

ORIGINAL ARTICLE

Influence of air-particle deposition protocols on the surface topography and adhesion of resin cement to zirconia

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Abstract

Objectives. This study evaluated the influence of air-particle abrasion protocols on the surface roughness (SR) of zirconia and the shear bond strength (SBS) of dual-polymerized resin cement to this ceramic. **Materials and methods.** Sintered zirconia blocks ($n = 115$) (Lava, 3M ESPE) were embedded in acrylic resin and polished. The specimens were divided according to the ‘particle type’ (Al: 110 μm Al_2O_3 ; Si: 110 μm SiO_2) and ‘pressure’ factors (2.5 or 3.5 bar) ($n = 3$ per group): (a) Control (no air-abrasion); (b) Al2.5; (c) Si2.5; (d) Al3.5; (e) Si3.5. SR (Ra) was measured 3-times from each specimen after 20 s of air-abrasion (distance: 10 mm) using a digital optical profilometer. Surface topography was evaluated under SEM analyses. For the SBS test, ‘particle type’, ‘pressure’ and ‘thermocycling’ (TC) factors were considered ($n = 10$; $n = 10$ per group): Control (no air-abrasion); Al2.5; Si2.5; Al3.5; Si3.5; Control_{TC}; Al2.5_{TC}; Si2.5_{TC}; Al3.5_{TC}; Si3.5_{TC}. After silane application, resin cement (Panavia F2.0) was bonded and polymerized. Specimens were thermocycled (6,000 cycles, 5–55°C) and subjected to SBS (1 mm/min). Data were analyzed using ANOVA, Tukey’s and Dunnett tests (5%). **Results.** ‘Particle’ ($p = 0.0001$) and ‘pressure’ ($p = 0.0001$) factors significantly affected the SR. All protocols significantly increased the SR (Al2.5: 0.45 ± 0.02 ; Si2.5: 0.39 ± 0.01 ; Al3.5: 0.80 ± 0.01 ; Si3.5: 0.64 ± 0.01 μm) compared to the control group (0.16 ± 0.01 μm). For SBS, only ‘particle’ factor significantly affected the results ($p = 0.015$). The SiO_2 groups presented significantly higher SBS results than Al_2O_3 (Al2.5: 4.78 ± 1.86 ; Si2.5: 7.17 ± 2.62 ; Al3.5: 4.97 ± 3.74 ; Si3.5: 9.14 ± 4.09 MPa) and the control group (3.67 ± 3.0 MPa). All TC specimens presented spontaneous debondings. SEM analysis showed that Al_2O_3 created damage in zirconia in the form of grooves, different from those observed with SiO_2 groups. **Conclusions.** Air-abrasion with 110 μm Al_2O_3 resulted in higher roughness, but air-abrasion protocols with SiO_2 promoted better adhesion.

Key Words: Adhesion, aluminum oxide, roughness, scanning electron microscopy, shear strength, silicon dioxide, yttria stabilized tetragonal zirconia

Introduction

Currently, ceramics that are based on zirconium oxide (hereon: zirconia) are being extensively studied because of their more favourable mechanical properties as opposed to other dental ceramics [1]. The polycrystalline tetragonal zirconia partially stabilized with yttria (Y-TZP) is composed of zirconium dioxide (ZrO_2) and displays a polymorphic structure that can present

different crystalline phases (monoclinic, tetragonal or cubic), depending on the temperature [2]. Yttrium oxide (Y_2O_3) is one of the most widely used stabilizers for this polycrystalline ceramic. When added (3–6%) to pure zirconia, it serves to stabilize zirconia at room temperature in the tetragonal phase, resulting in a crystalline material with high mechanical strength [3].

Common clinical failures associated with Y-TZP fixed dental prosthesis (FDP) are chipping of the

Table I. Brands, types, manufacturers and batch numbers of the materials used in this study.

