Aged Translucent Aesthetic Zirconia: Bond Strength Analysis

Bernardo Luiz Gallina[1] Mauro Carlos Agner Busato[2] Eliseu Augusto Sicoli[1] Veridiana Camilotti[3] Marcio Jose Mendonca[1]

[1]Department of Prosthodontics, Western State University of Paraná, Paraná, Brazil
[2]Department of Orthodontics, Western State University of Paraná, Paraná, Brazil
[3]Department of Restorative Dentistry, Western State University of Paraná, Paraná, Brazil

Address for correspondence Marcio Jose Mendonca, DDS, MS, PhD. Department of Prosthodontics, Western State University of Paraná, Paraná, Brazil (e-mail: dr.mendonca@uol.com.br).

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Abstract

Objective This study evaluates the bond strength of two compositions of aesthetic translucent zirconia (TZ).

Materials and Methods For this evaluation, test specimens were prepared from ICE Zirkon TZ and Prettau Anterior zirconia (PAZ) that were stored in distilled water at 37°C for two time periods: T1 (24 h) and T2 (90 days) to simulate aging. Two factors were evaluated for the samples—ceramic and aging time. The samples were subjected to tests of microshear strength and fracture type and were evaluated using scanning electron microscopy.

Results The results were analyzed using the D'Agostino test, analysis of variance, and Tukey's test ($p < 0.01$). Statistically significant differences were observed for ceramic type and aging time.

Conclusion The results showed that PAZ provides significantly superior performance to TZ at the two aging times evaluated.

Keywords
► bond strength
► microshear
► zirconia

Introduction

Zirconia-based ceramics are materials commonly used in indirect single-unit restorations, fixed partial dentures, indirect restorations, and, more recently, monolithic restorations. Zirconia has been extensively studied due to its biocompatibility, mechanical resistance, and aesthetic results, being widely used in dentistry. Several variations of zirconia have been developed, with each new material intended to provide some quality improvement over previous versions. Recently, the use of monolithic yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) for indirect restorations has been developed to overcome the problems of veneered zirconia fixed dental prostheses (FDPs).

Monolithic zirconia FDPs are milled from blocks using computer-aided design/computer-aided manufacturing (CAD/CAM) equipment and can be used either polished or glazed for enhanced aesthetic outcomes.[1,2] Monolithic zirconia FDPs have considerably enhanced strength and resistance to chipping.[3] Different types of monolithic zirconia have been developed to obtain a more aesthetic and translucent restoration and to match zirconia's success in physical properties.[4] Yttria (Y_2O_3) is the most commonly utilized stabilizer.[5] The higher translucency of Y-TZP materials is achieved with much lower alumina content compared with conventional zirconia and an increase in Y_2O_3 content to minimize low-temperature degradation.[6]

In addition, it has been reported that the improved translucency of monolithic zirconia was attained by the combination of a relatively fine grain size and the presence of optically isotropic cubic zirconia particles that reduce grain-boundary light scattering.[7] In spite of the advantages of monolithic zirconia restorations, bonding between adhesive resins and oxide ceramics still presents a challenge compared to that between adhesive resins and glassy matrix ceramics.[1,8,9] Zirconia is silica free, which makes it difficult to etch.[1,10,11] However, the greatest current challenge is to guarantee adequate bond strength with the resin cements used and to ensure satisfactory aesthetic results.

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There is no consensus on the best surface treatment to obtain optimum adhesive bond strength between zirconia and resin. Several methods have been suggested, including blasting with aluminum oxide,[12–14] tribochemical reaction,[12,15,16] laser irradiation,[13,16–18] and even the combination of all these.[14,16] Another way of increasing bond strength is to use monomers with phosphate in their composition. Furthermore, the combination of phosphate with aluminum blasting could be used.[14]

Resin cements are a good option for adhering parts, including zirconia,[10] although, over time, this bond may lose strength, cause retention loss, and/or increase microleakage, primarily for two reasons: temperature variations, which cause differential contraction and dilation between materials, and moisture, which can cause cement dissolution and chemical degradation of the bond between zirconia and cement/adhesive.[19] Therefore, aging in laboratory tests is important for simulating these conditions found in the oral environment,[20] with high interference in the long term.

