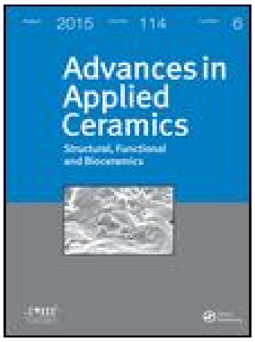

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Influence of different surface treatments on zirconia/resin shear bond strength using one-bottle universal adhesive

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ABSTRACT
This study was designed to evaluate the effect of different treatments on the zirconia/resin shear bond strength (SBS) using commercial one-bottle universal adhesive. Zirconia discs with different surface treatments (blank control; airborne-particle-abrasion; glazing) were bonded to the bovine enamel surfaces using one-bottle universal adhesive. All specimens were tested for SBS (MPa) before and after 10000 thermocycles. Statistically analysis were conducted by using one-way analysis of variance and multiple-comparison least significant difference tests (α = 0.05). Airborne-particle-abrasion group showed higher SBS (36.19 ± 11.86) than control group (14.98 ± 5.90) and glazing group (10.63 ± 5.39) ($p < 0.05$). After thermocycling test, the SBS significantly decreased for control group (8.84 ± 2.55) and glazing group (6.18 ± 2.78) while not for airborne-particle-abrasion group (41.5 ± 7.95). One-bottle universal adhesives combined with airborne-particle-abrasion showed quite high SBS of zirconia/resin, which was appropriate for bonding of zirconia restoration.

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KEYWORDS
Zirconia; adhesion; shear bond strength; surface treatments; one-bottle universal adhesive

Introduction

Zirconia is a metal oxide that was identified as a reaction product of heating the gem, zircon, by the German chemist Martin Heinrich Klaproth in 1789 [1]. Three crystalline forms exist in nature: monoclinic at low temperatures, tetragonal above 1170°C and cubic above 2370°C [2]. Over the last 10–15 years, yttria-partially stabilised tetragonal zirconia (YTZ) has been widely used in prosthetic dentistry, for example crowns and bridges fixed to implant abutments and prepared natural teeth [3], due to its high mechanical strength, stable chemical properties, nice esthetic appearance and long-lasting biocompatibility [4,5].

Long-term survival of YTZ restorations still relies on effective bonding [6]. In some instances, conventional cementation with a glass ionomer, which depends only on micromechanical retention, is used for high strength ceramic restorations. However, resin bonding is desirable which would lead to less micro-leakage, enhanced long-term fracture and fatigue resistance in oral environment [7]. Strong ceramic/resin bonding relies on micromechanical interlock and adhesive chemical bonding, requiring surface roughening for mechanical bonding and surface activation for chemical adhesion [2,8,9].

Compared to silica-based ceramics, which can be bonded after hydrofluoric acid etching and silanization, densely sintered zirconia has surface structures free of the glass phase [8]. This makes zirconia not easily etched or chemically functionalised using conventional treatments, and requiring very aggressive mechanical abrasion methods to increase surface roughness. Therefore, in order to achieve acceptable cementation, alternative attachment methods are required for zirconia ceramics. There are several methods used for surface roughening: grinding using abrasive paper (SiC or Al_2O_3), airborne particle abrasion using $Al_2 O_3$ particles ranging in size from 50 to 250 μm [4,10], grinding using a diamond bur, hot etching [11] and selective infiltration etching [12] and nano-structured alumina coating [13] and so on. Among these, airborne-particle abrasion has been commonly used to increase the surface roughness, while the Al_2O_3 particle size, blasting pressure [14] and blasting time have been discussed in studies [15]. There is still no consistent conclusion about the benefits or potential adverse effects of airborne particle abrasion procedure.