Brand	Type	Manufacturer	Batch number
Lava	Y-TZP ceramic block	3M ESPE, Seefeld, Germany	391266
Aluminum oxide	Aluminum oxide (110 μm)	Polidental Ltd., São Paulo, Brazil	21567
Rocatec Plus	Aluminum oxide coated with silica (110 μm)	3M ESPE	269078
ClearFill Ceramic Primer	MDP, HEMA, hydrophilic dimethacrylates, dl-camphorquinone, N, N-diethanol-p-toluidine, H ₂ O	Kuraray Co. Ltd, Osaka, Japan	00009C
Panavia F2.0	Paste A: silica, dimethacrylate monomer, functional acid MDP, photo-initiator, accelerator; Paste B: brown color, barium glass, sodium fluoride, dimethacrylate (DMA) monomer	Kuraray Co. Ltd.	340AA 062BA

veneering ceramic [4], framework fracture [4,5], secondary caries [4] and debonding [4,6]. The main reason for debonding is poor adhesion between the cement and zirconia [4,6]. Hydrofluoric acid etching followed by the application of silane coupling agent in silica-based ceramics is a well-established method to increase the adhesion of resin cement to such ceramics. However, this technique is not effective in zirconia because of its highly stable oxides that makes it resistant to acid etching [7]. Many researchers are engaged in the study of techniques to promote better adhesion between zirconia and resin cement. Several procedures have been suggested for this purpose such as silica deposition by plasma [8], selective infiltration etching [9], use of cements and adhesives based on 10-methacryloyloxy-decyl dihydrogenphosphate (MDP) [10], glaze application followed by etching [11] and air-borne particle abrasion [12]. Among all these methods, air-abrasion protocol is a simple, inexpensive technique that can be performed chairside [12]. In an attempt to increase the bond strength of resin cements to zirconia, particles of alumina (Al_2O_3) and alumina particles coated with silica (SiO_2) have been used employing different protocols [2,13–16]. Air-abrasion with alumina [15,17] or silica followed by silane application [17] seems to improve adhesion to zirconia. The efficacy of these procedures depends highly on the type of particles. The tribochemical silica coating technique has been shown to be more effective than air-abrasion with ordinary alumina, generating stable adhesion even after water storage for 6 months [17] or 37,500 thermal cycles for 150 days [14]. Yet, the effect of particle size and pressure during air-abrasion is not clearly known. For this reason, a well-defined pre-treatment protocol for zirconia frameworks is not defined so far [18].

It can be anticipated that the adhesion between resin cement and zirconia would be more durable when micromechanical retention is achieved since a rough

ceramic surface would allow the micromechanical interlocking of the resin cement through microretentions [19]. Accordingly, surface roughness of zirconia increases after air-particle abrasion or silica coating [18]. The objective of this study was to evaluate the influence of air-particle abrasion protocols on the surface roughness of zirconia ceramic and the adhesion of dual-polymerized resin cement to this ceramic. The null hypotheses tested were that surface roughness and shear bond strength of the resin cement would not be influenced by the air-particle abrasion protocols applied on zirconia.

Methods and materials

The materials used in this study and their respective brands, manufacturers and batch numbers are presented in Table I.

Ceramic block preparation

Sintered zirconia blocks (5.25 mm \times 5.25 mm \times 3 mm) (Lava, 3M ESPE, Seefeld, Germany) ($n = 115$) were sectioned using a diamond disc (KG Sorensen, Barueri, Brazil) and their surfaces were ground finished with silicon carbide papers of #600 to #1200 (3M, St. Paul, MN). The blocks were sintered in a specific furnace (Lava Furnace 200, 3M ESPE) according to the manufacturer's recommendations.

