

Effect of Airborne-Particle Abrasion Protocols and MDP-based Primer on the Bond Strength of Highly Translucent Zirconia

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Purpose: To evaluate the effects of airborne-particle abrasion and MDP (methacryloyloxydecyl dihydrogen phosphate)-based primer treatment on the strength of resin bonds to highly translucent zirconia.

Materials and Methods: Eight groups (n = 20 per group) of specimens were prepared with airborne-particle abrasion treatments (0.1-, 0.3-, or 0.6-MPa pressure) or not (untreated control) and MDP-based primer (treated) or not (untreated). Shear bond strength (SBS) tests were performed on the composite-to-ceramic bonded specimens either with or without thermocycling. After airborne-particle abrasion, the surface topography was evaluated by white light interferometry, and a phase analysis was conducted with x-ray diffraction (XRD). Surface roughness (Ra), surface energy (SE), and SBS measurements were statistically analyzed using either Tukey's HSD or the Kruskal-Wallis test, based on applicability. Lastly, the failure mode was observed by optical microscope and scanning electron microscope.

Results: Airborne-particle abrasion resulted in significantly larger Ra ($p < 0.05$), especially with higher treatment pressures. Treatment with MDP-based primer caused significantly higher SE and SBS than airborne-particle abrasion alone ($p < 0.05$), both with and without aging.

Conclusion: MDP-based primer can enhance the bond strength and reduce hydrolytic aging of the bonded interface for highly translucent zirconia, exceeding the effects of airborne-particle abrasion. It is recommended that MDP-based primer treatment be applied with a composite cement containing adhesive phosphate monomer.

Keywords: highly translucent zirconia, airborne-particle abrasion, MDP-based primer, shear bond strength, surface treatments.

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Zirconia is widely used in prosthodontics due to its excellent mechanical strength and machining accuracy.^{8,9} However, degradation of conventional zirconia with prolonged exposure to oral conditions (ie, long-term degradation: LTD) can reduce the strength.⁵ Moreover, conventional

zirconia is not suitable for monolithic anterior restorations because of its poor optical properties.¹¹ Highly translucent zirconia has been developed with higher transparency and more favorable resistance to aging, which is a consequence of increased yttrium oxide stabilizer and cubic phase.^{4,12,28}

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Bond strength is critically important for the clinical application and durability of zirconia restorations.^{17,38} Previous studies have established that airborne-particle abrasion is the most popular mechanical treatment for clinical applications of traditional zirconia.³⁰ Highspeed Al₂O₃ particles can clean and roughen the surface simultaneously, which increases the potential for mechanical interlocking and consequently improves resin bonding to zirconia.^{29,36}

In addition to the use of mechanical treatments, chemical treatments are also used to change the surface properties, and to enhance the chemical bonding between the resin cement and the zirconia surface.¹⁷ This can be achieved by applying adhesive monomers. There are many functional monomers, but only 4-META and MDP can form chemical bonds with zirconia and be firmly adsorbed onto the surface of zirconia.^{34,42} 4-META is a polar monomer of carboxylic acid esters, which can form a chemical bond with metal or metal oxide to improve its bond strength.²⁶ MDP is also a polar molecule with a hydrophilic phosphate group and a hydrophobic carbon chain structure.²⁰ In comparison to 4-META, MDP is reportedly more effective for strengthening resin bonds to zirconia.⁴² MDP can establish stable chemical bonds with zirconia in the form of hydrogen bonds or ionic bonds by hydrophilic phosphate groups, and then combine with the resin adhesive through the hydrophobic carbon chain structure to improve bond strength.^{20,27} Previous studies have also shown the effectiveness of combining airborne-particle abrasion with subsequent MDP treatment to improve the bond strength to conventional zirconia.^{40,43,45}

A number of studies have also evaluated the relationship between surface treatment and bond strength of highly translucent zirconia. Most scholars focused on the influence of chemical priming agents, especially MDP, and airborne-particle abrasion on bond strength, finding that these treatments had positive effects on the bond strength.^{3,13,22,42} However, few studies compared the contributions of sandblasting and MDP to the improvement of bond strength. Although Le et al²² found MDP-based cement combined with sandblasting produced more durable bonding than MDP-based cement treatment alone, they only used a single sandblasting-pressure parameter. Aung et al³ explored the effects of different alumina-blasting pressures on the bond strength with two MDP adhesives, but the bond strength after thermocycling was not included. Furthermore, neither of those studies evaluated the bond strength without MDP.^{3,22} Thus, it is not clear whether sandblasting or MDP treatment plays a more important role in improving bond strength to highly translucent zirconia.

