# Effect of Airborne Particle Abrasion at Different Sintering Stages on Surface Roughness and Bond Strength Between Resin Cement and Highly-Translucent Zirconia

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## Abstract

This study aimed to investigate the influence of airborne particle abrasion performed at different sintering stages (before, after, or before and after zirconia sintering) on surface roughness (Ra) and shear bond strength (SBS) of highly-translucent zirconia. One hundred and twenty translucent zirconia discs were prepared for measuring the surface roughness (n = 6) and shear bond strength (n = 12). The Ra values were determined after sintering stage by using a contact profilometer and the surface morphology was analyzed with scanning electron microscope (SEM). For SBS, specimens were divided into four groups according to the sintering stage of airborne particle abrasion performed: before sintering (BS), after sintering (AS), abrasion before and after zirconia sintering (BAS), and control (C). The resin cement cylinders (Panavia™V5) were prepared and bonded to zirconia discs. Bonded specimens were tested for SBS either being subjected to 24 hours water storage or thermocycling for 10,000 cycles (n = 12). The failure mode was determined by stereomicroscope. Data were statistically analyzed by one-way ANOVA, followed by the Tukey's HSD. The results show that BS group exhibited the highest Ra values ($p < 0.001$). After 24 hours of water storage, the AS groups achieved the highest SBS, followed by the BAS and BS group, respectively ($p < 0.05$). Thermocycling caused a significant decrease in SBS of both groups ($p < 0.001$). Airborne particle abrasion before sintering process (BS) enhanced the surface roughness, while reduced the shear bond strength. Combination of airborne particle abrasion before and after sintering (BAS) had a favorable effect on surface roughness and shear bond strength

***Keywords***: *airborne particle abrasion, surface roughness, shear bond strength, translucent zirconia*

## 1. Introduction

Zirconia has been widely used in prosthetic and operative dentistry, whether as a framework material or a fully anatomical alternative because of its excellent mechanical properties, biocompatibility, low thermal conductivity, chemical stability and acceptable natural tooth appearance (Peumans et al., 2000; Peampring, & Sanohkan, 2014; Giordano, & McLaren, 2010).

In effort to improve the optical properties, highly-translucent zirconia has been introduced into dentistry by using higher content of yttria, reducing of the alumina additive concentration, and raising the amount of cubic phase proportion. According to Pereira et al. (2018), translucent cubic zirconia presents lower mechanical properties due to the lack of transformation toughening from high content of cubic phase and low tetragonal phase particles.

Adhesive cementation of zirconia ceramic is one of the most essential factors for long-term clinical success. However, zirconia is an inert material (Manicone et al., 2007) with the lack of silica and the vitreous phase in its composition making it resistant to traditional ceramic bonding methods, such as hydrofluoric acid etching followed by silane coupling agent application (Gracis et al., 2015; Lüthy et al., 2006). To overcome this limitation, several surface roughening methods have been suggested to improve adhesion between zirconia and resin cements. Airborne particle abrasion (APA) with aluminum oxide ($Al_2O_3$) particles is one of the most applicable methods to roughen the zirconia surface. This surface modification can promote the adhesion by cleaning the zirconia surface, increasing the surface roughness and bonding area to produce micromechanical interlocks, and enhancing high surface energy and wettability.

Airborne particle abrasion, the stress-generating treatment, can alter the structural stability of zirconia particles which may lead the zirconia to be more prone to low-temperature degradation. As the microcracks progress and this process continues, these have an effect on failure of the restoration (Wolfart et al., 2007; Maruo et al., 2020). For this reason, one option is to conduct airborne particle abrasion of the pre-sintered zirconia before sintering. With this modification, the sintering procedure might reduce the monoclinic phase compared to abrasion after sintering, resulting in improved mechanical properties of zirconia ceramics. (Moon et al., 2011; Okutan et al., 2019)

Martins et al. (2019) studied about the influence of particle and air abrasion moment on Y-TZP surface characterization and bond strength. The results of this study showed that air abrasion with 50 µm alumina particles before sintering and before & after zirconia sintering provided higher roughness compared to air abrasion after sintering. For shear bond strength, air abrasion before sintering resulted in higher shear bond strength than air abrasion applied after sintering.

In contrast, Skienhe et al. (2018) reported that there was no significant difference in shear bond strength between pre-sintered and post-sintered air abrasion although the pre-sintered ones had the highest surface roughness. Hence, this study concluded that surface roughness was not associated with higher bond strength. Additionally, air abrasion with 50 µm alumina particles before sintering could be a productive surface treatment method due to the absence of monoclinic phase creation and minimal compressive stress.