Zirconia blocks were embedded in acrylic resin (Clássico Dental Products Inc., São Paulo, Brazil) using a silicone mold. After polymerization, each resin block was removed from the mold and the zirconia surfaces were ground finished using silicon carbide papers in grit sequence of #600, #1200, #1500 and #2000 (3M) under water cooling. They were then polished with a diamond paste (Diamond Excel Paste, FGM Dental Products, Joinville, Brazil) with a particle size of 10 μm and 3 μm , followed by colloidal silica (Struers OPS, Struers, Brisbane, Australia),

Table II. Experimental groups and group abbreviations for surface roughness (\pm standard deviation) test according to the main factors: particle type (2 levels) and pressure (2 levels).

Group abbreviations	Air-particle-abrasion		
	Particle type	Pressure (bar)	Surface roughness (μm)*
Control	—	—	0.16 ± 0.01
Al2.5	Al_2O_3	2.5	$0.45 \pm 0.02^{\text{C}}$
Si2.5	SiO_2	2.5	$0.39 \pm 0.01^{\text{D}}$
Al3.5	Al_2O_3	3.5	$0.80 \pm 0.01^{\text{A}}$
Si3.5	SiO_2	2.5	$0.64 \pm 0.01^{\text{B}}$

*Different capital letters in the last column denote statistical difference between groups (Tukey test; $p < 0.05$).
 $n = 3$ per group.

both on felt discs mounted on a polishing machine (Erios, PSK-2V, São Paulo, Brazil).

Air-particle abrasion protocols

Prior to air abrasion, the blocks were ultrasonically cleaned in 10% isopropyl alcohol for 5 min (Vitasonic II, Vita Zanhfabrik, Bad Säckingen, Germany) and placed onto gauze for 10 min to ensure complete alcohol evaporation. The specimens were randomly divided into the following experimental groups considering the particle type and pressure (Table II):

- Control group: The polished specimens acted as the control group.
- Group Al2.5: Zirconia specimens were air-abraded with $110 \mu\text{m}$ aluminium oxide (Polidental Ltd., Sao Paulo, Brazil) at 2.5 bar pressure.
- Group Si2.5: Zirconia specimens were air-abraded with $110 \mu\text{m}$ aluminium oxide coated with silica (Rocatec Plus, 3M ESPE) at 2.5 bar pressure.

In the groups Al3.5 and Si3.5 air-abrasion was employed at 3.5 bar pressure.

All air-abrasion protocols were performed using a chairside air-abrasion device (Dento-Prep, RØNVIG A/S, Daugaard, Denmark) attached to a metallic device [20], perpendicular to the surface of the ceramic blocks at a distance of 10 mm for 20 s with the specified pressure, according to the experimental group. Air-abrasion procedures were performed in circular movements to achieve a uniformly blasted surface.

Surface roughness (SR) analyses

Based on a previous study [21], the sample size was calculated using t -test considering a power of 99% and 5% of α error. A sample size of two specimens per group was decided but, to adjust the sample size due to differences in experimental design, three specimens were included in each group.

SR measurement of ceramics ($n = 15$, $n = 3$ per group) after air-abrasion were analyzed in a digital optical profilometer (Wyko NT 1100 Optical Profiling System, Veeco, New York, NY, USA.), which was connected to a computer. The data were analyzed using the specific software (Wyko Vision 32, Wyko, Veeco, USA). Measurements of 3D parameters were performed with a magnification of $\times 20$ and an area of $301.3 \times 229.2 \mu\text{m}$. Three measurements were made from each specimen. The roughness values (Ra) were obtained in micrometers.

Surface topography analysis

Surface topography of the specimens after air-abrasion protocols was analyzed at a magnification of $\times 5000$ using a Scanning Electron Microscope (SEM, Leo model 1430 VP, Zeiss, Cambridge, UK) equipped with digital software. Prior to gold sputtering, the specimens were cleaned in absolute ethanol ultrasonically (35 kHz) (Vitasonic II) for 10 min. Then the specimens were positioned on an aluminum stub and sputtered with a thin conductive layer of gold (50–100 Angstrom) by vapor deposition in an ion sputtering machine (Emitech K550X, Emitech, Ashford, UK).

