



# Effect of several repair techniques on the bond strength between composite resin and degraded Y-TZP ceramic

Efeito de várias técnicas de reparo na resistência de união entre resina composta e zircônia degradada

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## ABSTRACT

**Objective:** To evaluate the bond strength of different repair treatments for composite resin to aged Y-TZP ceramics. **Material and Methods:** Zirconia blocks were cut into smaller specimens, sintered according to manufacturer's recommendations (final dimensions of 4×4×3 mm), and randomly allocated into nine groups (n=15) according to the surface treatment and presence/absence of aging of the substrate (subjected to low-temperature degradation - LTD), as follows: without LTD (Control: without treatment; TBS: tribochemical silica coating + silane + adhesive); with LTD (Control-LTD: without treatment; TBS-LTD: TBS with hydrothermal degradation; MoS-LTD: Monobond S + adhesive; MoP-LTD: Monobond Plus + adhesive; MZP-LTD: Metal/Zirconia Primer + adhesive; USB-LTD: Single Bond Universal; AP-LTD: Alloy primer + adhesive). LTD was simulated in an autoclave (134 °C, 2 bar, 5 h). The ceramic blocks were embedded in PVC cylinders with a self-curing acrylic resin; each surface treatment protocol was performed; a composite resin cylinder (Ø: 3.25 mm and height: 3 mm) was then build-up using split metallic matrices. All the specimens were aged (thermocycling + storage in water for 90 days) and subjected to the shear bond strength test using a universal testing machine (1 mm/min). The failure mode was classified into four types: adhesive, composite resin cohesive fracture, ceramic cohesive fracture, and mixed. The bond strength values were subjected to Mann-Whitney test. **Results:** Only air-abraded samples (TBS and TBS-LTD) survived thermocycling. More than 80% of the samples of the other groups presented pre-test failures. TBS groups presented higher values of bond strength (3.94) compared to TBS-LTD (0.96). The predominant type of failure for the surviving samples were adhesive. **Conclusion:** Air particle abrasion is mandatory to improve the bond strength of the Y-TZP substrate; an aged substrate presents an even more unfavorable scenario for adhesion.

## KEYWORDS

Dental prosthesis repair; Hydrothermal degradation; Zirconia; Shear bond strength; Sandblasting.

## RESUMO

**Objetivo:** Avaliar a resistência de união ao cisalhamento gerada por diferentes técnicas de reparo em resina composta aderida em zircônia envelhecida (sujeita a degradação a baixa temperatura - LTD). **Material e Métodos:** blocos de zircônia foram seccionados em espécimes, sinterizados de acordo com o fabricante (dimensões finais de 4x4x3 mm), e aleatorizados em nove grupos (n=15) de acordo com o tratamento de superfície e presença/ausência de envelhecimento do substrato (LTD), conforme: sem LTD (Control: sem tratamento; TBS: tribosilicatização + silano + adesivo); com LTD (Control-LTD: Monobond S + adesivo; MoP-LTD: Monobond plus + adesivo; MZP-LTD: Meta/Zirconia primer + adesivo). LTD foi simulada em autoclave (134 °C, 2 bar, 5 h). Os blocos de cerâmica foram embutidos em cilindros de PVC com resina acrílica autopolimerizável; cada tratamento de superfície foi realizado; um cilindro de resina composta (Ø: 3,25 mm 3 mm de altura) foi confeccionado usando matrizes metálicas bipartidas. Todos os espécimes foram envelhecidos (termociclagem + armazenagem em água por 90 dias) e sujeitos ao teste de cisalhamento usando um equipamento de teste universal (1 mm/min). O modo de falha foi classificado em quatro grupos: adesivo, fratura coesiva em resina composta, fratura coesiva da cerâmica, e mista. Os valores de adesão foram sujeitos ao teste de Mann-Whitney. **Resultados:** somente as amostras jateadas (TBS e TBS-LTD) sobreviveram a termociclagem. Mais que 80% dos espécimes dos outros grupos apresentaram falhas pré-teste. Os grupos TBS apresentaram os maiores valores de resistência ao cisalhamento (3,94) comparado com TBS-LTD (0,96). Os tipos predominantes de falha para os espécimes sobreviventes foi adesiva. **Conclusão:** o jateamento é recomendável para aumentar a resistência adesiva à zircônia; um substrato envelhecido apresenta um cenário mais desfavorável de adesão.

