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Air Abrasion vs. Tribochemical Silica Coating: Effect on Translucent Zirconia Bond Strength

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ABSTRACT

*Objectives: This study evaluated the microshear bond strength between resin cement and three translucent zirconias (3Y-PSZ, 4Y-PSZ, and 5Y-PSZ) following air abrasion with aluminum oxide (Al2O3) or tribochemical silica coating (CoJet), before and after thermal cycling. 3Y-TZP was included as a control. Methods: Zirconia specimens were treated with air abrasion or CoJet, followed by the application of a zirconia primer and adhesive. Dual-cured resin cement cylinders were then bonded to the treated surfaces for microshear bond strength testing after 7 days in distilled water or after aging (30 days and 10,000 thermal cycles). Data were analyzed using three-way ANOVA (zirconia, treatment, and aging) and Tukey's test ($\alpha = 0.05$). Results: The triple interaction was not significant. Treatment*time and zirconia*time interactions were significant ($p<0.001$). 3Y-TZP exhibited higher bond strength at 7 days. Higher bond strength values were obtained for CoJet and Al2O3 air abrasion groups at 7 days (13.47 ± 3.55 MPa). All specimens presented adhesive failures. Conclusion: The type of zirconia significantly influenced the microshear bond strength. 3Y-TZP showed greater bond strength compared to translucent zirconias. Tribochemical coating promoted higher bond strength at both time points. Thermal cycling negatively affected the bond strength of all zirconias evaluated. Clinical relevance: The observed variations in bond strength among different translucent zirconias emphasize the importance of material selection for optimal clinical outcomes. Additionally, the positive influence of tribochemical silica coating on bond strength highlights its potential as a valuable surface treatment for enhancing the durability of zirconia-based restorations in the oral environment. However, the detrimental effects of thermal cycling on bond strength emphasize the need for careful clinical considerations when fabricating and cementing translucent zirconia restorations.*

INTRODUCTION

Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), especially the 3 mol% yttria zirconia (3Y-TZP), is a widely used dental material due to its excellent physical, mechanical, and biocompatible properties.[1] Zirconia restorations can be monolithic or veneered. Veneered restorations are prone to delamination, chipping, and residual thermal stresses from sintering.[2,3] Monolithic restorations avoid these issues, but 3Y-TZP's low translucency makes it unsuitable for this kind of restoration. Therefore, more translucent yttria-partially stabilized zirconias (Y-PSZ) have been developed.[4] Increased yttria content (3 to 5 mol%) alters zirconia structure, reducing the tetragonal phase and increasing the cubic phase, improving translucency but decreasing flexural strength.[4,5]

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Despite its advantages, monolithic zirconia presents bonding challenges with resin cement when compared to ceramics with a high glass matrix content.[6] Zirconia's resistance to acid etching and poor silane reactivity[7] require effective surface treatments for successful cementation. Various chemical and mechanical techniques, including abrasive oxides, tribochemical silica coating (CoJet, 3M), zirconia primers, MDP-containing adhesives, and experimental modifications, have been explored to enhance zirconia surface roughness and resin cement bonding for long-term strength.[8-13]

Optimal zirconia adhesion often requires combined mechanical and chemical surface treatments.[14] Tribochemical silica coating (CoJet) roughens and chemically modifies the surface with silica, promoting silane bonding.[15] CoJet blasting followed by a ceramic primer has been shown to significantly improve Y-TZP adhesion, even after aging.[16] Alternatively, functional monomers like methacrylate phosphoric acid and 10-methacryloxyethyl dihydrogen phosphate (MDP) increase bond strength after air abrasion.[17] MDP forms strong P-O-Zr bonds with zirconia, while its vinyl group copolymerizes with resin materials.[18]