The non-silica composition of zirconia makes it difficult to bond zirconia to tooth structures using traditional resin composite cements. Silica-coating techniques have been explored to utilise the chemical bonding between zirconia and resin provided by silanization, for example, tribochemical silica coating with the CoJet and Rocatec systems [16] and using plasma spray technique to deposit siloxane coating on ZrO_2 [17]. Another method is to fuse a thin layer of glass

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to zirconia, which makes zirconia with high acid resistance due to the absence of a glassy matrix could be etched by HF, and then silanization can be applied [18].

In recent years, zirconia primer containing functional phosphate monomer, such as 10-methacryloyloxydecyl-dihydrogen-phosphate (MDP), has been reported to attain P–O–Zr covalent bonds, which could improve wettability and then increase the shear bond strength (SBS) of zirconia/resin [4]. Surface roughening along with chemical adhesion is considered to be more effective in increasing bonding strength and improving the bond durability of zirconia/resin bonding interface [2,4,19].

With the development of adhesives in present, one-bottle universal adhesives have been developed to bond with almost all indirect restoration materials, including resin composites, zirconia-based and alumina-based ceramics, silica-based glass ceramics, alloys, enamel and dentin, which could simplify adhesion procedures [4,20]. Manufacturers claim that components such as MDP enable bonding to zirconia without the use of additional primers [4]. Studies [4,21] showed that application of one-bottle universal adhesives alone provided higher bonding strength to zirconia than the application of zirconia primers alone. Pitta J [22] reported that the universal adhesive group showed higher SBS, which was not affected by saliva contamination.

Scotchbond Universal Adhesive (3M ESPE, St. Paul, MN, USA) is a one-bottle solution that combined primer and adhesive, which can be used in all etching techniques, including total-etch, self-etch and selective-etch. It can be used for direct and indirect restorations (zirconia, alumina, glass ceramics and metals) without any extra primer because of the components of MDP and silane. The additional etching of enamel and dentin with Scotchbond Universal Etchant (3M ESPE), which is 32% by weight phosphoric acid etching gel, increases the adhesive strength. RelyX Ultimate (3M ESPE) is an adhesive dual-curing resin cement, which is used in combination with Scotchbond Universal Adhesive for the adhesive cementation of indirect restorations.

The aim of this in-vitro study was to evaluate the zirconia/resin bond strength after glazing and air-borne-particle-abrasion treatment using Scotchbond Universal Adhesive. The null hypothesis was that different zirconia surface treatments have no effect on improvement of bond strength and durability when one-bottle universal adhesives are used for bonding.

Materials and method

Preparation of zirconia samples

Zirconia was pre-sintered using YTZ powder (TZ-3YSB-E, Tosoh, Tokyo, Japan) at 1050°C. Ninety bar specimens (42 mm × 4 mm × 3 mm), 66 disc specimens (thickness: 4 mm; diameter: 4 mm) and 15 cubic specimens ($a = 10$ mm) were milled (Figure 1) and final sintered at 1450°C. Each specimen was polished with grit #180, #240, #600, #800, #1000, #1200 and #2000 SiC abrasive paper (NOON, Shanghai, China) consecutively under water-cooling, then cleaned ultrasonically in a deionised water bath for 10 min and gently air-dried.

Specimens were divided into three groups according to different surface treatments performed (bar specimens: $n = 30$; disc specimens: $n = 22$; cubic specimens: $n = 5$):

Group 1. Blank control

Group 2. Airborne particle abrasion with 50 μm Al_2O_3 particles applied for 10 s at 0.4 Mpa, 10 mm away from the spray mouth

Group 3. A layer of glazing paste (Upcera, Shenzhen, China) applied to the surface at 830°C for 8 min in a furnace (3G, LIRR, Luoyang, China), then 9.6% hydrofluoric acid (Pulpdent, Watertown, MA, USA) applied for 60 s, then rinsed and dried

Scanning Electron Microscopy (SEM) evaluation

In this test, two representative disc specimens were randomly selected from each group. The selected zirconia discs were rinsed with 95% ethanol and air-dried, mounted on metallic stubs, gold sputter coated for 80 s (E-104S, Hitachi, Tokyo, Japan), then evaluated under an SEM (S-4800, Hitachi, Tokyo, Japan) to assess the possible topography differences of the treated zirconia surfaces.

