The effects of airborne abrasion on micro-shear bond strength of resin cements on zirconia

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Objective: To evaluate the effect of various air abrasion treatments on micro-shear bond strength (µSBS) of zirconia bonded with different self-adhesive resin cements.

Methods: Fully-sintered Y-TZP block specimens (IPS e.max® ZirCAD, Ivoclar Vivadent) were prepared to 5 types of surface treatments: no treatment [CON], air abrasion with different particle sizes and pressures: 50 µm & 1 bar [50.1], 50 µm & 3 bar [50.3], 110 µm & 1 bar [110.1], and 110 µm & 3 bar [110.3]. Then, the specimens were divided into two subgroups for further cementation with self-adhesive resin cements (Rely X Unicem Aplicap, 3M ESPE and Panavia SA, Kuraray). These cements were placed into tygon tubes over the zirconia specimens. After water storage at 37°C for 24 hours, micro-shear bond strength (µSBS) was measured. Failure mode was observed under SEM. Statistical analysis was performed by ANOVA and Tukey's test (α = 0.05).

Results: The air abrasion treatment with various particle sizes and pressures affected µSBS (p<0.05). The higher pressure and larger particle size improved zirconia bond strength. Panavia SA performed superiorly in µSBS than Rely X Unicem (p<0.05).

Conclusions: The air abrasion with larger particle size (110 µm) & higher air pressure (3 bar) in combination with MDP-containing resin cement (Panavia SA) could create the optimal adhesion to zirconia.

Key words: air abrasion, micro-shear bond strength, resin cement, surface treatment, zirconia

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Introduction

Nowadays, an all-ceramic restoration becomes available in the dental industry and more popular than metal materials [1]. When all-ceramic prosthesis is selected in patients with high expectation of esthetics in posterior teeth, zirconia might be firstly considered in such case. Due to the limitation of other glass-ceramics with chipping or fractures at high loading areas, zirconia has been developed in this last decade to fulfill needs and replaced these limitations [2]. However, problems have been occurred regarding to the zirconia bonding.

Adhesive failure had been mostly detected at the zirconia-cement interfaces when the zirconia surfaces were not treated properly [3-4]. Unlike the glass ceramics, the reason why debonding tends to occur at zirconia-resin interface is due to the inertness and lacking of the glass particle in zirconia compositions. Therefore, a hydrofluoric treatment followed by silane application does not have any impact on zirconia [5,6,7].

Consequently, many studies have conducted numerous mechanical and chemical treatments to prepare zirconia surfaces for maximizing zirconia bond

strength and maintaining durable bond against the aging processes. Various studies emphasized on the mechanical treatments on zirconia: SIE (selective infiltration etching) [8], Tribochemical silica coating [3], Nd:YAG laser [9], or air abrasion. Also, the chemical retention to zirconia bonding was acquired by the applications of 10-MDP-containing agents [3-4] (10-methacryloyloxydecyl dihydrogenphosphate), silane coupling agents [10], Radio-Frequency plasma spraying [5], alloy primer [10], universal primer (Monobond® Plus) [11], and metal/zirconia primer [12]. Nevertheless, the mechanical retention was most likely strengthen the zirconia adhesion, which played a better role over the chemical retention [13]. One of the most common surface treatments claimed to improve the mechanical retention is the air abrasion with various particle sizes and pressures [10-11]. These variables should be performed thoroughly because an improper altitude such as an inadequate pressure significantly affected zirconia bonding [7,13]. Also, the treatment could yield some defects on the zirconia surface, which could lead to the micro-cracking, inducing the detrimental to bond strength and durability [8]. Therefore, the process should be carefully handled [14]. Still, an air abrasion has been widely chosen to perform due to many advantages: removing contaminations, increasing wetting, reducing surface tension, creating micromechanical interlocking, and providing long-term optimal bond strength to zirconia [10-11].

