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Adhesive Quality of Self-adhesive and Conventional Adhesive Resin Cement to Y-TZP Ceramic Before and After Aging Conditions

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Clinical Relevance

Chairside tribochemical silica coating and silanization on the YTZP surface appears to be essential to adhere this substrate to resin cements. Cleaning with isopropanol promotes weak and unstable resin adhesion.

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SUMMARY

Purpose: This study evaluated the adhesive quality of simplified self-adhesive and conventional

resin cements to Y-TZP in dry and aged conditions. Methods: Y-TZP ceramic blocks (N=192) (5 x 5 x 2 mm) were embedded in acrylic resin and randomly divided into two groups, based on surface conditioning: 96% isopropanol or chairside tribochemical silica coating and silanization. Conditioned ceramics were divided into four groups to receive the resin cements (Panavia F 2.0, Variolink II, RelyX U100 and Maxcem). After 24 hours, half of the specimens (n=12) from each group were submitted to shear bond strength testing (0.5 mm/minute). The remaining specimens were tested after 90 days of water storage at 37°C and thermocycling (12,000x, 5°C-55°C). Failure types were then assessed. The data were analyzed using three-way ANOVA and the Tukey's test (α=0.05). Results: Significant effects of ceramic conditioning, cement type and storage conditions were observed (*p*<0.0001). The groups cleaned using alcohol only showed low bond strength values in dry conditions and the bond strength was reduced dramatically after aging. Groups conditioned using silica coating and silanization showed higher bond strengths both in dry and aged conditions. A high number of specimens failed prematurely prior to testing when they were cleaned using 96% isopropanol. Conclusion: Overall, silica coating and silanization showed higher, stable bond strengths with and without aging. The durability of resin-ceramic adhesion varied, depending on the adhesive cement type.

INTRODUCTION

The increasing use of all-ceramic restorations has contributed to the development of ceramic materials with improved mechanical properties, such as densely sintered alumina and yttria-tetragonal zirconia polycrystal (Y-TZP) ceramics (hereon: zirconia).[1-2] However, the adhesion of resin cements to zirconia still presents a critical problem, especially for minimally invasive restorations.[3] The clinical success of reinforced ceramic restorations is directly dependent on the achievement of reliable bond strength between the cement and ceramic surfaces.[3] The reliable adhesion to ceramics principally requires surface conditioning of the ceramics.[4-9] Hydrofluoric acid etching and the application of a silane coupling agent do not improve the bond strength of resin cements to zirconia, because of the high crystalline content of such ceramics. The limited vitreous phase (below 1%) makes the resin cements resistant to acid etching.[4-9] As a consequence, alternative conditioning methods have been proposed. Several studies have shown that silica coating, followed by silanization, could be used to improve the bond strength of silica-based, glass-infiltrated alumina and zirconia ceramics.[10-13] In this conditioning system, the airborne particle abrasion of alumina particles coated with silica on ceramic surfaces (silica coating) creates a tribochemical effect on the surface. This layer then makes the ceramic surface chemically more reactive to silane coupling agents.[14] Özcan and Vallittu[11] showed that this kind of conditioning may significantly increase the bond strength of resin cements to high-alumina and zirconia ceramics when compared with airborne-particle abrasion with only alumina followed by silanization. Airborne particle abrasion may increase the surface area, clean the surface and result in micrometer scale roughness, thereby facilitating resin/ceramic micromechanical retention.[15-16] Nevertheless, the effect of a tribo-chemical silica coating on the bonding of zirconia to resin has been debated in the literature, since there is little information that has been published.[11,13,17]

Some resin-based cements, depending upon their wetting capacity and composition, have presented better bonding capacity than others.[18] Cement selection is an important step in achieving an effective bond strength to zirconia. Few conventional adhesive cements, such as phosphate monomer-based luting agents (Panavia 21, Panavia F 2.0, Kuraray Medical Inc, Osaka, Japan), have demonstrated satisfactory bond strength results to zirconia.[19-20] In order to simplify the cementation procedures, self-adhesive cements have been recently marketed.[21] These single-step resin cements contain a resin matrix packed with multifunctional acid methacrylates that also simultaneously react with the ceramic surfaces.[22-23] There is little information in the literature about the bonding of self-adhesive resin cement to zirconia.[21,24-25]

The analysis of different cement-ceramic adhesive joints should consider specimen aging with water storage and/or thermocycling, which may decrease bond strength.[5,9,11,26-28] The objectives of the current study were to evaluate the durability of bond strengths of conventional and self-adhesive dual-cured adhesive cements after thermocycling and water storage to zirconia after two different surface conditioning treatments, namely, cleaning with isopropyl alcohol and tribochemical silica coating and silanization. The hypotheses tested were: 1) resin cement with a MDP functional monomer would provide higher adhesion to zirconia ceramic; 2) silica coating and silanization would increase the cement-ceramic bond strength; 3) long-term water storage and thermocycling would decrease adhesion to zirconia for all cements.

