coupling agent (Espe-Sil, 3M ESPE) was applied on treated surface, Fifty cylinders of composite resin (Filtek Z250, lot n. 20061219, 3M ESPE) (4.5-mm diameter, 7-mm height) were produced using a template. They were immediately cemented to the treated ceramic surface with a self-adhesive resin cement (RelyX Unicem in capsules, lot n. 260028, 3M ESPE). An Aplicap capsule was activated and processed in a mixing machine. The resin cement was applied onto the composite surface for cementation to the ceramic surface. The cement excess was removed, and the specimens stored for 24 hours in distilled water at 37°C.

For shear testing, a knife-edge set-up was used. Every specimen was attached in the adapted device fixed in a universal testing machine (Instron 4302, Bucks, UK). The knife was kept as close as possible to the Y-TZP surface, and the load was applied perpendicular to the resin cylinder. Shear bond strength was tested at 0.5 mm/min. One-way ANOVA and Tukey's test ($p \leq 0.05$) were performed to analyze the data (Statistix 8.0 for Windows, Analytical Software Inc., Tallahassee, FL).

The fractured surfaces of all tested specimens were first analyzed with stereomicroscopy (200x) (Zeiss Discovery V-20, Carl Zeiss, Oberkochen, Germany) to assign the predominant failure mode: cohesive of cement; or adhesive at cement/ceramic interface. Specimens with representative fractures were chosen for microscopic analysis. The specimens selected for further evaluation were mounted on a metallic stub, sputter coated with gold (DESK II, Denton Vacuum, Moorestown, NJ) and observed under a scanning electron microscope (1000x) (JEOL–JSM–6360, JEOL, Tokyo, Japan).

Results

One-way ANOVA indicated a statistically significant difference in shear bond strength among the groups ($p = 0.0019$). The mean shear bond strengths are plotted in Figure 1.

All tested specimens had predominantly adhesive fractures between the ceramic and resin cement. Residues of resin cement were noted on the ceramic surface. Fractures always occurred at the ceramic/cement interface. No complete cohesive fracture at the ceramic or composite cylinders was noted.

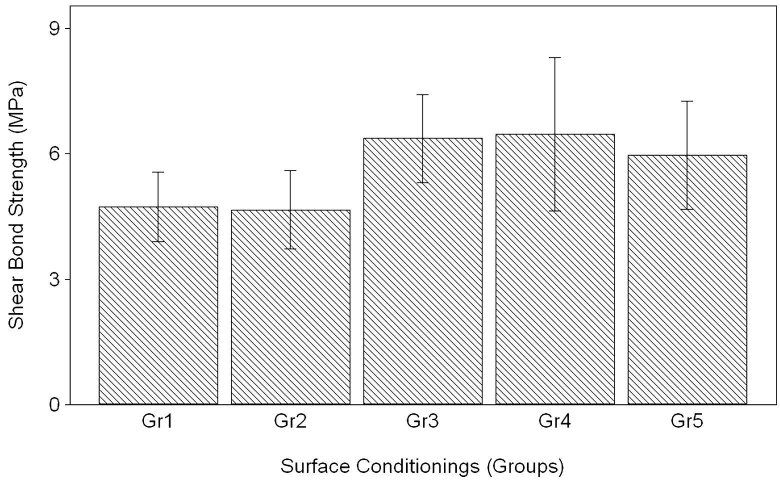

Figure 1 Means and standard deviations of the shear bond strength data (MPa): Gr1 = 4.7 ± 0.8,[b] Gr2 = 4.6 ± 0.9,[b] Gr3 = 6.4 ± 1.0,[a] Gr4 = 6.5 ± 1.8,[a] Gr5 = 6 ± 1.3[ab] (similar letters mean statistical similarity).

Discussion

The current study found higher bond strength results with air abrasion using Al_2O_3 before sintering of the Y-TZP (Gr4 and Gr3), when compared to the nonconditioned control Gr1 (as recommended by manufacturer) and tribochemical silica coating (Gr2). The results obtained with air abrasion using 50-μm Al_2O_3 before (Gr3) and after (Gr5) sintering were statistically similar. Airborne abrasion with Al_2O_3 particles before sintering may become an option in the future if this does not damage the strength properties of the Y-TZP ceramics; however, previous studies on the bonding of different resin cements to Y-TZP ceramic have noted a significant reduction of bond strength after aging.[13-16] Blatz et al[14] obtained shear bond strength values from 16 MPa to 20 MPa in dry conditions, but the bond strengths were significantly reduced after aging (0 to 17 MPa). Özcan et al[15] noted low values (8 to 12 MPa) when the specimens were thermocycled. Özcan et al[16] observed zero values for bond strength after long-term storage. The results in this study, using RelyX Unicem, are lower than the results of Luthy et al[11] (39.2 MPa) in a dry condition. In addition, low values of resin bond strength to Y-TZP ceramic were obtained from the current study, and it may be clinically unacceptable. Thus, future resin bond studies evaluating the effect of these conditioning methods on the mechanical properties of ceramics must be conducted.

The bond strength results observed in this study may have an important clinical impact. Since Y-TZP ceramic surface conditioning (air abrasion before sintering,) would be carried out during the fabrication of the restoration in the laboratory, the cementation procedures of Y-TZP frameworks for crowns or FPDs are simplified. Future studies about the effects of these surface conditionings on Y-TZP ceramic's mechanical properties (flexural strength, Weibull's modulus, fatigue resistance, time of life) should be made. Further controlled clinical trials may also indicate the relevance of the improved resin bond to Y-TZP on the clinical performance of restorations made of feldspar-veneered Y-TZP frameworks.

Strong degradation at the Y-TZP ceramic/resin cement interface has been noted when the specimens are submitted to the severe aging condition.[2-5] Ozcan et al[16] found no adhesion (0 MPa, debonding during aging) between Y-TZP ceramic air abraded with Al_2O_3 and different resin cements (Panavia F2.0, Multilink, Superbond C&B, Quadrant Posterior Dense). Blatz et al[14] noted a decrease in the tensile bond strength values between Y-TZP and two resin cements (adhesive application + Relyx ARC, adhesive application + Panavia F) after long-term aging. Hence, this study did not measure the effects of aging condition on resin bond, a limitation of this investigation.

This study used the shear test to assess the bond strength between resin and Y-TZP ceramic. Many studies using stress distribution analyses reported that some bond strength tests do not appropriately stress the interfacial zone.[27-32] For instance, shear tests have been criticized for developing nonhomogeneous stress distributions at the bonding interface, inducing either an underestimation or a misinterpretation of the results, since failure often starts in one of the substrates and not at the adhesive zone.[29-34] On the other hand, microtensile tests allow better alignment of the specimens and a more homogeneous distribution of stress, in addition to a more sensitive comparison or evaluation of bond strengths.[35-38] A recent investigation[39] showed that either shear or microtensile bond strength tests can be used to evaluate the resin bond to highly crystalline ceramics.

Conclusions

Within the limitations of this study, the following could be concluded:

(1) Additional surface treatment with air abrasion before Y-TZP ceramic sintering provided a significant increase in bond strength ($p < 0.05$).
(2) Tribochemical silica coating before sintering was not effective as a surface treatment to improve bond strength ($p > 0.05$).

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