The purpose of this study was to evaluate the effects of airborne-particle abrasion and MDP-based primer on the resin bond strength to highly translucent zirconia. The hypotheses tested were: 1. the contributions of airborne-particle abrasion and the MDP-based primer to the surface properties of highly translucent zirconia are not significantly different, and 2. airborne-particle abrasion and the MDP-based primer can influence resin bond strengths to highly translucent zirconia. The results should identify an optimum surface conditioning method for highly translucent zirconia.

MATERIALS AND METHODS

Specimen Preparation

The materials used in this study are listed in Table 1. Specimens (N=160, 5 mm x 5 mm x 5 mm) of highly translucent zirconia Multilayer AT (Aidite Technology; Qinhuangdao, China) were sectioned and sintered according to the manufacturer's instructions. Composite cylinders (N = 160, 3 mm diameter x 3 mm high) were prepared from Filtek Z350 (3M Oral Care; St Paul, MN, USA) in a 3D-printed mold (Project MJP 3600 Series, 3D Systems; Rock Hill, SC, USA) and light cured (Elipar S10, 3M Oral Care) with an intensity of 1200 mW/cm². All the zirconia specimens and composite cylinders were polished with 800-grit silicon carbide papers (Struers; Copenhagen, Denmark) under water cooling, and were then ultrasonically cleaned (KQ-50B, Shumei; Kunshan, China) for 5 min in absolute ethanol, followed by another 5 min in distilled water.

The specimens were randomly divided into 4 groups according to the treatment pressure (untreated: AU; 0.1 MPa: A1; 0.3 MPa: A3; or 0.6 MPa: A6). The airborne-particle abrasion treatments were achieved with a commercial unit (Easyblast model, Bego; Bremen, Germany) using 110- μ m alumina particles (Korox 110, Bego). Airborne-particle abrasion was performed for 20 s at a distance of 10 mm with the nozzle oriented perpendicular to the sample surface. The sandblasted specimens were ultrasonically cleaned once again, then air dried. Each group was randomly divided into 2 subgroups (n=20), one in which the bonding surface was coated with methacryloxydecyl phosphate (MDP)-based primer (A1M, A3M, A6M), while the other remained only particle-abraded. The description of the groups is shown in Table 2.

The resin composite cylinders were bonded to the treated ceramic surfaces using a commercial self-adhesive resin cement (RelyX U200, 3M Oral Care).³¹ The composite-to-ceramic luting procedures recommended by the manufacturer were performed maintaining a constant axial load of 0.5 kg at room temperature (23 \pm 1°C) during the first 5 min of cement self-curing. Any excess cement was removed. An additional 40 s of light irradiation was applied from each side of the blocks to ensure optimal polymerization.⁷ Thereafter, the bonded specimens were stored in distilled water for 24 h at 37°C.³⁹ Finally, half of the specimens in each subgroup were subject to shear bond strength (SBS) testing immediately after the 24 h period. The other half was subject to thermocycling for 5000 cycles (at 5°C and 55°C, with 30 s dwell time and 2 s transfer time) before conducting the SBS test.^{1,32}

Surface Roughness

The surface topography of each specimen was evaluated by white light interferometry (Rtec Instruments; San Francisco, CA, USA) before applying MDP-based primer. The average surface roughness (Ra) was measured using commercial software matched to the instrument (Gwyddin 2.30, Czech Metrology Institute; Brno, Czech Republic). Five Ra readings were obtained at different locations of the treated surfaces from 10 samples of each group, and then averaged.



Table 1 Chemical composition and application mode of the materials tested

Material	Manufacturer	Main composition	Batch No.
Multilayer AT	Aidite (Qinhuangdao) Technology Co; Qinhuangdao, China	< 90.67wt% ZrO ₂ , 9.28wt% Y ₂ O ₃ , 0.05wt% Al ₂ O ₃	W160107 ATA2m-1
Korox 110	Bego; Bremen, Germany	110- μ m alpha corundum (Al ₂ O ₃)	1360651
RelyX U200	3M Oral Care; St Paul, MN, USA	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives	647271
Single Bond Universal	3M Oral Care	MDP, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane	81030A
Filtek Z350	3M Oral Care	Bis-GMA, UDMA, PEG-DMA, TEG-DMA, and bis-EMA resins, 78.5 wt% silica filler	N825205

MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: hydroxyethyl methacrylate; bis-GMA: bisphenol A glycidyl methacrylate; UDMA: urethane dimethacrylate; PEG-DMA: Poly (ethylene glycol) dimethacrylate; TEG-DMA: trimethylene glycol-dimethacrylate; bis-EMA: ethoxylated bisphenol A glycidyl methacrylate.