Flexural strength testing

Each bar specimen was oriented in a holding jig with the modified surface subjected to tensile forces. The bars were loaded to failure using a steel knife edge rounded to a radius of 0.8 mm, in a 3-point apparatus in a universal testing machine (Instron, MA, USA) with at 0.5 mm/min speed. The breaking loads were recorded, and flexural strengths were calculated using the following formula:

$$\sigma_f = \frac{3FL}{2bd^2}$$

F: breaking load
L: test span in millimetre
b: width of specimen
d: thickness of specimen

Wettability test

The hydrophilicity of the treated zirconia was examined using the sessile drop method by dropping 2 μL

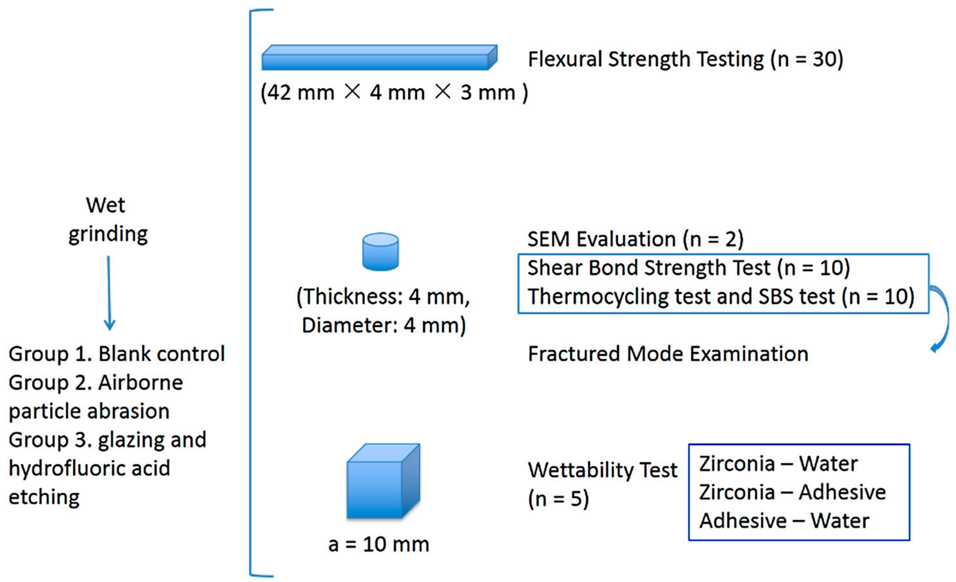

Figure 1. Flow chart of sample preparation and grouping.

deionised water on the cubic specimens. The surface wettability of zirconia to resin was also examined by replacing water with the Scotchbond Universal Adhesive. Instant photography was performed with a CCD camera to capture images of the droplet. The static contact angles between the liquid drop and zirconia were measured by the angular method. Then all specimens were cleaned ultrasonically in 95% ethanol for 5 min and gently air-dried.

Scotchbond Universal Adhesive was then applied to the zirconia cubes surfaces with a brush for 20 s, light-cured for 10 s after air-blowed to form an adhesive film. The static contact angles were also measured immediately by dropping deionised water.

SBS test and fractured mode examination

Bovine enamel free of caries and cracks was sectioned to 6 mm × 6 mm using diamond burrs (TR11, Mani, Tochigi, Japan) and stored in 0.5% chloramine solution at 4°C. The enamel was embedded in self-curing resin in a Teflon mold to form a cylindrical specimen (diameter: 20 mm; length: 40 mm). After solidification of the self-curing resin, the enamel was polished with SiC abrasive paper (step by step from grit #180, #240, #360, #600, #1000 to #2000) under flowing water. The specimens were discarded when the area of the exposed enamel was less than 6 mm × 6 mm.