## PALAVRAS-CHAVE

Reparo em prótese dental; Degradação hidrotérmica; Zircônia; Resistência de união ao cisalhamento; Jateamento.

## INTRODUCTION

Yttria-stabilized polycrystal tetragonal zirconia (Y-TZP) consists of a polycrystalline ceramic material characterized by high flexural strength, fracture toughness, and fatigue resistance [1]. However, it exhibits high opacity, thus requiring a veneering material made of a vitreous-based ceramic for improved aesthetic results [2]. Unfortunately, the fractures or delamination/chippings of this veneer ceramic have been recognized as the major complication of this type of restoration [2]. In several cases, even the exposure of the infrastructure material may occur after chipping [3]. Hence, in such scenarios, the use of intra-oral methods of repair (based on the use of composite resins) may be a conservative alternative that can increase the lifetime of this restoration until it finally undergoes a real need of replacement [4].

Although zirconia consists of a chemically biological inert material (which is a beneficial characteristic), it presents a clinical challenge in a restorative dentistry context, which is the difficulty in achieving adequate adhesion [1]. The gold-standard surface treatment for enhanced adhesion on porcelain materials (glass-ceramics) involves etching with hydrofluoric acid (5% to 10%) followed by the application of a silane-bonding agent, which, when associated with resin cements, results in increased bond strength [5]. Nevertheless, this traditional adhesive technique is not effective for zirconia-based ceramics, due to its crystalline nature and non-reactive surface, which renders them resistant to the effects of etching with hydrofluoric acid [6].

Therefore, zirconia-based ceramics require alternative methods of surface modification for adhesion. Accordingly, numerous techniques have been proposed in the literature. Among them, the most commonly used technique is tribochemical treatment [7], which includes air abrasion with aluminum particles coated with silica followed by the application of a silane-based primer; or air abrasion with alumina particles [4] followed by surface activation with adhesion promoters (primers). Both alternatives

are based on simultaneously obtaining a chemically reactive surface (chemical adhesion) and an increased surface roughness, which would propitiate mechanical interlocking [1].

However, some studies showed that the air abrasion of Y-TZP ceramics with particles may lead to material degradation owing to the introduction of micro-cracks in the material surface by phase transformation ( $t \rightarrow m$ ), which may lead to posterior catastrophic fracture under mechanical loading/stimuli [8]. Therefore, other alternatives were proposed based solely on the use of chemical agents (primers) for zirconia, focusing on the improvement of chemical adhesion. However, previous literature still shows decreased stability of bonding after storage and thermocycling, used for artificially inducing cement hydrolysis and degradation of bonding interface [9].

As mentioned above, Y-TZP ceramics is considered a biologically inert material; however, although this fact appears undeniable [10], it is also true that this material exhibits a metastable characteristic, which leads to phase transformations that can be triggered by different stimuli. Such phase transformation mechanism is the main reason for an immediate increase in the mechanical properties but also for the long-term degradation due to a process known as low-temperature degradation (LTD) leading to a superficially increased roughness, changes of the material density, and finally compromising the mechanical properties [11].

Thus, after installment, the final prosthetic restoration becomes exposed to the harsh oral environment. In a scenario where this restoration results in a chipping fracture and a clinician may attempt a repair technique using a composite resin, it is important to consider that Y-TZP ceramic is probably present in a degraded stage, with the presence of m-phase content on its surface. Thus, clinicians have to consider and choose the best protocol for bonding with such surface, although literature is scarce and data is lacking to guide such choices. Hence, the present study aimed to evaluate: (1) the influence

of different treatments of Y-TZP ceramics hydrothermally degraded in the bond strength to composite resin; (2) the adhesion between specimens treated with primers with and without phosphate monomers - MDP (without air abrasion). The null hypotheses were: (1) The surface pretreatment will not improve the adhesion of composite resin to Y-TZP ceramic; (2) The LTD will not affect the bond strength of composite resin to Y-TZP ceramic.

## MATERIAL AND METHODS

### Sample size calculation

A sample calculation using a bicaudal test was performed in OpenEpi program (Version 2) to estimate the number of specimens required for the bond strength test. The estimate was based on the findings of Vanderlei, et al. [12], considering the difference between groups with means  $7.9 \pm 2.6$  and  $13.3 \pm 4.1$  MPa, because these were the groups presenting statistically significant difference and the smallest difference between the means. Applying these parameters, 80% power, and a significance level of 5%, seven specimens per group were determined to be necessary. However, we decided to adopt 15 specimens per group in this study to obtain a safety margin.