Another important point to take into account is the translucency of the material, given that, in a clinical setting, once the piece is positioned, the cement needs to be reached by light from the photoactivator to be activated. In this context, the cementation of highly opaque zirconia presents a challenge because those types are most susceptible to failure.[16,21,22] Therefore, it is necessary to develop types of zirconia that are more translucent, allowing photoactivation by light through the element, and that can provide favorable aesthetics. A minimum energy density of 4000 mJ/cm^2 is required to ensure that the bond between dentin and adhesive is not affected.[23] Prettau Anterior zirconia (PAZ) aims to combine the physical properties of zirconia with high translucency to meet the aesthetic needs of anterior teeth. In previous studies, PAZ showed greater light irradiation through different thicknesses, benefiting aesthetics, and cementation, when photoactivated cements were used.[4,24] Moreover, further studies are needed to evaluate the mechanical properties of this new material and to investigate whether these properties satisfy the minimum clinical and mechanical parameters.

Therefore, this study compared the microshear bond strength of two forms of aesthetic translucent zirconia (TZ): PAZ and ICE Zirkon TZ, both of which were previously aged.

Materials and Methods

The sample was calculated using a family *F* probability, with a repeated families design, with interaction within and among the factors. The effect size of 0.15, type 1 (α) error of 0.01, and analysis power of 0.95 chosen resulted in 44 sample units (test specimens [TSs]), with 11 samples per experimental group. GPower software (version 3.1.9.2, University of Düsseldorf, Germany) was used for sample calculation.

The materials used in this study are listed in ►**Table 1**. A total of 44 zirconia samples were produces, with 22 samples of 3 mm × 5 mm × 10 mm Zirkonzahn PAZ (Gais, Bolzano, Italy) in A1 color, through presintered blocks cut using a wet milling CAD/CAM machine (Zirkonzahn M1, Gais, Bolzano, Italy), and another 22 TZ samples (Zirkonzahn, Gais, Bolzano, Italy) in A1 color, measuring as much as PAZ, also presintered and cut in the CAD/CAM Zirkonzahn M1 Wet-Zirkonzahn milling machine (Zirkonzahn, Gais, Bolzano, Italy) at a final temperature of 1,500°C at a heating rate of 8°C/min for 2 h. The blocks were blasted over the entire surface using aluminum oxide of 100 μm at 3.5 Bar for 5 s. Forty-four circular PVC pipes (Tigre do Brasil S/A, Rio Claro, Brazil) of dimension 2 cm × 5 cm were used to fix the blocks.

Table 1 List of the materials used

Material	Manufacturer	Composition
Zirconia Prettau Anterior	Zirkonzahn, Gais, Bolzano, Italy	ZrO_2, Y_2O_3 < 12%, Al_2O_3 < 1%, SiO_2 < 0.02%, Fe_2O_3 < 0.01%, Na_2O < 0.04%
ICE Zirkon Translucent Zirconia	Zirkonzahn, Gais, Bolzano, Italy	ZrO_2, Y_2O_3 4–6%, Al_2O_3 < 1%, SiO_2 < 0.02%, Fe_2O_3 < 0.01%, Na_2O < 0.04%
Monobond Plus	Ivoclar Vivadent AG, Liechtenstein	Silane methacrylate, methacrylate phosphate, methacrylate, bisphenol glycidyl methacrylate (Bis-GMA), and triethylene glycol dimethacrylate (TEG-DMA)
RelyX Unicem U200 Resin Cement	3M ESPE Dental, St. Paul, MN, USA	Base: Methacrylate monomers, methacrylate phosphoric acid, silanized particles, initiator components, stabilizer Catalyst: Methacrylate monomers, alkaline particles, initiator components, silanized particles, stabilizer, and pigments

The zirconia block was placed on a glass plate within the tubes, and chemically activated acrylic resin Vip Flash (Dental Vip, Pirassununga, Brazil) was poured in sandy phase into the plastic tube until it was completely filled. Subsequently, all samples were cleaned with distilled water for 30 s and 37% phosphoric acid (FGM, Florianópolis, Brazil) for 30 s to remove residues and then washed again with distilled water for 30 s. Monobond Plus (Ivoclar Vivadent AG, Liechtenstein) was actively applied with a microbrush (Vigodent, Rio de Janeiro, Brazil) for 10 s, and after 5 min, cement with a matrix made of silicone by express addition (3M ESPE Dental, St Paul, Minnesota), measuring 1 mm × 5 mm × 10 mm and containing three cylindrical holes of 1-mm diameter (►**Fig. 1**), was applied.