Zirconia bonding depends on not only the surface treatments, but also the selection of resin luting types [1,4]. Consequently, proper lutings determine the longevity of indirect restorations especially in all-ceramics [15]. Indeed, resin cements are the material of choice for ceramic bonding due to many desired properties required in all-ceramic restorations such as: great marginal sealing, improved fracture toughness, low solubility, low marginal leakage, high compressive strength, great retention, and varieties of colors & translucency, inhibiting crack formation and superior strengthening effect [16-18]. Some studies claimed that both conventional and self-adhesive resin cements performed similarly in term of the bond strengths as long as they both contained suitable compositions that chemically reacted to zirconia

oxide [4,19]. The 10-MDP (10-methacryloyloxydecyl dihydrogenphosphate) component in resin cements offered the chemical bond to zirconia restorations and increased surface wettability and maintained adequate chemical bond strength [8-9,16,20].

Even though, many studies on airborne abrasion treatments have been published as previously mentioned, there is limited knowledge regarding the effects of definite methods for the air abrasion protocols on zirconia surface [21]. Also, there has not been many studies regarding to bonding of IPS e.max ZirCAD (Ivoclar Vivadent, Schaan, Liechtenstein). Therefore, this present study aimed to find the proper air abrasion treatments and selection of self-adhesive resin cements to optimize the adhesion of IPS e.max ZirCAD. The null hypotheses were: (1) there were no significant difference on µSBS on zirconia surfaces luting with different resin cements, and (2) There were no significant differences on µSBS of pretreated zirconia with different particle sizes and air pressures of air abrasion.

Material & Methods

Pre-sintered Y-TZP block with size of 15.4×19×39 mm^3 (IPS e.max® ZirCAD, Ivoclar Vivadent, Schaan, Liechtenstein) were cut into smaller dimensions of 2.5×4.5×15 mm^3 by low speed diamond saw (Isomet, BUEHLER, Illinois, USA). The specimens were ground-finished with a 180 SiC papers on 300 rev/min for 10 seconds under running water by grinding machine (Buehler Metaserv, Buehler Germany) in order to stimulate as-milled surface, then cleaned in ultrasonic cleanser (Q210H, L&R manufacturing, USA) with 95% ethanol for 2 minutes to eliminate the debris particles. Then, the specimen were sintered to its final density in the furnace (Sirona in Fire HTC, Sirona Dental Systems, USA) at 1500°C with the total period of 7 hours.

The zirconia specimens were divided into 5 groups (n=20 in each group) according to the different air abrasion treatments with aluminum oxide particles (Al$_2$O$_3$) (Cobra, Renfert, Hilzingen, Germany). The specimens were perpendiculary and uniformly

sandblasted across the specimens by sandblast machine (Bash M.B.L., Dentalfarm, Torino, Italy) in a circular motion, equipped with the nozzle of 1.2 mm. in diameter, 5-mm distance from the nozzle, for 10 seconds with various particles sizes and pressures: 50 μm & 1 bar [50.1], 50 μm & 3 bar [50.3], 110 μm & 1 bar [110.1], and 110 μm & 3 bar [110.3]. No treatment group [CON] represented as controls, which represented the zirconia surface after milling. After sandblasting, the specimens were cleaned in ultrasonic cleanser (Q210H, L&R manufacturing, USA) with 95% ethanol for 5 minutes, then cleaned with Steam Clean Machine (Evolution steam line, EV4, Silfradent, Italy) for 5 seconds, and dried with the compressed air for 30 second. Surface topography in each group (n=1) was evaluated under Scaning Electron Microscope (SEM: JSM-5410LV, JEOL Ltd.,Tokyo, Japan) images with magnification of 2,000.

Micro-shear bond strength test

All treated specimens were subdivied to 2 subgroups (n=10) as the self-adhesive resin cement used. The cementation were proceeded immediately after the surface treatments to prevent any possible contamination. RelyX™ Unicem Aplicap™ and Panavia™ SA CEMENT (Table 1). were loaded into the plastic tube (Tygon®, Norton Performance Plastic Co, Cleveland, OH, USA) (Ø 0.8 mm, height 1 mm). After that, the specimens were photo-cured by LED light-curing unit, Bluephase® (Ivoclar Vivadent, Schaan Leichtenstein) for 20 seconds with 1200 mW/cm^2 prior to the water storage at 37°C for 24 hours. Voids or gaps were evaluated under Polarize light microscope examination at the magnification of 25 (E400 POL), which was excluded before the bonding test. The interfacial areas of each luting cylinder were measured by the digital vernier caliper (± 0.1 mm^2) (Model CD-15CW, Mitsutoyo Corp., Japan).

