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Effect of surface pre-treatments on the zirconia ceramic–resin cement microtensile bond strength

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ABSTRACT

Objective. To evaluate the influence of different surface treatments on the microtensile bond strength of resin cement to zirconia ceramic.

Materials and methods. Twelve cylinder-shaped (⌀ 12 × 5.25 mm high) blocks of a commercial zirconium-oxide ceramic (Cercon® Zirconia, DENTSPLY) were randomly divided into 4 groups ($n = 3$), based on the surface treatment to be performed: (1) airborne particle abrasion with 125 μm Al_2O_3 particles (S); (2) selective infiltration etching (SIE); (3) experimental hot etching solution applied for 30 min (ST) and (4) no treatment (C). Paradigm MZ100 blocks (3M ESPE) were cut into twelve cylinders of 4 mm in thickness. Composite cylinders were bonded to conditioned ceramics using a resin cement (Calibra®, DENTSPLY), in combination with the proprietary adhesive system. After 24 h bonded specimens were cut into microtensile sticks and loaded in tension until failure. Bond strength data were analyzed with Kruskall–Wallis and Dunn's Multiple Range test for multiple comparisons ($p < 0.05$). Failure mode distribution was recorded and the interfacial morphology of debonded specimens was analyzed using a scanning electron microscope (SEM).

Results. Bond strength values achieved after SIE and ST treatment were significantly higher than after S treatment and without any treatment ($p < 0.05$). Premature failures were mostly recorded in the S group.

Significance. Conditioning the high-strength ceramic surface with SIE and ST treatments yielded higher bond strengths of the resin cement than when zirconia ceramic was treated with airborne particle abrasion or left untreated.

1. Introduction

The use of partially stabilized zirconium dioxide ceramics to fabricate metal-free esthetic restorations has increased in recent years, thanks to their excellent physical properties and optimal biocompatibility [1–6]. Several studies reported that zirconia-based ceramics may achieve better mechanical resistance than feldspathic, leucite, and lithium disilicate ceramics, especially when restoring posterior teeth [7–13]. Clinically, chipping of veneering porcelains and loss of retention are the most frequently reported complications of zirconia-based ceramics [14]. Particularly, poor retention may be ascribed to incorrect tooth prepara-

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tion and inadequate luting technique or cement selection [15,16].

Although zirconia restorations can be cemented with zinc phosphate or modified glass-ionomer cements [17], it has been reported that resin-based luting agents are the most appropriate materials for the purposes of marginal seal, retention, and fracture resistance [18]. Resin cements containing 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate) have been considered as the materials of choice, since the phosphate ester monomers are capable of a chemical interaction with the hydroxyl groups of the ZrO_2 ceramic [18,19].

Previous studies have investigated different chemo-mechanical surface treatments aimed at optimizing the cement/zirconia bonding mechanism [20–23]. The rationale of these conditioning processes lies in increasing the surface area available for bonding to obtain strong and durable restorations [24–26]. Sandblasting has been widely applied for increasing ceramics surface roughness and thus the surface area available for bonding. Up to date, the combination of sandblasting and 10-MDP monomer based resin is the recommended method of bonding to zirconia frameworks [27–30].

However, the outcome of this procedure may be affected by variables such as particle size and application distance. Particularly, excessive particle size and reduced application distance may induce crack initiation, possibly reducing the ceramic long-term mechanical properties [31–34].

Novel surface treatments have been proposed in order to improve zirconia/resin cement bonds, such as the selective infiltration etching (SIE). This treatment is based on the application of a low viscosity melting glass on the surface of zirconia, that create abraded and porous surfaces improving bond strength [35].

More recently, an experimental hot chemical etching solution (ST), composed by HCl and Fe_2Cl_3 in methanol, previously used for conditioning metal and/or alloys has been applied on zirconia ceramics with the result of improving their average surface roughness [36–38]. However, it has not yet been verified whether such increase in superficial roughness promotes the adhesion of the luting agent.

Therefore, the aim of this study was to evaluate the influence of different surface treatments on the bond strength between a commercially available partially-stabilized zirconia ceramic and a resin cement. The tested null hypothesis was that there were no statistically significant differences in the microtensile bond strengths measured at the zirconia ceramic–resin cement interface following different pre-treatments of the zirconia ceramic surface.

2. Materials and methods

Twelve cylinder-shaped (12 mm diameter, 5.25 mm height) Cercon® zirconia sintered ceramic blocks (DETREY DENTSPLY Ceramco, York, USA) were used for the study. Specimens were polished with SiC abrasive papers (grit # 600, 1000, 1200 and 2000). Final polishing was carried out on nylon cloths using 1- and 0.50 μm grit diamond pastes. Specimens were sonicated in deionized water for 5 min and randomly assigned to four equally sized experimental groups, according to the surface treatment performed on zirconia:

(1) *Selective infiltration etching procedure*. Specimens were coated with a thin layer of an infiltrating agent containing low temperature melting glass and additives (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.%)). 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, heated again up to 750 °C for 20 min (increasing T intervals 60 °C/min), and finally cooled at room temperature. Remnants of the infiltrating agent were dissolved immersing ceramic discs in an ultrasonic bath with 5% hydrofluoric acid solution for 30 min (SIE) [31].
(2) *Experimental etching solution*. A hot acidic solution containing HCl and Fe_2Cl_3 in methanol was heated up to 100 °C. The zirconia specimens were immersed in the solution for 30 min (ST) [36,37].
(3) Sandblasting with 125 μm Al_2O_3 particles for 10 s at 0.4–0.7 MPa from a distance of 20 mm (S).
(4) No pretreatment (C).

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

Resin composite blocks (Paradigm MZ100, 3M ESPE, size 14; batch # 20060213) were cut by means of a water-cooled diamond saw (Isomet 1000, Buelher, Lake Bluff, IL) into 12 cylinders of 4 mm in height.

The intaglio surface of each composite block was ground with 180-grit SiC paper, cleaned with ethanol and gently air-dried.

A dual-cure resin cement (Calibra™ DeTrey DENTSPLY; batch # 080910) was used in combination with the proprietary adhesive (XP Bond, DENTSPLY, batch # 0810003096) for luting the composite disc to the conditioned ceramic surface. The resin composite surface was etched with 37% phosphoric for 15 s, washed thoroughly for 1 min under tap water. Then a thin layer of adhesive (XP bond Adhesive) was applied on zirconia and composite surfaces, it was dried with a gentle airflow and polymerized for 20 s. Zirconia and composite blocks were luted using a resin luting material (Calibra) that was applied on zirconia surface; a seating pressure of 1 kg was maintained over the specimens during the first 5 min of cement autocure.

Then, light irradiation (Vip, Bisco, Schaumburg, Illinois, USA; Output: 500 mW/cm^2) was performed for 40 s on each side of the block to ensure optimal polymerization. Bonded specimens were stored in a laboratory oven at 37 °C and 100% relative humidity for 24 h.

2.1. Microtensile bond strength test

Ceramic–composite bonded specimens were cut vertically into 2 mm-thick slabs with a slow-speed diamond saw (Isomet). Each slab was serially sectioned into 2.0 × 2.0 mm sticks. From every ceramic–composite bonded specimen a number of sticks variable between 16 and 9 was obtained.

Each stick was glued with cianoacrylate (Super Attack gel Henkel Consumer Adhesives, Avon, Ohio, USA) to the free-sliding components of a Girardeli's jig, and loaded in tension