

ORIGINAL ARTICLE

Effect of air abrasion particles on the bond strength of adhesive resin cement to zirconia core

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Abstract

Objective. To evaluate the effect of air abrasion with different particles of different sizes and forms on the shear bond strength of adhesive resin cement to zirconia core. **Material and methods.** Sixty zirconia core disks were produced and sintered. The specimens were divided into six equal groups for application of air abrasion procedures. The surfaces of the specimens were treated with one of five air abrasion particles: 30 µm silica-coated aluminum oxide particles; 1–3 µm synthetic diamond particles; 110 µm aluminum oxide particles; 30–50 µm synthetic diamond particles; and 60–80 µm cubic boron nitride particles. The remaining 10 specimens were untreated and served as controls. Composite resin disks were cemented to each of the zirconia core specimens. All specimens were stored in distilled water at 37°C for 24 h and thermocycled for 6000 cycles. The shear bond strength was measured using a universal testing machine at a crosshead speed of 1 mm/min. Data were statistically analyzed by one-way ANOVA with Tamhane tests ($\alpha = 0.05$). The effect of the air abrasion procedures was examined using scanning electron microscopy. **Results.** Air abrasion with different materials affected the bond strength ($P < 0.001$). The highest bond strengths were obtained by air abrasion with 30–50 µm synthetic diamond particles; the lowest bond strengths were obtained in the control group ($P < 0.001$). **Conclusion.** Air abrasion with 30–50 µm synthetic diamond particles, 60–80 µm cubic boron nitride particles and 110 µm aluminum oxide particles showed higher bond strength values than other methods.

Key Words: Air abrasion, cubic boron nitride, surface treatment, synthetic diamond particle, zirconia core

Introduction

At the beginning of the 1990s, oxide ceramics that contain only little or no silica were introduced into restorative dentistry. By definition, oxide ceramics contain <15 wt.% silica. i.e. only a small or no glass phase [1]. Current dental oxide ceramics consist mostly of alumina, magnesia, zirconia and yttrium tetragonal zirconia polycrystal (Y-TZP; zirconia) [1]. Y-TZP is one of the most commonly used oxide ceramics for conventional and resin-bonded fixed partial dentures (FPDs) and complete coverage crowns [1–6]. Zirconium oxide restorations can be cemented using conventional methods or resin cements [1,2,7–11]. Although superior in terms of mechanical performance (strength, toughness, fatigue resistance), bonding of resins to these materials is more difficult than it is for conventional silica-based dental ceramics [3,10,12].

Non-treated zirconium oxide ceramic is a relatively inert substrate with low surface energy and wettability [4]. To create a reliable bond between the ceramic material and adhesive resins, an increasing number of surface pretreatment methods are being introduced [3,4,7,13]. Common treatment options are grinding with diamond burs, air abrasion with aluminum oxide (Al_2O_3), tribochemical silica-coating (CoJet, Rocatec), acid etching with hydrofluoric acid, coupling with silane, plasma spraying with hexamethyldisiloxane, applying a low-fusing porcelain pearl layer and combinations of any of these methods [2,5,7,12–17]. Although etching the inner surfaces of conventional silica-based dental ceramics with hydrofluoric acid or the application of the most-often used silane-coupling agent, 3-methacryloxypropyltrimethoxysilane (MPS), was found to be an effective conditioning method for bonding resin composite [1,9,15,18], these methods showed an unsatisfactory resin bond

to oxide-based dental ceramics [1,4,9,10,19]. Because the composition and physical properties of high-strength ceramic materials differ substantially from those of silica-based ceramics, these specific ceramics require very aggressive mechanical abrasion methods or alternative bonding techniques to increase surface roughness and achieve strong, long-term durability [4,9,10,12,19].

High-strength ceramics are compact materials, making them difficult to grit-blast [19]. Airborne abrasion seems to be a prerequisite to achieve high bond strengths and durable bonds to zirconium oxide ceramics [20] and this process could be applied either in the laboratory or chair-side, using large- or small-sized particles [19]. Different sizes of abrasive alumina particles between 50 and 110 μm are usually used and it was stated that airborne particle abrasion increased long-term shear bond strength significantly, regardless of the abrasive particle size [21]. Tribochemical silica coating followed by silanization has been used to improve the bond strength of resins to oxide-based ceramics [2,7] and varying results have been obtained [1,2,8,15,16,19,20,22,23].