According to the existing data, few studies (Abi-Rached et al., 2015; Martins et al., 2019) have demonstrated increased surface roughness and improved bond strength after receiving a combination of airborne particle abrasion before and after zirconia sintering, which is considered as another viable procedure. Moreover, there is still no consensus in the studies about the effect of airborne particle abrasion and sintering protocols on translucent zirconia.

Thus, the purpose of the present study was to examine the surface roughness and shear bond strength of resin cement to translucent zirconia after airborne particle abrasion, performed at different sintering stages (before, after, or before and after zirconia sintering). The null hypotheses were that (1) the surface roughness and shear bond strength of translucent zirconia are not affected by airborne particle abrasion performed at different sintering stages, and (2) the bond strength after airborne particle abrasion, performed at different sintering stages is not influenced by thermocycling.

## 2. Objectives

1) To investigate the effect of airborne particle abrasion performed at different sintering stages on surface roughness and shear bond strength of highly-translucent zirconia.
2) To compare the shear bond strength after abrasion at different sintering stages, both before and after thermocycling.

## 3. Materials and Methods

### *3.1 Specimen preparation*

A total of 120 zirconia disc specimens were milled from pre-sintered zirconia blocks (Prettau® 4 Anterior®, lot no. ZC0394A, Zirkonzahn, South Tyrol, Italy) with a final dimension after milling of 14.8 mm. in diameter and 1.5 mm in thickness.

The prepared specimens for measuring the surface roughness (n = 6) and shear bond strength (n = 12) were randomly assigned into four groups according to the airborne particle abrasion, as follows in Table 1.

**Table 1** Test groups of the study

| Group code | Airborne particle abrasion | |
|---|---|---|
| | **Before sintering** | **After sintering** |
| C | – | – |
| BS | 20 s | – |
| AS | – | 15 s |
| BAS | 20 s | 15 s |

Abbreviations: C, control; BS, air-abraded before sintering; AS, air-abraded after sintering; BAS, air-abraded before and after sintering.

The airborne particle abrasion was performed with 50 µm $Al_2O_3$ particles (Renfert, Hilzingen, Germany), using special devices to standardize a perpendicular distance (10 mm) between the zirconia surface and a tip of the airborne particle abrasion unit, for 20 seconds and 15 seconds at pressures of 0.5 and 2.8 bar for abrasion before and after sintering, respectively. Prepared specimens were sintered in a furnace (Zirkonofen 600, Zirkonzahn, Italy), according to the manufacturer's instructions. The final dimensions of the specimens (12 mm diameter, 1.2 mm thickness) were verified using a digital caliper after a sintering shrinkage of around 19%. Materials used in the present study and the components are listed in Table 2.

**Table 2** Materials used

| Material | Composition | Manufacturer |
|---|---|---|
| Prettau® 4 Anterior® (Translucent zirconia) | $ZrO_2$ (main component), $Y_2O_3$ max. 12%, $Al_2O_3$ max. 1%, $SiO_2$ max. 0.02%, $Fe_2O_3$ max. 0.01% | Zirkonzahn (South Tyrol, Italy) |
| Panavia™ V5 (Resin cement) | Paste A: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated fluoroalminosilicate glass filler, colloidal silica<br>Paste B: Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated alminium oxide filler, accelerators, dl-Camphorquinone, pigments | Kuraray Noritake (Okayama, Japan) |
| CLEARFIL™ Ceramic Primer Plus (Primer) | 3-Methacryloxypropyl trimethoxysilane (3-MPS)<br>10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP), ethanol | Kuraray Noritake (Okayama, Japan) |
| Ivoclean (Cleaning paste) | Zirconium oxide, water, polyethyleneglycol, sodium hydroxide, pigments, additives | Ivoclar Vivadent (Schaan, Liechtenstein) |

Abbreviations: Bis-GMA, bisphenol A-glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; 3-MPS, 3-methacryloxypropyl trimethoxysilane; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate

### *3.2 Surface Roughness (Ra) and Topography*

The surface roughness (Ra) of all specimens (n = 6) was determined after the sintering stage by using a contact profilometer (Talyscan 150, Taylor Hobson Ltd., Leicester, UK). A total of five readings were recorded with a traveling distance of 2 mm across the surface around each point of occlusal contact, using a cutoff value ($\lambda_c$) of 0.25 mm and a stylus speed of 0.5 mm/s. The mean of these five surface roughness measurements was used as the outcome value for each group.