The specimens were attached on the testing device using the cyanoacrylate adhesive (Model repair II Blue, Dentsply-Sankin, Japan) on the universal testing machine (Lloyd™ Testing Machine, Model LR 10K, Lloyd Instruments, Fareham Hanth, UK). The wire of 0.2 mm-diameter were looped around luting cylinder, which were aligned to parallel for the accuracy of μSBS. The crosshead speed (1 mm./min) was loaded until failure and recorded.
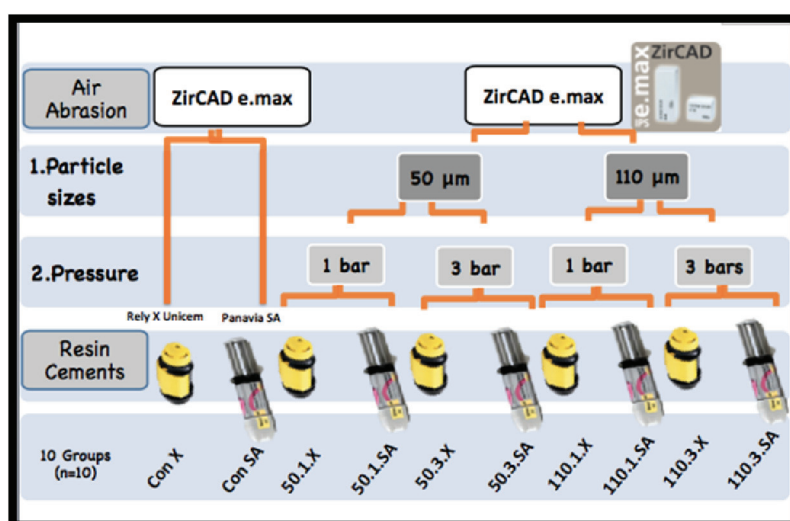

Figure 1. Experimental diagram

Table 1. Materials used in this study

Materials	Composition	Manufacturer	Instructions
IPS e.max ZirCAD Batch # (T42432)	ZrO_2, $Y2O_3$, HfO_2, Al_2O_3	Ivoclar Vivadent; Schaan, Liechtenstein	-
RelyX™ Unicem Aplicap™ (3M ESPE, St. Paul, MN, USA) Batch # (568880)	Powder: Alkaline (basic) fillers, Silanated fillers, Initiator components, Pigments Liquid: Methacrylate monomers containing, phosphoric acid groups (phosphoric ester), Methacrylate monomers, Initiator components, Stabilizers	3M ESPE (St Paul, MN, USA)	Triturated for 10 seconds
Panavia™ SA CEMENT (Kuraray, Osaka, Japan) Batch # (620080)	PASTE A: 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Bisphenol A diglycidylmethacrylate (Bis-GMA), Triethyleneglycol dimethacrylate, Hydrophobic aromatic dimethacrylate, Silanated barium glass filler, Silanated colloidal silica, dl-Camphorquinone, Benzoyl peroxide, Initiators PASTE B:Bisphenol A diglycidylmethacrylate (Bis-GMA), Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Silanated barium glass filler, Silanated colloidal silica, Surface treated sodium fluoride, Accelerators, Pigments	Kuraray Noritake, Osaka, Japan	Equal amounts of pastes were extruded, and then hand-mixed for 10 seconds

Failure mode analysis

The fractured patterns were evaluated under polarized light microscope (E400 POL) at the magnification of 10. Any unclear patterns were sputtered coated with gold-palladium (E-1030, Hitachi, Tokyo, Japan) and re-examined under SEM at the magnification of 90-100x. The fracture patterns were classified as cohesive failure, adhesive failure and mixed failure.