In dentistry, one alternative air abrasion material, synthetic diamond particles, which were used to increase the surface roughness and surface area, was previously applied onto the glass-infiltrated aluminous oxide ceramic (In-Ceram; Vita Zahnfabrik, Germany) surface [13].

The search for alternative super-hard materials, some of which might even be harder than diamond, has stimulated high-pressure research for >50 years. High-pressure synthesis on an industrial scale is applied to obtain synthetic diamonds and cubic boron nitride (CBN), which are the super-hard abrasives of choice for cutting and shaping hard metals and ceramics [24,25]. CBN is the hardest material presently available, next to diamond, and may be another alternative material for air abrasion.

Owing to excessive air abrasion-induced chipping or a high loss of ceramic material [12], a non-destructive, simple method for treating the ceramic surfaces

would be very useful, especially if it was compatible with existing adhesive bonding techniques (silane + resin cement) [10]. However there is limited knowledge on the effects of different size and mechanical properties of air abrasion particles on resin bond strength to zirconium oxide-based ceramic.

The purpose of this study was to evaluate the effects of alternative air abrasion particles of different sizes and forms on a zirconia core that could be used to obtain a reliable bond between the ceramic material and adhesive resin cement. The research hypothesis tested was that the shear bond strengths after air abrasion with different particles varying in size and form would not be different.

Material and methods

The materials used in this study are presented in Table I. Sixty zirconium oxide core specimens (10-mm diameter; 2-mm thickness) were produced by a copy-milling system (Zirconzahn, Bruneck, Italy) using prefabricated blanks of zirconium oxide (ICE Zircon Translucent; Zirconzahn) and then sintered according to the manufacturer's instructions. Zirconium oxide cores were embedded in the centers of autopolymerizing acrylic resin blocks (Meliodent; Heraeus Kulzer, Armonk, NY). Zirconium oxide core surfaces were ground-finished with a 1200-grit silicone carbide abrasive paper (3M ESPE, St. Paul, MN) for 10 s on a 300 rev/min grinding machine (Buehler Metaserv, Buehler, Germany) under running water, ultrasonically cleaned for 3 min in ethanol and deionized water and air-dried. Subsequently, all specimens were divided into six groups, each containing 10 specimens, for air abrasion procedures. One of the groups served as a control and no surface treatment was applied (Group C). The other groups involved air abrasion with 30 μm silica-coated aluminum oxide particles (SiO_x ; CoJet Sand; Group Co); 1–3 μm synthetic diamond particles (Micron+MDA; Group D1); 110 μm Al_2O_3 particles (Korox110; Group K); 30–50 μm synthetic diamond particles

Table I. Materials used in the study.

Material	Type	Batch no.	Manufacturer
Ice Zircon	Yttrium oxide partially stabilized with tetragonal polycrystalline structure (Y-TZP)	ZA9110M	Zirconzahn SRL, Bruneck, Italy
Micron + MDA M13	1–3 μm synthetic diamond particles	1165546	Elementsix, Shannon, Ireland
Micron + MDA M3050	30–50 μm synthetic diamond particles	2670483	Elementsix, Shannon, Ireland
ABN600	60–80 μm CBN	2739835	Elementsix, Shannon, Ireland
Korox 110	99.6% 110 μm Al_2O_3	681128	BEGO, Bremen, Germany
CoJet Sand	30 μm silica-coated Al_2O_3 particles	172562	3M ESPE, Seefeld, Germany
Panavia F 2.0	Dual-polymerized adhesive resin cement	41233	Kuraray, Okayama, Japan

(Micron+MDA; Group D2); and 60–80 μm CBN particles (ABN600; Group CBN). The air abrasion procedure was performed using an intraoral air abrasion device (Microetcher; Danville Engineering Inc, San Ramon, CA) at an air pressure of 3 bar for 15 s at a distance of ≈ 10 mm. Before the bonding procedure, zirconium oxide core specimens (except for those in Group Co) were ultrasonically cleaned in ethanol and deionized water for 3 min and air-dried.

Sixty composite resin disks (Filtek Z250; 3M ESPE, Seefeld, Germany) were fabricated by compacting the material into a polytetrafluoroethylene mold (Isoflon, Diemoz, France) with a hole in the center (6-mm diameter; 2-mm thickness). Composite resin was incrementally condensed into the mold to fill it up and each layer was light-polymerized for 40 s at a distance of 1 mm using a light-polymerizing unit (Astralis 3; Ivoclar Vivadent, Schaan, Liechtenstein) with an output power of 600 mW/cm². One composite resin block was fabricated for each specimen.