Random specimens from these four groups were analyzed with scanning electron microscope (JSM-6000, JEOL Ltd., Tokyo, Japan) to observe surface microstructure at 500× magnification with an accelerating voltage of 10.0 kV and 15.0 kV.

### *3.3 Bonding Procedure and Shear Bond Strength test (SBS)*

All the specimens were embedded in auto-polymerizing acrylic resin for shear bond strength test (SBS). Before receiving the primer, zirconia discs were cleaned with Ivoclean (Ivoclar Vivadent, Schaan, Liechtenstein) for 20 seconds and rinsed thoroughly with a water spray and the bonding surface was dried with oil-free air. CLEARFIL™ Ceramic Primer Plus (Kuraray Noritake Dental Inc., Okayama, Japan) was applied to the zirconia specimens and then blown dry with air, according to the manufacturer's recommendations. The cylindrical translucent molds (Tygon® tubing, Saint-Gobain, Courbevoie, France) were centrally positioned over the zirconia surface and filled with the Panavia™ V5 dual-cure resin cement (Kuraray Noritake Dental Inc., Okayama, Japan). Subsequently, the cement was light-cured for 40 seconds using a dental light-curing unit on the top surface (Elitedent, Rolence Enterprise Inc., Taoyuan, Taiwan) at a light intensity of 1,400 mW/cm$^2$ and the molds were gently removed to expose resin cement cylinders (2.0 mm internal diameter × 1.0 mm thickness) bonded to the zirconia surface.

For the non-thermocycled groups (n = 12 per group), the specimens were stored in distilled water at 37°C for 24 hours to record the immediate bond strength. The other groups (n = 12 per group) underwent thermocycling for 10,000 cycles between two water baths, one at 5°C and the other at 55°C, with a dwell time of 30 seconds, in accordance with ISO 10477:2020.

The shear bond strength was measured on a universal testing machine (AGS-X, Shimadzu Corporation, Kyoto, Japan) with a knife-edged blade parallel to the bonded surfaces at a crosshead speed of 1 mm/min until failure occurred. The load at fracture was recorded in Newtons (N) for SBS calculation by dividing the recorded load by the surface area, as shown in the following equation:

$$\text{Shear bond strength} = \frac{\text{Failure load (N)}}{\text{Surface area (mm}^2\text{)}}$$

The bonded cross-sectional area was calculated using the formula for the area of a circle, $A = \pi r^2$, where $r = 1$ mm (half of the diameter of the resin cement cylinder).

All failed samples were analyzed using a stereomicroscope (Euromex Ltd., Gelderland, Netherlands) to assess the fracture pattern. The failure modes were classified as adhesive (complete zirconia surface visible or debonding of the resin cement from the zirconia surface), cohesive (partial fracture of the resin cement on the zirconia surface) and mixed failure (a combination of adhesive and cohesive failure).

### *3.4 Statistical analysis*

Data were analyzed using SPSS version 26 statistical software (SPSS Inc., Chicago, IL, USA). The Shapiro-Wilk and Levene tests were performed for the assessment of normal distribution and homogeneity of variances, respectively. The Shapiro-Wilk tests for both data (Ra and SBS) were satisfied, while the Levene tests were violated ($p < 0.05$) for SBS. Therefore, surface roughness (Ra) data were analyzed by One-way analysis of variance (ANOVA) followed by Tukey's HSD test. The analysis of SBS data was performed by one-way ANOVA and Dunnett's T3 test. The comparison between non-thermocycled groups and thermocycled groups was also evaluated by one-way ANOVA. The statistical significance level was set at 0.05 for all tests.

## 4. Results and Discussion

### *4.1 Results*

Table 3 indicates the means and standard deviations of Ra values and SBS in MPa. Among all groups, the highest Ra values were observed in the BS group, while the C group presented the lowest Ra values ($p < 0.001$). The BAS group showed significantly higher Ra values compared to the AS group ($p < 0.001$).

After 24 hours of water storage, all the treated groups exhibited higher SBS than the control group ($p < 0.001$). The AS group achieved the highest SBS values, followed by the BAS and BS group, respectively.

After thermocycling for 10,000 cycles, the specimens (n = 12) from the control group (without airborne particle abrasion) did not survive and exhibited pre-test failures. SBS values in the other groups were measurable but significantly decreased compared to specimens stored in water for 24 hours ($p < 0.001$). There were no significant differences between the BAS and AS groups, or between the BAS and BS groups ($p > 0.05$). However, the AS group showed higher SBS than the BS group ($p < 0.05$).

