



Effect of the bonding strategy on the tensile retention of full-contour zirconia crowns



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ABSTRACT

This study evaluated the effect of distinct bonding strategies on the retention of full-contour zirconia ceramic (Y-TZP, FCZ) crowns, and it characterized some physicochemical and mechanical properties of FCZ ceramic and its corresponding glazing system. To evaluate retention strength, dies were made with a dentin-analogue material to simulate a prepared tooth. FCZ crowns were manufactured using CAD-CAM technology and allocated into groups according to the bonding strategy: no ceramic treatment (PF – Panavia F cementation), glaze (GL), tribochemical silica coating (CJ), CJ + GL, and piranha solution followed by glaze (PS + GL). The specimens were subjected to thermocycling and storage in distilled water for 100 days before the retention tests. FCZ presented a porosity volume fraction of 0.2%, an apparent density of 6.06 g/cm³, Vickers hardness of 12.4 ± 0.07 GPa, and fracture toughness of 5.54 ± 0.24 MPa m^{1/2}. SEM revealed a homogeneous microstructure composed of submicron-sized grains. XRD identified mainly zirconia's tetragonal phase. Glaze powder morphology was observed to be irregular, with a nanometric particle size, and a diffraction pattern characteristic of an amorphous material with several peaks of leucite. The PF and GL groups had higher retention values. The majority of the groups presented pre-test bonding failures, and two catastrophic failures of the FCZ-crown (GL and PF groups) were noted. The use of an MDP-containing resin cement or glaze application might improve retention of the FCZ crowns.

1. Introduction

Zirconia has gained increased attention as a dental restorative material due to its proven biocompatibility, mechanical strength, and its use in Computer-Aided Design/Computer-Aided Machining (CAD/CAM) technology [1–3]. Superior mechanical properties, particularly those as a consequence of the transformation-toughening mechanism, have also contributed to zirconia's popularity. Induced by mechanical stress, the phase transformation from tetragonal to monoclinic (t → m) produces an increased grain volume of 3–4% [1–3]. The restricted volume expansion results in the development of compressive stresses, which oppose crack propagation within the ceramic and along its surface [1–3]. The transformation-toughening mechanism can be controlled by adding stabilizing oxides or dopants, such as yttrium oxide, thus yielding the dental zirconia most commonly used—3-mol% yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP) [1–3].

Clinically, 3Y-TZP ceramic is employed as the core material for fixed partial dentures (FPDs) or single crowns, with a veneering ceramic layered or pressed onto the occlusal surface. However, clinical studies continue to report failures, such as debonding, chipping, or fracture of the veneering ceramic [4–7]. These failures can be attributed to multiple factors, such as the differences in material properties (e.g., the coefficient of thermal expansion [CTE] between the zirconia core and the veneering ceramic), the cooling rate and geometry of the bilayer ceramic structure, and difficulty in promoting a strong, reliable bond between the zirconia surface and tooth structure. To prevent such failures, full-contour 3Y-TZP zirconia crowns (FCZ) without veneering porcelain have been developed [8]. These are specifically indicated for the posterior areas of the mouth, where esthetics are not the primary focus and for cases with limited occlusal and palatal space [9,10].

Despite significant improvement in bonding between zirconia and resin cement, it is difficult to precisely predict clinical outcomes,

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because more frequently, the bond is compromised if its internal (in-taglio) surface is not properly treated. Zirconia has a high crystalline content that makes hydrofluoric acid (HF) unable to effectively attack its surface [11,12]. Several studies have investigated alternative bonding strategies to improve the bond strength of resin cements to zirconia [11–19]. Particularly, studies have focused on surface treatments using airborne particle abrasion with aluminum oxide (Al_2O_3) or aluminum oxide particles modified by silica. However, the effects of airborne particle abrasion can affect the mechanical properties of zirconia, resulting in phase transformation ($t \rightarrow m$) and long-term deleterious effects [20,21]. To avoid side effects from particle abrasion, studies have investigated the application of a thin layer of a vitreous material (i.e., glaze) on the zirconia internal surface [13–16]. Glaze is a vitreous material, sensitive to HF etching, which can make the zirconia surface chemically reactive and mechanically retentive [15]. An alternative approach would be a chemical treatment using the piranha solution (PS), which is a mixture of sulfuric acid and hydrogen peroxide (3:1), known as a strong oxidizing solution that not only makes most surfaces hydrophilic by hydroxylation, but also improves the hydroxylation and bond strength of adhesive monomers [17].

Therefore, the aims of this investigation were to: 1) characterize some physicochemical and mechanical properties of FCZ ceramic; 2) characterize its corresponding glaze, which needs to be considered when working with monolithic zirconia restorations; and 3) evaluate the effects of distinct bonding strategies on the retention of FCZ crowns.

2. Materials and methods

2.1. FCZ preparation

FCZ ceramic bars (3Y-TZP, Lot#P02286, Diazir[®], Ivoclar-Vivadent, Amherst, NY, USA) were cut into blocks ($10 \times 10 \times 3 \text{ mm}^3$) using a diamond blade mounted on a precision saw machine (ISOMET 1000, Buehler Ltd., Lake Bluff, IL, USA) [2]. The ceramic blocks were sintered at 1500°C for 2 h in a high-temperature furnace (Lindberg Blue M, Asheville, NC, USA). Samples were polished with 9, 6, 3, and $1 \mu\text{m}$ ceramographic cloth and diamond pastes (Préparations Diamantées Mecaprex, Grenoble, France). All samples were immersed in isopropyl alcohol and cleaned for 5 min in an ultrasonic bath (Vitasonic, Vita Zahnfabrik, Bad Säckingen, Germany) [2].