METHODS AND MATERIALS

Zirconia Block Production and Experimental Groups

Blocks from partially sintered zirconia (N=192) (In-Ceram YZ 2000 cubes 40/15, Vita Zanhfabrik, Bad

Säckingen, Germany, Batch #21970) were sectioned in a customized cutting machine using a diamond-coated cutting disc (Microdont, São Paulo, Brazil, #34570) (7.5 x 7.5 x 2.5 mm). The blocks were sintered using the manufacturer's instructions in a VITA ZYRcomat furnace (Vita Zanhfabrik). As 20%-25% shrinkage occurs during sintering, the blocks presented dimensions approximately 5 x 5 x 2 mm at the end of the sintering. The blocks were then embedded in acrylic resin with a 5 x 5-mm free surface for adhesion. The specimens were ground finished from 400- to 1200-grit silicon carbide paper using a rotating metallographic polishing machine under water-cooling.

The specimens were randomly divided into 16 groups (n=12 specimens/group), depending on the two surface treatments, four cement types and two storage conditions (Table 1). Prior to the bonding procedures, all of the specimens were cleaned ultrasonically for five minutes in water (VITA sonic, Vita Zanhfabrik).

Zirconia Surface Conditioning

The exposed ceramic surfaces of half of the specimens (n=96) were cleaned with 96% isopropanol for 30 seconds and allowed to evaporate for 30 seconds. The remaining specimens (n=96) were treated with the tribochemical silica coating method (CoJet System, 3M ESPE, Seefeld, Germany, Batch #351794). Initially, the zirconia surfaces were air-abraded with 30 µm silica-coated alumina particles using an intraoral air abrasion device (air-abrasion parameters—pressure: 2.8 bars; distance: 10 mm; duration: 15 seconds). The conditioned surfaces were then silanized with an MPS silane (ESPE Sil, 3M-ESPE, Batch #10926) and left to air dry for five minutes before adhering the cements.

Application of Resin Cements

Four different resin cement systems, namely, Panavia F 2.0 (Kuraray Medical Inc, Batch #51205), RelyX U100 (3M ESPE, Seefeld, Germany, Batch #306228), Maxcem (Kerr, Orange, CA, USA, Batch #453145) and Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein, base: Batch #K43442, catalyst: Batch #K56289) were applied to the exposed zirconia surfaces.

For bonding procedures, a metal template was placed on the conditioned zirconia surface. Each resin-cement was manipulated according to the manufacturer's instructions and inserted onto the ceramic surface using the central cylindrical hole (diameter: 3 mm, height: 3 mm). Photopolymerization (Elipar FreeLight 2/3M ESPE, 900 mW/cm^2) was performed according to each manufacturer's recommendations.

Storage Conditions and Shear Testing

A randomly selected half of the specimens from each group was tested after 24 hours of storage in distilled water at 37°C (non-aged groups). The remaining specimens (aged groups) were stored in distilled water at 37°C for 90 days, then subsequently subjected to 12,000 thermal cycles between 5°C and 55°C.

The specimens were attached to an adapted device fixed in the Universal Testing Machine (EMIC DL-1000, EMIC, São José dos Pinhais, PR, Brazil). A knife-edge blade with a 45° inclination at the tip was used for shear testing. The blade was kept as close as possible to the substrate surface, and the load was applied perpendicular to the adhesive interface (crosshead speed: 0.5 mm/minute). The shear bond strength was recorded in N/mm^2 (MPa).