Composite resin disks were cemented to the specimen surfaces with dual-polymerized adhesive resin cement (Panavia F 2.0; Kuraray Co Ltd, Osaka, Japan) containing the 10-Methacryloyloxydecyl dihydrogen phosphate (MDP). For cementation, equal amounts of a dual-polymerized resin-luting agent paste base and catalyst were mixed and applied to the composite resin block with a plastic spatula. Each composite disk was bonded to a zirconium oxide core specimen under a load of 500 g. The excess resin cement was removed by means of a brush. The resin cement was then light-polymerized for 20 s with a curing light (Astralis 3). A glycerin gel (Oxyguard II; Kuraray Co Ltd) was applied on the cement layer for 10 min. The specimens were washed with an air-water spray, stored in distilled water at 37°C for 24 h and thermocycled for 6000 cycles between $5 \pm 2^\circ\text{C}$ and $55 \pm 2^\circ\text{C}$ with a dwell time of 30 s.

A universal test machine (Lloyd LRX; Lloyd Instruments PLC., Fareham, UK) was used for the shear bond strength test at a crosshead speed of 1 mm/min. Each specimen surface was parallel to the direction of the force during the shear bond strength test. Force was applied to the zirconium–composite interface. The shear bond strength values were calculated in megapascals (MPa) by dividing the failure load (N) by the area of the composite resin ($\text{N}/\pi r^2$). Data were statistically analyzed. The Kolmogorov–Smirnov test showed that the data followed a normal distribution ($P > 0.05$). A homogeneity of variance test was done using Levene's test ($F = 0.475$, $P > 0.05$). Means and standard deviations (SDs) of bond strengths were calculated and mean values were compared by ANOVA (SPSS 12.0; SPSS Inc., Chicago, IL), followed by a multiple-comparisons test performed using a Tamhane test ($\alpha = 0.05$).

To assess the mode of failure (cohesive, adhesive or mixed failure at the ceramic or the composite–resin

surface), the specimen surfaces were examined visually.

To evaluate the effects of air abrasion on the surface morphology of zirconium oxide core ceramic, six additional samples were treated with the same experimental protocol as described previously. All specimens were coated with gold using a sputter coater (S150B; Edwards, Crawley, UK) and examined under a field-emission scanning electron microscope (JSM-6335F; JEOL, Tokyo, Japan) at 20 kV. Air abrasion particles were also examined using scanning electron microscopy (SEM). The SEM photomicrographs were developed at a magnification of $\times 250$ for visual inspection.

Results

The means and SDs of the shear bond strength values are presented in Table II. One-way ANOVA revealed significant differences between the air abrasion particles ($P < 0.001$). The bonding strength depended on the type and particle size of the air abrasion particles. Air abrasion with 30–50 μm synthetic diamond particles showed a stronger bond between the resin cement and zirconium oxide core and no significant differences were found when compared with 60–80 μm CBN and 110 μm Al_2O_3 particles ($P > 0.05$). There were no significant differences between air abrasion with 1–3 μm synthetic diamond particles and silica-coating with 30 μm SiO_x ($P > 0.05$). The lowest shear bond strength value was obtained in the untreated control group ($P < 0.05$).

Visual examination of the fractured surfaces showed that mixed and adhesive fractures were the most common types of failure for the air-abraded groups. Pure zirconia and pure composite resin cohesive failures were not observed. The control group specimens exhibited 100% adhesive failures on average between the zirconia core and the resin-luting cement. Adhesive failures between the zirconia core and resin-luting cement occurred on average in 60%

Table II. Mean (\pm SD) values of shear bond strength.

Group	Surface treatment method	MPa (\pm SD)*
C	Control	13.9 (± 1.6) ^a
Co	Air abrasion with 30 μm SiO_x	23.6 (± 1.7) ^b
D1	Air abrasion with 1–3 μm synthetic diamond particles	22.7 (± 2.0) ^b
K	Air abrasion with 110 μm Al_2O_3	30.5 (± 1.3) ^c
D2	Air abrasion with 30–50 μm synthetic diamond particles	31.0 (± 2.1) ^c
CBN	Air abrasion with 60–80 μm CBN	29.7 (± 2.2) ^c

*Values indicated with the same letter did not differ significantly in the Tamhane test ($P > 0.05$).

of Group Co, D1 and K specimens, 40% of Group D2 specimens and 50% of Group CBN specimens (Table III).

