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Short communication

Influence of different surface treatments on surface zirconia frameworks

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ABSTRACT

Objectives: To evaluate the effect of different chemo-mechanical surface treatments of zirconia ceramic in the attempt to improve its bonding potential.
Methods: Sintered zirconium oxide ceramic discs (Lava^{TM} Ø10 mm × 1 mm height) were treated with ($n = 4$): (1) airborne particle abrasion with 125 μm Al_2O_3 particles; (2) 9.5% HF acid etching; (3) selective infiltration etching (SIE); (4) experimental hot etching solution applied for 10, 30 and 60 min; (5) no treatment.

Ceramic discs surfaces were analyzed by atomic force microscopy (AFM) recording average surface roughness measurements of the substrate. Data were statistically analyzed by Kruskall–Wallis analysis of variance and Mann–Whitney tests ($\alpha = 0.05$). The same discs were used for bi-dimensional zirconia ceramic surface characterization with scanning electron microscope (SEM).
Results: Ceramic surface treatments significantly influenced surface topography and roughness ($p < 0.001$). Bi-dimensional changes in ceramic surface morphology were assessed on a nanometric scale. The experimental hot etching solution improved surface roughness, independently from the application time.
Conclusion: Zirconia conditioning with the experimental hot etching solution may enhance ceramic roughness and improve the surface area available for adhesion allowing the formation of micromechanical retention. The influence of this surface treatment with regard to bond strength of zirconia needs to be addressed.

1. Introduction

Interest for using high-strength zirconium oxide ceramics for the fabrication of computer-manufactured full coverage crowns and bridge frameworks is growing in recent years, due to their improved mechanical properties in comparison to more conventional alumina or lithium disilicate-based ceramics.[1,12,13]

Although this aspect has been recognized as a crucial factor for ensuring reliable clinical performance, the definition of a suitable luting strategy that guarantees clinical retention and optimal marginal fit, is still a matter of concern.

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Different cement types have been proposed for luting zirconia (i.e. traditional cements, self-adhesive cements, GICs) however, 10-MDP-based resin luting agents seem to be the most appropriate, thanks to the chemical interaction between the hydroxyl groups of the passive zirconium oxide surface coating and the phosphate ester monomers of the MDP.[2–4]

Ceramic/resin cement bonds may be more effective and durable if associated with micromechanical retentions: the achievement of roughened ceramic surfaces may allow the resin cement to penetrate and flow into these microretentions, thus creating a stronger micromechanical interlock.[5–8]

Although several surface treatments have been recently investigated both under *in vitro* and *in vivo* conditions, concerns still exist regarding the selection of the most appropriate zirconia surface pre-treatment. Neither hydrofluoric acid etching nor silanization result in a satisfactory resin bond to zirconia, because of the high crystalline content and the limited vitreous phase (below 1%) of this high-strength core ceramic.[11,14–20,25]

Airborne particle abrasion was also employed in the attempt to enhance the surface area available for bonding[21–23]: although an improvement in the average surface roughness has been recorded on a micrometer scale, the treatment appeared inadequate to establish reliable ceramic/cement bonds.[20]

Alternative technologies are advocated in the attempt to change these high-strength ceramic cores into more retentive substrates. Selective infiltration etching (SIE) has been recently proposed achieving promising results in terms of bond strengths values at the zirconia–resin cement interfaces.[9] This treatment, that is based on the principle of the heat-induced infiltration process, may determine zirconia crystal rearrangements as well as intergrain nano-porosities formation where low-viscosity resinous materials may flow and interlock after polymerization.[4,11]

On the other hand, considering the metallic nature of pure zirconium, it can be assumed that treatments originally performed for conditioning metals or alloys may be somewhat beneficial for etching zirconium dioxide crowns or bridge frameworks. A hot chemical solution has been proposed to etch the wings of Maryland bridges, roughening the surface and promoting retention.[10] The possibility of performing this procedure in the dental office as well as in the laboratory, makes this technique potentially useful for treating zirconia.

The aim of this study was to evaluate the surface topography and changes in average surface roughness (R_a) provided by different zirconia ceramic surface treatments. The null hypothesis to be tested is that the conditioning treatments do not modify the ceramic surface roughness and morphology.

2. Materials and methods

Twenty-eight cylinder-shaped (Ø10 mm × 1 mm height) zirconium oxide ceramic discs (Lava™; 3M Espe, Seefeld, Germany) have been selected for the study. Specimens were polished with SiC abrasive papers (grit# 600, 1000, 1200 and 2000). Final polishing was carried out with nylon cloths using 1- and 0.50-μm grit size diamond pastes.

Specimens were sonicated in deionized water for 30 min and were divided into five groups ($n = 4$) according to the surface treatment performed: Group (1) airborne particle abrasion (S) with 125 μm Al_2O_3 particles applied for 10 s at 60–100 psi; Group (2) 9.5% hydrofluoric acid etching (HF) for 90 s; Group (3) selective infiltration etching procedure (SIE), modifying the treatment previously proposed by Aboushelib et al.[9] Specimens were coated with a thin layer of an infiltrating agent containing low temperature melting glass and additives. They were heated up to 750 °C for 1 min using a computer-programmed electrical induction furnace (Austromat 3001; Dekema Dental-Keramikofen, Freilassing, Germany), cooled reaching 650 °C for 1 min, and heated again up to 750 °C for 20 min (increasing T intervals 60 °C/min), then cooled at room temperature. Remnants of the infiltrating agent have been dissolved immersing ceramic discs in a ultrasonic bath with 5% hydrofluoric acid solution (30 min); Group (4) experimental hot etching solution (ST) that was heated up to 100 °C and applied for: (a)10 min, (b) 30 min and (c) 60 min according to a protocol previously proposed by Ferrari for conditioning the wings of Maryland bridges[10]; Group (5) no treatment.

Experimental groups and the chemical composition of the different conditioning agents are summarized in Table 1.

After being treated, specimens were rinsed with tap water for 1 min, ultrasonically cleaned in a deionized water bath for 30 min and gently air-dried.

2.1. Atomic force microscope (AFM) evaluation

Zirconia discs were evaluated under an atomic force microscopy (AFM, Multimode Nanoscope IIIa, Digital Instruments,

Table 1 – Experimental groups and the chemical composition of the different conditioning agents tested in the study.

Group	Treatment	Chemical composition
1	Airborne particle abrasion	125 μm Al_2O_3 particles
2	Hydrofluoric acid etching	9.5% HF
3	Selective infiltration etching	SiO_2 (65 wt.%); Na_2O (15 wt.%); Al_2O_3 (8 wt.%); Li_2O (3 wt.%); B_2O_3 (4 wt.%); CaF_2 (5 wt.%)
4	Exp. hot etching solution (a) 10 min (b) 30 min (c) 60 min	Methanol (800 ml); 37% HCl (200 ml); and ferric chloride (2 g)
5	No treatment	