Statistical analysis

The means and standard deviations of µSBS (MPa) were evaluated by Kolmogorov Smirnov test (K-S test) to determine the distribution of the data. Levene's test was then used to test the homogenous of variance. Two-way ANOVA and Tukey's test were used to determine statisticaly differences among the µSBS to examine the effects of particle sizes, pressures on each resin cement (p<0.05). T-test was used to compare the µSBS between self-adhesive resin cement in any surface treatement. For the failure modes, the results were analyzed by chi-square test (p<0.01).

Results

For SEM assessment of surface topography, the irregularities of horizontal-line pattern produced by the SiC paper were shown in the SEM image (Figure. 3a). After the air abrasion with different particle sizes and pressures, the irregularities of polished-line pattern were less visible to air-abraded specimens. Then, some defects were more apparent after the air abrasion treatments (Figure. 3b-f). Although, the high pressure completely eliminated the initial horizontal-line pattern, the high impact of deep grooves also mostly presented as well (Figure. 3c&f). Whereas, the specimens encountered with the lower pressure of 1 bar inadequately eliminated the initial irregularities. These presented rougher surfaces and the horizontal-line patterns were partially visible (Figure. 3b&d).

Figure 2 showed the means and standard deviations of µSBS with two self-adhesive resin cements. All each independent factor (pressure, particle size) had a significant effect on µSBS (p<0.05). Moreover, the interactions between particle size & pressure were significantly affected (p = 0.043).

Obviously, all of the air-abraded zirconia groups were significant higher in the bond strengths than the non-treated one. Panavia SA cement performed significantly higher bond strength than Rely X Unicem cement, regardless of particle sizes or pressure. The highest bond strength was found in the zirconia treated with 3-bar air pressure and 110 µm-sized particle when cemented with Panavia SA (31.001 ±3.5633 Mpa),
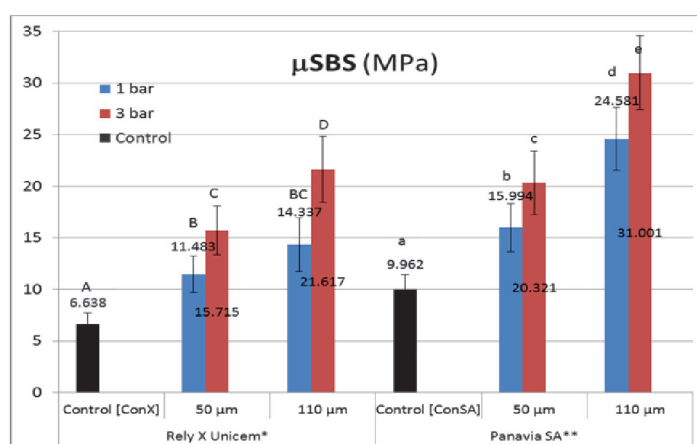
Figure 2. Means µSBS (µm) of cemented zirconia surfaces with different surface treatments (n=10). The different capital letters represented the significant differences within Rely X Unicem groups, while the different small letters represented the significant differences within Panavia SA groups (p<0.05). There was significant difference on µSBS between 2 resin cements* (p<0.0001).
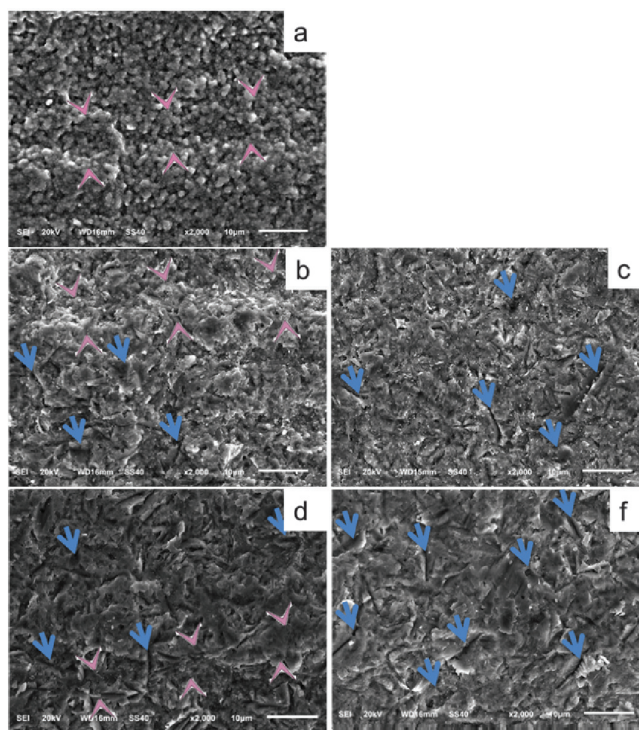
Figure 3. Representative SEM micrographs of zirconia surface with different air abrasion treatment (a) No any surface treatment (control). The zirconia grains are detectable. The horizontal-line pattern created by 180-grit SiC paper was found (purple pointers). (b) 50-μm particle size and 1-bar pressure. The zirconia grains are less obvious after the air abrasion procedure. Grooves (blue arrows) appeared. (c) 50-μm particle size and 3-bar pressure. Grooves are more deepen compared to the surfaces encountered with the lower pressure. (d) 110-μm particle size and 1-bar pressure. The existing horizontal lines and wider ranges of grooves were found. (e) 110-μm particle size and 3-bar pressure. No existing horizontal lines with wider ranges or grooves were found