Failure Analysis

The fractured surfaces of all tested specimens were analyzed under an optical microscope (Mitutoyo TM-505, Kanagawa, Japan) at 200x magnification. Specimens with representative failures were chosen for scanning electron microscope (SEM) analysis. The selected specimens were mounted on a metallic stub, sputter coated with gold (Denton Vacuum, DESK II, Denton Vacuum, LLC, Moores-town, NJ, USA) and observed

Table 1: *Groups Considering the Type of Resin Cement, Surface Treatment and Storage Conditions (non-aging vs aging)*

Resin Cement	Y-TZP Surface Treatment	Storage Condition	Groups*
Panavia F 2.0	96% isopropanol	Non-aging	G1
		Aging	G2
	CoJet System	Non-aging	G3
		Aging	G4
Variolink II	96% isopropanol	Non-aging	G5
		Aging	G6
	CoJet System	Non-aging	G7
		Aging	G8
RelyX U100	96% isopropanol	Non-aging	G9
		Aging	G10
	CoJet System	Non-aging	G11
		Aging	G12
Maxcem	96% isopropanol	Non-aging	G13
		Aging	G14
	CoJet System	Non-aging	G15
		Aging	G16

**n=12*

under the SEM (1000x) (JEOL-JSM-6360, JEOL Ltd, Tokyo, Japan).

Statistical Analysis

Statistical analysis was performed using SPSS 11.0 software for Windows (SPSS Inc, Chicago, IL, USA). Bond strength data (MPa) were submitted to three-way analysis of variance (three-way ANOVA) with bond strength as the dependent variable and cement type (four levels), surface conditioning (two levels) and aging conditions (two levels: dry vs thermocycling) as the independent variables. Multiple comparisons were made using the Tukey's test. *P*-values less than 0.05 were considered to be statistically significant in all tests. Specimens that failed prematurely during the aging conditions were considered as 0 MPa for statistical analysis.

RESULTS

Bond Strength Analysis

Significant effects of the cement type (Variolink II = U100 > Panavia F 2.0 > Maxcem) (p<0.0001), surface conditioning methods (SiO_x > alcohol) (p<0.0001) and storage conditions (TC > dry) (p<0.0001) were observed on the bond strength to zirconia (three-way ANOVA) (Table 2). Mean bond strength values recorded per test group are summarized in Table 3 and Figure 1.

Table 2: *Three-way ANOVA Results of the Shear Bond Strength Data*

Source	DF	SS	MS	F-value	p-value*
Cement	3	1607.4	535.8	25.29	0.0000
Surface conditioning	1	14110.8	14110.8	666.12	0.0000
Storage	1	586.3	586.3	27.68	0.0000
Cement*Surface conditioning	3	1580.3	526.8	24.87	0.0000
Cement*Storage	3	1329.8	443.3	20.93	0.0000
Surface Conditioning*Storage	1	1492.3	1492.3	70.44	0.0000
Cement*Surface Conditioning*Storage	3	1470.8	490.3	23.14	0.0000
Error	176	3728.3	21.2		
Total	191	25906.0			

*p<0.05

Table 3: *Mean Values (MPa) and Standard Deviations of the Bond Strength Obtained for the Different Resin Cement, Ceramic Surface Conditioning With and Without Aging*

	Panavia F 2.0	Variolink II	RelyX U100	Maxcem
		96% Isopropanol		
Non-aging	***Gr1***: 5.87 ± 4.35^{de}	***Gr5***: 0.52 ± 0.62^{e}	***Gr9***: 3.64 ± 2.18^{de}	***Gr13***: 0.52 ± 1.26^{e}
Aging	***Gr2***: 1.22 ± 1.22^{e}	***Gr6***: 0^{e}	***Gr10***: 0^{e}	***Gr14:*** 0^{e}
		Silica Coating		
Non-aging	***Gr3***: 12.31 ± 2.99^{bc}	***Gr7***: 14.83 ± 4.79^{b}	***Gr11***: 13.46 ± 5.46^{bc}	***Gr15***: 13.99 ± 5.61^{bc}
Aging	***Gr4***: 17.19 ± 8.57^{b}	***Gr8***: 35.62 ± 5.81^{a}	***Gr12***: 30.48 ± 5.90^{a}	***Gr16***: 7.60 ± 9.59^{cd}

Identical letters indicate no statistically significant differences (p>.05).