Specimens for shear bond strength (SBS) test

Prior to silane application, the blocks were also ultrasonically cleaned in 10% isopropyl alcohol for 5 min (Vitasonic II) and placed onto gauze for 10 min to ensure complete alcohol evaporation, except for SiO_2 groups that had their surfaces only sprayed with air after sandblasting, as suggested by the manufacturer.

For the SBS test, zirconia blocks were randomly divided into 10 groups ($n = 100$, $n = 10$ per group), according to the particle type (Al and Si), pressure (2.5 and 3.5 bar) and thermocycling (TC, with and without) factors: Control (no air-abrasion); Al2.5; Si2.5; Al3.5; Si3.5; Control_{TC}; Al2.5_{TC}; Si2.5_{TC}; Al3.5_{TC}; Si3.5_{TC} (Table III).

Silane coupling agent (Clearfil Ceramic Primer, Kuraray, Japan) was applied to the air-abraded zirconia surfaces in all groups including the control group with a microbrush (Dentsply, New York, NY) and left to react for 5 min, according to the manufacturer's recommendations. Then, resin cement (Panavia F2.0, Kuraray) was bonded to the silanized zirconia surfaces with the aid of a silicone mold (diameter: 3.5 mm, height: 3 mm). The lower orifice of the silicone mold was positioned in the center of cementation surface of the ceramic, so that the entire layer of cement stayed in contact with the ceramic. With the aid of a plastic spatula, the base paste and catalyst paste were manipulated until homogenization of the cement. The cement was inserted in the silicone mold and photo-polymerized using the incremental technique (three layers of 1 mm each). Each layer of

Table III. Experimental groups for shear bond strength (SBS; \pm standard deviation) test according to the main factors: particle type (2 levels), pressure (2 levels) and thermocycling (2 levels).

Groups	Particle type	Pressure (bar)	Thermocycling	SBS (MPa)
Control	—	—	Without	3.67 ± 3.0^B
Al2.5	Al ₂ O ₃	2.5	Without	4.78 ± 1.86^B
Si2.5	SiO ₂	2.5	Without	7.17 ± 2.62^A
Al3.5	Al ₂ O ₃	3.5	Without	4.97 ± 3.74^B
Si3.5	SiO ₂	3.5	Without	9.14 ± 4.09^A
Control _{TC}	—	—	With	NT
Al2.5 _{TC}	Al ₂ O ₃	2.5	With	NT
Si2.5 _{TC}	SiO ₂	2.5	With	NT
Al3.5 _{TC}	Al ₂ O ₃	3.5	With	NT
Si3.5 _{TC}	SiO ₂	3.5	With	NT

*Different capital letters in the last column denote statistical difference between groups (Tukey test; $p < 0.05$). NT, Not tested due to pre-test failures.

$n = 10$ per group.

cement was photo-polymerized for 40 s (XL 3000, 3M ESPE; light intensity= 600 mW/cm²).

Thermocycling

The specimens from groups Control_{TC}, Al2.5_{TC}, Si2.5_{TC}, Al3.5_{TC} and Si3.5_{TC} were subjected to thermocycling (TC) (Nova Etica, São Paulo, Brazil) for 6000 cycles at $5\text{--}55^\circ\text{C} \pm 1^\circ\text{C}$ in water. The time of immersion in each bath was 30 s and transfer time between the two baths was 2 s.

The specimens from groups that were not subjected to TC were stored in distilled water at 37°C for 24 h prior to the SBS test.

Shear bond strength test

The SBS test was performed in the Universal Testing Machine (EMIC model DL-1000, São José dos Pinhais, Brazil) according to ISO 10477:2004 Test 7.7 [22]. A metallic device was used to position the specimen in the testing machine so that the ceramic–cement interface was perpendicular to the horizontal plane. A knife-shaped device was placed on the load cell (100 kgf) of the testing machine and the ceramic–cement interface was loaded at a constant cross-head speed of 1 mm/min until debonding.