SEM photomicrographs of air abrasion particles are presented in Figure 1. Synthetic diamond and CBN particles were geometric and angular in shape, whereas Al_2O_3 and SiO_x particles were sharper, amorphous or triangular. SEM images of treated zirconia core surfaces are presented in Figure 2. The topographic patterns differed between the specimens air-abraded with Al_2O_3 , SiO_x , synthetic diamond and CBN particles. The SEM images showed that the air abrasion procedure modified the surface topography of the zirconia core by increasing the irregularities on the surface. Air abrasion with 30–50 μm synthetic diamond, 60–80 μm CBN and 110- μm Al_2O_3 particles created a more irregular and rougher surface than other particles when compared with the control surface.

Discussion

The data support rejection of the hypothesis that shear bond strengths after air abrasion with different particles varying in size and form would not be different. Air abrasion with different materials with different particle sizes and forms affected the shear bond strength ($P < 0.05$).

Independent variables that will affect the results of air abrasion include particle size, air abrasion pressure, particle shape, the incidence angle of the particles and wet versus dry particles [3]. In contrast to a previous study [26], which claimed that increasing the particle size increased the bond strength, in the present study air abrasion with larger particles (110 μm Al_2O_3) did not result in significant differences when compared with small particles (30–50 μm synthetic diamond and 60–80 μm CBN). Also, Phark et al. [21] reported that the particle size (50 or 110 μm Al_2O_3) did not result in significant differences with respect to shear bond strength values before and after artificial

aging [21]. However, Sen et al. [13] reported that air abrasion of glass-infiltrated porous aluminum oxide core (In-Ceram Alumina) surfaces with 1–3 μm synthetic diamond particles created higher surface roughness and bond strength values than 50 μm Al_2O_3 particles. In the present study, the mean bond strength value (22.7 ± 2 MPa) on zirconia core after air abrasion with 1–3 μm synthetic diamond particles was similar to that obtained in the study of Sen et al. (21.6 ± 3.6 MPa).

In previous studies, shear bond strength values after air abrasion with 110 μm Al_2O_3 particles ranged between 16–50 [8,9,19,21] and 15–27 MPa [2,9,19] after silica coating with 30 μm SiO_x .

It has been stated that the use of larger-sized abrasive particles causes more intensive surface abrasion, because the wear of the surface increases in proportion to the square of the diameter of the abrasive particle [27]. Tsuo et al. [28] showed that increasing the alumina particle size (50, 75, 100, 150 μm) resulted in an increase of surface roughness but did not affect the shear bond strength of resin cement to zirconium oxide ceramic before thermocycling. After 5000 thermocycles, the shear bond strength decreased significantly regardless of the mean particle size of sandblasting [28]. Although the particle sizes of synthetic diamond (30–50 μm) and CBN (60–80 μm) were small, higher bond strength values were obtained than with air abrasion with 110 μm Al_2O_3 particles. In addition, similar bond strength values were obtained with 1–3 μm synthetic diamond abrasives and 30 μm silica-coated Al_2O_3 particles. This result may be due to the hardness and particle shape of diamond and CBN particles. The hardness of Al_2O_3 particles was lower than that of synthetic diamond abrasives and CBN. According to Mohs' scale, the hardnesses of Al_2O_3 , CBN and synthetic diamond abrasive are 9, 9.9 and 10, respectively [24]. Zirconium oxide is a more ductile material than these materials and its surface hardness is lower (Mohs hardness 8.5) [24,27]. Harder particles affected the shear bond strength of the resin-luting cement and the surface topography of zirconium oxide core more than 110 μm Al_2O_3 particles.

In a previous study [2], a silica-coating technique with 30 μm SiO_x increased the bond strength of the resin cement to glass-infiltrated zirconia-reinforced ceramic (In-Ceram Zirconia) (21.6 ± 3.6 MPa) when compared to air abrasion with 125 μm Al_2O_3 (15.7 ± 2.9 MPa). In contrast to the present study, In-Ceram Zirconia surfaces were air-abraded with 125 μm Al_2O_3 particles before air abrasion with 30 μm SiO_x . It was thought that the additional micro-mechanical retention after this procedure might have increased the shear bond strength. Also, the presence of the glassy phase in oxide ceramics favors better siloxane bonds [15,16,19]. Kern & Thompson [16] found long-term durable bond strengths with silica/

Table III. Failure types.

Group	Type of failure (%)		
	AD	CO	MI
C	100	–	
Co	60	–	40
D1	60	–	40
K	60	–	40
D2	40	–	60
CBN	50	–	50

AD = Adhesive failure; CO = cohesive failure; MI = mixed fracture at the ceramic or composite resin surface.

