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The potential of novel primers and universal adhesives to bond to zirconia





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ABSTRACT

Objectives: To investigate the adhesive potential of novel zirconia primers and universal adhesives to surface-treated zirconia substrates.

Methods: Zirconia bars were manufactured ($3.0 \text{ mm} \times 3.0 \text{ mm} \times 9.0 \text{ mm}$) and treated as follows: no treatment (C); air abrasion with $35 \,\mu\text{m}$ alumina particles (S); air abrasion with $30 \,\mu\text{m}$ silica particles using one of two systems (Rocatec or SilJet) and; glazing (G). Groups C and S were subsequentially treated with one of the following primers or adhesives: ZP (Z-Prime Plus), AZ (AZ Primer); MP (Monobond Plus); SU (ScotchBond Universal) and; EA (an Experimental Adhesive). Groups Rocatec and SilJet were silanized prior to cementation. Samples form group G were further etched and silanized. Bars were cemented (Multilink) onto bars of a silicate-based ceramic ($3.0 \text{ mm} \times 3.0 \text{ mm} \times 9.0 \text{ mm}$) at 90° angle, thermocycled (2.500 cycles, 5–55 °C, 30 s dwell time), and tested in tensile strength test. Failure analysis was performed on fractured specimens to measure the bonding area and crack origin.

Results: Specimens from group C did not survive thermocycling, while CMP, CSU and CEA groups survived thermocycling but rendered low values of bond strength. All primers presented a better bond performance after air abrasion with Al₂O₃ particles. SilJet was similar to Rocatec, both presenting the best bond strength results, along with SMP, SSU and CEA. G promoted intermediate bond strength values. Failure mode was predominately adhesive on zirconia surface combined to cohesive of the luting agent.

Conclusions: Universal adhesives (MP, SU, EA) may be a considerable alternative for bonding to zirconia, but air abrasion is still previously required. Air abrasion with silica particles followed by silane application also presented high bond strength values.

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1. Introduction

The microstructural characteristics of zirconia-based ceramics make resin bonding challenging¹. Improved adhesion increases retention and improves the marginal sealing of fixed partial dentures (FPDs). The adhesive cementation procedure for silica-based ceramic surfaces is well defined, and involves etching with hydrofluoric acid (HF) and silanization for chemical bonding. For zirconia substrates, the appropriate clinical protocol is still under controversial discussion.

Several procedures for adhesive cementation on zirconia have been investigated. Silica-coating through air abrasion associated to application of silane coupling agents has been largely investigated,^{2–8} and resulted in higher bond strength to composite resins compared to other surface treatments, whether in air, water or after thermocycling.^{2–5} Air abrasion with aluminium oxide roughens the ceramic surface and increases the surface area for bonding, providing mechanical retention.⁶ However, surface damage could lead to immediate clinical consequences or premature failure.^{9,10} For small sizedparticles (35µm) Scherrer et al.⁷ showed improvements in mechanical strength of air abraded yttria stabilized polycrystalline tetragonal zirconia (Y-TZP) specimens after mechanical ageing in humid environment.

Primers play an important role in adhesive procedures, especially for zirconia-based ceramics, since the untreated surface of zirconia is not readily reactive to conventional silane agents. Silane-based (y-methacryloxypropyltrimethoxy-silane) primers are capable of bonding both to inorganic and organic surfaces, and their use has been proven to increase the bond strength between resin cements and zirconia ceramics after surface treatment (silica deposition).^{11,12}

Another important component that may be present in primers is the 10-methacryloxydecyl dihydrogen phosphate monomer (MDP). This molecule was firstly added to resin cements (e.g. Panavia F, Kuraray, Japan) for bonding to crystalline ceramics having low glass content, resulting in improved adhesion.^{6,13,14} Organo-phosphate monomers contain polymerizable functional groups (e.g. methacrylates), which can polymerize with the matrix of methacrylate-based dental resin cements, composites and adhesives.¹⁵ The formation of a covalent bond between the oxygen, phosphorus and zirconia (P–O–Zr) was confirmed in a recent study.¹⁶

The existence of such a wide variety of primers makes it difficult for clinicians to choose the correct system for specific clinical situations. Following a simplified strategy, new universal adhesives were developed to be used with multiple restorative materials. They present both silane and phosphoric monomers in their composition. Such chemical agents may promote adhesion to surfaces based on silica and/or metallic oxides. This approach is particularly interesting for repairs in veneered zirconia crowns, as one single agent can be used for coupling to both zirconia and porcelain exposed surfaces.