Failure analysis

After debonding, failure types were analyzed using a stereomicroscope (Stemi 2000-C, Carl Zeiss, Göttingen, Germany) at $\times 100$ magnification.

Failure types were classified as follows: (A) adhesive failure between the ceramic–cement; (B) cohesive

Table IV. Results of two-way analysis of variance (ANOVA) and the interaction terms for surface roughness (Ra) measurements depending on particle type and pressure.

Effect	DF	SQ	p
Particle type	1	0.035 21	0.0001*
Pressure	1	0.267 01	0.0001*
Particle type \times Pressure	1	0.008 01	0.0001*
Error	8	0.000 80	
Total	11	0.311 03	

* $p < 0.05$.

failure in the ceramic; (C) cohesive failure in the cement and (D) mixed failure (adhesive failure together with cohesive failure in the cement).

Statistical analysis

SR data were analyzed using two-way ANOVA (two levels: particle type and pressure) followed by Dunnett test using a statistical software package (Statistix 8.0 for Windows, Analytical Software Inc, Tallahassee, FL). SBS data were analyzed using three-way ANOVA (three levels: particle type, pressure and thermocycling) followed by Tukey's and Dunnett tests ($\alpha = 0.05$).

Results

Surface roughness

Both the particle type ($p = 0.0001$) and pressure ($p = 0.0001$) significantly influenced the roughness results (Table IV). Interaction terms were also significant ($p = 0.0001$).

All protocols significantly increased the SR compared to the control group (Dunnett) (Table II). The mean SR for the group Al3.5 ($0.80 \pm 0.01^A \mu\text{m}$) was significantly higher than those of other groups (Tukey's test) (Table II).

Surface topography analysis

Specimens in Al2.5 and Al3.5 groups presented defects in the form of grooves and chips on their surfaces, indicating that these protocols damage the ceramic surface. Representative photomicrographs of each group and 3D images of these surfaces obtained by digital optical profilometer are presented in Figures 1A–F.

Shear bond strength

While particle type significantly affected the SBS results ($p = 0.015$), pressure did not ($p = 0.398$).