### Sample preparation

With the aid of a cutting machine (IsoMet® 1000 / Buehler, Lake Bluff, USA), ceramic blocks of Y-TZP (VITA In-Ceram 2000 YZ cubes for inLab, Bad Sackingen, Germany) were cut, thus obtaining 135 specimens (4 mm height) with an adhesive surface area of approximately  $5 \times 5$  mm, which were subsequently polished with Sof-Lex discs (3M/ESPE) and 1200-grit silicon carbide papers so that their surfaces were regularized and any defect introduced by cutting was removed. The specimens were sintered according to the manufacturer's recommendations (heat rate 1:  $60^\circ\text{C}/\text{min}$  up to  $700^\circ\text{C}$ ; heat rate 2:  $60^\circ\text{C}/\text{min}$  up to  $1300^\circ\text{C}$ ; heat rate 3:  $40^\circ\text{C}/\text{min}$  up to  $1530^\circ\text{C}$ ; dwell time: 25 min, followed by slow cooling by opening the furnace at temperatures

below  $400^\circ\text{C}$ ), resulting in the final dimensions of approximately  $4 \times 4$  mm of bonding area  $\times 3$  mm (height) and subsequently subjected to ultrasonic cleaning with isopropyl alcohol for 10 min.

After the preparation, 105 specimens were subjected to LTD in an autoclave (Sercon HS1-0300 n11560389/1, Mogi das Cruzes, Brazil), at  $134^\circ\text{C}$ , 2 bar pressure for 5 h, which corresponded to approximately 15–20 years *in vivo* [13]. The monoclinic phase content should not exceed the maximum of 25% for Y-TZP implants to be considered suitable for biomedical purposes after aging under this protocol [14].

The remaining specimens (45) were not subjected to LTD. Subsequently, the specimens were divided into 9 groups (Table 1), considering the factor surface treatment (Table 2).

### Shear bond strength test (SBS)

Each specimen of Y-TZP was embedded with acrylic resin of chemical polymerization (VIPI Flash, VIPI, Pirassununga, Brazil), inside of a plastic cylinder (h = 14 mm and  $\varnothing = 25$  mm). The specimens were fixed with a double-sided tape onto a glass plate. Subsequently, the plastic cylinder was positioned and the acrylic resin was placed on the plastic cylinder, but leaving a surface of ceramic to the adhesion of acrylic resin.

Specimens surfaces were treated according to the description presented in Table 2. All the groups received a layer of adhesive (Adper Scotchbond Multi-purpose Plus Adhesive, 3M ESPE, St Paul, USA), except the single bond universal group (SBU, 3M ESPE, St Paul, USA). Subsequently, split metallic matrices ( $\varnothing = 3.25$  mm and height = 3 mm) were positioned on the treated zirconia surface, and the composite resin (Filtek Z350 XT, 3M ESPE, St Paul, USA) was inserted into the matrices (two increments: approximately 1.5 mm and 10 s of curing in the first increment and 1.5 mm and 40 s of curing in the second increment) (Radii-cal, SDI; Bayswater, VIC, Australia - power output of  $1200 \text{ mW}/\text{cm}^2$ ). The circular cross-section adhesive area was  $8.3 \text{ mm}^2$ .

The shear test was performed using a universal testing machine (EMIC DL-2000 Pinhais, Brazil) at a speed of 1 mm/min, and the load was applied perpendicular to the adhesive interface until the occurrence of the failure. The test was conducted with a chisel device. The bond strength was calculated as  $R = F/A$ , where R is the resistance (MPa), F is the charge required to break the specimen (N), and A is the interfacial area of the specimen (mm<sup>2</sup>).

### Thermocycling

The groups were subjected to thermocycling (number of cycles: 10,000; temperature: 5 °C–55 °C with 30 s in each bath and 4 s between baths, Nova Etica, Sao Paulo, Brazil) and subsequently stored in distilled water at 37 °C for 90 days before the specimens were shear tested.