Uncertainties remain regarding the effectiveness of air particle abrasion and CoJet followed by a zirconia primer application for improving the adhesion of various zirconia to resin cement for immediate and long-term bonding. These uncertainties are related to the differences in the chemical compositions and mechanical properties of zirconias.[19-21] The air abrasion can affect each type differently. Conventional zirconia exhibits improved immediate mechanical strength due to transformation toughening triggered by different mechanical treatments on the zirconia surface.[22] However, translucent zirconia experiences a diminished capacity for this toughening mechanism, potentially leading to a critical reduction in mechanical strength after air abrasion surface treatments.[23,24]

While there is extensive literature on established surface treatment protocols for conventional zirconia,[16] and on the comparative bond strength of different translucent zirconia types,[19,23,24] limited research exists that directly compares the influence of surface treatments (Al_2O_3 air abrasion vs. CoJet) on the bond strength of various zirconia-based materials. This study aims to evaluate the microshear bond strength between resin cement and four types of dental zirconia (3Y-TZP, 3Y-PSZ, 4Y-PSZ, and 5Y-PSZ) treated with either standard air abrasion with Al_2O_3 or tribochemical silica coating, before and after aging. The following null hypotheses were established: (1) the bond strength between the various zirconia types will not differ significantly; (2) aging in water and thermocycling will not have a significant effect on the bond strength of the evaluated zirconias; and (3) there will be no significant difference in bond strength between Al_2O_3 and CoJet treatments.

MATERIAL AND METHODS

Four dental zirconias for computer-aided design/computer-aided manufacturing (CAD/CAM) restorations were used in this study: 3Y-TZP, 3Y-PSZ, 4Y-PSZ, and 5Y-PSZ (*Table 1*). Presintered zirconia blocks were sectioned into 1.5 mm thick slices (n = 6) using diamond discs under water cooling in a metallographic cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). To ensure uniform thickness (1.25 ± 0.05 mm) and prevent humidity-related defects during sintering, the samples were flattened with 320-grit sandpaper and subsequently dried in a desiccator. All the materials were sintered in a dental zirconia furnace for 2 hours in air at the following temperatures: 3Y-TZP at 1450°C, 3Y-PSZ at 1530°C, 4Y-PSZ at 1450°C, and 5Y-PSZ at 1450°C. Following sintering, the final thickness of the samples was 1.0 ± 0.05 mm, measured using a digital caliper with 0.01 mm accuracy.

The zirconia slices were embedded in self-curing acrylic resin (Jet, Artigos Odontológicos Clássico Ltda, São Paulo, Brazil) within PVC cylinders (1.2 cm in height and 2.5 cm in diameter). The zirconia surfaces were abraded with 600-grit sandpaper to remove any residual acrylic resin and establish a flat, uniform surface for the bond strength test. The specimens were then meticulously cleaned with 70% alcohol for 30 s, followed by rinsing with distilled water and drying with oil-free compressed air.

The zirconia surfaces were subjected to air abrasion using either 100 µm aluminum oxide (Al_2O_3) or 30 µm silica-coated aluminum oxide (CoJet) particles at a pressure of 3.5 bar for 10 s, maintaining a distance of ~2 cm from the surface. A layer of zirconia primer (MZ Primer, Angelus, Londrina, PR, Brazil) was applied for 3 min, followed by a layer of adhesive (Ambar, FGM, Joinville, SC, Brazil) actively applied for 10 s, air-thinning for 10 s to remove excess adhesive. The adhesive was light-cured for 10 s using an LED light-curing unit (Radii-cal, SDI, Bayswater, Victoria, Australia) at 1200 mW/cm².

For the microshear bond strength test, polyvinyl siloxane molds (Variotime EasyPutty, Kulzer, Hanau, Germany) were used. These molds contained six cylindrical cavities measuring 1.3 mm in diameter and 1 mm in height. The molds were carefully positioned on the pretreated zirconia surfaces. The cavities were filled with dual-cure resin cement (Allcem Core, FGM, Joinville, SC, Brazil) and the cement cylinders were then light-cured for 60 s using an LED light-curing unit (Radii-cal). After 10 min, the molds were removed, resulting in resin cement cylinders bonded to the zirconia surfaces with a bonding area of 1.8 mm². Half of the specimens were tested after 7 days of storage in distilled water at 37°C. The other half of the specimens remained stored in distilled water for 30 days and then underwent thermocycling (10,000 cycles of 30 s each at 5°C and 55°C) (OMC200, Odeme, Luzerna, SC, Brazil).

