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Bond Strength between Different Zirconia-Based Ceramics and Resin Cement before and after Aging

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Abstract: The objective of this study was to evaluate the bond strength of different stabilized zirconias with resin cement and evaluate the susceptibility to thermal aging of the adhesive interface. Zirconia discs (Vita Zahnfabrik, Bad Säckingen, Germany) were obtained: 3Y-TZP first generation (translucent), 3Y-TZP third generation (high-translucent), 4Y-PSZ (super-translucent), and 5Y-PSZ (extra-translucent). Each disc had its surface polished with a standardized protocol. The specimens were cleaned and sintered according to the manufacturer's recommendation (conventionally: ~12 h). However, 3Y-TZP groups were subdivided into subgroups and sintered following the speed sintering process (~80 min). After their sintering shrinkage, the dimensions of the final discs were 12 mm × 2 mm. The specimens were blasted with 50 µm aluminum oxide (1 cm distance, 2 bar pressure, and 2 s/cm^2), cleaned, and silanized with an MDP primer. After the surface treatment, a resin cement cylinder was built on the ceramic surface (Ø = 1 mm; h = 2 mm). Half of the specimens of each group were subjected to a microshear bond strength test in a universal testing machine after 24 h of cementation, while the other half were subjected to thermocycling prior to the bond strength test (6000 cycles; 5 °C–55 °C, 30 s for each bath). Bond strength data were submitted to two-way ANOVA and Tukey's test (95%), as well as Weibull analysis, to determine adhesive reliability. Bond strength was statistically different among the materials, and only 3Y-TZP third generation and 4Y-PSZ were not affected by thermal aging. The speed sintering method was statistically similar to the conventional process for 3Y-TZP first generation. However, 3Y-TZP third generation showed higher immediate bond strength when speed sintered. The Weibull modulus was superior for conventional 3Y-TZP third generation and 4Y-PSZ. In this study, thermal aging caused a degradation of the adhesive interfaces of 3Y-TZP first generation and 5Y-PSZ with the resin cement; however, it did not affect the interfaces of 3Y-TZP third generation and 4Y-PSZ. The speed sintering method did not affect the long-term bond strength with the resin cement. Adhesive reliability was superior for 3Y-TZP third generation and 4Y-PSZ.

Keywords: zirconia; dental materials; resin cements; microshear bond strength

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1. Introduction

Several dental ceramics have been developed with different compositions, microstructures, and indications. Efforts are directed concerning the development of biomaterials with adequate mechanical properties combined with favorable aesthetics. Zirconia is a polycrystalline ceramic widely used in dentistry that can present different translucency levels and compositions [1,2].

Zirconia is used in several dental specialties [3], and its mechanical properties are already well-established in the scientific literature as one of the strongest restorative materials [4,5]. Its clinical limitations are bond strength and translucency, which are inferior to reinforced glass-ceramics. Due to its polycrystalline microstructure, which allows it to achieve exceptional mechanical properties, its present increased opacity and decreased adhesive capacity compromising its indication for the rehabilitation of anterior teeth [1,6].

During the evolution of minimally invasive dentistry, dental preparations with selective wear and rational tooth removal emerged. Therefore, proper adhesion has become the primary factor of attention when considering rehabilitation with indirect restorations [1,6]. This promotes the bonding of the restorative material to the remaining dental element, ensuring the clinical longevity of the prosthetic treatment even when it does not have extra macro-retention [7–9]. Zirconia requires specific surface treatments to ensure adhesion with resin cements, mainly because acid etching is ineffective for this material [7]. Different surface treatment protocols have been used, such as surface abrasion with alumina particles followed by the application of primers or cements based on MDP (10-methacryloyloxydecyl dihydrogen phosphate) [8–17]. However, how it can affect the bond strength with the new generation of high-translucent zirconia materials is not clear. Despite that, clinical cases with high-translucent zirconia in the anterior areas have been reported as a new therapeutic modality [12,18].