The treated surfaces of the zirconia discs were cemented to the enamel surfaces with the bond system according to the manufacturer's instructions. First, the enamel surfaces were etched with Scotchbond Universal Etchant (3M ESPE) for 15 s, rinsed and dried. Next, the Scotchbond Universal adhesive was applied to the zirconia discs and enamel surfaces with a brush for 20 s, and air-blowed for 5 s. Then, the zirconia specimens were cemented on the surface of the enamels using RelyX Ultimate adhesive resin cement and compressed at a constant load of 20 N for 20 s, and light-cured for 20 s after excess cement wiped off.

All of the specimens were stored in distilled water for 24 h after bonding step. Half of the specimens ($n = 10$) were subjected to an SBS test after 1-day storage, and half were tested after thermocycling. The thermocycling test consisted of 10,000 thermocycles alternately in water at 5 and 55°C, each with a dwelling time of 30 s.

The SBS test was performed according to ISO11405 (2003) using universal testing machine (Instron). The shear force was applied parallel to the interface of the bonding surfaces at a speed of 0.5 mm/min until bonding failed. The shear force was recorded automatically at the point of failure. The SBS was calculated as follows:

$$\text{SBS}\ \ (\text{MPa}) = \text{load}\ (\text{N})/\text{area}\ \ (\text{mm}^2).$$

The fracture modes were determined with stereomicroscope (Olympus, Tokyo, Japan) at 10× magnification as: ZR-failure at zirconia/resin interface; ER-failure at enamel/resin interface; MM-mixed mode and CF-cohesive fracture inside the resin cement.

The main materials, their manufacturers and compositions are listed in Table 1.

Statistical analysis

One-way ANOVA in combined with least significant difference (LSD) tests performed by the SPSS (version 20.0, IBM SPSS, Chicago, IL, USA) were used to analyse difference in flexural strength, contact angles and SBS among groups ($\alpha = 0.05$), after normal distribution and homogeneity of variance were checked

Table 1. The main materials, their manufacturers and compositions.

Material	Manufacture	Main composition
Zirconia	TOSOH	Zirconium dioxide, Yttrium trioxide, Hafnium dioxide
Scotchbond Universal Adhesive	3M ESPE	MDP, dimethacrylate resins, HEMA, ethanol, water, silane treated silica, 2-propenoic acid, copolymer of acrylic and itaconic acid, initiators, silane
RelyX Ultimate	3M ESPE	Silane treated glass powder, 2-propenoic acid, 2-methyl, 1,1′- [1-(hydroxymethyl) 1,2-ethanediyl] ester, reaction products with 2-hydroxy-1,3-propanediyl dimethacrylate and phosphorus oxide, TEGDEMA, silane treated silica, oxide glass chemicals, sodium persulfate, tert-butyl peroxy-3,5,5-trimethylhexanoate, copper (II) acetate monohydrate, substituted dimethacrylate, 1,12-dodecane dimethacrylate, silane treated silica, -benzyl-5-phenyl-barbic-acid, calcium salt, sodium p-toluenesulfinate, 2-propenoic acid, 2-methyl-, [(3 methoxypropyl)imino] di-2,1-ethanediyl ester, calcium hydroxide, titanium dioxide
Scotchbond Universal Etchant	3M ESPE	32% (wt%) phosphoric acid
Glazing paste	Upcera	Silicon dioxide
HF	Pulpdent	9.6% hydrofluoric acid

Note: According to the manufacturers' instruction and Passia N's introduction [20].

using the Kolmogorov–Smirnov test and Levene tests, respectively.

Results

SEM images of each group are shown in Figure 2. The SEM images showed that the polished zirconia had a relative smooth surface with scratches caused by the wet grinding. After airborne-particle-abrasion treatment, the zirconia surfaces were rough with irregularly distributed grooves. The glazing group showed a relative uniform glazing surface with some entrapped air bubble inclusions.

