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Influence of surface treatments on topography and bond strength of densely-sintered zirconium-oxide ceramic

Gabrielle Alencar Ferreira Silva [a], Elidineide Cruz da Luz [a], Frederico dos Reis Goyatá [b], Lais Regiane da Silva Concilio [c], Ana Christina Claro Neves [c], Rafael Pino Vitti [c,*], Leonardo Gonçalves Cunha [a]

[a] Department of Restorative Dentistry, University of Bahia, Salvador, Bahia, Brazil
[b] Department of Prosthodontics, Federal University of Minas Gerais, Belo Horizonte, Minas Gerais, Brazil
[c] Department of Prosthodontics, University of Taubaté, Taubaté, São Paulo, Brazil

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ABSTRACT

The aim of this study was to evaluate the effect of surface treatments on the roughness and bond strength of dental materials containing MDP to zirconium oxide ceramic. Forty square-shaped zirconium-oxide ceramic blocks (Lava Zirconia, 3M-ESPE) were treated as follows: (CT) polished only; (SB) sandblasting (110 μm aluminum oxide particles) or (SC) silica coating (110 μm particles). Roughness of treated surface was measured using a profilometer (Ra) and by atomic force microscope (AFM). Two resin luting agents were used after silane application: self-adhesive (Rely X U200, 3M-ESPE) and dual cure (Rely X Ultimate, 3M-ESPE). The samples were submitted to microshear bond strength test. The failure analysis was performed. Data were submitted to ANOVA and Tukey test ($\alpha=0.05$). Bond strength results ranged from 20.44 (CT+Ultimate) to 34.37 MPa (SC+U200) after 24 h and from 12.03 (CT+Ultimate) to 27.44 MPa (SC+U200) after 12 months of storage with SC statistically superior to the other treatments. Mean values of roughness varied from 0.07 (CT) to 0.85 μm (SC). The both resin luting agents showed similar results to all surface treatment groups. Silica coating provided the best treatment of the ceramic surface.

1. Introduction

The introduction of computer-aided design/computer-aided manufacture (CAD/CAM) systems in dentistry resulted in a significant increase in the use of all-ceramic restorations. This technology enabled the dental laboratory to control the fabrication process, allowing the production of high quality rehabilitations with a known production schedule, and also reducing the technician's time [1].

Zirconium oxide represents one of the primary reinforced ceramic substrates used in the CAD/CAM process. The popularity of this material is related to its superior properties, such as high flexural strength, biocompatibility, and esthetics [1,2]. In addition, short-term clinical trials and lifetime predictions reveal favorable success rates for zirconia restorations [3], with an acceptable marginal fit of zirconia rehabilitations produced by milling systems [4,5]. However, many questions remain about the optimal cementation process for this ceramic substrate.

The achievement of reliable adhesion to ceramics requires surface pre-treatments. Bonding to glass-ceramics normally is obtained by etching with hydrofluoric acid. This step promotes dissolution of the glassy phase, creating a rough surface, which favors adherence that relies on mechanical interlocking [6]. However, the polycrystalline zirconia material does not contain a glassy phase, and cannot be modified by hydrofluoric acid etching [7]. As a consequence, alternative conditioning methods have been proposed. Some previous studies reported that airborne abrasion may increase the surface area, resulting in acceptable micrometer scale roughness facilitating resin/ceramic micromechanical interlock formation [8,9]. However, no chemical alteration is achieved in the ceramic surface using this treatment.

In addition, the use of silane coupling agents to enhance the bond of resin composites to silica-based ceramics is well accepted in the dental literature [10]. However, due to the low content of silica (below 1%) in the chemical structure of zirconia, this kind of treatment does not affect adhesion to zirconia [11]. In this sense, silica coating the ceramic surface may be a promising method to promote the bonding of acid-resistant ceramics to resin. It has been suggested that this surface treatment can increase the silica

* Correspondence to: University of Taubaté, Rua dos Operários, 09, CEP: 12020-270, Taubaté, SP, Brazil.
E-mail address: rafapvitti@gmail.com (R.P. Vitti).

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Table 1
Composition and application mode of each material used.

Material	Composition	Application
Rely X U200 (3M-ESPE)	Base: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, initiators, stabilizers, rheological additives. Catalyst: methacrylate monomers, alkaline fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives.	1. Mix cement (20 s) 2. Apply mixture 3. Light activation (20 s)
Rely X Ultimate (3M-ESPE)	Base: methacrylate monomers, radiopaque silanated fillers, initiator, stabilizers, rheological additives. Catalyst: methacrylate monomers, radiopaque alkaline fillers, initiators, stabilizers, pigments, rheological additives, fluorescence dye, dual-cure activator for single bond universal adhesive.	1. Mix cement (20 s) 2. Apply mixture 3. Light activation (20 s)

content on ceramics and metals enhancing the bond to resins via a silane coupling agent [3,12–14].