Another approach that resulted in strong bond strength to zirconia was the selective infiltration etching, in which a molten glass is infiltrated between the boundaries of the surface grains, creating a 3-dimensional network,¹⁷ or heating

of metal/zirconia primers.¹⁸ Coating the inner surface of zirconia with a silica based ceramic was also recommended for adhesive procedures.^{2,3,19,20} The silica-based ceramic is fused to zirconia during firing, and a protocol for cementation of glass ceramics is then applied: hydrofluoric acid etching followed by silane application.²⁰

The purpose of this study was to evaluate the effect of different primers, adhesives, and different surface treatments on the bond strength to zirconia after thermal ageing. The null hypothesis is that neither surface treatments or primers/ adhesives influence the bonding to zirconia.

2. Materials and methods

2.1. Zirconia specimens production

Yttrium-stabilized zirconium dioxide (Vita In-Ceram YZ for inLab, VITA Zahnfabrik, Bad Säckingen, Germany) was used as the test substrate for the bonding experiments. Bar-shaped specimens (225 units) were cut from pre-sintered blanks, cleaned and sintered to the final dimensions of $3.0 \text{ mm} \times 3.0 \text{ mm} \times 9.3 \text{ mm}$ (thickness, width and length, respectively) in a sintering oven (EVA 1700; Linn High Therm, Eschenfelden, Germany) at 1530 °C for 2 h (heating rate: 600 °C/h).

2.2. Zirconia priming and surface treatments

Ninety bars were used as control (as-sintered state) and the same number of bars had their bonding surface modified by air abrasion with $35 \,\mu$ m Al₂O₃ particles (Hasenfratz, Assling, Germany) (2.8 bar pressure, 7 s, 10 mm distance). For these both surface treatments, five primers were tested and compared to no primer application (n = 15): Z-Prime Plus (Bisco Inc.), AZ Primer (Shofu Inc.), Monobond Plus (Ivoclar Vivadent), Scotchbond Universal (3M ESPE) and an Experimental Adhesive (Voco). The composition of the primers and adhesives is described in Table 1.

In order to evaluate the effect of silica coating, additional groups were tested (n = 15) using silica particle air abrasion (Rocatec and SilJet groups, Table 1) and porcelain glaze layering. The first silica coating groups received air abrasion with 30 μ m silica modified Al₂O₃ particles from 2 different commercial brands: Rocatec Soft (3M ESPE) followed by silane application (Porcelain Primer, Shofu Inc.); and Siljet (Danville, San Ramon, USA) followed by silane application (S-Bond, Danville). The porcelain glaze layering (Vita Akzent, Vita Zahnfabrik, Bad Säckingen, Germany) was applied in additional samples (n = 15), with subsequent firing (900 °C, 1 min), etching (5% hydrofluoric acid – HF (Vita Ceramics etch, Vita Zahnfabrik)) and silanization for 60 s (Porcelain Primer, Shofu Inc.). Tested groups are described in Table 2.

After air abrasion and glaze application, and before the application of primers/adhesives and/or cementation, all bars were subjected to ultrasonic cleaning (5 min, isopropanol 70%) and drying.

The surface roughness (R_a -value) of the zirconia bars was measured (n = 5) in the following conditions: as-sintered, air abraded with 35- μ m Al₂O₃ particles, air abraded with 30- μ m SiO₂ particles (in both Rocatec[®] and SilJet[®] systems) and

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