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Table 1. Composition of the materials used in this study.

Zirconia	Manufacturer	Composition
3Y-TZP	Vipi Block Zirconn, Vipi, Pirassununga, Brazil	Zirconium oxide stabilized with 3 mol % yttria
3Y-PSZ	Zpex, Tosoh Corporation, Tokyo, Japan	Partially stabilized zirconia powder with uniform dispersion of 3mol% yttria
4Y-PSZ	Zpex 4 Tosoh Corporation, Tokyo, Japan	Partially stabilized zirconia powder with uniform dispersion of 4 mol% yttria
5Y-PSZ	Zpex Smile, Tosoh Corporation, Tokyo, Japan	Partially stabilized zirconia powder with uniform dispersion of 5 mol% yttria
MZ Primer	Angelus, Londrina, Brazil	P-HEMA, methacrylate acid, PMDM, benzoyl peroxide, acetone
Ambar	FGM, Joinville, Brazil	MDP, methacrylics, photoinitiator composition, co-initiators and stabilizers
Allcem CORE dual	FGM, Joinville, Brazil	Base paste: Bis-GMA, Bis-EMA and TEGDMA, camphorquinone, co-initiators, barium- aluminum-silicate glass microparticles and particles of silicon dioxide, and inorganic pigments Catalyst paste: methacrylate monomers, dibenzoyl peroxide, stabilizers, and barium-aluminum-silicate glass
Al_2O_3	Polidental Ltd, São Paulo, Brazil	Alumina particles oxide (100 µm)
CoJet	3M Oral Care, St. Paul, USA	Alumina particles coated with silica (30 µm)

P-HEMA: polyhydroxyethylmethacrylate, PMDM: pyromellitic Dimethacrylate, MDP: 10-methacryloyloxydecyl dihydrogen phosphate, Bis-GMA: bisphenol-A glycidyl methacrylate, Bis-EMA: bisphenol A diglycidyl methacrylate ethoxylated, TEGDMA: triethylene glycol dimethacrylate.

Microshear bond strength tests were performed using a universal testing machine (DL 2000, EMIC, São José dos Pinhais, PR, Brazil) equipped with a 50 kgf load cell at 0.5 mm/min until fracture occurred. Before testing, the device was meticulously aligned to ensure the 0.2 mm diameter steel wire loop was positioned as close as possible to the bonded interface. The bond strength was calculated by dividing the maximum load (in Newtons) by the bonded area (1.8 mm²) and expressed in megapascals (MPa). Following testing, the specimens' surfaces were examined under a stereomicroscope (SZX9, Olympus, Tokyo, Japan) at up to 57x magnification to categorize the failure pattern. Failures were classified as adhesive, cohesive, or mixed.

The mean bond strength of all resin cement cylinders bonded to a single zirconia slice was calculated. The normality and homogeneity of the data variances were assessed using the Shapiro-Wilk and Levene tests, respectively. A three-way ANOVA was then performed to statistically analyze the influence of zirconia, surface treatment (Al_2O_3 or CoJet), and storage period on the bond strength, followed by Tukey's post-hoc test. A significance level of 5% was used in all analyses. Post-hoc power analysis was performed, and observed power values between 0.902 and 1 were obtained.

RESULTS

Table 2 summarizes the microshear bond strength results for all groups. Statistical analysis revealed significant effects for zirconia, surface treatment, and storage time ($p < 0.0001$). The interaction zirconia*surface treatment showed no statistically significant differences ($p = 0.246$). The interactions zirconia*storage time ($p = 0.001$) and surface treatment*storage time ($p < 0.001$) were statistically significant. The triple interaction was not statistically significant ($p = 0.569$).