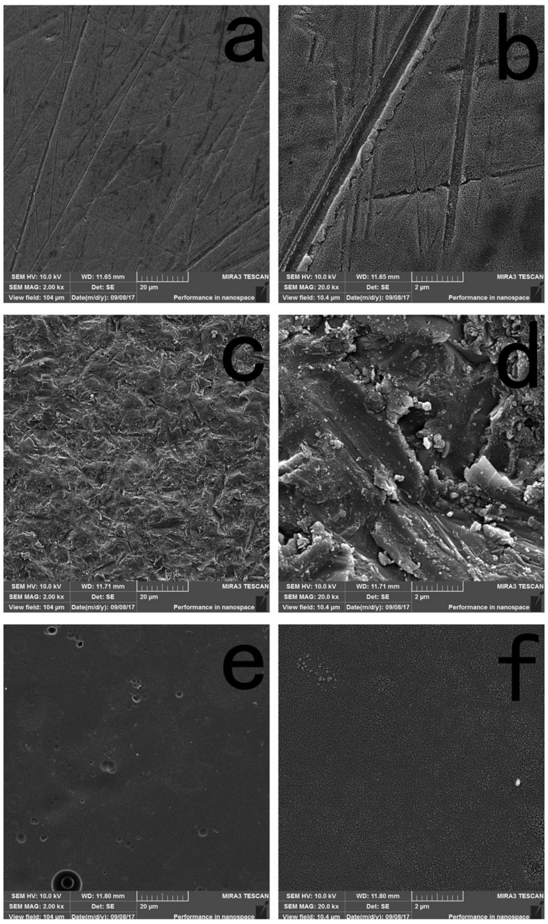

Figure 2. SEM photomicrographs of zirconia ceramic discs after different surface treatments. (a,b) Blank control group; (c, d) Airborne-particle-abrasion group; (e, f) Glazing group.

The mean values and standard deviations (SD) of the average flexural strength of different groups are listed in Table 2.

The images of wettability test are shown in Figure 3. The analysis of contact angles is listed in Table 3. The contact angles of zirconia after airborne-particle-abrasion treatment were similar to the control group both for zirconia–water and zirconia–adhesive, while glazing group showed lower contact angles for water and no difference for adhesive. After applying a layer of adhesive, the contact angles significantly decreased for each group.

The SBS of each group is listed in Table 4. The airborne-particle-abrasion group had significantly higher SBS than the control group and glazing group not only in the immediate test but also after thermocycling ($p < .01$). The bond strengths were significantly decreased after the thermocycling test in control ($p = .01$) and glazing ($p = .043$) groups, while no significant difference was detected in airborne-particle-abrasion group ($p = .257$).

The calculation of the different failure modes of each group is listed in Table 5. CF (cohesive fracture) in the resin cement and ER (failure at enamel/resin interface) were never observed and most of the failure modes were ZR (failure at zirconia/resin interface).

Discussion

Naichuan Su [7] assumed that larger volume of alumina powder led to lower loss of the material on the abraded surface. On the contrary, other studies [23] demonstrated that larger alumina particles would cause larger damage based on substantial particle abrasion damage that was 4 μm deep in the zirconia. In this study, fine alumina particle and mild condition were adopted. SEM images showed a rough

Table 2. Flexural strength (MPa) of zirconia with different surface treatments.

Surface treatment	Mean	SD
Control group	1255.66	286.62
Airborne particle abrasion[a]	1552.75	205.37
Glazing	1167.34	312.02

[a]indicated significant differences ($p < .05$).