In order to improve the translucency of dental zirconia, different microstructural modifications have been made to zirconia materials, involving the temperature and time parameters of the sintering cycles, the amount of alumina, the grain size, and the structure of the ceramic [10,11]. Additionally, an increase in translucency accompanies the increase in the amount of yttria, the stabilizing agent of the tetragonal phase. Therefore, these translucent 4Y- or 5Y-zirconias have lower mechanical properties (flexural strength, fatigue strength, hardness, and fracture toughness) than conventional 3Y zirconia [13]. Generally, microstructure modifications affect the adhesive behavior of the materials. Nevertheless, due to the recent development of translucent zirconia, the literature only demonstrates that the application of an MDP-based primer results in an improvement of the adhesion of translucent zirconia [14–19]. Studies on the adhesive performance and aging of interfaces are extremely important to predict their long-term survival [20–22].

Thus, the aim of this study was to evaluate the bond strength of different stabilized zirconia with resin cement and to evaluate the susceptibility to thermal aging of the adhesive interface. The null hypothesis was that there would be no difference between zirconia with different translucency levels for initial and long-term bond strength.

2. Materials and Methods

2.1. Specimens Preparation

Four different zirconia discs with different translucency levels and chemical compositions were used (Table 1):

- Translucent zirconia—3Y-TZP: 1st generation of 3 mol% Yttria-Tetragonal Zirconia partially stabilized (YZ T, Vita Zahnfabrik, Bad Säckingen, Germany);
- High-translucent zirconia—3Y-TZP: 3rd generation of 3 mol% Yttria-Tetragonal Zirconia partially stabilized (YZ HT, Vita Zahnfabrik, Bad Säckingen, Germany);
- Super-translucent zirconia—4Y-PSZ: 4 mol% Yttria-partially stabilized zirconia (YZ ST, Vita Zahnfabrik, Bad Säckingen, Germany), and;
- Extra translucent zirconia—5Y-PSZ: 5 mol% Yttria-partially stabilized zirconia (YZ XT, Vita Zahnfabrik, Bad Säckingen, Germany).

Table 1. Chemical composition of the zirconias that were used in the present study.

Components (Weight in %)	3Y-TZP 1st Generation	3Y-TZP 3rd Generation	4Y-PSZ	5Y-PSZ
ZrO_2	90–95	90–95	88–93	86–91
Y_2O_3	4–6	4–6	6–8	8–10
HfO_2	1–3	1–3	1–3	1–3
Al_2O_3	0–1	0–1	0–1	0–1
Pigments	0–1	0–1	0–1	0–1

Legend: ZrO_2—zirconia oxide; Y_2O_3—yttrium oxide; HfO_2—hafnium oxide; and Al_2O_3—aluminum oxide.

The discs were submitted to a circular sample cutter to obtain cylinders measuring 18 × 16 mm. Then, the cylinders were fixed in a precision cutting machine (ISOMET 1000, Buehler, Lake Bluff, IL, USA) with a diamond disk under constant cooling to obtain circular specimens measuring 3 mm thickness.

Subsequently, the sample surfaces were regularized and polished with sandpaper of increasing granulation (#600, #800, and #1200). The final dimensions of the discs were 16 mm in diameter and 2.5 mm in thickness. The total number of specimens per group (n = 20) was based on the sample size calculation through power analysis, considering 95% reliability for the difference among the groups.

The discs were cleaned with an ultrasonic bath in isopropyl alcohol and distilled water for 2 min. After cleaning, the samples were sintered according to the manufacturer's protocol to obtain the final mechanical and optical properties. For 3Y-TZP, two different sintering protocols were used to make additional groups. The sintering parameters are presented in Table 2, except for the speed sintering programs that are not available from the manufacturer (total time: 80 min).

Table 2. Parameters of the sintering cycles indicated for each of the zirconias used in the present study.

Programs	$T_{initial}$ °C	$\nearrow$ min.	$\nearrow$ °C/min.	T_{max} °C	$\rightarrow$ min.	$\searrow$ T_{final} °C
3Y-TZP 1st gen	25	88:32	17	1530	120:00	200
3Y-TZP 3rd gen	25	83:49	17	1450	120:00	200
4Y-PSZ	25	188:08	8	1530	120:00	200
5Y-PSZ	25	356:15	4	1450	120:00	200

The steps above are: initial temperature, time to reach the maximum temperature, rate cooling, maximum temperature, sintering time (time in T_{max}), and final temperature (opening oven).