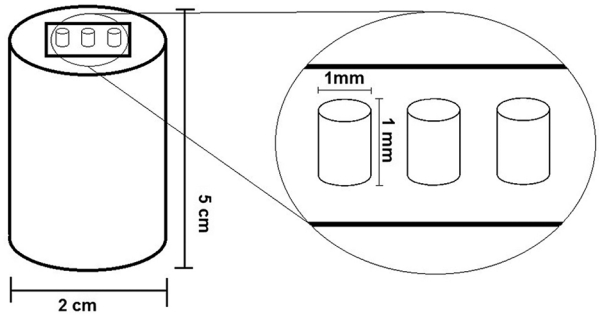

Fig. 1 Schematic representation of the experimental sample.

The matrix was placed on the surface of the blocks for construction of the TSs used in the microshear strength test. Self-adhesive resin cement (RelyX Unicem U200; 3M ESPE Dental, St Paul, Minnesota) was handled as per the manufacturer's instructions and inserted into the silicone matrix. This was placed on the zirconia samples and overlaid with a polyester strip held under digital pressure by a previously trained operator for 5 s; excess cement was removed.

Then, photoactivation was carried out for 20 s with Bluephase photopolymerizer (Ivoclar Vivadent AG, Liechtenstein) at a power of 1,200 mW/cm^2, directly on the polyester matrix; the matrices were removed. The photoactivator used was calibrated at every 10 uses to verify the intensity of the light supplied, using a radiometer (RD7; Ecel Indústria e Comércio Ltda, Ribeirão Preto, Brazil). The groups were divided into 11 TZ samples (TZ1 group) and 11 PAZ samples (PAZ1 group), which were maintained in distilled water at 37°C for 24 h. The remaining 11 TZ samples (TZ2 group) and 11 PAZ samples (PAZ2 group) were maintained for 90 days in distilled water at 37°C.

The TSs underwent microshear bond strength test in a universal test machine (EMIC DL-200 MF; EMIC, São José dos Pinhais, Brazil). With a metallic wire (Morelli; Sorocaba, Brazil) with a diameter of 2 mm placed around the TS, the loading force was applied at the base of the TS at a traction speed of 0.5 mm/min until fracture occurred (►**Fig. 2**).[25,26]

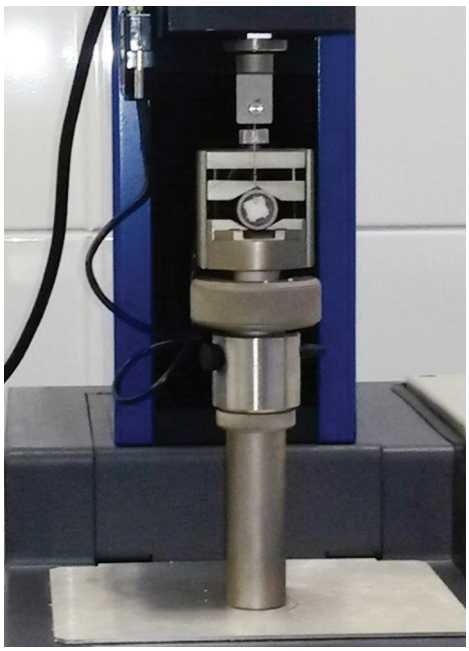

Fig. 2 Sample fixed to universal test machine.

The samples were analyzed using an Olympus SZX7 stereomicroscope (Tokyo, Japan) at ×7 magnification. The fracture pattern was classified (by a single evaluator) in adhesive and mixed types.

Representative fractured beams from each group were desiccated, mounted in aluminum stubs, and sputter-coated with gold to analyze the morphology of the fracture patterns with a scanning electron microscope (SEM, JEOL–6390 LV, Tokyo, Japan).

The mean MPa data (arithmetic mean calculated from three values obtained from the same block) were analyzed using Bioestat 5.3 software (Instituto de Desenvolvimento Sustentável Mamirauá; Tefé, Brazil). For this, the values were initially subjected to the D'Agostino test. The data were found to be normally distributed and thus were subjected to factorial analysis of variance (ANOVA) and Tukey's post hoc test ($p < 0.01$).

Results

Factorial ANOVA of ceramic brand × aging time showed statistically significant differences for both factors, but not for the interaction between them (►**Table 2**).

The two types of material showed significantly different bond strength [MPa; ►**Fig. 3** and ►**Table 3**], with the PAZ group showing the highest resistance to microshear after both 24 h and 90 days aging. The analysis of the same material showed a significant decrease after the storage period.