Tables 3-5 present the results of the significant double interactions. Table 3 shows that 3Y-TZP exhibited the highest bond strength after 7 days of storage. The translucent zirconia groups presented lower bond strength values after 7 days, with statistically similar values among them. After aging, the bond strength decreased for all groups and became statistically similar regardless of the zirconia type. In Table 4, statistically significant differences were observed across all groups tested. The highest bond strength values were found in the groups treated with either CoJet or Al_2O_3 after 7 days of storage. The lowest values were observed in the groups following the aging. The data shown in Table 5 shows no statistically significant differences in bond strength between the surface treatments across the zirconia types.

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Table 2. Means and standard deviations according to zirconia, surface treatment, and aging.

Zirconia	Surface treatment	Bond strength (MPa)	
		7 days	After aging
3Y-TZP	Al_2O_3	11.19 ± 4.02	1.23 ± 0.31
	CoJet	17.50 ± 1.49	5.24 ± 3.26
3Y-PSZ	Al_2O_3	4.94 ± 0.98	0.96 ± 0.23
	CoJet	12.57 ± 1.60	3.76 ± 1.46
4Y-PSZ	Al_2O_3	4.79 ± 1.43	0.90 ± 0.18
	CoJet	11.95 ± 1.41	2.04 ± 1.57
5Y-PSZ	Al_2O_3	5.39 ± 1.34	0.57 ± 0.21
	CoJet	9.83 ± 2.20	1.36 ± 0.73

Table 3. Means and standard deviations according to zirconia and aging.

Zirconia	Bond strength (MPa)	
	7 days	After aging
3Y-TZP	14.34 ± 4.57 ^{Aa}	3.23 ± 3.20 $^{c\ Ba}$
3Y-PSZ	7.50 ± 3.96 ^{Ab}	2.36 ± 1.87 $^{c\ Ba}$
4Y-PSZ	8.37 ± 4.12 ^{Ab}	1.47 ± 1.34 $^{c\ Ba}$
5Y-PSZ	7.61 ± 3.07 ^{Ab}	0.96 ± 0.72 $^{c\ Ba}$

In each row, values followed by the same uppercase letters are statistically similar ($p > 0.05$). In each column, values followed by the same lowercase letters are statistically similar ($p > 0.05$).

Table 4. Means and standard deviations according to surface treatment and aging.

Surface treatment	Bond strength (MPa)	
	7 days	After aging
Al_2O_3	7.09 ± 3.97 ^{Ab}	0.95 ± 0.36 ^{Bb}
CoJet	13.47 ± 3.55 ^{Aa}	3.34 ± 2.75 ^{Ba}

In each row, values followed by the same uppercase letters are statistically similar ($p > 0.05$). In each column, values followed by the same lowercase letters are statistically similar ($p > 0.05$).

Table 5. Means and standard deviations for bond strength according to zirconia and surface treatment.

Zirconia	Bond strength (MPa)	
	Al_2O_3	CoJet
3Y-TZP	6.21 ± 5.99	11.37 ± 6.93
3Y-PSZ	2.95 ± 2.26	8.16 ± 4.98
4Y-PSZ	2.85 ± 2.35	7.00 ± 5.53
5Y-PSZ	2.98 ± 2.78	5.59 ± 4.86

All tested specimens exhibited adhesive failures, regardless of the zirconia, surface treatment, or aging. Pre-test failures were categorized into two main groups: those occurring during the aging period and those happening during specimen handling before the microshear bond strength test. While these failures were documented, they were excluded from the final analysis. The pre-test failure rates during the aging period ranged from 13% to 59% for the Al_2O_3-treated specimens, with the remaining failures occurring during handling. In contrast, the CoJet-treated groups displayed pre-test failure rates ranging from 0% to 60% during aging (*Figure 1*).