**Table 3**: The means and standard deviations (SD) of surface roughness (n = 6) and shear bond strength (n = 12)

| Group | Ra (µm) | SBS (MPa) | |
|---|---|---|---|
| | | **24 h** | **10,000 TC** |
| C | 0.26 (0.03) $^{\alpha}$ | 3.08 (0.77) $^{A,a}$ | 0 $^{A,b}$ |
| BS | 1.07 (0.04) $^{\beta}$ | 10.78 (1.47) $^{B,a}$ | 1.27 (0.35) $^{B,b}$ |
| AS | 0.51 (0.05) $^{\gamma}$ | 17.84 (1.04) $^{C,a}$ | 1.77 (0.38) $^{C,b}$ |
| BAS | 0.77 (0.06) $^{\delta}$ | 15.19 (1.15) $^{D,a}$ | 1.85 (0.74) $^{BC,b}$ |

Abbreviations: MPa, megapascal; SBS, shear bond strength; 24 h, 24 hours water storage; TC, thermocycling.
Note: Different lowercase Greece letters and uppercase superscript letters within the same column indicate significant differences ($p < 0.001$). Different lowercase superscript letters within the same row indicate significant differences ($p < 0.001$).

The representative SEM images of different topographic surface structures are shown in Figure 1. A smooth surface was produced by polishing (Figure 1a) in the control group, while the specimens treated with airborne particle abrasion exhibited a visibly rougher surface (Figure 1b-d). The BS group (Figure 1b) displayed a more micro-retentive pattern than the others. The BAS group also showed more surface irregularities than the AS group.

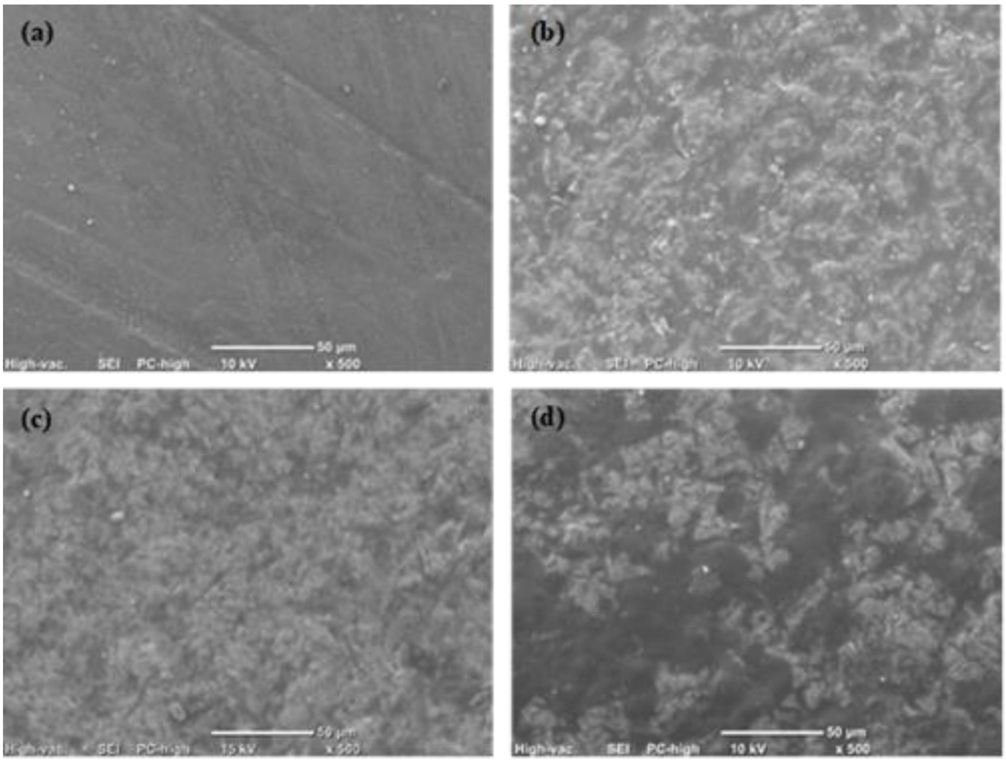


**Figure 1**: Representative SEM images showing surface structure of (a) control; (b) BS group; (c) AS group; (d) BAS group

Figure 2 shows the distribution of failure modes after 24 hours of water storage. Adhesive failure at the fractured zirconia/resin cement interface was predominantly found in all groups. The percentage of mixed failure mode (a combination of adhesive and cohesive failure) that occurred in the AS, BAS, and BS groups was 41.67%, 25%, and 16.67%, respectively. No cohesive failure was observed in any of the experimental groups. After thermocycling for 10,000 cycles, all debonded specimens exhibited a 100% adhesive failure at the zirconia surface.

