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Bonding strategies to full-contour zirconia: Zirconia pretreatment with piranha solution, glaze and airborne-particle abrasion

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ABSTRACT

This study aimed to evaluate the effect of various zirconia surface pretreatments on the adhesion between full contour 3Y-TZP zirconia and glaze, and the shear bond strength (SBS) between glazed/3Y-TZP and resin cement. Specimens were allocated into groups: GL-glaze; AL+GL-sandblasting with Al_2O_3+GL; CJ+GL-tribochemical silica coating (Cojet®/CJ)+GL; PS+GL-piranha solution+GL; and CJ. Adhesion between 3Y-TZP and GL was evaluated using the scratch test. Surface topography and glaze thickness were evaluated by using a scanning electron microscope (SEM). For SBS, glazed/3Y-TZP surface was etched with hydrofluoric acid and a silane was applied. For CJ only the silane was applied. Samples were tested after 24 h (24 h wet) or after 15,000 thermal cycles and 90 days storage (thermocycled). After SBS, the type of failure was classified as: adhesive, mixed or cohesive. The data were analyzed using two-way ANOVA and Tukey's test. SEM analysis after scratch test revealed circular cracks in the GL group and conformal cracks in the others groups. SEM micrographs suggested that zirconia specimens submitted to airborne-particle abrasion presents rougher and porous surface when compared to surfaces treated with GL and PS. The glaze layer was approximately 1.86 µm thick in all groups. After 24 h, SBS test showed highest values for AL+GL and CJ+GL and were significantly higher when compared to the GL group. Differences were not significant between PS+GL and the other groups. After aging (thermocycling+storage), groups GL and CJ presented no statistically significant difference compared to 24 h and aged AL+GL, CJ+GL and PS+GL groups. The predominant type of failure was mixed. 3Y-TZP surface treatment with glaze application could be considered as an alternative treatment, since it yielded a similar resin bond strength without the need for airborne-particle abrasion.

1. Introduction

Zirconia is widely used as a dental restorative material due to its proven biocompatibility and superior mechanical properties. In clinical dentistry, to achieve reliable and durable bond strength between silica-based ceramics and resin cements, the use of hydrofluoric acid (HF) followed by the application of a silane coupling agent are currently widely accepted as important steps [1–3]. Among acids, HF is the most commonly used and has been shown to improve the bonding ability between the glassy matrix of silica-based ceramics and resin cement [2,4–6]. HF reacts with the ceramic glass matrix by forming hexafluorosilicates [5,7] thus effectively removing the matrix to expose the crystalline structure and create a rough and retentive surface that increases the mechanical interlocking with the resin cement [5,7]. The next step is the use of a silane coupling agent such as methacryloxypropyl trimethoxysilane (MPS) to improve the chemical bonding between ceramic and resin cement. Briefly, silane molecules react with water resulting in silanol groups (-Si-OH) and form the methoxy groups (-Si-O-CH_3). The silanol group reacts with the silica present on the ceramic surface by forming a siloxane network (-Si-O-Si-O-) [8–10]. However, this micromechanical/chemical bonding mechanism is not effective on 3Y-TZP. This is because HF is unable to successfully erode the highly crystalline surface of these ceramics and therefore does not create the desired microretentive surface topography [11–13]. Thus, discovering the best way to improve bonding and durability of zirconia restorations has been a common goal of researchers.

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Table 1
Full-contour zirconia surface treatments applied for each group.

Groups	Surface treatment
GL	A low-fusing porcelain glaze was applied onto the ceramic surface with a brush, dried at 37 °C for 2 h, and sintered following the manufacturer's instructions.
AL + GL	Zirconia specimens were air-abraded with 50 µm aluminum oxide particles (Al_2O_3) (Batch #3150313, Patterson Dental Supply, Inc., Saint Paul, MN, USA) for 30 s, under 2.8 bars and from a distance of approximately 10 mm. A low-fusing porcelain glaze was applied onto the ceramic surface with a brush, dried at 37 °C for 2 h, and sintered following the manufacturer's instructions.
CJ + GL	Zirconia specimens were silica-coated using particle abrasion with 30 µm silica-coated aluminum oxide particles (Cojet®-Sand, 3M ESPE AG, Seefeld, Germany; Lot # 501661) for 30 s, under 2.8 bars and from a distance of approximately 10 mm. A low-fusing porcelain glaze was applied onto the ceramic surface with a brush, dried at 37 °C for 2 h, and sintered following the manufacturer's instructions.
PS + GL	Zirconia specimens were chemically pretreated with Piranha Solution 3:1- Sulfuric acid (H_2SO_4) /30% H_2O_2. Zirconia samples were immersed for 30 min in the solution, rinsed with distilled water for 5 min, and immersed in distilled water for 20 min. A low-fusing porcelain glaze was applied to the ceramic surface with a brush, dried at 37 °C for 2 h, and sintered following the manufacturer's instructions.
CJ	Zirconia specimens were air-abraded with 30 µm silica-coated aluminum oxide particles (Cojet) (Cojet®-Sand, 3M ESPE AG, Seefeld, Germany; Lot # 501661) for 30 s, under 2.8 bars and from a distance of approximately 10 mm.