Considering the sintering shrinkage of 20% of the volume, the discs showed final dimensions of 12 mm in diameter and 2 mm in thickness.

2.2. Surface Conditioning and Resin Cement

Following the manufacturer's surface treatment protocol, all samples were blasted with 50 μm Al_2O_3 at 1 cm distance, 2 bar pressure, and 2 s/cm^2. The samples were cleaned with an ultrasonic bath in isopropyl alcohol and distilled water for 2 min, and then silane containing MDP (Monobond N, Ivoclar Vivadent, Schaan, Liechtenstein) was applied to the surface. The base and catalyst pastes of resin cement (Variolink N, Ivoclar Vivadent, Schaan, Liechtenstein) were mixed, and the material was placed in silicone matrices (diameter of 1 mm and 2 mm height) with the aid of an injection syringe (Centrix, Nova DFL, Rio de Janeiro, Brazil) and light cured for 60 s (1200 mW/cm^2—Radii Cal, SDI, Victoria, Australia). The silicone matrices were removed after the cement polymerization, and then a resin cement cylinder was obtained on the ceramic surface (Ø = 1 mm and h = 2 mm). Half of the samples were placed in distilled water and subjected to a microshear test after 24 h. The other half of the specimens were subjected to thermal aging for 6000 cycles [23–25] with baths of 5 °C and 55 °C, with 30 s for each bath. After thermocycling, the aged specimens were also subjected to the microshear test.

2.3. Microshear Bond Strength Test

The microshear test was performed in a universal testing machine (DL-1000 EMIC, São José dos Campos, Brazil), and the load was applied to the cylinder base using a steel wire loop (0.3 mm in diameter) at a 0.5 mm/min speed and using a load cell of 250 N until failure (Figure 1). The bond strength was calculated by the formula:

$$R = F/A$$

where R is bond strength (MPa); F is force (N); and A is interface area (mm^2).