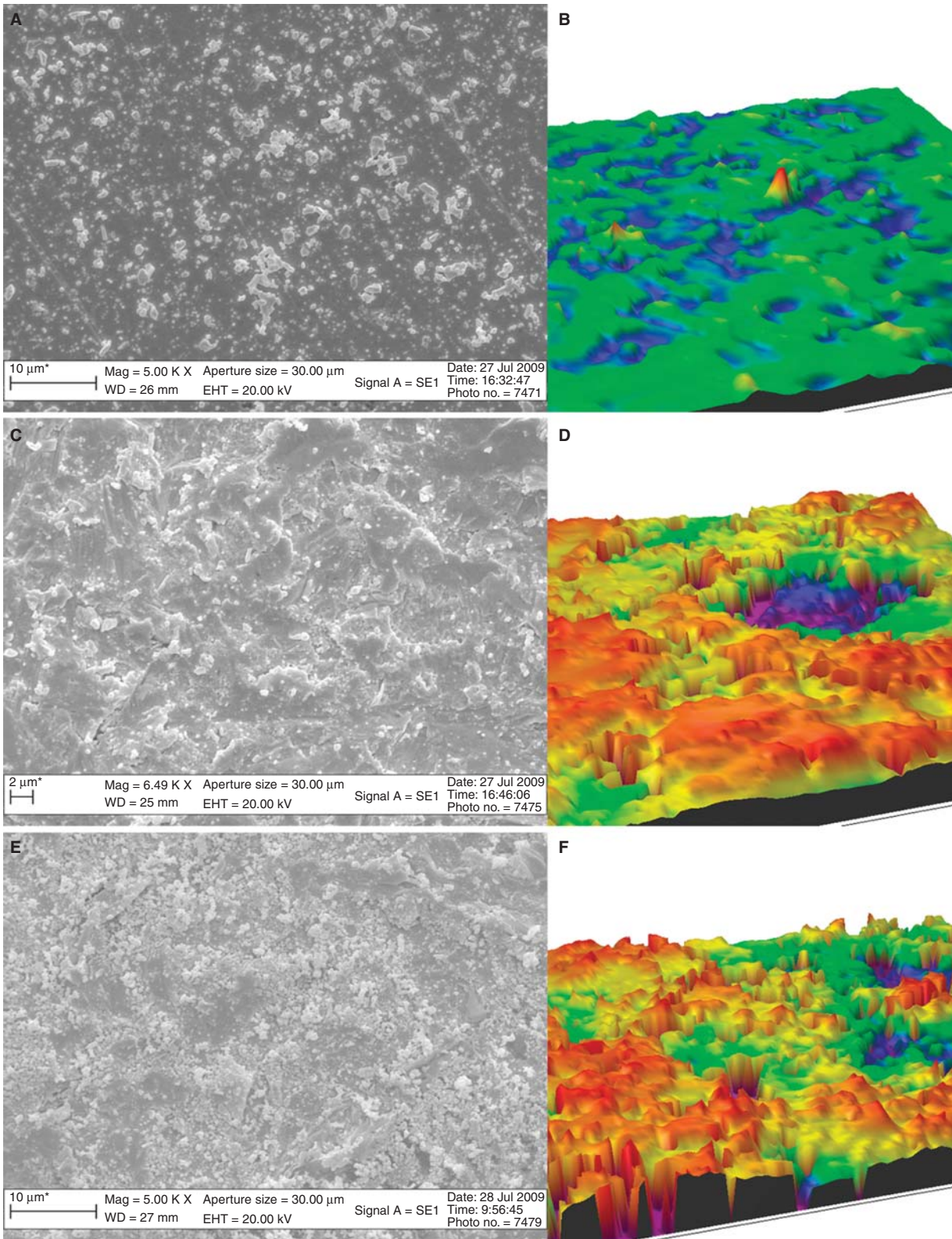


Figure 1. (A) Scanning Electron Microscopy (SEM) photomicrograph ($\times 5000$) and (B) 3D graphic representation of the surface topography of the non-abraded control zirconia; (C) SEM photomicrograph ($\times 5000$) and (D) 3D graphic representation of the surface topography of zirconia air-abraded with Al_{3.5}; (E) SEM ($\times 5000$) and (F) 3D graphic representation of the surface topography of zirconia air-abraded with Si_{3.5}. Note the more aggressive topography change after air-abrasion with Al_{3.5} compared to control and Si_{3.5}.

Table V. Results of two-way analysis of variance (ANOVA) and the interaction terms for shear bond strength (MPa) depending on particle type and pressure.

Effect	DF	SQ	<i>p</i>
Particle type	1	72 055	0.0150*
Pressure	1	7 781	0.3980
Particle type × Pressure	1	5 296	0.4846
Error	24	252 142	
Total	27		

* $p < 0.05$.

Interaction terms were not significant ($p = 0.4846$) (Table V).

Groups Si2.5 and Si3.5 presented the highest SBS values, whereas Al2.5 and Al3.5 the lowest (Tukey's test) (Table III). When the experimental groups were compared to the control group, only groups Si2.5 and Si3.5 promoted significantly higher bond strength ($p < 0.05$) (Table III).

All groups showed pre-test (PTF) failures during TC (Table VI). Thus, the thermocycling factor was not considered in the statistical analysis.

Failure analysis

Failure types were predominantly mixed (Score D) in all groups (Table VII). Only in the control group, failure types were exclusively adhesive (Score A). None of the groups showed cohesive failures in the ceramic (Score B).