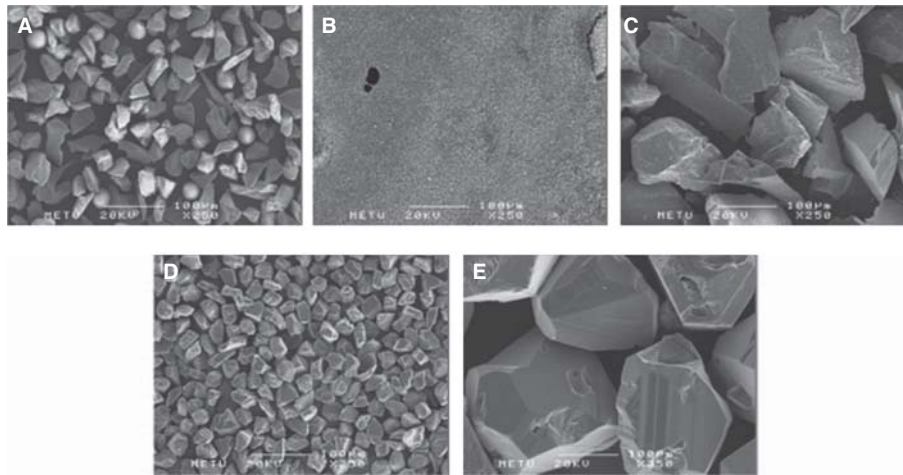


Figure 1. SEM images of the air abrasion particles: (A) 30 μm SiO_x ; (B) 1–3 μm synthetic diamond particles; (C) 110 μm Al_2O_3 ; (D) 30–50 μm synthetic diamond particles; (E) 60–80 μm CBN.

silane coating to glass-infiltrated alumina but not to zirconium oxide [8,29,30]. Blatz et al. [22] reported that this may be due to the industrial zirconium oxide used in those studies; this may differ from commercial zirconium oxide systems in terms of physical properties and surface hardness as well as morphology [22].

Although some studies have demonstrated good results with tribochemical treatment [2,7,22], for denser alumina or zirconium oxide ceramic surfaces silica coating seems to be less suitable, resulting in reduced bond strength [1,8,15,29,31]. This result may be related to the fact that the silica coverage originating from the coating particles appeared, to a large extent, not to be really firmly attached to the hard zirconium oxide surface compared with systems with a glassy structure. Therefore, the weakly attached silica layer seems to be the weak link when bonding to silica-coated densely sintered oxide ceramics [1,31]. More recent studies [2,9,19] showed that a high-strength, reliable resin bond to alumina

and zirconium oxide ceramics was achieved with silica coating (Rocatec, CoJet) and by using an adhesive phosphate monomer containing a silane-coupling agent and an adhesive phosphate monomer-containing resin composite cement (Panavia EX, Panavia F2.0, RelyX UniCem).

Another variable which was not investigated in the present study was the operating air pressure used in air abrasion [27]. In previous studies, operating pressures ranging from 2.5 to 10 bar were used for air abrasion with Al_2O_3 , and pressures of 1.5–4.5 bar were used for silica-coating procedures [2,4,7–9,15,19,21,27,30–34]. In several studies on zirconium oxide ceramic bonding, air abrasion with 50–110 μm alumina particles at 2.5 bar was used to condition the ceramic surface in order to increase the surface roughness, as well as to clean and activate the surface [8,14,15,18,19,22,29,30]. Reducing the pressure during air abrasion might be recommended to reduce the negative influence of high-pressure air