### Analysis of failure mode

All the samples were analyzed under stereomicroscopy (Discovery V20, Carl-Zeiss, Gottingen, Germany) with increased magnification (10-60x) to identify the type of failure, which were classified into: 1) adhesive (ADES): fracture at the interface between adhesive/primer and ceramic; 2) composite resin cohesive fracture (COES-CR); 3) ceramic cohesive fracture (COES-cer); 4) mixed (MIST): adhesive fracture combined with composite cohesive fracture.

### Statistical analysis

The nominal values of bond strength were recorded in MPa and tabulated in a statistic software (SPSS version 21, IBM, Chicago, Illinois, USA). Specimens that failed during the thermocycling were assigned a bond strength value of 0.01 MPa. In order to verify the normality of distribution, we used the Shapiro–Wilk test. The bond strength data were not parametric; therefore, the values were subjected to the Mann–Whitney test (5%).

## RESULTS

Only TBS and TBS-LTD groups presented specimens that survived to thermocycling. All

specimens of the remaining groups (those not air abraded) failures during the aging. More than 50% of specimens of TBS and TBS-LTD had SBS values after thermocycling. Hence, we assigned a bond strength value of 0.01 MPa to the other half of the specimens of these groups. The mean values and pre-test failure numbers (%) are presented in Table 3.

## DISCUSSION

The null hypothesis (1) was rejected because air-abraded groups (TBS-LTD, TBS) exhibited statistical difference in relation to the other groups. All the groups that were not previously air abraded exhibited high incidence of spontaneous debonding during thermocycling (Table 3). This can be due to the weak bonding between Y-TZP and composite resin, highlighting the importance of the surface pretreatment of zirconia. Besides, it also has to be considered that the aging protocol adopted herein (thermocycling for 10,000 cycles + storage for 90 days) may consist in an extremely harsh aging scenario, especially in comparison to protocols in the previous literature (fewer cycles or less time, rarely associated); we emphasize that we opted for this protocol exactly due to its harsh characteristics, to elucidate a potential to enhance adhesion even in such environment.

Recently, many adhesion studies of ceramics are being published with thermal aging protocols above to the one recommended by ISO 11405:2003 (thermocycling test comprising of 500 cycles in water between 5 °C and 55 °C, starting after 20 h to 24 h storage in water at 37 °C and/or long-term test after six-month storage in water at 37 °C). In the current study we used 10,000 cycles and storage for 90 days. The use of such protocol could be the most probable reason for the low values obtained on shear bond strength test. It is stated that thermocycling and the water storage are aggressive environments to adhesive interfaces that degrades the bond strength [15] and the long artificial aging is important to water saturation in composites, leading to hydrolytic degradation of the adhesive interface [16]. According to Sarret, et al. [17]

most of water absorption of composites occurs at the first two months of storage. So, although such aging protocol did not mimic clinical environment it consists on a valid tool to assess adhesion stability, in this sense the performance observed was very concerning and more studies should be made to attempt on optimization of such outcome.

The air abrasion with alumina particles coated with silica under pressure modifies the zirconia surface, thus promoting an increase of roughness [4] and chemical compatibility with adhesive primers [1]. Thus, possibly, the absence of mechanical interlocking on non-air-abraded groups favored water penetration (hydrolysis of adhesive interface) causing debonding failures [18].

In the current study, traditional/conventional (silane–Monobond S), MDP (ester phosphate groups—Monobond Plus, Alloy Primer and Single Bond Universal), and phosphonic acid acrylate (MZP) primers were used without air abrasion of the surface as pretreatment. In such groups the adhesive interaction forces are formed between functional monomers and metal ions [19]. As zirconia ceramics consist of metal oxides, adhesive monomers may interact with these oxides forming chemical bonds, such as van de Waals forces or hydrogen bonds on the adhesive interface (resin–zirconia) and improve the surface wettability of zirconia [20].

The groups that were not air-abraded (AP-LTD, Control-LTD, USB, Control, MoP-LTD, MoS-LTD, and MZP-LTD), no matter the monomer type, exhibited high percentage of pre-test failures during thermocycling (Table 3) indicating that the absence of mechanical interlocking promoted by air abrasion negatively affected the adhesion between the composite resin and aged Y-TZP. These results are in accordance with Pereira, et al. [21]. Yang, et al. [25] showed that the initial bond strength of zirconia specimens without air abrasion and chemically treated with phosphate primers was improved; however, after long-term storage, the chemical bonds were not water resistant. Hence, the mechanical pretreatment may be essential to

achieve adequate adhesion in association with a chemical step (primer) [4,26,27].