Discussion

Based on the results of this study, since particle type and pressure significantly affected the surface roughness of zirconia, and particle type significantly

Table VI. Number (*n*) of specimens produced, percentage (%) of pre-test failures (PTF) during thermal cycling (TC) or water storage and number of tested specimens (TE).

Groups	<i>n</i>	<i>n</i> (%) of PTF	<i>n</i> (%) TE
Control	10	4 (40)	6 (60)
Al2.5	10	5 (50)	5 (50)
Si2.5	10	5 (50)	5 (50)
Al3.5	10	5 (50)	5 (50)
Si3.5	10	4 (40)	6 (60)
C _{TC}	10	10 (100)	0
Al2.5 _{TC}	10	10 (100)	0
Si2.5 _{TC}	10	10 (100)	0
Al3.5 _{TC}	10	10 (100)	0
Si3.5 _{TC}	10	10 (100)	0

Table VII. Incidence of failure types (%) for the experimental groups.

Groups	Failure types			
	A	B	C	D
Control	—	—	—	100
Al2.5	—	—	20	80
Si2.5	—	—	10	90
Al3.5	—	—	10	90
Si3.5	—	—	20	80
Control _{TC}	100	—	—	—
Al2.5 _{TC}	100	—	—	—
Si2.5 _{TC}	100	—	—	—
Al3.5 _{TC}	100	—	—	—
Si3.5 _{TC}	100	—	—	—

Score A: adhesive failure between the ceramic-cement; Score B: cohesive failure in the ceramic; Score C: cohesive failure in the cement; and Score D: mixed failure (adhesive failure together with cohesive failure in the cement).

influenced the bond strength of the resin cement, the null hypotheses could be rejected.

The surface roughness values for experimental groups showed statistical differences. The surface analysis by optical profilometry indicated higher roughness values when zirconia surfaces were air-abraded with 110 μm Al₂O₃ particles under 3.5 bar pressure followed by 110 μm alumina particles coated with silica under 3.5 bar. The high precision of an optical profilometer being able to detect topographical changes less than 0.1 nm increases the reliability of the measurements. SEM analysis showed more defects on zirconia surface with the use of 110 μm Al₂O₃ particles that may be attributed to sharp morphology of the individual particles in this sand type [23]. On the contrary, coating of alumina particles with the silica using sol-gel technologies reduced the sharp morphology of the alumina particles, possibly also reducing the impact of the particles on the zirconia surface. Furthermore, under both 2.5 and 3.5 bar pressure, Al₂O₃ generated surface roughness being statistically higher than with SiO₂. Although the particle size was similar, namely 110 μm , this finding indicates that the variation in particle morphology is of importance. These results were similar to those reported in an *in vitro* investigation where atomic force microscopy was used to detect the surface roughness [24]. Whether surface damage created with 110 μm Al₂O₃ particles is detrimental for clinical success of zirconia FDPs needs to be verified in clinical studies.

In this study, all air-abrasion protocols provided micromechanical retention and thereby better bond strength than the control group, supporting the findings of previous studies [16,25,26]. Interestingly,

however, high surface roughness obtained with Al_2O_3 under both 2.5 and 3.5 bar pressure did not necessarily yield to higher bond strength. This implies that chemical aspect of physico-chemical conditioning was more favorable for SiO_2 [10,14,15]. Although tribochemical conditioning using Rocatec Plus necessitates the use of 3-methacryloxypropyltrimethoxysilane (MPS) silane according to the manufacturer, and MDP-based silane was used which is the recommendation of the resin cement tested. Phosphate ester groups in this silane bond directly to the surface oxides of zirconia and the methacrylate group makes covalent bonds with the resin matrix of the cement [27,28]. In a previous study [7], slightly better results were obtained in dry conditions when an MPS silane was used in combination with bis-GMA cement. However, also in that study [7], after aging conditions, practically no adhesion was achieved. This implies that both MPS and MDP-based silanes do not provide hydrolytically stable interfaces with the resin cement and zirconia. Higher values were also reported by Akgungor [14] when the zirconia was treated with CoJet and silanization (27.1 ± 3.2 MPa) than alumina airborne-particle abrasion (23.3 ± 2.2 MPa). On the other hand, Yang et al. [15] observed that when the zirconia was air-abraded with alumina and followed by MDP-containing primers application, higher values of bonding to dental zirconia ceramic (39.3 MPa) were provided.