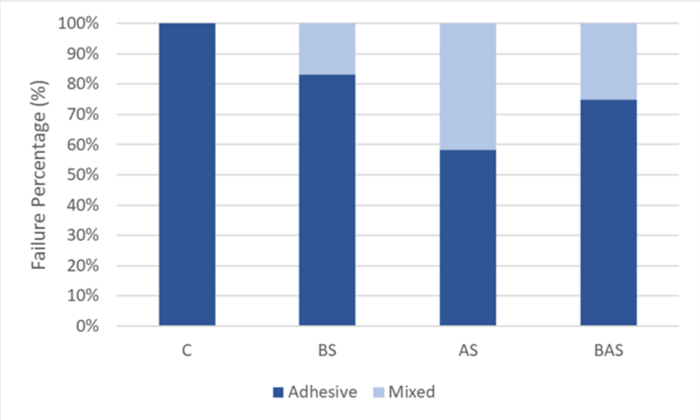


**Figure 2:** The distribution of failure mode (%) after 24 hours of water storage

***4.2 Discussion***

This study was designed to evaluate the effect of airborne particle abrasion performed at different sintering stages on surface roughness and shear bond strength between resin cement and translucent zirconia. Based on the findings, airborne particle abrasion applied at different sintering stages influenced surface roughness and shear bond strength. Therefore, the null hypotheses were rejected.

A contact profilometer was used in this study as the retention of laser energy by non-contact laser profilometer is difficult on white opaque surface of zirconia discs. Profilometry results showed that all treated specimens had higher Ra values when compared to the control group. Airborne particle abrasion applied to the zirconia specimens at pre-sintered stage (BS) exhibited the roughest surface, followed by the BAS and AS groups. Similar results were reported by other studies, where airborne particle abrasion before sintering process resulted in rougher surfaces than post-sintered abrasion even though the pre-sintered group was abraded with a reduced pressure (0.5 bar) (Abi-Rached et al., 2015; Okutan et al., 2019; Yilmaz, & Okutan, 2021). This might be attributed to the effect of harder alumina particles that simply embed in the soft and lower hardness of non-sintered zirconia surface in the green stage (Monaco et al., 2013). On the contrary, the lower Ra value obtained from the AS group could be due to the increased hardness of fully sintered zirconia (Monaco et al., 2013). Meanwhile, the use of airborne particle abrasion both before and after sintering in combination (BAS) created a significantly rougher surface than the AS group. These results are consistent with the study by Yilmaz and Okutan (2021), where different zirconia materials were airborne particle abraded using the same $Al_2O_3$ particle size.

Regarding the shear bond strength, the highest SBS values were observed for the AS and BAS groups compared to the BS and control groups, after 24 hours of water storage. These results are consistent with a study by Abi-Rached et al. (2015) which also showed that the BAS provided the highest SBS followed by the AS. Meanwhile, the BS group showed lower SBS than both, despite having their significantly roughest surface. Increased surface roughness should provide a more extensive area for adhesion. The researchers stated that the prominent edge morphology of the BS group probably impaired the wettability of zirconia. In contrast, the study performed by Martins et al. (2019) revealed the SBS results of the 50 µm $Al_2O_3$ treated specimens before sintering were higher than those of the AS and BAS groups, respectively. The authors concluded that the sintering process after airborne particle abrasion, in the BS group, induced the reverse transformation ($m \rightarrow t$), which reduced the proportion of the monoclinic phase.

The morphological evaluation in the SEM showed that the smooth surface of control group resulted in a smaller bonding surface area, which is related to the lower SBS. On the other hand, the SBS result of the BS group was low despite the fact that the prominent surface irregularities and greater Ra were observed.

Skienhe et al. (2018) reported the negative correlation between Ra and SBS as well. A possible explanation is that the infiltration of resin cement might not have been achieved all the way down into the deeper pits and grooves created by the BS. Abi-Rached et al. (2015) also concluded that airborne particle abrasion before zirconia sintering resulted in rougher surface but probably impaired the wettability. Moreover, a surface that is too deep and narrow produced by airborne particle abrasion would reduce the penetration of adhesives or resin cements (Lee et al., 2015). However, this assumption should be further investigated. The surface morphologies observed in the BAS (Figure 1d) showed a more microretentive pattern compared with the AS group, corroborating the Ra results in the present study. A study conducted by Kurtulmus-Yilmaz et al. (2019) also reported that the surface topography of post-sintered groups exhibited superficial depression when compared with pre-sintered groups.