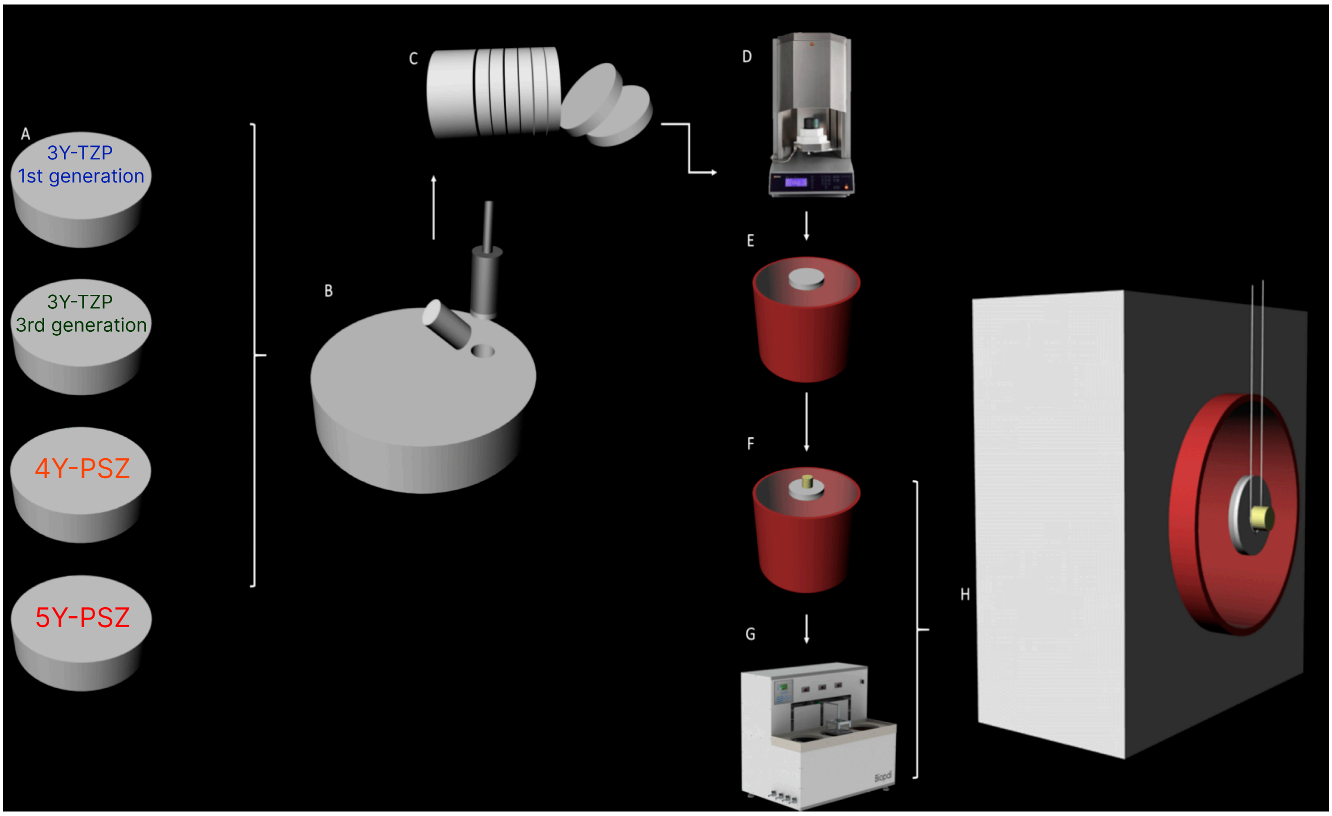

Figure 1. A–H—Graphical summary of the sample preparation process until testing. A—Selection of four translucent zirconias; B—circular cutting bur; C—section of the cylinder in thinner specimens; D—sintering the specimens; E—placing the specimens in a horizontal plane; F—fabrication of the resin cement cylinder on the ceramic surface; G—thermal aging of half of the samples and; H—microshear test to obtain the bonding strength.

2.4. Failure Analysis

All fractured surfaces were examined under a stereomicroscope (Stereo Discovery V20, Zeiss, Göttingen, Germany), and the types of failure were classified as: A—adhesive at the interface between ceramic and cement; B—cohesive of the ceramic; C—cohesive of the cement; and D—mixed (adhesive at the interface between ceramic and cement + cohesive failure of the cement). Representative images were observed via scanning electron microscopy (Vega 3, Tescan, Brno, Czech Republic).

2.5. Data Analysis

Minitab statistical software (Minitab LLC., State College, PA, USA) was used. The adhesive strength data in MPa were tested for normality for each of the groups. Then, relevant statistics were performed to compare the factors "material" and "aging". After confirming normality, the parametric test was adopted, using a two-way analysis of variance (ANOVA) and Tukey's test, both with a significance level of 5%.

To determine the adhesive reliability, a Weibull analysis was performed using the least squares method, also with a significance level of 5%. The Weibull modulus determines the structural homogeneity of the adhesive interfaces (reliability), and with this analysis, the characteristic bonding strength was also calculated, which is the strength where 63.2% of the adhesive interfaces will fail [26].

3. Results

A descriptive statistical analysis was performed, which is represented in Figure 2. After the inferential analysis with two-way ANOVA (material and aging), it was possible to observe that there was no statistical difference among the materials ($p = 0.551$). However, there was a statistical difference among the groups after aging ($p = 0.001$).