Zirconia surface treatments can be divided into "traditional" and "new" [14] with traditional treatments including the use of restorative materials containing MDP (phosphate ester monomers), airborne-particle abrasion, tribochemical silica coating (Rocatec or Cojet, 3M ESPE) and zirconia primers [14]. On the other hand, "new treatments" include the use of selective infiltration etching [15–17], glaze-on technique, hot etching solution and chemical treatments (piranha solution (PS)-a mixture of 3:1 sulfuric acid:30% hydrogen peroxide and up to 40% HF) [14].

Airborne-particle abrasion has been shown to improve the bonding ability between 3Y-TZP and resin cements and the glaze-on technique has been suggested as an alternative for airborne-particle abrasion treatment due to potential ceramic weakening effects associated with zirconia tetragonal to monoclinic (t → m) phase transformation. The glaze-on technique with multi-phase glaze containing a major lithium disilicate phase results in a zirconia surface amenable to etching and adhesive bonding and therefore can be an alternative treatment for zirconia [18]. A similar method described as "internal coating technique" uses a silica-based ceramic on the zirconia surface followed by silane application to improve bond strength [19]. In addition, some studies have reported using a chemical treatment with a PS [20,21], which is known as a cleaning reagent and a strong oxidizing solution that hydroxylates most surfaces and makes them hydrophilic thus improving their hydroxylation and bond strength to adhesive monomers [14,20,21].

The durability of a glaze-coating technique could be influenced by its adhesion to the substrate and depends on the properties of the substrate and the coating [22,23]. There is a lack of information regarding the bonding ability between 3Y-TZP and glaze, which can affect long-term stability of the technique. One way to evaluate glaze-zirconia bonding is by using the scratch test, which determines the cohesive and adhesive properties of thin films during coat detachment [22,23]. The test is conducted by applying a progressive or constant force on the substrate through a diamond stylus at constant speed so as to gently damage/scratch the coat [24].

In order to avoid problems related to chipping of the veneering ceramic and limited occlusal and palatal space, full-contour zirconia (FCZ) without the veneering ceramic has been developed [25–28]. In clinical practice, dental restoration treatments using FCZ can be improved by adequate treatment of the zirconia surface. Therefore, the aims of the present study were to evaluate the effect of different zirconia pretreatments on 1) the adhesion between 3Y-TZP and glaze, and 2) the SBS between glazed/3Y-TZP and resin cement. Two hypotheses were tested in this study: 1) zirconia pretreatment via airborne-particle abrasion, tribochemical silica coating and PS improves the glaze-coating adhesion to the FCZ and to the resin cement; 2) After aging (i.e., thermocycling and water storage) the zirconia surface pretreatment combined with a glaze application would provide better results of shear bond strength when compared to the single treatments (CJ and GL).

2. Materials and methods

2.1. Preparation of FCZ specimens

One hundred and fifty FCZ ceramic bars (3Y-TZP, Lot #P02286, Diazir®, Ivoclar-Vivadent, Amherst, NY, USA) were cut into blocks (10 × 10 × 3 mm^3) using a diamond-wafering blade mounted on a precision saw machine (ISOMET 1000, Buehler Ltd., Lake Bluff, IL, USA). The ceramic blocks were sintered at 1500 °C for 2 h in a high-temperature furnace (Lindberg/Blue M, Kendro Laboratory Products, Inc., Asheville, NC, USA) according to the manufacturer's instructions. The ceramic blocks were then finished with SiC paper (LECO Corporation, St. Joseph, MI, USA), cleaned in an ultrasonic bath containing isopropyl alcohol for 5 min, rinsed with water, air-dried, and randomly distributed into five groups (Table 1).

2.2. Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM, JSM-6390, JEOL, Tokyo, Japan) was used to analyze the ceramic surface morphology after each conditioning method (Table 1). The specimens were mounted onto a metallic stub and submitted to sputter coating with gold.

2.3. Scratch test

The bonding ability between FCZ and glaze (N = 3; except for the CJ group – without glaze application) was assessed by the scratch test (ASTM C1624-05) [24] using a 200 µm spherical Rockwell C diamond stylus with a progressive vertical load that increased linearly from 0 to 30 N at a constant speed. After the test, the critical damage was viewed through the SEM and the images were qualitatively analyzed.

2.4. Glaze thickness

Glaze-coated FCZ ceramic bars were vertically mounted in epoxy resin after applying the glaze. The zirconia/glaze interface was evaluated through SEM and the glaze thickness was measured using Image J software (National Institutes of Health, Bethesda, MD, USA).

2.5. Shear bond strength test and failure analysis

The glaze-modified FCZ surface was etched with 5% HF (Lot #R53559, Ivoclar Vivadent) for 90 s, rinsed for 60 s and air-dried. A silane agent (Lot #R50513, Monobond Plus, Ivoclar-Vivadent) was then applied with a brush and was left undisturbed for 1 min, and the solvent was air-dried. Resin cement buttons (~ 2.2-mm in height and 2.38-mm in diameter) were fabricated using a specially fabricated jig (Ultradent Products, Inc., South Jordan, UT, USA) and a cylindrical Teflon mold; they were then placed over each zirconia specimen. Resin cement (Lot #R25959, Multilink® Implant, Ivoclar-Vivadent) was applied to the

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