Table 2 Experimental groups with the various treatment conditions

Sandblasting pressure (MPa)	Untreated		0.1		0.3		0.6	
MDP-based primer (no/yes)	No	Yes	No	Yes	No	Yes	No	Yes
Code	AU	AUM	A1	A1M	A3	A3M	A6	A6M

AU: untreated (no sandblasting, no MDP); AUM: no sandblasting, with MDP; A1: sandblasting pressure 0.1 MPa, no MDP; A3: sandblasting pressure 0.3 MPa, no MDP; A6: sandblasting pressure 0.6 MPa, no MDP. A1M: sandblasting pressure 0.1 MPa, MDP coating; A3M: sandblasting pressure 0.3 MPa, MDP coating; A6M: sandblasting pressure 0.6 MPa, MDP coating.

X-ray Diffraction

Before the application of MDP-based primer, phase analysis was conducted on three samples randomly selected from each group to observe the effect of airborne-particle abrasion on the phase transformation. The phase was identified by x-ray diffraction (XRD) using Cu-K α radiation. The specimens were scanned from 10 degrees to 120 degrees (2 θ), with a step size of 0.026 and a step time of 17.34 s/step. The XRD data were analyzed using Jade 6.5 software (Materials Data; Livermore, CA, USA).

For the monoclinic-cubic A zirconia, the monoclinic phase content was calculated according to Toraya et al:³⁷

$$X_m = [I_m(-111) + I_m(+111)] / [I_m(-111) + I_m(+111) + I_c(111)]$$

In the equation, the terms $I_m(-111)$, $I_m(+111)$ and $I_c(111)$ represent the integrated intensity of the peaks diffracted in the monoclinic planes $m(-111)$ and $m(+111)$, and the cubic plane $c(111)$, respectively.

Contact Angle and Surface Energy

Three specimens were randomly selected from each subgroup to evaluate its contact angle and surface energy (SE). On the specimens coated with MDP-based primer, a layer of

oxygen inhibitor (GC; Tokyo, Japan) was applied and light cured for 20 s before the test.

Two different test liquids were used for the contact angle measurements: a polar liquid water and the dispersive liquid diiodomethane. Contact angles were measured with an optical contact angle instrument (DSA25, Kruss; Hamburg, Germany), using sessile drops placed on the surfaces of the zirconia specimens. One droplet of each solvent was applied per specimen, and the contact angles of the droplets were calculated by averaging values of the left and right sides. The SE was automatically calculated by a supporting software. In this test, the SE of Y-TZP was calculated as the sum of the two components as follows:

$$\gamma_s = \gamma_{sp} + \gamma_{sd}$$

where γ_s represents the SE of the Y-TZP, γ_{sp} represents the polar component of the SE of the Y-TZP and γ_{sd} represents the dispersive component of the SE of Y-TZP.

Shear Bond Strength

To measure shear bond strength (SBS), the specimens were placed in a universal testing machine (5565, Instron; Norwood, MA, USA) and shear loading was applied parallel

Table 3 Mean and standard deviation of average roughness Ra recorded after airborne-particle abrasion

Pressure (MPa)	Untreated	0.1	0.3	0.6
Ra (μm)	0.16 ± 0.02^a	0.30 ± 0.03^b	0.57 ± 0.06^c	0.75 ± 0.04^d
Different superscript lowercase letters indicate significant differences ($p < 0.05$).				

to the adhesive interface until fracture occurred. The specimens were loaded at a crosshead speed of 0.5 mm/min until failure.⁴⁶ The load-displacement response was automatically converted to a shear stress-strain curve with Bluehill universal software (Instron). The SBS was calculated according to the formula: $\text{SBS} = F/A$, where F is the maximum load (N) and A is the bonding area of the sample (mm^2). The SBS of pre-test failures (PTF) were recorded as 0 MPa; the percentage of PTFs that occurred in each group was also recorded.

Fractography

After failure, the debonded ceramic surfaces were examined using optical microscopy to determine the failure mode.²¹ In addition, the fractured interfaces were observed using scanning electron microscopy (SEM). Based on the observations, the failures were categorized as either adhesive failure between ceramic and cement, cohesive failure in the cement, or mixed failure containing both adhesive and cohesive failure components.

Statistical Analysis

Statistical analysis of the results was performed using commercial software (SPSS 20.0, IBM; Armonk, NY, USA) with the level of significance set at $\alpha = 0.05$. The Kolmogorov-Smirnov test and Levene's test were first performed to confirm the normality and equal variance assumptions of the data; the results showed that only the X_m and SE data were normally distributed with equal variance. Thus, one-way ANOVA was used to analyze the influence of surface treatments on the X_m and SE, followed by Tukey's HSD test for multiple comparisons. For the Ra and SBS data obtained from each group, a Kruskal-Wallis test was conducted to analyze the influence of surface treatments and for multiple comparisons.