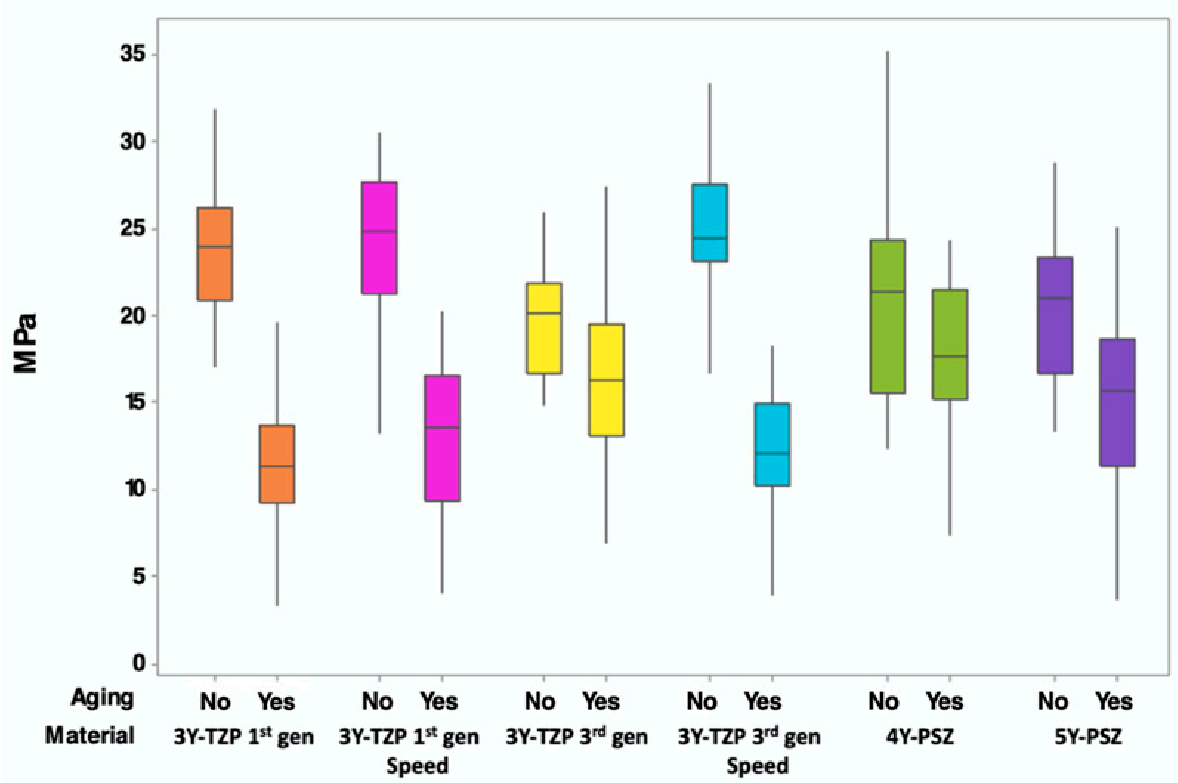

Figure 2. Boxplot of the average bond strength of all studied groups.

Table 3 shows the results of the Tukey test (95%). Speed-sintered 3Y-TZP third generation, 3Y-TZP first generation (conventional and speed), and 5Y-PSZ showed lower bond strength results after aging. Speed-sintered 3Y-TZP third generation showed higher bond strength than conventionally sintered 3Y-TZP third generation without aging, whereas 3Y-TZP first generation and aged 3Y-TZP third generation did not show statistical differences depending on the sintering mode (conventional x speed).

Table 3. Mean, standard deviation, and Tukey's test (95%) of bond strength values according to the groups.

Aging	Group	Mean ± SD	Grouping (Tukey 95%) *						
No	3Y-TZP 3rd gen Speed	24.38 ± 5.41	A						
	3Y-TZP 1st gen Speed	24.02 ± 5.00	A	B					
	3Y-TZP 1st gen	23.99 ± 3.92	A	B					
	4Y-PSZ	21.15 ± 6.09	A	B	C				
	5Y-PSZ	20.39 ± 4.21	A	B	C				
	3Y-TZP 3rd gen	19.28 ± 4.48		B	C	D			
Yes	4Y-PSZ	17.52 ± 4.22			C	D	E		
	3Y-TZP 3rd gen	16.32 ± 5.18			C	D	E	F	
	5Y-PSZ	14.68 ± 6.15				D	E	F	G
	3Y-TZP 1st gen Speed	12.76 ± 4.35					E	F	G
	3Y-TZP 3rd gen Speed	12.14 ± 3.90						F	G
	3Y-TZP 1st gen	11.15 ± 3.87							G

* Different letters indicate $p < 0.05$.

Regarding the Weibull analysis (Table 4), the characteristic strength did not show any statistical difference among the groups. However, according to the interposition of confidence intervals, it is possible to observe that the conventional sintered 3Y-TZP third generation, 4Y-PSZ, and 5Y-PSZ groups had a higher Weibull modulus, but 5Y-PSZ was also similar to the other groups.

Table 4. Data from the Weibull analysis.