Some studies described interesting adhesion results between MDP-based primers (10-methacryloxydecyl dihydrogen phosphate monomer) and Y-TZP ceramics owing to the supposed adhesive interaction of the ester phosphate groups with the zirconia metal oxides [28]; however, this phenomenon was not observed in the current study. Amaral, et al. [27] achieved higher percentage of specimens cemented with MDP primers that survived thermocycling; however, the bonding values were lower compared to those of the air-abraded specimens, partially in accordance with our results. Spontaneous failures during aging were also observed by Kern, Barlooi and Yang [20] and Cristoforides, et al. [4].

A conventional MPS-based primer (methacryloxypropyltrimethoxy-silane, Monobond S) was also used in the current study and exhibited high losses during thermocycling. It has been demonstrated that the use of conventional silane results in an increase in the bonding strength between Y-TZP surfaces air-abraded with alumina particles coated with silica; however, this mechanism is not sustained on surfaces without silica deposition [1,24].

Regarding the use of phosphonic acid acrylate-based primer (Metal/Zirconia Primer), according to the manufacturer, this product promotes adhesion between ceramics oxides and methacrylate composites. In the current study, this primer did not promote a stable adhesion, resulting in debonding during aging, which was also observed by Cristoforides, et al. [4].

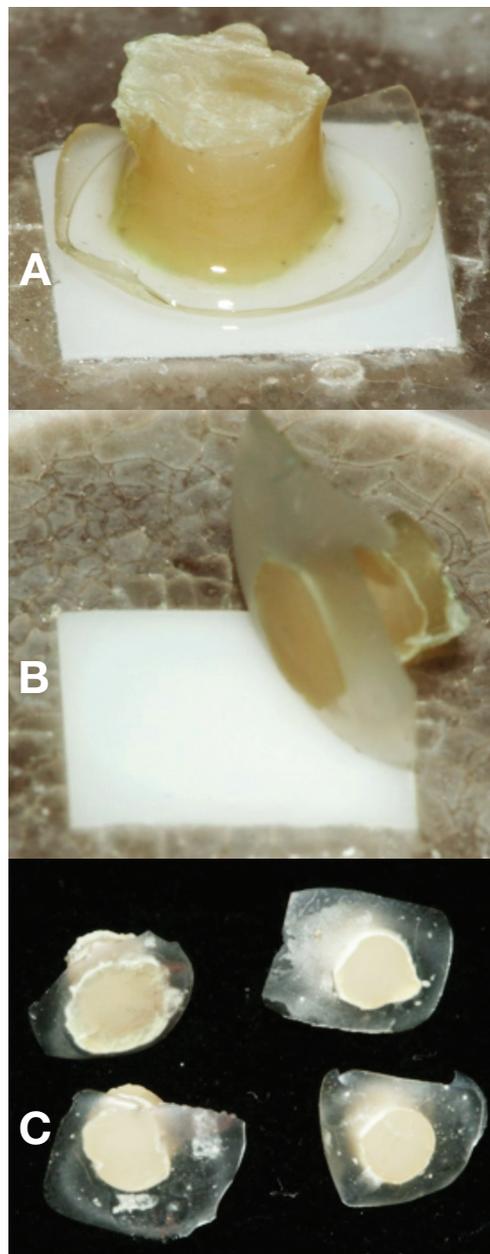
Another important aspect is that the low-temperature degradation (LTD) affected the bond strength negatively, as observed by comparing TBS and TBS-LTD (Table 3), and hence, the null hypothesis (2) was rejected. It is stated that, after some stimuli (such as aging), a phase transformation ( $t \rightarrow m$ ) on superficial grains may occur [11], leading to a volume increase (3 to 4%) around these grains and the introduction of superficial residual stress

(compression) on the surface of zirconia [23]. This mechanism can have either a positive or negative effect on the properties of Y-TZP, depending on the intensity of the stimuli and *m*-phase content post-transformation [29].