while the control group in Rely X Unicem performed the lowest bond strength (6.638 ±1.076 MPa).

Considering the particle size, all groups treated with the 110 μm-sized particle produced significantly higher bond strength than the 50 μm-sized particle, regardless of the air pressures and resin cements (p<0.05). Furthermore, the 3-bar air pressure also produced significantly higher bond strength than the 1-bar air pressure, regardless of the particle size and resin cement.

The fracture mode distribution was outlined in Figure 4. All groups revealed significantly different on fracture mode patterns (p<0.05). The most failure mode was the adhesive failure between resin cement and zirconia surface. Those groups cemented with Rely X Unicem (with same particle sizes and pressures) presented higher adhesive failure pattern than those

cemented with Panavia SA. The mixed failure was found more when the pressure increased in both resin cements. However, there were no significant differences in failure mode when the particle size was increased.

Figure 5 showed SEM image (90x-100x) on the right side that represented a fracture specimen of ConX with the clear visual of an adhesive failure pattern. The zirconia surface revealed more than 75% throughout the surface. On the left side, the image of the light microscope (10X) correlated to the SEM image (Figure 4a-b). The representative of cohesive pattern was presented in ConSA specimen. The remnant cement exhibited more than 75% (Figure 4c-d). The other specimen of ConSA was calibrated the two different surfaces, which neither unveiled more than 75% of zirconia nor cement surface (Figure 4e-f). Therefore, it was categorized as the mixed failure pattern.