RESULTS

Surface Roughness

The average Ra measurements for the zirconia specimens are presented in Table 3. All the treated experimental groups exhibited a higher Ra after airborne-particle abrasion than the corresponding untreated groups ($p < 0.05$). Higher treatment pressures caused significantly larger Ra ($p < 0.05$).

Phase Analysis

Representative XRD patterns for the highly translucent zirconia used here are shown in Fig 1. As expected, none of the

groups exhibited the monoclinic phase, with or without airborne-particle abrasion ($X_m = 0$). All the experimental treated groups showed an asymmetrical broadening and left shift of the peaks, which indicated deformation of the zirconia lattice as a consequence of airborne-particle abrasion.⁴⁸

Contact Angles and SE

Figure 2 shows representative images of the contact angle measured using water and diiodomethane. Results from the corresponding calculation of SE are shown in Fig 3. As evident from these values, the MDP-based primer resulted in a significantly higher SE ($p < 0.05$). Interestingly, before the application of MDP-based primer, the SE of the untreated specimens was significantly higher than those of the airborne-particle-treated groups ($p < 0.05$). However, after the application of MDP-based primer, there was limited difference among the four groups. The treatment pressure had no obvious effect on the SE of the airborne-particle-treated groups. Both the polar and dispersive components of the SE of zirconia were significantly affected by the MDP-based primer ($p < 0.05$).

SBS

The immediate SBS are shown in Table 4. Without MDP-based primer, the untreated group showed the lowest SBS ($p < 0.05$). Among the airborne-particle-treated groups, group A3 showed the highest SBS ($p < 0.05$). With exception of group A3, the MDP-based primer significantly improved the SBS ($p < 0.05$). The SBS of the untreated groups increased the most with primer treatment. After the application of MDP-based primer, there was no significant difference among the untreated and airborne-particle-treated groups. No PTF occurred during the immediate SBS test.

A summary of SBS after aging along with the calculated reduction compared with immediate SBS is presented in Table 5; a comparison of the SBS of the immediate and thermocycled groups is shown in Fig 4. Thermocycling caused a significant decrease in SBS for all groups ($p < 0.05$) evaluated. Furthermore, all of the experimental groups that received MDP-based primer treatment exhibited significantly higher SBS after aging than did the controls ($p < 0.05$). PTFs only occurred in AU (100%) and A1 (10%) groups. In contrast, the 0.3 MPa and 0.6 MPa airborne-particle-treated groups achieved the highest SBS ($p < 0.05$). All of the experimental groups that received MDP treatment showed similar SBS ($p > 0.05$).

Fractographic Analysis

According to the fractographic analysis of the specimens that failed during SBS testing, the fracture surfaces were

Fig 1 XRD patterns for the AU, A1, A3 and A6 groups of zirconia specimens.

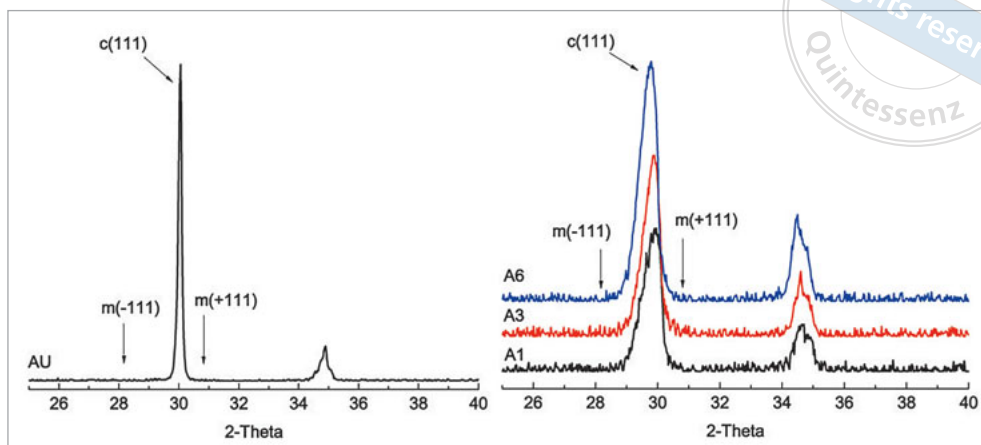


Fig 2 Images of water and diiodomethane droplets used for measurement of contact angles for the different surface treatments.