►**Fig. 4** presents descriptive analysis of the fractures, with the PAS group displaying more mixed fractures than TZ at both aging times. In both materials, mixed fractures are reduced after 90 days.

►**Figs. 5**–**8** show SEM analysis of the most representative samples from each experimental group.

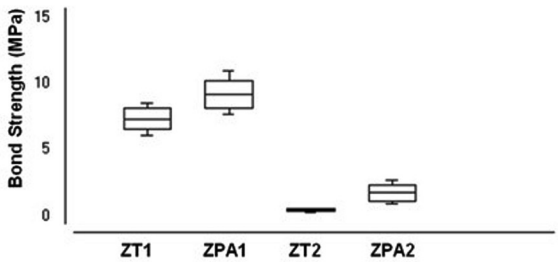

Fig. 3 Box-plot graph of the bond strength of the experimental groups (MPa).

Table 2 Factorial ANOVA for the experimental groups

Sources of variation	Degrees of freedom	Sum of squares	Mean squares	Calculated F	Critical F
Ceramic Brand	1	30.2685	30.2685	47.107	< 0.0001
Time	1	581.4255	581.4255	904.8773	< 0.0001
Interaction	1	1.5252	1.5252	2.3737	0.1276
Error	40	25.7018	0.6425		

Discussion

The PAZ samples showed significantly greater microshear bond strength compared to TZ samples, regardless of the immersion time. This is attributed to PAZ having higher yttrium oxide (Y_2O_3) and hafnium oxide (HfO_2) contents (12% of each) compared with TZ (6% Y_2O_3). This makes zirconia more stable, an important factor for maintaining the chemical bonds between zirconia, adhesive, and cement, which might help ensure intact phosphate bonds in the long term.[27]

Table 3 Mean values of resistance to microshearing of the experimental groups, followed by the respective standard deviations (MPa) and statistical analysis

Material/Time	24 hours	90 days
ZT	7.35 (± 0.76) [Aa]	0.45 (± 0.11) [Ab]
ZPA	9.16 (± 1.03) [Ba]	1.74 (± 0.57) [Bb]

Different letters in the same row or column mean statistically significant differences. Capital letters for columns and lower case letters for rows ($p < 0.01$).

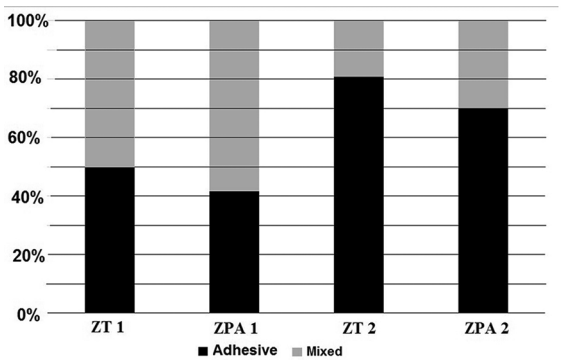

Fig. 4 Analysis of the frequency of fracture types (%).

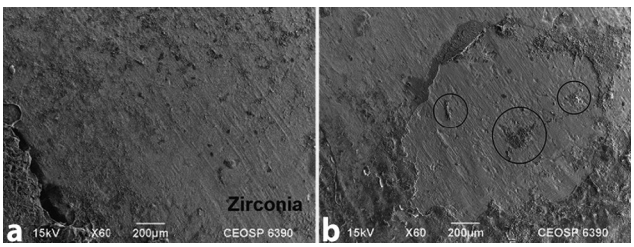

Fig. 5 Scanning electron micrograph of the ZT1 group. **(a)** Adhesive failure: It may be noted that no fragments of resin cement adhered to the surface of the zirconia. **(b)** Mixed failure: It is noted that cement adhered to the surface of the zirconia, as can be seen in the encircled areas.

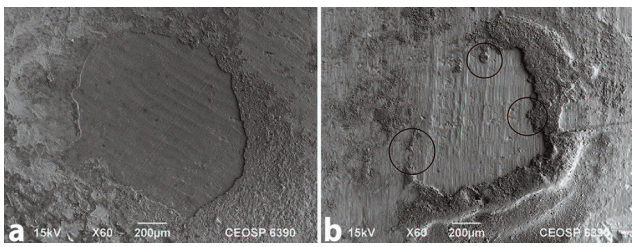

Fig. 6 Scanning electron micrograph of the ZPA1 group. **(a)** Adhesive failure: No fragments of resin cement adhered to the surface of the zirconia. **(b)** Mixed failure: It is noted on the surface of the zirconia resin cement adhered on the edges and part of the center of the surface where the cement post was located, as can be seen in the encircled areas.