2.2. FCZ microstructural characterization

Density and porosity of the sintered ceramics were determined using an immersion method based on the Archimedes principle [2]. The polished sintered ceramic blocks received a thermal treatment in order to reverse the phase transformation ($m \rightarrow t$) produced during ceramographic procedures and reveal the post-sintering microstructure using a scanning electron microscope (SEM, XL30, Phillips, Eindhoven, The Netherlands) [2]. The zirconia grain diameter was measured using the ImageJ software (National Institutes of Health, Bethesda, MD, USA). X-ray diffraction (XRD) (DMAX 2000, model Multiflex, Rigaku Corporation, Tokyo, Japan) analysis was performed to identify the crystalline phase (tetragonal/cubic/monoclinic) by using $\text{Cu}/\text{K}\alpha$ radiation with a scan range of $5\text{--}90^\circ$, step of 0.02° , and a counting time of 8 s/step.

2.3. FCZ mechanical test

Hardness was determined by the Vickers hardness test (VMT-7, Buehler). Briefly, 3 samples were embedded in bakelite and polished with a ceramographic cloth and diamond suspensions (Préparations Diamantées Mecaprex). A 10 kgf load was applied for 15 s by the indenter, and the impressions were taken from 6 areas per specimen. The values were calculated using Eq. (1), where “P” is the applied load (N), “d” is the diagonal length (m), and “ α ” is the angle between the opposite faces of the indenter (136°) [2,20].

$$Hv = \frac{\alpha P}{d^2} \quad (1)$$

Fracture toughness (K_{IC}) was calculated by observing crack type, dimensions of the indentations, and cracks on the ceramics during indentation using an optical microscope (PMG3, Olympus, Tokyo, Japan). The crack type was identified by investigating the indented surface polished with a diamond paste, following previously established criteria and equations [21,22].

2.4. FCZ glaze preparation

Diazir[®] full-contour glaze paste (Lot#P4VM, Ivoclar-Vivadent) was dried in an Electric Muffler Oven (model 1857, Fornitec Indústria e Comércio Ltda, São Paulo, Brazil) at 200°C to obtain the glaze powder.

2.5. Glaze morphological and chemical characterizations

Powder particle size distribution was evaluated using Dynamic Light Scattering (DLS, Brookhaven Instruments Corporation, Holtsville, NY, USA) and the data were analyzed using dedicated software (ZetaPlus Particle Sizing Software Ver.4.02, Brookhaven Instruments Corporation). The glaze mineral phase identification was performed by X-ray diffraction. The diffraction pattern was interpreted by comparing it with the ICDD (International Centre for Diffraction Data, Newtown, PA, USA) card files. Morphology of the obtained glaze powder was carried out via SEM (Tabletop, Model TM 3000, Hitachi High-Technologies, Krefeld, Germany).

2.6. Epoxy resin dies preparation

Epoxy resin (NEMA Grade G10 glass-reinforced epoxy plastic laminates, Accurate Plastics, Inc., Yonkers, New York, USA) dies were prepared (Centro para Inovação e Competitividade do Cone Leste Paulista, Parque Tecnológico de São José dos Campos, SP, Brazil) to simulate a single-tooth FCZ crown, i.e., a base of 8 mm (diameter) \times 6 mm (height), preparation of 6 mm in height, rounded shoulder finish line, and 20° of total occlusal convergence angle [23].

2.7. FCZ crown preparation

FCZ disks were cut into blocks using a diamond blade mounted on a precision saw machine (ISOMET 1000, Buehler). The blocks presented the same dimensions as the commercial blocks for the CAD-CAM system IPS e.max[®] ZirCAD, (MO C15L, Ivoclar-Vivadent). All the blocks were finished with SiC papers under water-cooling to standardize the surfaces. A milling mandrel was glued to each FCZ block with cyanoacrylate gel, resulting in a CAD-CAM block that could be attached to the milling unit. FCZ crowns were milled with retentions (Fig. 1) on the occlusal surface [18,19,24] using the InLab MC XL Milling Unit (Sirona Dental Systems, Bensheim, Germany) and designated software (Inlab 3.60). After milling, all crowns were washed with distilled water, dried in an oven (Pyro-oven, H.D. Justi Company, Philadelphia, PA, USA), and sintered (Programat S1, Ivoclar-Vivadent) following the manufacturer's instructions.

2.8. Bonding strategy

Prior to the bonding protocol, the FCZ crowns were cleaned in alcohol in an ultrasonic bath for 5 min, rinsed in DI water, and dried with oil-free canned air. The samples were allocated into 5 groups ($N = 12/\text{group}$) based on the bonding strategy (Table 1): CJ, GL, PF, CJ + GL, and PS + GL. For surface pretreatment, G10 dies were etched with HF (Lot# R53559, Ivoclar-Vivadent) for 60 s, and a thin layer of a silane coupling agent (Lot# R50513, Monobond Plus, Ivoclar-Vivadent) was applied [23].

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