DISCUSSION

The development of 4Y-PSZ and 5Y-PSZ enhanced the translucency of monolithic zirconia restorations; however, they exhibit lower fracture strength and fracture toughness compared to 3Y-TZP due to the increased yttria content and the presence of a cubic phase.[4] Interestingly, properties like hardness and modulus of elasticity remain comparable to 3Y-TZP.[4] Given the relatively recent introduction of translucent zirconia, limited research has investigated its bond strength to resin cements compared to 3Y-TZP.[25,26] Kown *et al.*[25] reported similar bond strengths for 5Y-PSZ and 3Y-TZP after treatment with air abrasion and MDP primer application. However, their study also revealed that 5Y-PSZ presented nearly half the flexural strength and lower fracture toughness compared to 3Y-TZP, suggesting a higher susceptibility to surface defect formation during procedures like air abrasion.[25] Accordingly, the present study demonstrated that translucent zirconias exhibited lower bond strength to resin cement compared to 3Y-TZP. This finding rejects the initial hypothesis, which proposed no significant difference in bond strength between the zirconia types tested. The observed lower bond strength values for translucent zirconia warrant further investigation.

Surface pre-treatment is a well-established method to improve the bond strength between resin cement and zirconia, regardless of its translucency. These treatments aim to achieve two main goals: increasing the surface roughness of the zirconia for improved mechanical interlocking and promoting a stronger chemical bond between the zirconia and the cement.[27,28] Increased

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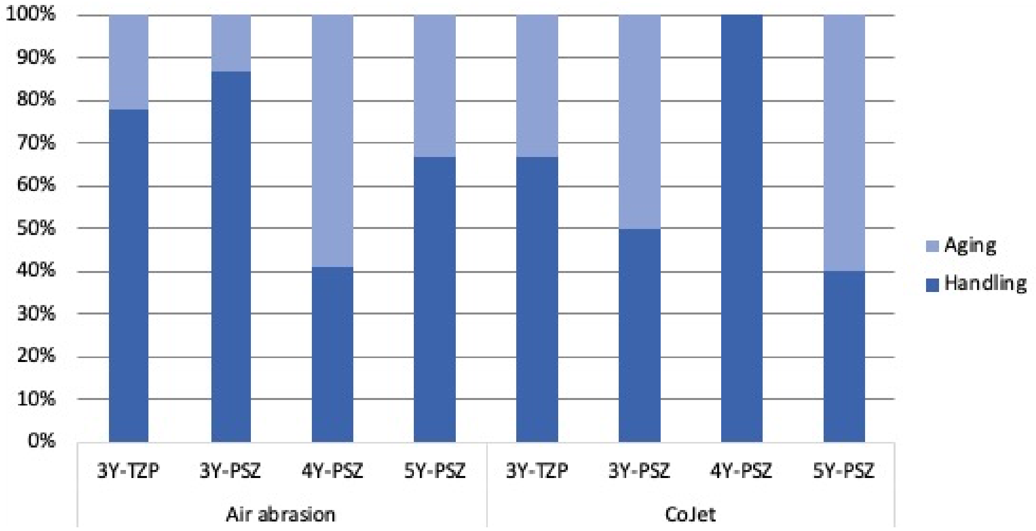

Figure 1: Pre-test failure rate during aging or handling for each group evaluated.

surface roughness creates a larger bonding area, which is particularly beneficial for ceramics.[29] While air abrasion with Al_2O_3 is a popular technique for micro-mechanical bonding, it can introduce micro-cracks that compromise the mechanical strength of highly translucent zirconia.[30] A review suggested tribochemical treatment as a promising method to significantly improve adhesion to zirconia.[17] Furthermore, Bielen *et al.*[7] demonstrated that tribochemical silica coating on Y-TZP zirconia resulted in superior bond strength compared to Al_2O_3 air abrasion, especially after aging. Our results also confirmed that tribochemical silica coating yielded higher bond strength values compared to Al_2O_3 air abrasion. Therefore, the hypothesis that there would be no significant difference between conventional air abrasion and tribochemical coating was rejected, as CoJet presented higher bond strength values.