Different luting agents have also been proposed, such as self-adhesive cements or cements containing adhesive phosphate methacrylate monomers in their compositions (10-methacryloyloxydecyl dihydrogen phosphate – MDP). A previous study has shown that these monomers can react with the ceramic surface with no pretreatment, which provides bond strength equal to or higher than the bond strength produced by conventional resin cements [15].

Therefore, the aim of the present study was to evaluate the effect of surface treatments on the roughness and bond strength of cements to zirconium-reinforced ceramic substrates. The first tested hypothesis was that the surface treatment can influence the topography of the ceramic surface and the bond strength of luting agents to the ceramic substrate. The second hypothesis was that difference on bond strength would observed between adhesive and self-adhesive luting agents.

2. Material and methods

2.1. Specimen preparation

Densely sintered yttria-stabilized (Lava, 3M-ESPE, St. Paul, MN, USA) samples were used in the present study. Forty eight blocks were confectioned following manufacturer's instructions, with dimensions of $5 \times 5 \times 2$ mm.

Ceramic blocks were individually embedded in acrylic resin (Vipi Flash, Vipi, São Paulo, Brazil) using a standard cylindrical silicon matrix (Arotec Ind e Com, São Paulo, Brazil). The surface of the ceramic blocks was wet-ground with aluminum oxide sandpaper of 600, 1200, and 1500 grit in a horizontal Polishing Machine (Aropol, ArotecInd e Com, São Paulo, Brazil) and all blocks were cleaned in ultrasonic equipment for 5 min in water bath.

Subsequently, the ceramic blocks were randomly divided into three groups ($n=4$), according to the following surface treatments:

Group CT: no surface treatment (control).
Group SB: sandblasting using 110 μm aluminum oxide particles for 10 s, applied perpendicularly to the surface at a 10 mm distance and pressure of 30 psi, followed by ultrasonic cleaning for 5 min.
Group SC: silica coating using particles of 110 μm (Rocatec, 3M-ESPE, St Paul, USA). The Rocatec-Sand was blasted for 10 s applied perpendicularly to the surface at a 10-mm distance and pressure of 30 psi.

After that, one coat of a silane agent (Rely X Ceramic Primer, 3M-ESPE, St Paul, USA) was applied, and a period of 60 s waited to dry. Acrylic plastic tubes (1 mm internal diameter and 3 mm in height) were used to produce the luting agent samples. Four tubes were positioned on each treated ceramic surface and then filled with one of two luting agents tested: an adhesive resin cement containing MDP monomer (Rely X Ultimate, 3M-ESPE, St Paul, USA), or a self-adhesive resin cement (Rely X U200, 3M-ESPE, St Paul, USA). Each cement was used following the manufacturer's instructions (Table 1).

Table 2
Mean bond strength values (MPa) and failure mode to each surface treatment and luting agent after 24 h and 12 months of storage.

	24 h	12 months	Failure Mode					
			Adhesive		Mixed		Cohesive	
			24 h	12 m	24 h	12 m	24 h	12 m
CT+U200	22.52 (3.3) bc, A	13.72 (2.84) c, B	11	13	5	3	0	0
CT+Ultimate	20.44 (4.9) c, A	12.03 (1.98) c, B	11	13	5	3	0	0
SB+U200	26.23 (3.7) b, A	19.65 (2.64) b, B	9	11	7	5	0	0
SB+Ultimate	26.91 (3.8) b, A	19.25 (2.65) b, B	8	10	8	6	0	0
SC+U200	34.37 (3.0) a, A	27.44 (2.85) a, B	8	9	7	7	1	0
SC+Ultimate	33.54 (4.8) a, A	26.58 (2.64) a, B	7	10	9	6	0	0

Data with different lowercase letters in column and capital letters in row are statistically different ($p < 0.05$).

A total of 16 samples were confectioned for each tested group. Cement samples were cured for 40 s using a LED curing unit (EliparFreeLight 2, 3M-ESPE, St Paul, USA) at irradiance of 1200 mW/cm^2. The irradiance was frequently checked using a radiometer (RD7, Ecel, Ribeirão Preto, Brazil).

Bonded samples were stored in distilled water (37 °C) for 24 h and 12 months before a microshear bond strength test was performed at a crosshead speed of 1 mm/min in a universal testing machine (Versat 2000, Panambra, São Paulo, Brazil). Microshear test was performed using a wire loop, held close to the bonded area to avoid torqueing. Results were obtained in Kgf and converted to MPa by dividing the failure load by the cross-sectional area of each sample.

Results were tested for normality test, and then subjected to ANOVA and post-hoc Tukey test with a significance level of 5%.

2.2. Failure mode evaluation

The analysis of the entire bonding area of the debonded surfaces of all samples was performed by two blinded examiners using an Optical Stereomicroscope (VEB Leipzig, Germany) under magnification of 100×. Images were analyzed using specific software (VEB Jena, Germany) and classified as to the characteristic of failure as follows: cohesive failure in ceramic, cohesive failure in the luting agent, adhesive failure, or mixed (adhesive and cohesive in cement). When there was disagreement during an evaluation, the two examiners made the decision by consensus.