Thermocycling, which causes repeated thermal expansion and fatigue at the interface, is the most common test applied to mimic the oral conditions and investigate the bond durability (Comino-Garayoa et al., 2021; Gale, & Darvell, 1999). After thermocycling these specimens, the current SBS results were consistent with studies showing that thermocycling reduced the SBS of resin cement to zirconia (Ehlers et al., 2015; Yagawa et al., 2018; Shen et al., 2023). Thermocycling for 10,000 cycles in this study exhibited a drastically decreased SBS when compared to the 24 hours' water storage. This decrease may be attributed to thermal stress developed at the resin-zirconia interface, rather than the hydrolytic degradation of the P-O-Zr bond during thermocycling (Xie et al., 2016). The specimens from the control group represented pre-test failures during thermocycling. It can be concluded that airborne particle abrasion and MDP-containing primer application are essential factors for a long-term durable bond. However, all specimens in this study were subjected only to thermal stress in water on exposed cement cylinders. Therefore, the results of clinical restorations could be different during exposure to artificial saliva instead.

According to the failure mode analysis, the control group and the groups that underwent thermocycling represented only adhesive failure and showed the lowest SBS. However, after 24 hours of storage in water, combination of adhesive and cohesive failure (mixed failure mode) predominantly occurred in the AS and BAS groups which presented the highest SBS.

The zirconia material used in the present study contains 12% yttrium oxide in its composition. This high-translucent zirconia possesses a reduced grain size of yttria and a greater amount of cubic phase, which may deteriorate the mechanical properties due to the small changes in the crystal structure. The average SBS results obtained from this zirconia were consistent with the study performed by Zhang et al. (2021). These researchers examined the SBS of translucent zirconia (UPCERA TT) to a resin cement using 50 µm $Al_2O_3$ particles at a pressure of 2 bar and observed mean SBS values between 13.08 – 18.68 MPa. The surface of translucent zirconia material in this study was airborne particle abraded using 50 µm $Al_2O_3$ particles under 2.8 bar after zirconia sintering, as applied in previous studies (Abi-Rached et al., 2015; Martins et al., 2019; Martins et al., 2020; Ebeid et al., 2017). Zhao et al. (2020) recommended that air pressure up to 3 bar increases the SBS of sintered translucent zirconia. In addition, using airborne particle abrasion combined with MDP-containing primer such as Clearfil Ceramic Primer Plus produced higher bond strength of resin cement to zirconia than those observed by Okutan et al. (2019) (2.35-4.92 MPa), in which no MDP-containing primers were used.

Based on the results of this study, although the airborne particle abrasion before sintering provided the highest Ra value, this procedure seems to lower the bond strength. On the other hand, the combination of airborne particle abrasion before and after sintering (BAS) had acceptable performance in both Ra value and bond strength when compared with the other procedures (AS and BAS). Thus, the BAS method could be considered as an alternative surface treatment to the translucent zirconia apart from the common procedure that usually applied after zirconia sintering (AS).

The current study was performed under *in vitro* conditions and mainly concerned the bond strength of abraded translucent zirconia. One of the limitations of this study was that the mechanical properties were not evaluated. The preparation of resin cement cylinders from Tygon® tubing mold may produce excessive cement and affect the bond strength. Moreover, the effect of clinical simulation by fabricating the prosthesis on bonding ability and mechanical behavior to assess clinical performance requires further investigation. In further studies, x-ray diffraction analysis (XRD) should be implemented to determine any structural changes of crystalline phase in translucent zirconia. Other surface roughness parameters should be examined to assess resin penetration.

## 5. Conclusion

Within the limitations of this study, the following conclusions can be drawn:

1) Airborne particle abrasion with 50 µm $Al_2O_3$ particles applied to highly-translucent zirconia before the sintering process (BS) created an extensive area for adhesion leading to enhanced surface roughness.
2) Shear bond strength was remarkably decreased after thermocycling.
3) The combination of airborne particle abrasion before and after sintering (BAS) had a favorable effect on surface roughness. Its SBS result was also compatible with the highest SBS values obtained from the airborne particle abrasion after sintering (AS).

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