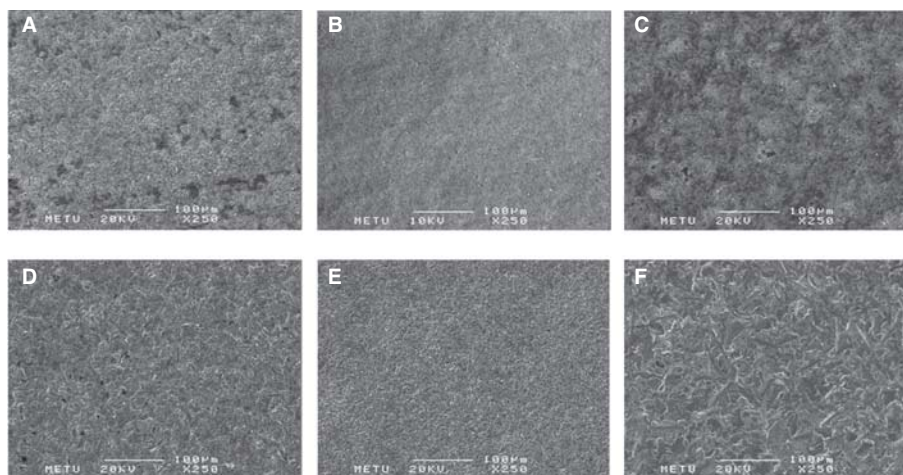


Figure 2. SEM images of the zirconia core surface after different surface pretreatments: (A) control; (B) air abrasion with 30 μm SiO_x ; (C) air abrasion with 1–3 μm synthetic diamond particles; (D) air abrasion with 110 μm Al_2O_3 ; (E) air abrasion with 30–50 μm synthetic diamond particles; (F) air abrasion with 60–80 μm CBN.

abrasion on the mechanical properties of zirconium oxide ceramic.

Yang et al. [20] concluded that using a self-adhesive luting resin composite, air abrasion at 2.5 bar or the combination of low-pressure air abrasion (0.5 bar) and priming with MDP-containing primers seems to be useful to achieve durable long-term bonding to zirconium oxide ceramic. The results of numerous laboratory studies with glass-infiltrated alumina ceramic and also with densely sintered alumina or zirconium oxide ceramics indicate that when a low air-abrasion pressure is used, primers containing MDP monomer are necessary for durable zirconia-ceramic resin bonding [1,8,16,22,23,29,35].

Other techniques for the superficial treatment of zirconium oxide ceramics have been described; these include plasma spraying with hexamethyldisiloxane, fusing glass pearls to the zirconium oxide surface, new patented modified ceramic surfaces (NobelBond; Nobel Biocare, Göteborg, Sweden) and Nd:YAG laser treatment [17,36–38]. These treatments improved the bond strength of resin cements to the zirconium oxide surface [36–38]. However, with the exception of laser treatment, the other methods have to be used during the manufacturing process, which makes them less suitable for daily practice. In particular, an intraoral test of a dental restoration is regularly needed in clinical dentistry [1].

Air abrasion was found to be an efficient way of including a tetragonal to monoclinic (T-to-M) phase transformation on the Y-TZP surface and, therefore, increasing the flexural strength of the ceramic [6,11]. In a previous study, Qeblawi et al. [11] stated that air abrasion with 50 μm Al_2O_3 and wet hand-grinding showed higher flexural strength than silica-coating and controls. Also, the smaller particle size may have been less effective at inducing the T-to-M phase transformation on the surface of the Y-TZP [11]. In the present study, the effects of the size and type of the sandblasting particles on the flexural strength of the zirconium oxide core were not evaluated: this is one of the limitations of the study.

The other limitation of our study may be the artificial aging method. Some studies of the resin bond to zirconium oxide apply long-term water storage (150–180 days) and high numbers of thermal cycles (10 000–37 500) to ultimately test the durability of the bonds [8,20–22,29,34,39,40]. The ISO TR 11450 standard (1994) indicates that 500 cycles in water at 5–55°C is an appropriate aging regimen [11]. According to previous studies [6,15,11,41], the number of cycles in this study was arbitrarily set at 6000. The thermocycling parameters were chosen according to the estimation that 6000 \times 5–55°C thermocycles represent the situation during 5 years in the oral cavity [41]. Thermocycling with 6000 cycles was well

above the recommended cycle number according to the ISO [15].

The results of this study were based on the use of a single cement and cannot be generalized to other resin cements. Surface roughness and volume loss from the zirconium oxide core surfaces were not investigated in the present study. Atomic force microscopy might be used in a further study to investigate the changes on the surfaces. Despite the possible negative outcomes of surface treatments on the mechanical properties of zirconium oxide materials, the application of resin cements to untreated surfaces apparently results in low bond strength, which is unable to resist water storage. Additionally, in a long-term clinical study with alumina and zirconia FPDs [42], the authors noted that fractures only occurred at untreated sites, never at air-abraded surfaces. Although several scientific studies are currently available, clinical studies are necessary to evaluate the long-term behavior of zirconium oxide restorations and to establish which materials and techniques should be recommended for luting these restorations.

Within the limitations of this study, the following conclusions can be drawn. Air abrasion with different particles produced significantly different ($P < 0.001$) shear bond strength values. The highest bond strength values were obtained with application of 30–50 μm synthetic diamond particles. Further evaluation of the effect of alternative air abrasion particles on bond durability is required before making any clinical recommendations.

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