Discussion

Nowadays, dental zirconia is used extensively in dentistry as it acquires many superior properties such as higher toughness, greater flexural strength, and better-disguised discolored abutments, comparing to glass ceramics [2,17]. Nonetheless, the bonding to zirconia is crucial as no efficient protocol to treat an intaglio of zirconia fixed prosthesis prior to cementation is convinced. Obviously, both mechanical and chemical pre-treatments to zirconia surfaces have been studied inevitably to render durable bond strength [22]. However, no many studies have studied about the µSBS of various variables of air-abraded-treated zirconia to self-adhesive resin cements. Consequently, this present study was
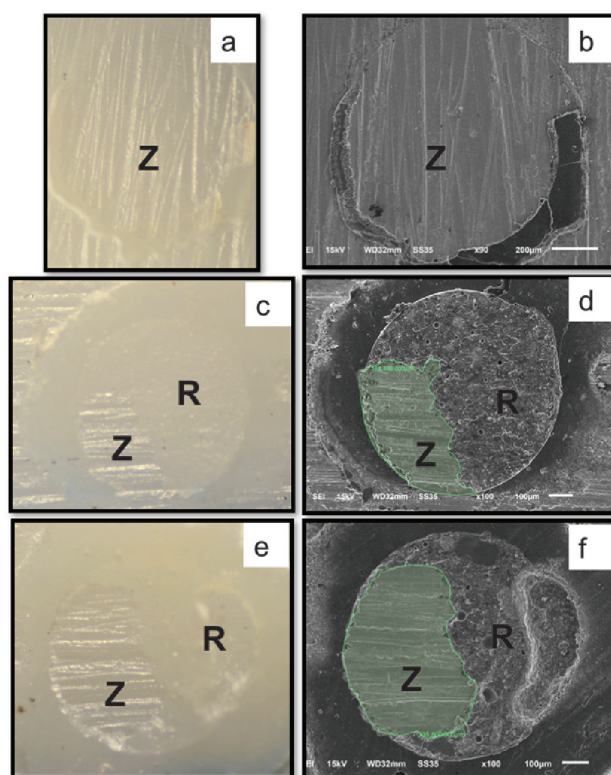
Figure 4. Representative light microscope (10X) and SEM micrograph (90-100X) of each fractured pattern. (a,b) adhesive failure pattern, (c,d) cohesive failure pattern, (e,f) mixed failure. The letter "Z" stands for zirconia surface and "R" stands for attached cements on the bonding interface.
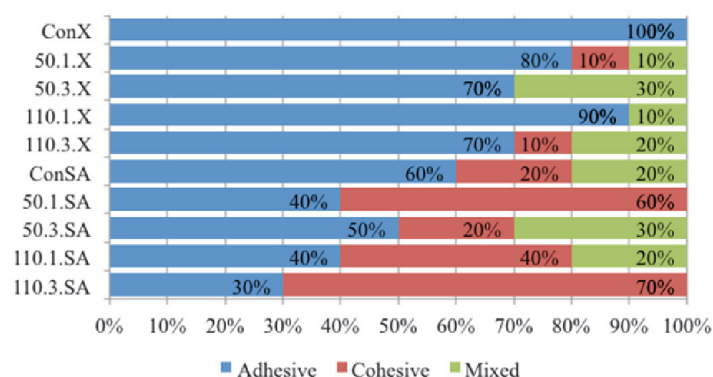
Figure 5. The fracture mode distribution in all group tested

conducted to establish the effect of various airborne abrasion protocols on zirconia in micro-shear bond strength with different self-adhesive resin cements.

Moreover, it was known that selecting types of resin cement was mandatory for longevity of zirconia bonding [4]. Our study demonstrated that Panavia SA, MDP-containing cement, provided significantly higher µSBS than Rely X Unicem at 24 hours, regardless of any air abrasion treatments. Therefore, our first hypothesis, there were no significant difference in µSBS between different luting agents on zirconia surface, was rejected (p<0.05). Many studies highly favored the performance of Panavia over Rely X Unicem in the long-term bond strength [3,8,16]. The main reason was due to the component of MDP, bonding directly to the metal oxides [21,23], which positively affected the adhesion. The monomer increased surface wettability and created methacrylate cross-linkages & siloxane bond with OH group of zirconia [4,13,20]. The other favorable property was the greater flexural strength of Panavia SA (81 MPa) compared to Rely X Unicem (48 MPa), which positively affected the bond strength.

In this study, the µSBS of surface-treated zirconia was significant higher than non-treated groups as anticipated [24], which was consistent to the study of Gome et al. (2013). This study conducted the µTBS of pretreated Cercon with various air abrasion treatments. It found that the microbar of untreated specimens failed prematurely prior to the conduction of the bond strength test [20]. Consequently, such mechanical treatment was mandatory for the zirconia bonding [16]. Nonetheless, great care must be taken not to perform excessive pressures or particle sizes, so such treatment must be performed wisely within normal range to prevent any damage [25].