The increase of residual stresses in response to the volume expansion promoted by phase transformation may inhibit the crack propagation and improve the flexural strength of Y-TZP [23]; however, as the monoclinic phase transformation mechanism proceeds towards the bulk of the Y-TZP ceramic [30], the internal flaws and effects of degradation can be critical. It is known that LTD produces phase transformation within a layer of 6 to 20  $\mu\text{m}$  on the surface of zirconia, changing the roughness and texture of the surface<sup>3</sup> owing to grain pull-out [22]. Moreover, the air abrasion promotes a topographic alteration within a layer of approximately 15  $\mu\text{m}$  [31] on the surface of zirconia.

Thus, it is possible that the compressive layer introduced by phase transformation on the surface (compressive stress layer) makes the penetration of silica-coated alumina particles difficult (TBS-LTD group); or that the air-abrasion of transformed grains had resulted in a silica deposition on grains that were not firmly adhered to the core material, potentially being easily detached and leading to decreased adhesion. This needs further investigation. In contrast to these results, Galvão Ribeiro, et al. [24] evaluated the effect of airborne-particle abrasion (sandblasting with  $\text{Al}_2\text{O}_3$  or silica-coated  $\text{Al}_2\text{O}_3$  particles) and a bonding agent on the shear bond strength at the zirconia ceramic–composite resin interface to find that hydrothermal aging did not influence the bonding.

Despite the present inhomogeneous stress distribution, the shear bond test is the most commonly used method and an acceptable alternative to bonding tests, principally compared to the microtensile bond strength test of zirconia specimens because cutting procedures can cause damage to the adhesive interface [4]. In relation to the mode of failure (Table 4), the majority of specimens exhibited adhesive failures for both after shear bond strength test (Figures 1A-B: fracture at the interface between adhesive/primer and ceramic) as during thermocycling (Figure 1C).



**Figure 1** - Representative images of the failure analysis. A. Alloy Primer specimens group. It is possible to observe the adhesive layer detached of the zirconia surface. B. Single Bond Universal specimen group. Adhesive layer adhered on composite resin cylinder. C. Adhesives spontaneous failures occurred during the aging. Note the adhesive layer attached on the composite cylinder.

Clinically, a zirconia crown with veneer chipping would present an occlusal surface with a porcelain/vitreous ceramic exposed. As vitreous ceramics are etchable by hydrofluoric acid, the repair procedure would involve different conditionings on the exposed zirconia

and vitreous ceramics surfaces. Hence, it is possible for the clinical adhesion to be more efficient owing to the etchable ceramic. Further, in a clinical situation, the Y-TZP framework would exhibit some degree of roughness caused

by machining, which promotes the mechanical interlocking with the repair composite. Further studies to simulate the real condition of a veneered zirconia crown, which is chipped and repaired, subjected to mechanical fatigue and survival analysis are required.

**Table 1** - Experimental design of the study.

Group (n=15)	Surface Treatment	LTD
Control -*	without treatment + adhesive (Scotchbond, 3M ESPE)	Yes
TBS - LTD	Cojet TBS cojet + silane (Ceramic primer, 3M ESPE) + adhesive (Scotchbond, 3M ESPE)	Yes
MoS	Monobond S (Ivoclar Vivadent) + adhesive (Scotchbond, 3M ESPE)	Yes
MoP	Monobond Plus (Ivoclar, Vivadent) + adhesive (Scotchbond, 3M ESPE)	Yes
MZP	Metal/Zirconia Primer (Ivoclar, Vivadent) + adhesive (Scotchbond, 3M ESPE)	Yes
TBS*	Cojet TBS cojet (3M ESPE) + silano (Ceramic primer, 3M ESPE) U adhesive (Scotchbond, 3M ESPE)	No
SBU	SB universal (3M ESPE)	Yes
AP	Alloy primer (Kuraray) + adhesive (Scotchbond, 3M ESPE)	Yes
Control +*	Without treatment + adhesive (Scotchbond, 3M ESPE)	Yes

\* Evaluation of the adhesive effect in the adhesion to zirconia.

Abbreviations: Control-: negative control group; TBS – LTD: tribochemical silica coating with low temperature degradation group; MoS: Monobond S; MoP: Monobond Plus; MZP: Metal/Zirconia Primer; TBS: tribochemical silica coating; SBU: Single bond universal; AP: Alloy Primer; Control +: positive control group.