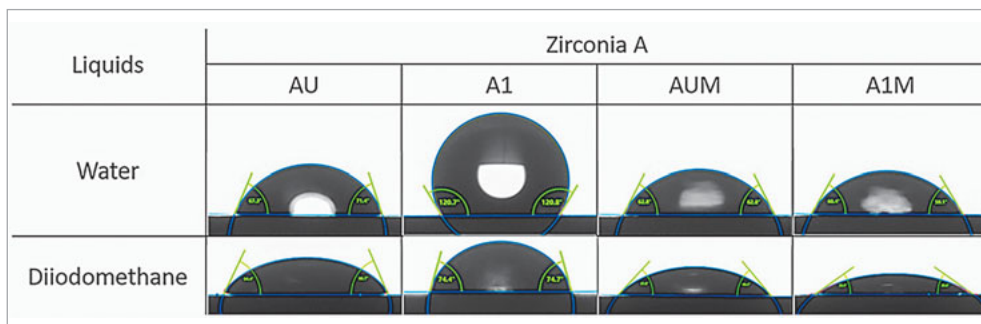
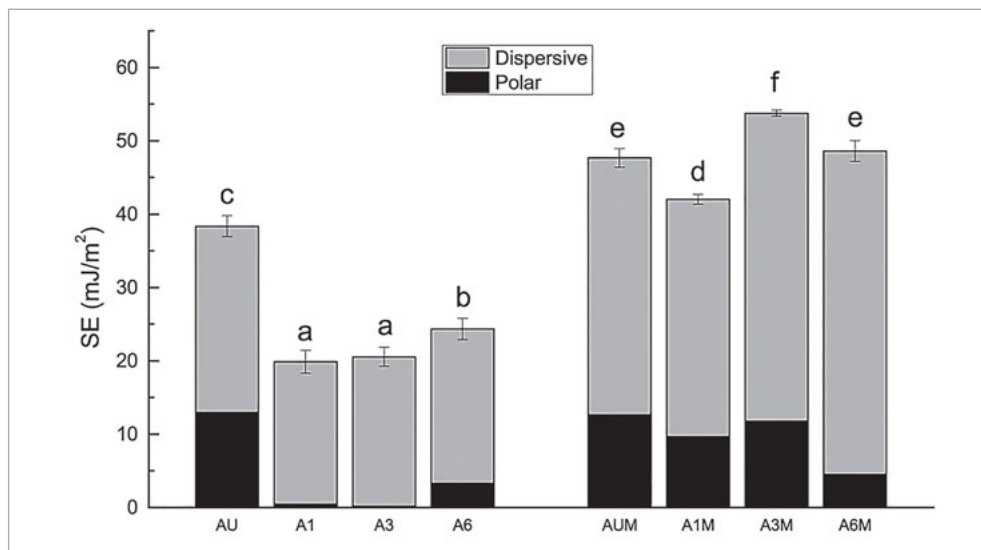


Fig 3 The SE of the highly translucent zirconia with different surface treatments. Different lowercase letters show significant differences ($p < 0.05$).



categorized as adhesive, cohesive, or mixed failures (Fig 5). The percentages of failure modes observed in the various groups are shown in Fig 6.

Without thermocycling, all groups primarily showed a combination of cohesive and mixed failures, with a range of 60%~100%, except the AU and A1 groups. Furthermore, the MDP-based primer treatment significantly reduced the percentage of adhesive failures in the AUM, A1M and A3M

groups with respect to the control groups. Except for group A3, thermocycling resulted in an increase in the percentage of adhesive failures of all control groups with a range of incidence of 40%~100%. Whereas the AU and A6 groups showed adhesive failures exclusively, overall, the experimental groups exhibited a relatively low percentage of adhesive failures (10%~30%), even after thermocycling. The exception was group A6M, which showed 50% adhesive failures.

Table 4 Mean and standard deviation of immediate SBS measurements

		Non-MDP treatment	MDP treatment	Increase (%)
No airborne-particle abrasion		4.41 (2.75) ^{Aa}	15.90 (2.36) ^{Ba}	260
Airborne-particle abrasion	0.1 MPa	11.24 (2.40) ^{Ab}	15.17 (4.22) ^{Ba}	35
	0.3 MPa	15.79 (1.22) ^{Bc}	16.85 (3.13) ^{Ba}	7
	0.6 MPa	11.81 (2.51) ^{Ab}	17.16 (3.37) ^{Ba}	45

Different superscript uppercase letters indicate significant differences in the same row ($p < 0.05$); different superscript lowercase letters indicate significant differences in the same column ($p < 0.05$).

Table 5 Mean and standard deviation of SBS measurements after aging

		Non-MDP treatment	Decrease (%)	MDP treatment	Decrease (%)
Non-airborne-particle abrasion		0 (0) ^{Aa}	100	10.01 (2.57) ^{Ca}	37
Airborne-particle abrasion	0.1 MPa	2.04 (1.13) ^{Ab}	82	11.56 (2.51) ^{Ba}	24
	0.3 MPa	4.82 (2.02) ^{Ac}	70	9.07 (2.50) ^{Ba}	46
	0.6 MPa	3.20 (0.86) ^{Abc}	73	9.83 (3.05) ^{Ba}	43

Different superscript uppercase letters indicate significant differences in the same row ($p < 0.05$); different superscript lowercase letters indicate significant differences in the same column ($p < 0.05$).