Table 4: *Number of Specimens Per Group (n), Number of Pretest Failure (PTF) and Failure Types of the Debonded Specimens*

Study Factors						Failure Types			
Cements	**Ceramic Conditioning**	**Storage**	**Groups**	**n**	**PTF**	**Adhes**	**C-cer**	**C-cem**	**Mix**
PAN	**alcohol**	**Non-aging**	**Gr1**	12 (100%)	2	9 (75%)	0	0	3 (25%)
	cleaning	**Aging**	**Gr2**	12 (100%)	4	12 (100%)	0	0	0
	silica	**Non-aging**	**Gr3**	12 (100%)	0	3 (25%)	0	1 (8.33%)	8 (66.67%)
	coating	**Aging**	**Gr4**	12 (100%)	2	12 (100%)	0	0	0
VAR	**alcohol**	**Non-aging**	**Gr5**	12 (100%)	3	12 (100%)	0	0	0
	cleaning	**Aging**	**Gr6**	12 (100%)	12	12 (100%)	0	0	0
	silica	**Non-aging**	**Gr7**	12 (100%)	0	5 (41.67%)	0	0	7 (58.33%)
	coating	**Aging**	**Gr8**	12 (100%)	0	10 (83.33%)	0	0	2 (16.67%)
U100	**alcohol**	**Non-aging**	**Gr9**	12 (100%)	0	12 (100%)	0	0	0
	cleaning	**Aging**	**Gr10**	12 (100%)	12	12 (100%)	0	0	0
	silica	**Non-aging**	**Gr11**	12 (100%)	0	5 (41.67%)	0	0	7 (58.33%)
	coating	**Aging**	**Gr12**	12 (100%)	0	5 (41.67%)	0	4 (33.33%)	3 (25%)
MAX	**alcohol**	**Non-aging**	**Gr13**	12 (100%)	9	12 (100%)	0	0	0
	cleaning	**Aging**	**Gr14**	12 (100%)	12	12 (100%)	0	0	0
	silica	**Non-aging**	**Gr15**	12 (100%)	0	6 (50%)	0	0	6 (50%)
	coating	**Aging**	**Gr16**	12 (100%)	7	12 (100%)	0	0	0
			Total	**192**		**151 (78.65%)**	**0**	**5 (2.60%)**	**36 (18.75%)**

Failure between ceramic and cement (ADHES); cohesive failure of cement and ceramic (MIX); cohesive failure of the ceramic (C-cer); cohesive failure of the cement (C-cem).
Panavia F2.0; VAR= Variolink II; U100= Relyx U100; MAX= Maxcem

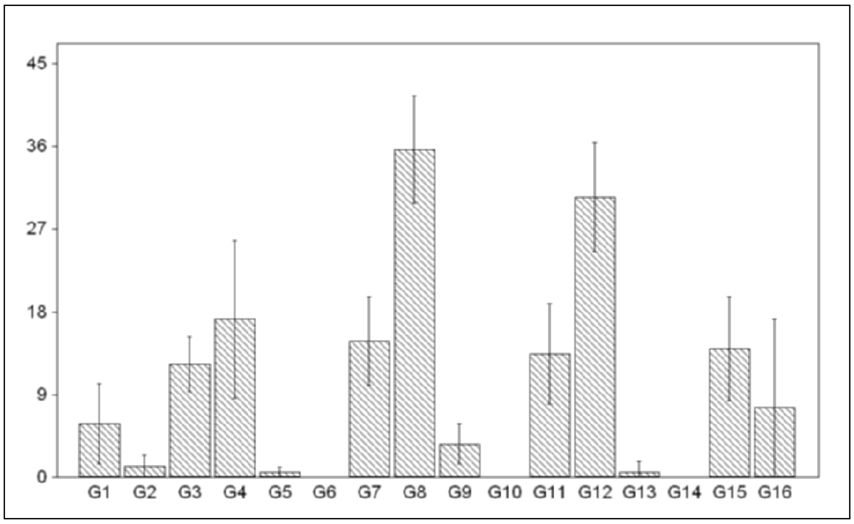

Figure 1. *The mean bond strength values (MPa) and standard deviations for the resin cement and surface conditioning method with and without aging.*

In the non-aged groups with both surface-conditioning methods (96% isopropanol vs silica coating), no significant difference in bond strength was found between the resin cements ($p>0.05$). In the silica- coated and silanized groups, regardless of the aging conditions, Variolink II and RelyX U100 attained significantly higher bond strengths when compared to Panavia F 2.0 and Maxcem ($p<0.05$). In these test conditions, there was no significant difference between the mean bond strengths of Panavia F 2.0 and Maxcem ($p>0.05$), where the latter showed a significant decrease after aging.