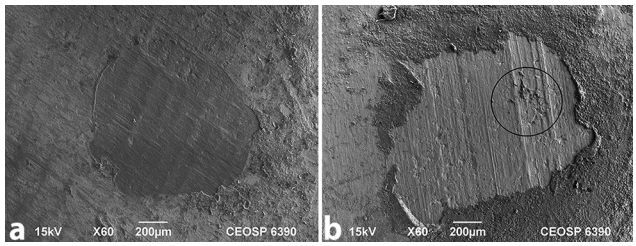

Fig. 7 Scanning electron micrograph of the ZT2 Group. **(a)** Adhesive failure: No resin cement adhered to the surface. **(b)** Mixed failure: There is a single area where resin cement is present in the encircled area.

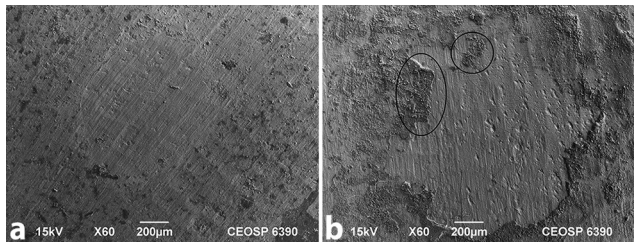

Fig. 8 Scanning electron micrograph of the ZPA2 group. **(a)** Adhesive failure: No resin is present on the surface, but some dirt appears. **(b)** Mixed failure: Areas are highlighted where resin can be observed after the microshear tests.

In the present study, the samples were aged by immersion in distilled water rather than by thermocycling, because a meta-analysis by Inokoshi et al[28] observed a stronger correlation between storage failure at 100% humidity and stable temperature ($r = -0.4$) than in thermocycling ($r = -0.15$). When using thermocycling to simulate aging, large numbers of cycles (100,000) have been suggested, so that a relevant effect can be found on the bonding surface, equivalent to approximately 3 months storage at 100% humidity, with similar results reported regarding the adhesion of enamel and dentine.[29] Considering the amount of time required to complete so many thermocycles, and the equipment needed for the process, aging in distilled water proves to be of great value due to its positive results and easy applicability.

In addition to analyzing the bond strength, the samples were subjected to fracture type analysis. The reduction of mixed fractures after prolonged storage, as shown in ►**Fig. 3**, probably occurred due to inherent degradation of resin cement and in the bond between the materials, directly affecting the results. Similar findings were observed in other studies that employed different forms of surface treatment.[13,16] Cohesive fractures were not found but were reported in other studies that subjected zirconia to microshear tests.[30]

Various surface treatments aim to increase the surface roughness[12,13,18] or the chemical bond between the components.[12,14,16,31] However, greater surface roughness is necessary to increase the total bond area, a characteristic that is easy to achieve and use in the case of ceramic elements.[28] Therefore, media blasting was used in this study. In addition, the application of adhesives containing monomers with methacryloyloxydecyl dihydrogen phosphate (MDP) increases the chemical

bond between material and cement,[32] becoming relevant in the choice of protocol employed.[22] This type of treatment shows the best laboratory results[28] and was therefore used here.

The SEM analysis revealed that a certain amount of resin cement remained adhered to the samples that suffered mixed failures.[27] No resin cement was present on the surface of the zirconia in the adhesive fracture; so in these cases, the failure occurred at the zirconia/cement interface, suggesting that there was insufficient chemical bond between the materials.[27] Comparing images between the different groups, there was no clear difference between the two materials at the same aging time. Comparison of the different aging times revealed more resin adhered in the zirconia in the samples that were aged for 24 h, compared to those aged for 90 days. This is likely due to greater degradation of the interface between the materials with longer aging time.

The use of an in vitro protocol has limitations for predicting the behaviors of materials in vivo. Nevertheless, it was possible to observe superior mechanical behavior for PAZ than for TZ, which suggests the use of PAZ in the routine of dentistry practices, due to its superior aesthetics and mechanical resistance and due to the support technology involved in this type of oral rehabilitation, provided by materials, software, and the CAD/CAM system, providing aesthetic and mechanical predictability for PAZ crowns.

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Conflicts of Interest
None.

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