DISCUSSION

The present study was conducted to evaluate the influence of airborne-particle abrasion and application of an MDP-based primer on the resin bond strength to highly translucent zirconia. According to the results, the first hypothesis was rejected and the second hypothesis was accepted. Airborne-particle abrasion and the MDP-based primer significantly influenced the surface properties of the highly translucent zirconia used here. The effects of the two treatments were different (Tables 4 and 5). Furthermore, the airborne-particle abrasion and the MDP-based primer caused significant changes to the resin bond strengths to the highly translucent zirconia.

With respect to the control groups, the Ra of the airborne-particle-treated zirconia and the immediate SBS were significantly higher than those of the untreated groups (Tables 3 and 4). The repeated impact of the high-speed particles caused localized surface deformation, which effectively changed the surface topography, increased the effective bonding area, and enhanced the potential for mechanical interlocking between the ceramic substrate and resin, all of which contribute favorably to bond strength.³⁶ Also, airborne-particle abrasion can remove the contamination layer, which also enhances the bond strength.¹⁶

Interestingly, the immediate SBS did not uniformly increase with the increase of airborne-particle-abrasion pressure. There was an increase in Ra (Tables 3 and 4) and a significant increase in the immediate SBS with an increase in treatment pressure from 0.1 MPa to 0.3 MPa. With fur-

ther increase in pressure to 0.6 MPa, the Ra increased further. It appeared that the bonds to the zirconia samples underwent degradation through microleakage at the bonding surface, which was accompanied by a sharp reduction of the SBS (Table 4). In addition, the post-aging SBS of the zirconia samples showed trends similar to those of the immediate SBS (Table 5). In conclusion, in the absence of any other surface treatment, airborne-particle abrasion with 0.3 MPa pressure can be recommended for bonding to zirconia.

Although airborne-particle abrasion resulted in a significant increase in SBS for highly translucent zirconia, the SBS of these treated groups after thermocycling was still very low (Table 5, Fig 6). Pre-test failures occurred in the control groups after thermocycling, with a high percentage of adhesive failures. This suggests the resin bonds have poor resistance to hydrolytic aging with mechanical treatment alone (Table 5, Fig 6).⁴⁰

In comparison to the SBS of the control groups, the experimental groups that received MDP-based primer showed significantly higher SBS, regardless of whether or not they were thermocycled. In addition, there was less decline in the SBS after thermocycling for those groups that received primer treatment (Tables 4 and 5). MDP is an adhesive phosphate monomer now widely used for the surface treatment of conventional zirconia. Its effectiveness in enhancing bond strength to conventional zirconia has been demonstrated in many studies.^{17,24,41,42} MDP is a polar molecule with a hydrophilic phosphate group at one end and a hydrophobic carbon chain at the other end.^{20,27} The hydrophobic

Fig 4 The immediate (not thermocycled) and aged (thermocycled) SBS after different surface treatments.