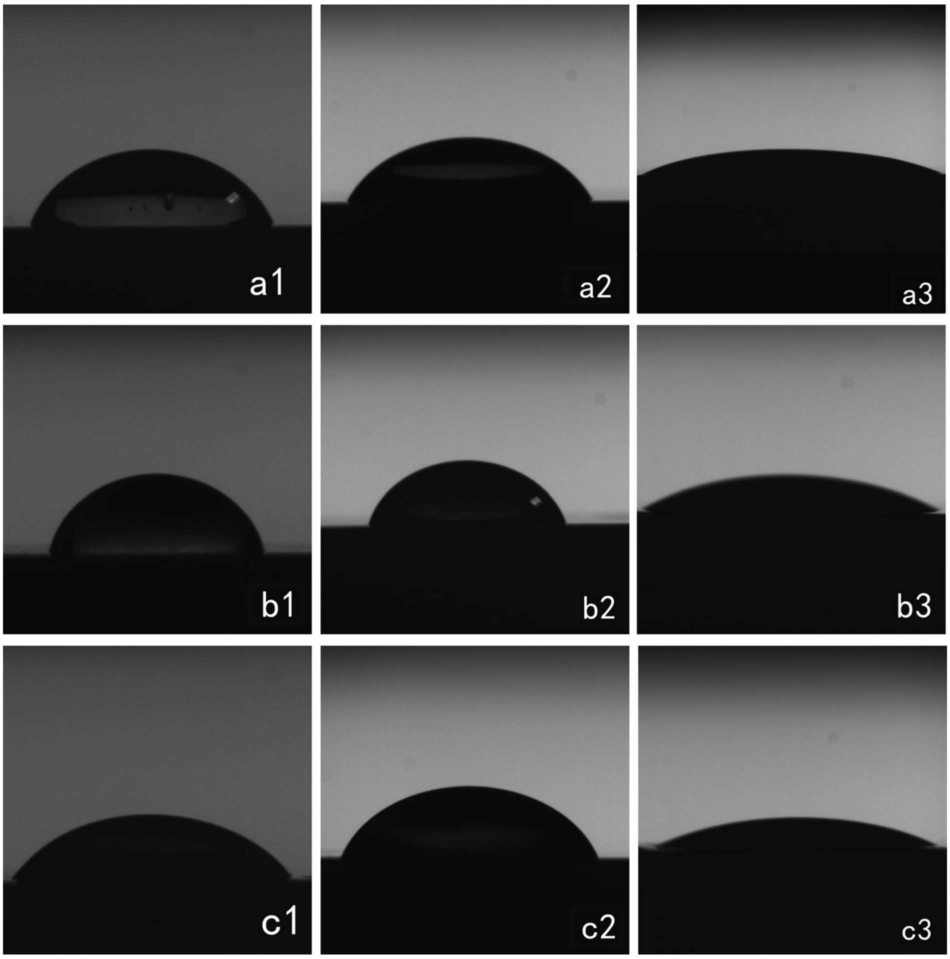

Figure 3. Images of contact angle test of different surface treatments (a. Control group; b. Airborne-particle-abrasion group; c. Glazing group; 1. Zirconia–water; 2. Zirconia–adhesive; 3. Adhesive–water).

surface with irregular grooves distributed, and no obvious cracks were observed. For glazing group, SEM images showed relative smooth and uniform surfaces with some entrapped air bubble inclusions at low magnification.

The airborne-particle-abrasion group showed higher flexural strength, which was consistence with the conclusions of most studies [15]. Airborne-particle abrasion protocols applied to zirconia surfaces have been shown to induce protective compressive residual stresses from the t–m phase transformation, thereby initially increasing the flexural strength [24]. Although, small cracks, flaws and defects may exist within the transformation layer, they were probably healed by the 4% volume increase in the grains during the phase transformation to resist fracture. With the progression of the monoclinic phase transformation from the surface to the bulk of the zirconia specimen, microcracks and tensile residual stresses may develop and decrease the flexural strength; however, it seems that the depth reached by the transformed layer was not great enough to allow microcracks to reduce the flexural strength [15]. Airborne-particle-abrasion treatment would remove a significant amount of

Table 3. Contact angles (°) analysis of different groups (Mean ± SD).