In this study, air-abrasion protocols were applied for 20 s based on the results of some preliminary studies. No statistically significant difference on the bond strength between the two different pressures, 2.5 and 3.5 bar, regardless of particle type. This may change when zirconia surfaces are air-abraded for prolonged durations.

An important factor that influences bond strength is the aging in with thermocycling, which is often used in *in-vitro* studies to simulate the worse-case clinical conditions. There is no consensus on a relevant protocol for artificial aging by thermocycling. In general, average temperatures of 5°C and 55°C have been used as the lower and upper temperature in the water tanks [29]. The ISO 11405 norm indicates that 500 cycles in water at 5–55°C is an appropriate method for aging resin–tooth interfaces [30]. Moreover, Gale and Darvell [29] reported that 10,000 cycles corresponds to ~ 1 year of *in vivo* function. In the present study, 6000 thermocycles with an immersion time of 20 s in each bath corresponds to a period of ~ 7.2 months of clinical use.

Previous studies have generally shown reduced bond strength of resin cement to zirconia after different artificial thermocycling periods. Compared to water storage at constant temperature only, thermocycling has a greater impact on the bond durability between the zirconia and resin cements [31,32]. Kern and Wegner [31] evaluated the bond strength to zirconia after 150 days of water storage only or water storage followed by thermocycling [32]. In another

study, significant reduction in adhesion was reported after artificial aging for 180 days combined with thermocycling (12 000 cycles, 5–60°C) [33]. The authors concluded that air-abrasion combined with the use of an MDP-based cement resulted in more durable adhesion, demonstrating only cohesive failures in the cement. In the present study, specimens of all groups subjected to thermocycling showed spontaneous debondings. Thus, the thermocycling factor could not be analyzed. On the other hand, the specimens tested without aging conditions presented mainly cohesive failure of the cement or mixed failure type. These types of failures indicate some degree of adhesion that does not surpass the cohesive strength of zirconia. Exclusively adhesive failures observed in the control group imply the necessity of physico-chemical surface conditioning of zirconia.

In a previous study, no adhesion was obtained after air-abrasion with alumina and silanization following thermocycling [15]. This poor adhesion was attributed to the hydrolysis of Al-O-Si in aqueous conditions [34,35]. The results of this study contradict with what was found by Amaral et al. [20], who reported stable adhesion to zirconia even after thermocycling when the surface was coated by silica. It has to be noted that, in that study, microtensile test was used. It is most probable that the aging effect of thermocycling in the resin–zirconia interface in the bonded specimens are less than those prepared for shear test. This aspect warrants further investigation.

Clinical success of bonded FDPs relies on the adhesion of the resin cement to both the restoration and the dental tissues. Thus, further *in vitro* and *in vivo* studies should be developed with the aim of clarifying the influence of surface conditioning methods on the adhesion of resin cements to both zirconia and tooth substrates.

Conclusions

From this study, the following could be concluded:

- (1) Air-abrasion with 110 μm alumina or 110 μm alumina particles coated with silica increased surface roughness and shear bond strength of the MDP containing resin cement to zirconia compared to the control group.
- (2) The use of alumina particles coated with silica revealed less damage on zirconia and showed increased bond strength compared to air-abrasion with alumina.
- (3) Increasing blasting pressure from 2.5 to 3.5 bar increased the surface roughness values but it did not affect the mean bond strength of the resin cement to zirconia.
- (4) After 6000 thermocycles, spontaneous debondings in all groups indicates that adhesion to zirconia is prone to aging.

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