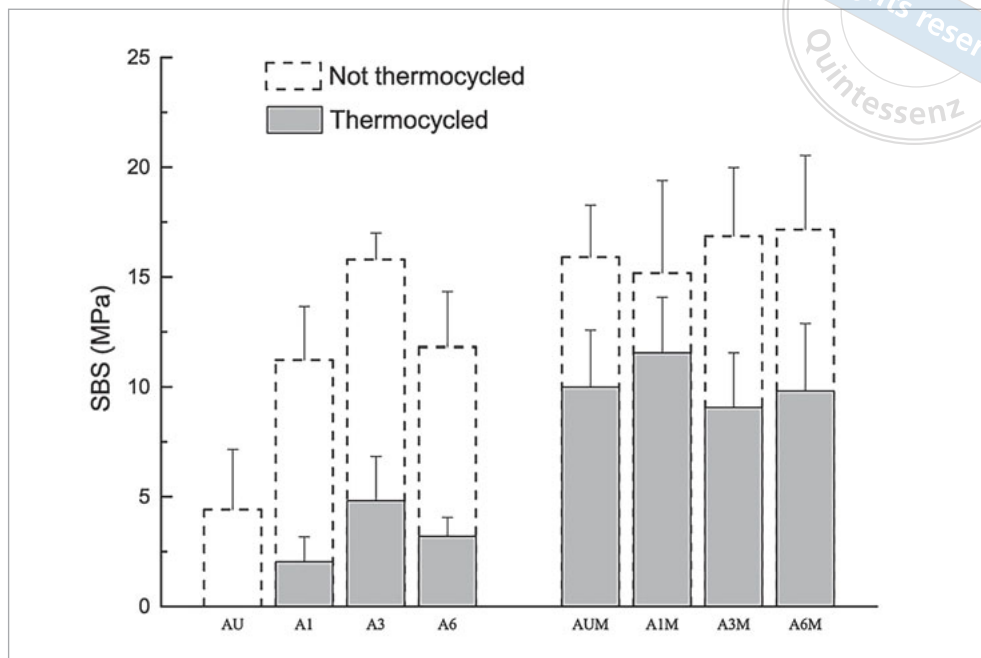
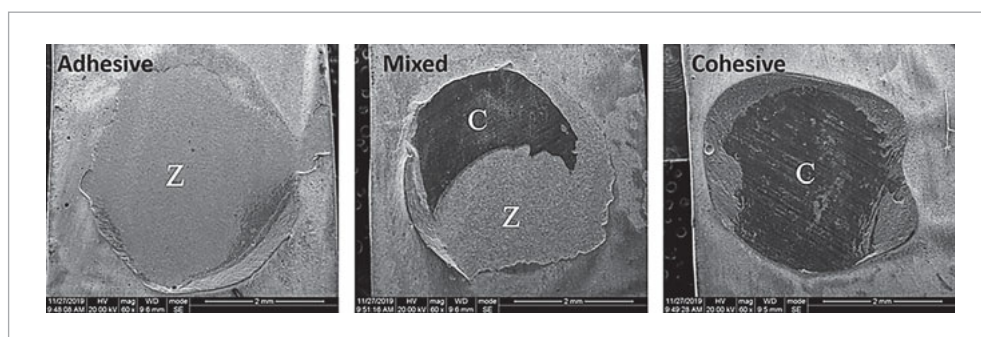


Fig 5 Representative SEM images for selected fracture surfaces (Z: zirconia; C: cement).



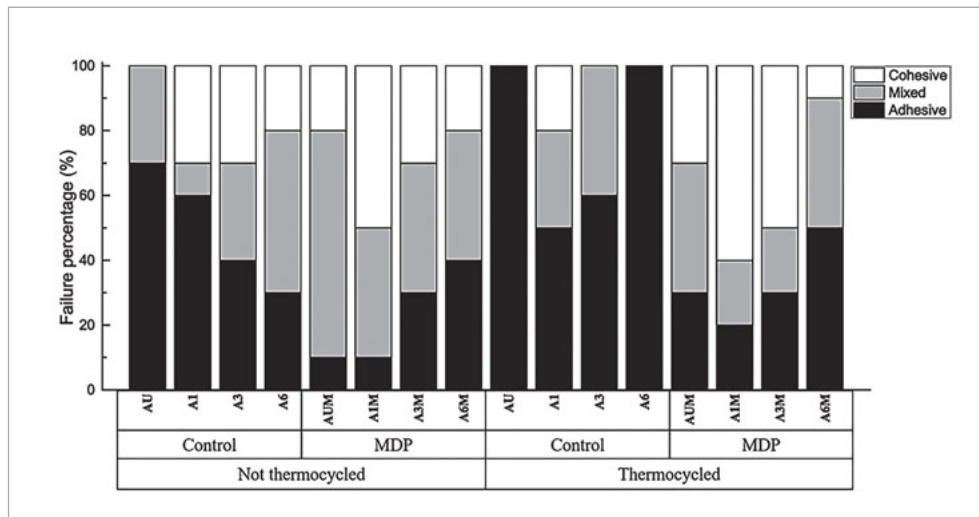
carbon chain can firmly bond with the resin cement, and the hydrophilic phosphate group can form a stable chemical bond with zirconia through hydrogen or ionic bonds.^{6,27} The main chemical composition of both highly translucent zirconia and conventional zirconia is zirconium oxide. As such, MDP was also effective at improving the bond strength to the highly translucent zirconia (Tables 4 and 5).^{4,11,42}

In addition to forming a chemical bond with zirconia, the MDP-based primer can also improve the surface wettability and SE of zirconia through the silane component.^{20,23} Indeed, the results showed an obvious increase in wettability and SE after the application of MDP-based primer (Figs 2 and 3). The higher wettability and SE can promote greater flow and infiltration of resin cement, increase the opportunity for chemical bonding, and the extent of micro-interlocking between the zirconia and resin cement, which enhances the bond strength.^{20,40} In comparison to the MDP solution with a single component, multifunctional universal primers containing more functional components (eg, silane) are re-

portedly more favorable for bonding to zirconia.^{20,23,24} As such, the multifunctional Single Bond Universal was chosen as the experimental MDP-based primer in the current study.