In the bond strength test with zirconia, most studies evaluated the bond strength by polishing zirconia specimens with 600 or 1200-grit SiC paper to create flat surfaces and eliminate excess or uneven spots on zirconia surfaces prior to the bond strength test. Therefore, their control surfaces were considered the smoothest compared to other air abraded groups [24]. Nevertheless, our present study attempted to evaluate the bond strength

of an actual milled zirconia surface, which matched the roughness of the zirconia surfaces polished with the 180-grits of SiC paper on our pilot study. For this reason, the irregular surface of the control group in this present study was more obvious compared to some of the air-abraded groups.

As results has shown, both larger particle size and higher pressure positively affected on the bond strength (Figure 2). Thus, our second hypothesis, there are no significant difference in µSBS affected by either in different particle sizes or air pressures of air abrasion, was rejected (p<0.05). The 110 µm-sized particle increased significantly the bond strength values compared to the 50 µm-sized particle. According to our SEM micrographs on treated zirconia surfaces, the grooves were more prominently in 110 µm-sized particle groups than the 50 µm-sized particle groups (Figure 3b, d/ c, f). This characteristic would increase the more mechanical retention to rougher zirconia surfaces. Unlike this present study, Valentino et al. (2012) evaluated the influence of different surface treatment for promoting a bond between zirconia-based ceramic (Cercon Smart Ceramics, Degudent, Hanau, Germany) and a dual-cure resin luting agent (Enforce, Dentsply, Caulk,Milford, DE, USA). No significant differences in surface morphology and µSBS between the air abrasion with particle sizes of 50 and 110 µm was found [26].

Also, the 3-bar air pressure also produced significantly higher bond strength than the 1-bar air pressure, regardless of the particle size and resin cement. While, the 1-bar air pressure was not enough to create the adequate retention on zirconia surfaces [27], which was consistent to our SEM micrograph of surface topography (Figure 3b,d). Therefore, the treated zirconia with any particle sizes in 3-bar air pressure (50.3 & 110.3) provided higher bond strength than those in 1-bar air pressure (50.1 & 110.1).

The higher pressure of 3 bar and larger particle size of 110 µm were more capable to remove the superficial defects and the horizontal line pattern created by the initial polishing of 180-grit SiC paper (Figure 3). When the Al_2O_3 particle attacked on the zirconia surface, the larger particle size and higher air pressure, the more

kinetic energy presented. This energy could transfer to the rough surface, and groove, chamfering the sharp angle and subsequently shallowing the deep groove, smoothening and leveling out these rough surfaces [28-29]. This shallow groove may facilitate the resin infiltration on this treated zirconia, increasing the adaptation of resin cement on surface. Therefore, this protocol could increase the bond strength. Moreover, this protocol was more capable to remove contamination, increase surface wettability [24].

The adhesive failure was mostly found in the non-treated zirconia. The air-abraded zirconia, on the other hand, demonstrated greater amounts of the cohesive and mixed failures [20]. Moreover, Oyague et al. (2009) found the bond strengths of air-abraded specimens remained stable after 6 months water storage due to the micromechanical interlock of resin penetration into roughened zirconia [4]. Furthermore, Panavia SA (MDP-containing cement) also presented less in adhesive failure, because the MDP was proposed to chemically interact with oxides of zirconia [3,8,20].

This study selected performing µSBS test in order to avoid hassle and error of the specimen preparation [22], because µTBS tended to cause the pre-mature failure during cutting the microbars [19]. Furthermore, unstable temperatures and moisture could degrade the zirconia bonding [6,30]. Therefore, the clinical simulation of thermocycling should be conducted for the future research in order to investigate long-term bond strengths on zirconia bonding.

Conclusion

1. MDP-containing resin cement significantly provided higher µSBS to zirconia bonding than non-MDP resin cement.

2. The different air abrasion treatments on the zirconia surfaces and zirconia bonding with two resin cements affected on the bond strength. The zirconia treated by the air abrasion with larger particle size (110 µm) & higher air pressure (3 bar) and cemented with MDP-containing resin cement (Panavia SA) created the optimal adhesion to zirconia.

Acknowledgement

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