Material	Weibull Modulus	CI Modulus	Characteristic Resistance (σ_0)	CI * σ_0
3Y-TZP 1st gen	2.31 ± 0.23 B	1.89–2.81	17.44 ± 1.02	15.54–19.58
3Y-TZP 1st gen Speed	2.57 ± 0.26 B	2.11–3.14	18.64 ± 0.98	16.80–20.67
3Y-TZP 3rd gen	3.94 ± 0.40 A	3.22–4.82	19.12 ± 0.65	17.87–20.45
3Y-TZP 3rd gen Speed	2.40 ± 0.24 B	1.97–2.93	18.33 ± 1.03	16.41–20.48
4Y-PSZ	3.84 ± 0.35 A	3.19–4.60	20.63 ± 0.73	19.24–22.12
5Y-PSZ	3.06 ± 0.32 AB	2.48–3.77	18.51 ± 0.81	16.98–20.17

* CI indicates the confidence interval.

For the failure analysis, no cohesive failures of the zirconia or resin cement were found. However, adhesive failures and mixed failures were observed, in which there was a cohesive failure of the resin cement despite being predominantly adhesive. Representative specimens were observed in SEM to exemplify the two types of failure (Figure 3). It was not possible to identify any failure pattern according to the study groups. However, the failures were mostly 100% adhesive, with sporadic and similar mixed failures among all groups.