Aside from primers, some dual-curing resin cements also contain adhesive phosphate monomers like MDP, and often show much better bonding effectiveness than do other cements.^{14,31} The resin cement used in this study (RelyX U200) is also a dual-curing resin containing adhesive phosphate monomers, for which the manufacturer does not suggest application of a primer coating before bonding.³¹ Nevertheless, the results of this study showed that the combination of MDP-based primer and dual-curing resin cement significantly improved the SBS (Tables 4 and 5), which is consistent with a previous study.⁴⁰ Using resin cement alone, it is difficult to penetrate into grooves of the bonding surface because of its high viscosity and the poor wettability of zirconia. That leads to limited micro-interlocking and chemical bonding, which result in poor bond strength between the cement and zirconia (Tables 4 and 5).^{2,40} The

Fig 6 The percentage of fracture surfaces exhibiting adhesive, cohesive or mixed failure patterns after shear bond strength (SBS) testing.



high flowability of MDP-based primer and repeated brushing during its application increased the opportunities for chemical bond between zirconia and MDP.^{6,40} Furthermore, as previously indicated, the MDP-based primer improved the wettability and SE of zirconia, which facilitate the flow and penetration of resin cement to micro-interlock with the zirconia substrate (Figs 2 and 3).²³ Therefore, a combination of multifunctional MDP-based primer and resin cement containing adhesive phosphate monomers is recommended to obtain high bond strength to highly translucent zirconia.

According to the results of immediate and post-aged SBS, all of the experimental groups with MDP-based primer showed similar immediate and post-aging SBS with or without airborne-particle abrasion (Tables 4 and 5), which is consistent with previous studies involving conventional zirconia.^{15,19,33,35} This is because the enhancement achieved by the MDP-based primer was so strong that it masked the influence of airborne-particle abrasion.^{15,19} Hence, there is a limit to the effectiveness of abrasion treatments. An MDP-based primer combined with primer resin cement containing adhesive phosphate monomer is adequate to establish a durable bond.

A contamination layer with saliva or blood that develops during the try-in procedure is very difficult to avoid in the oral cavity.¹⁶ Concerning the potential contamination of bonding substrates during clinical practice, airborne-particle abrasion is considered necessary for coarsening and cleaning the bonding surface, as well as strengthening the bond durability.^{44,46,47} But the airborne-particle abrasion treatment with 0.6 MPa pressure harmed the zirconia, as evidenced by 50% adhesive failures in group A6M (Fig 5). The phase structure of the zirconia mainly consisted of the stable cubic phase, and there was no obvious monoclinic phase observed.²⁵ Thus, for the highly translucent zirconia used here, high treatment pressure can cause deep microcracks and defects. This promotes microleakage due to the lack of $t \rightarrow m$ phase transformation, which induces hydrolysis of the bonding interface and causes a reduction in the

bond stability.^{10,18,48} Thus, the airborne-particle abrasion should be conducted at pressures less than 0.6 MPa for highly translucent zirconia. 0.1 MPa is sufficient to obtain a stable bond (Tables 4 and 5).³⁰

In summary, although airborne-particle abrasion can improve the bond strength to highly translucent zirconia, its overall effectiveness is unstable due to the potential for hydrolytic degradation. To increase the bond durability, it is necessary to combine airborne-particle abrasion with an MDP-based primer treatment.³⁰ To obtain the highest bond strength, it is recommended that airborne-particle abrasion be conducted first with 0.1 MPa pressure, followed by application of an MDP-based primer and a resin cement containing adhesive phosphate monomer. One limitation of the current study is that it only evaluated the importance of conditioning methods involving highly translucent zirconia by a simplified aging treatment in the laboratory. Further studies should be conducted that involve experimental conditions more akin to those of the real oral environment, for instance, including bacteria, pH and mastication, and then complemented with clinical experiments to verify the laboratory results.

CONCLUSIONS

1. Airborne-particle abrasion increased the resin bond strength to highly translucent zirconia with up to 0.3 MPa treatment pressure. However, the SBS underwent a substantial decrease after thermocycling.
2. Application of an MDP-based primer increased the bond strength and effectively reduced the hydrolytic aging of the bonded interface for highly translucent zirconia, exceeding the effects of airborne-particle abrasion.
3. Considering the application of the results to clinical practice, to obtain high bond strength to highly translucent zirconia, it is recommended that MDP-based primer be applied with a resin cement containing an adhesive phosphate monomer.

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Clinical relevance: To achieve high bond strength to highly translucent zirconia, it is recommended that MDP-based primer treatment be applied with a resin cement containing adhesive phosphate monomer.