Chemical pre-treatment with materials containing is recognized as a method to increase the bonding between Y-TZP zirconia and resin cement.[31] This is because MDP promotes a stronger chemical bond between the zirconia and the resin-based materials.[29] In this study, an adhesive system containing MDP was used for bonding. Shimizu *et al.*[32] reported that highly translucent zirconia treated with an MDP-containing primer (PZ Primer) exhibited higher bond strength compared to untreated samples. Similarly, the present study used a zirconia primer (MZ Primer) containing primarily pyromellitic dimethacrylate monomer (PMDM) before applying the adhesive. Pilo *et al.*[33] investigated the surface chemistry changes induced by two different zirconia primers, one containing PMDM (Danville Z Bond) and another containing MDP (Z-Prime Plus). It was observed that the MDP-based primer formed a thicker, amorphous film of zirconium phosphate salt, while the PMDM-based primer created a thinner film of zirconium carboxylate salt. These differences in film composition and water solubility between the carboxylate and phosphate salts may influence the bond strength and long-term durability of the adhesive interface with zirconia.[33] Supporting this evidence, a systematic review analyzing the *in vitro* durability of various 3Y-TZP cementation protocols to enamel concluded that surface pre-treatment with functional monomer-based primers and resin cements significantly improves bond strength.[34]

In addition to bond strength, the fracture patterns of the specimens were examined. The absence of resin cement residue on the zirconia surfaces indicated adhesive failure. This suggests that stress concentration occurred primarily at the interface during microshear testing. Similar findings of adhesive failures have been reported in other studies using various surface pre-treatment techniques.[28]

This study revealed a detrimental effect of aging on bond strength values, regardless of the zirconia type or surface treatment method. Consequently, the hypothesis that aging in water and thermocycling wouldn't affect bond strength was rejected due to the observed significant decrease in bond strength values after aging. Previous research suggests that thermocycling and water storage significantly influence the interfacial fracture energy of zirconia.[8] This is because the bonded interface's inherent fragility allows water infiltration, which dramatically weakens the bond. The difference in thermal expansion coefficients between zirconia and resin cement leads to the introduction of thermal stresses at the interface during thermocycling. These cyclic stresses contribute to the observed reduction in bond strength, potentially weakening the bonded interface.[8] The present study observed a significant decrease in bond strength values after aging, alongside pre-test failures that occurred either during thermocycling or specimen handling before the bond strength test.

A systematic review on zirconia adhesion identified water immersion or thermocycling as the most frequent methods for simulating the *in-vivo* aging process of the adhesive interface.[17] However, some studies have used a combined approach of water storage and thermocycling, leading to a more pronounced decrease in bond strength values.[35] The combination of these two approaches, as demonstrated in the present study, may have resulted in the most significant degradation of the adhesive interface observed.

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The use of a single light-curing unit (LCU) is a factor to consider, as the output and spectra of these devices can vary, possibly affecting resin cement polymerization and bond strength. Although future studies with other curing units are recommended to assess generalizability and curing protocol influence, the consistent use of the same device across all groups minimizes its impact on relative comparisons between zirconia types and surface treatments.

In vitro studies have limitations in directly translating their findings to the complex oral environment. While Y-TZP restorations have demonstrated promising clinical outcomes,36 newer materials like monolithic translucent zirconia require long-term clinical validation through further research. Despite previous suggestions that conventional cementation protocols and surface treatments might be effective for translucent zirconia, achieving long-term bond strength,[37] this study observed lower bond strength values to resin cement for translucent PSZ zirconia compared to Y-TZP. Therefore, the use of translucent zirconia in restorations should be approached cautiously, despite their good aesthetics. Translucent zirconia restorations offer aesthetic advantages, but their lower bond strength warrants further investigation and potentially different clinical considerations compared to 3Y-TZP.

CONCLUSIONS

It can be concluded that the composition of the zirconia-based materials significantly influences bond strength, with 3Y-TZP exhibiting higher bonding compared to translucent zirconia. Aging negatively affected bond strength for all zirconias. However, CoJet surface pre-treatment consistently increased bond strength, regardless of zirconia or aging.

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