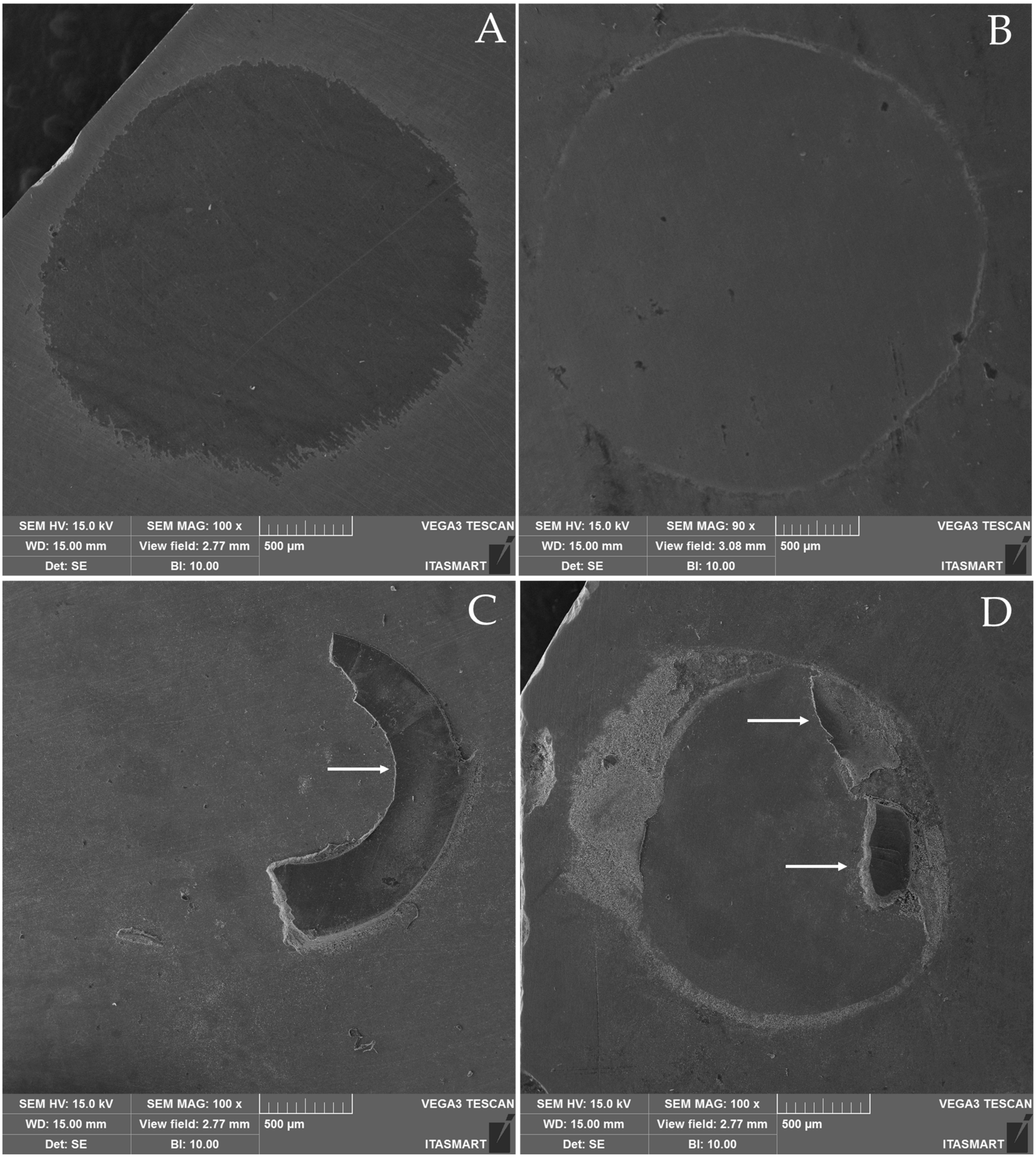

Figure 3. Representative SEM images of failure types. (**A**,**B**) Examples of adhesive failures on speed-sintered 3Y-TZP third generation and 5Y-PSZ, respectively. (**C**,**D**) Examples of mixed failures, in which the arrows point to the cohesive failure fractions of resin cement in conventionally sintered 3Y-TZP third generation and 4Y-PSZ, respectively.

4. Discussion

According to the analysis of the results, it was possible to observe that the only material that showed similar bond strength before and after aging was 4Y-PSZ; all other groups had lower bond strength values after thermocycling. It was also possible to observe that the sintering protocol, whether speed or conventional, did not affect the long-term bond strength of 3Y-TZP, since the values obtained were statistically similar. Thus, the results obtained demonstrate that the hypothesis of this study can be considered rejected, since there was a difference in the bond strength between the different ceramics and the resin cement.

Another investigation [27] evaluated 3Y-TZP through a shear bond strength test before and after thermal aging with 10,000 cycles, using a similar cementation procedure as used in this study. However, the average bond strength values obtained for 3Y-TZP were slightly below the values obtained herein, being 18.11 MPa before and 6.58 MPa after aging. This difference can be explained by the other group's use of a macroshear test rather than the microshear test used here; the macroshear test has a higher probability of including defects at a larger adhesive interface [28], and the study implemented a higher number of thermal cycles than in the present study. Therefore, although these two factors contributed to the obtainment of lower bond strength values, both investigations showed a pattern of decrease in bond strength after thermocycling for this material.

The third generation of 3Y-TZP presents a reduction in the number and size of alumina (Al_2O_3) grains and, consequently, their reallocation in the ceramic structure, causing an increase in translucency when compared to the first generation [1]. This zirconia can also be sintered in two modes, with it being possible to observe that in the conventional sintering there was no thermal degradation of the adhesive interface as there was in the speed sintering mode, in which the value of the bond strength was reduced after the thermocycling. The results of the bonding strength of the third generation of 3Y-TZP after aging obtained in this study corroborate a previous report that reported adequate bond strength for this dental biomaterial [29].

The 4Y-PSZ, called super-translucent zirconia (according to the manufacturer), had no indication of sintering in speed mode, only with the conventional method. This 4Y-PSZ differs from the first and third generations of 3Y-TZP in its chemical composition, and has greater translucency than previous generations; this is achieved through the introduction of an optically isotropic cubic phase with a higher content of yttria for its stabilization [30–32]. This zirconia showed similar bond strength before and after aging. Franco-Tabares et al. [27] also found lower values as an average of its resistance after thermocycling (5.99 MPa), although they used the same justification regarding the number of cycles used and the specificity of the chosen test, maintaining the pattern of decrease after thermal aging for 4Y-PSZ.

The 5Y-PSZ, or extra-translucent zirconia, can only be used with the conventional sintering mode. It is the most translucent zirconia among the available generations. It has 5% mol of yttria with reduced grain size and a greater amount of cubic phase [1], to the detriment of its mechanical strength, which, due to the small amount of the transformation of crystals, is lower than that of the previous generations. The average bond strength obtained from this zirconia in the present study corroborates values obtained by others [27,30]. After the thermal aging procedure, there was a reduction in the bond strength values for 5Y-PSZ from 20.39 MPa to 14.68 MPa (≈28%), which corroborates the values that can be found in the literature [31].