**Table 2** - Surface treatments used in the study as well their compositions, brands and use mode.

Treatment	Composition	Manufacturer	Use*
Tribochemical silica coating	Al <sub>2</sub> O <sub>3</sub> particles coated with silica (30µm)	Cojet Sand - 3M ESPE	2.8 bar pressure, 7 s, 10 mm distance + RelyX Ceramic Primer.
Monobond S	Alcohol solution of silane methacrylate.	Ivoclar Vivadent	Applied on zirconia surface with a microbrush, wait for 60 seconds and dispersed with a strong stream of air
RelyX Ceramic Primer	3-MPS, ethyl alcohol, water	3M ESPE	Applied with a microbrush, wait for 5 seconds before drying with an oil-free air stream
Monobond Plus	Ethanol, 3-trimethoxysilylpropyl methacrylate, 10-MDP, sulphide methacrylate	Ivoclar Vivadent	Applied on zirconia surface with a microbrush, wait for 60 seconds and dispersed with a strong stream of air
Single Bond Universal	10-MDP monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	3M ESPE	Applied with a microbrush for 20 s, dried with a gentle air stream for 5 s to evaporate solvent, and light cured for 10 s.
Metal/Zirconia Primer	Phosphonic acid acrylate, dibenzoyl peroxide, methylsobutylketone, tert-butyl alcohol	Ivoclar Vivadent	Applied with a microbrush, wait for 180 s and dispersed with a strong stream of air for 5 s.
Alloy Primer	10-MDP, VBATDT, acetone	Kuraray	Applied with a microbrush, wait for 10 s and gently air dry for 5 s.
Adper Scotchbond Multi-purpose Plus Adhesive	Bis-GMA, HEMA, tertiary amines, photoinitiator	3M ESPE	Applied with a microbrush and light cure for 10 s.
Filtek Z350 XT	Bis-GMA, TEG-DMA, UDMA, PEG-DMA, bis-EMA, Zirconium/Silicium cluster	3M ESPE	An increment of 1.5 mm and 10 s of curing and other of 1.5 mm and 40 s of curing.

\* After the surface treatment, all groups received the adhesive application (Adhesive component of the Adper Scotchbond Multi-purpose Plus except Single Bond Universal group).

Abbreviations: 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; VBATDT: 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine-2,4-dithione; 3-MPS: 3-methacryloyloxypropyl trimethoxysilane; Bis-GMA: bisphenol A diglycidyl methacrylate; TEG-DMA: triethyleneglycol dimethacrylate; bis-HEMA: ethoxylated bisphenol-A dimethacrylate; UDMA: urethane dimethacrylates; PEG-DMA: poly(ethylene glycol) dimethacrylate.

**Table 3** - Shear bond strength values means (MPa) ± standard deviation (SD) and pre-test failures.

Groups	Means ± SD	Pre-test failures during thermocycling – No. (%)
TBS	3.94 ± 2.64 A	1 (7)
TBS - LTD	0.96 ± 1.84 B	8 (50)
AP	-	13 (87)
Control -	-	15 (100)
USB	-	13 (87)
Control +	-	15 (100)
MoP	-	12 (80)
MoS	-	13 (87)
MZP	-	14 (93)

The groups that presented more than 50% of pre-test failures were not included in the statistical analysis.

**Table 4** - Failure mode classification during the thermocycling and after shear bond strength

Groups	LTD	Failure Mode (%)			
		ADES*	COES-CR*	COES-CER*	M*
Control –	Yes	15 (100)	-	-	-
TBS	Yes	15 (100)	-	-	-
MoS	Yes	15 (100)	-	-	-
MoP	Yes	15 (100)	-	-	-
MZP	Yes	15 (100)	-	-	-
TBS - LTD	No	15 (100)	-	-	-
ZP	Yes	15 (100)	-	-	-
AP	Yes	14 (93)	1 (7)	-	-
Control +	No	14 (93)	1 (7)	-	-

\* ADES: fracture at the interface between adhesive/primer and ceramic.

COES-CR: Composite resin cohesive fracture.

COES-CER: Ceramics cohesive fracture.

M: adhesive fracture combined with composite resin cohesive fracture.

## CONCLUSION

- The air abrasion were fundamental for the adhesion between composite resin and aged and non-aged Y-TZP.

- It suggests that an aged Y-TZP substrate is an even more challengeable scenario for achieving adhesion to resinous materials, given the low bond strengths.

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