Surface treatment	Zirconia–water	Zirconia–adhesive	Adhesive–water
No treatment	$60.8 \pm 3.85^{b\ A}$	58.9 ± 3.85^{B}	22.0 ± 2.21^{B}
Airborne particle abrasion	$61.6 \pm 8.33^{b\ A}$	62.5 ± 6.68^{B}	19.2 ± 5.39^{B}
Glazing	$46.9 \pm 5.08^{a\ A}$	56.3 ± 5.84^{B}	16.7 ± 4.72^{C}

^{a,b} indicated significant differences within a column ($p < .05$).
^{A,B,C}indicated significant differences within a row ($p < .05$).

Table 4. The SBS (MPa) of zirconia with different surface treatments (Mean ± SD).

Surface treatment	Immediate SBS	SBS after Thermocycling
No treatment	$14.98 \pm 5.90^{b,A}$	$8.84 \pm 2.55^{b,B}$
Airborne particle abrasion	36.19 ± 11.86^{a}	41.50 ± 7.95^{a}
Glazing	$10.63 \pm 5.39^{b,A}$	$6.18 \pm 2.78^{b,B}$

^{a,b}indicated significant differences within a column ($p < .05$).
^{A,B}indicated significant differences within a row ($p < .05$).

Table 5. The failure modes count of each group before and after thermocycling test.

	ZR		MM	
Surface treatment	Before	After	Before	After
No treatment	8	9	2	1
Airborne particle abrasion	6	7	4	3
Glazing	8	9	2	1

Note: ZR-failure at zirconia/resin interface; MM-mixed mode.

material from restorations [25], which could affect clinical adaptation. Hence, airborne-particle-abrasion treatment should avoid the fragile margins [7]. The flexural strength of glazing group showed no significant difference with control group in this study. In previous study, flexural strength was found to be increased after glazing compared to the control group according to Ketaki JC's [26] results. However, Lai X [27] reported that glazing would impair the strength for the super-translucent dental zirconia. Oblak's [28] study also showed that for glazed zirconia bridges the fracture might originate from the outer surface of the glaze and a clear relationship was found between the number and size of pores in the glaze layer and the fracture load.

The observed failure mode was mainly at zirconia/resin adhesive interface and no cohesive failure was observed, which was consistent with previous studies [7,22]. This phenomenon indicated that sufficient bonding existed between bovine enamel and adhesive resin cement by universal adhesive. At the same time, when the SBS reached a relatively high level, the percentage of mixed mode increased. Only adhesive failure at the interface occurred when the bonding strength was at a relatively low level, which was consistent with Naichuan Su's [7] results. It could be concluded that the reliability of zirconia restorations mainly depended on the zirconia/resin interface.

Good wettability is crucial for obtaining adhesion of restorations with resins [3]. Previous studies pointed that high surface energy and wettability were normally achieved by airborne-particle-abrasion treatment of zirconia [7,29], and silanization improved the wetting of the zirconia surface which facilitated the adhesion between sandblasted zirconia surfaces and resin cement [3]. Chuang SF's [21] report also showed that both the water and adhesive contact angles were the highest on the untreated zirconia, and decreased on primer treated groups, that MDP treated groups presented super hydrophilicity (contact angle < 10°). Otherwise, in this study, the wettability of zirconia after airborne-particle-abrasion treatment was similar to the control group, while after applying a layer of adhesive, the contact angles significantly decreased which may facilitate the bonding of resin cement. The ToF-SIMS analysis [4,21] confirmed that the MDP application attained P–O–Zr bonds, which changed the wettability of zirconia to water and resin monomer, and improved resin–zirconia bond durability. These results reminded that the adhesive should be evenly spread to the adhesive surfaces especially the marginal areas, and shouldn't simply depend on the air spray blowing. The use of one-bottle universal adhesive could facilitate the spread of resin cements.