In addition to the restorative material, the durability of a cement layer is an important aspect for defining restoration longevity since the degradation of the adhesive interface promotes failure between the restorative material and the dental element [20]. Several studies indicate that thermocycling is an excellent method for evaluating the effectiveness of long-term adhesion [21,22]. The microshear test is one of the possible methods to be used when testing the bond strength of dental materials [32,33]. It is schematically equal to the shear test, but is performed on a smaller scale. Consequently, the force incident on the material is more concentrated, which gives more specificity and reliability to the obtained data. This test has widespread use due to its simplicity and the ease of specimen preparation, and has a considerable advantage over the micro-tensile test, with no incorporation of residual stresses during sample processing [23,24]. In the present study, the device used to apply force on the resin cement was a 0.3 mm-diameter steel wire instead of a chisel, since the chisel produces a higher stress concentration during the incidence of force, which may lead to the underestimation of bond strength results [34].

Speed sintering (80 min) was used in this study for 3Y-TZP groups. Kaizer and collaborators [35] showed that speed sintering modifies the microstructure, physical properties, and wear behavior. As the temperature increases, the grains are sintered, and the existing pores are reduced by diffusion [36,37]. Therefore, the density of zirconia is also changed depending on the sintering cycle [37–39]. Furthermore, the higher the temperature and the longer the sintering time, the larger the grain size [10]. These microstructural changes alter properties such as flexural strength, contrast ratio, and translucency [38]. Given this background, it would be possible to expect a different bond strength. The present study showed that the adhesive behavior was similar for this zirconia regardless of the sintering mode, and more studies are needed to draw conclusions regarding the comparison of bond strength data between different sintering protocols.

The Weibull modulus represents the structural reliability. In this study, when based on the bond strength values, it represents the reliability of the adhesive interface. The characteristic resistance represents the probability that 63.2% of the interfaces will fail [26,36]. These estimates are widely used for the analysis of ceramic materials, as it becomes possible to determine the reliability according to the intrinsic and/or superficial defects of these materials. However, this estimate is not widely used in bond strength studies, despite being valuable for predicting the behavior of adhesive interfaces. Based on the present results, it can be noted that 4YPSZ, 5Y-PSZ, and 3Y-TZP third generation zirconia with conventional sintering obtained the highest Weibull modulus compared to other zirconia generations, confirming their promising adhesion, where the higher the Weibull modulus, the greater the adhesive reliability.

According to the findings of this study, it is possible to observe that the long-term bond strength of polycrystalline zirconia-based materials can be affected by the translucency level of the material. The limitations of this study include the fact that it is an in vitro study. Therefore, methods that can partially simulate oral conditions were chosen, such as thermal aging and the microshear test. Tensile and shear stress are found in the adhesive interface of ceramic and teeth, and any method that considers only one of these stress types is limited; however, the majority of data in the literature were obtained by methods using isolated tensile or shear stress [38,39]. Among all zirconias subjected to microshear, speed-sintered 3Y-TZP third generation without thermal aging obtained the highest bond strength. Among the aged samples, 4Y-PSZ obtained the highest bond strength values. It is recommended to use the speed cycle when possible, in this case only for both 3Y-TZP, since this presented similar bond strength to materials sintered by the conventional cycle. The results indicate that the restoration's processing time can be greatly optimized with the use of these materials. Amongst the materials evaluated, 4Y-PSZ had the highest stability in long-term bond strength with the resin cement. However, 5Y-PSZ, despite being affected by aging, had similar bond strength with the resin cement to 4Y-TZP after aging.

5. Conclusions

On the basis of all the information provided by this study, and even with its limitations, it can be concluded that:

1. The sintering mode (speed or conventional) did not affect the long-term bond strength between 3Y-TZP and resin cement;
2. The long-term bond strength of polycrystalline zirconia-based materials is different depending on the zirconia translucency level;
3. Conventionally sintered 3Y-TZP third generation and 4Y-PSZ have higher adhesive reliability than the other evaluated zirconia materials.

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