Haifeng Xie [4] reported that one-bottle universal adhesives combined with alumina sandblasting showed comparable SBS but more susceptible to hydrolysis when compared to tribochemical silica coating with silanization. However, the given SBS result of each group in that study was no more than 14 MPa. Barragan G [30] also reported that zirconia surface treated with airborne-particle-abrasion combined Z-Prime Plus (Bisco, Schaumburg, Illinois, USA) showed a statistically significant increase in bond strength values (23.2 ± 4.1) when compared to the untreated surface. However, the SBS of the airborne-particle-abrasion group in those studies were much lower than the present study, which was probably due to the mixed failure mode that the SBS lowered because of the fracture of resin cement. The failure in the present study mainly happened at zirconia/resin interface, which also indicated a success bonding between resin and enamel. Sari F [18] found that regardless of the application of sandblasting, glaze layer and HF application followed by silanization provided higher bond strength between composite and monolithic zirconia than the untreated and sandblasted monolithic zirconia followed by zirconia primer. However, Moradabadi A [31] reported that airborne particle abrasion obtained highest SBS, while the mean SBS were significantly decreased by applying a glaze layer on zirconia surfaces but could be improved after etching process. In this study, zirconia surfaces treated with airborne-particle-abrasion showed significant higher SBS than untreated group, which was consistence with most of the studies [4,18,30]. However, the SBS was not improved for glazing group followed by HF etching combined with universal adhesive. More studies need to be done to explore the influence of glazing on zirconia/resin adhesion, which might be crucial for the adhesion of orthodontic bracket on zirconia restorations.

The latest generation of universal adhesives, containing phosphate monomer MDP, made bonding of zirconia without prior use of zirconia primers possible, which significantly simplify the clinical operating steps. Llerena-Icochea AE [32] confirmed that the commercially available universal adhesives indicated for bonding to zirconia showed the highest bonding values, while the concentration of MDP on adhesives was not significant. The existence of MDP offered chemical adhesion between zirconia and resin cement, while surface roughing was also crucial for bonding. However, Passia N [20] found that the application of RelyX Ultimate resin cement alone could improve the zirconia/resin tensile bond strength (TBS) before and after aging, while the universal primer Scotchbond Universal did not increase TBS significantly. Despite the results, the manufacturer did not recommend the use of RelyX Ultimate without Scotchbond Universal as a primer to keep the procedure consistent with the procedure needed for other restoration materials. Even though the use of adhesive might not improve the TBS, according to the results of wettability in the present study a layer of adhesive might facilitate the spread of the resin cement. Meanwhile, in this study, bond with Scotchbond Universal and RelyX Ultimate after airborne particle abrasion showed nice immediate SBS and thermocyling resistance, while the SBS significantly decreased in control group and glazing and hydrofluoric acid etching group after thermocycling, which might due to the smooth surface of zirconia and lack of mechanical retention.

Conclusions

Within the limitations of this in-vitro study, the following conclusions are drawn:

1. Zirconia surfaces treated with airborne particle abrasion improved the thermocycling resistance of zirconia/resin bonding, based on suitable procedure and avoiding marginal damage.
2. Glazing and hydrofluoric acid etching treatment could not enhance the bonding of zirconia/resin interface, and showed low thermocycling resistance.
3. The application of universal adhesive could improve the wettability of zirconia surface.
4. One-bottle universal adhesives combined with airborne-particle-abrasion treatment of zirconia showed quite high SBS of zirconia/resin and nice thermocycling resistance, while the untreated and glazing group followed by adhesive application showed much lower SBS. Airbone-particle-abrasion treatment followed by universal adhesive was appropriate for bonding of zirconia restoration, which could be easily used in clinical work.

Disclosure statement

No potential conflict of interest was reported by the authors.

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