Failure Analysis

Table 4 summarizes the number of premature failures before testing and the failure modes of the debonded specimens, depending on the surface conditioning, cement type and aging conditions.

After 90 days of water storage and thermocycling (aging) in the 96% isopropanol-treated groups, all of the specimens cemented with Variolink II, U100 and Maxcem failed prematurely. In these conditions, only four specimens from Panavia F 2.0 failed prior to testing. The Maxcem groups showed the highest percentage of premature failures.

The incidence of adhesive failures increased in all groups after aging. Few pure cohesive failures were detected only in the two silica-coated subgroups (Table 4). After 24 hours, mixed failures were more common in this group, where remnants of the cement were visible (Figure 2a); but after using only 96% isoprapanol exclusively, adhesive failure was observed before and after aging (Figure 2b).

DISCUSSION

The results of the current study show that the bond strength was influenced by different surface conditioning methods, choice of resin-luting cement and storage conditions. Air-abraded specimens with tribochemical silica coating + silanization (CoJet-Sand, CoJet System, 3M ESPE) showed the highest bond strengths. Previous studies have shown that the application of a modified bis-GMA resin-luting agent containing the adhesive phosphate monomer 10-methacryloyloxydecyl dihydrogen phosphate (MDP) (Panavia 21, Panavia F 2.0, Kuraray Medical Inc) is an important factor in providing a durable, long-term resin bond to zirconia ceramic.[16,19,29-30] This kind of cement in the current study did not report a higher bond strength when luted after a silica coating, but it showed bond durability after 90 days of water storage and thermocycling. Also in the current study, zirconia pretreated surfaces were not

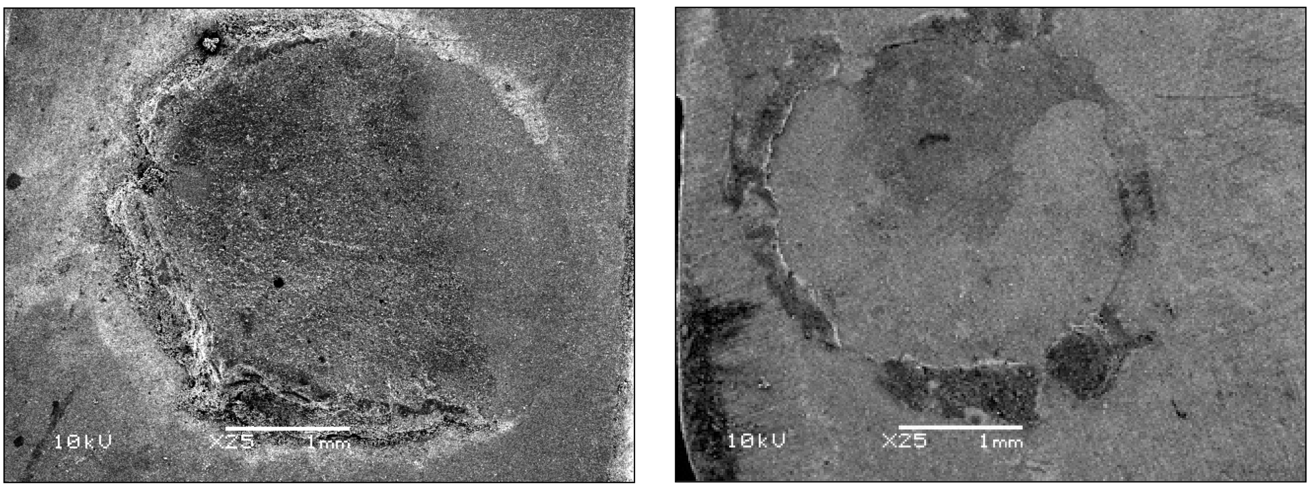

Figure 2. *Representative micrographs of the debonded surfaces: Figure 2a–specimen cemented with RelyX U100, mixed failure; Figure 2b–adhesive failure of a specimen when Maxcem was used (25x).*

bonded to human dentin, because the purpose of the investigation was to analyze the cement-ceramic interface only.

In the current study, the aged group that was thermocycled, stored in water for 90 days and cleaned with 96% isopropanol resulted in low bond strengths for all resin cements. After aging, the bond strength to Y-TZP ceramic was shown to be low and not stable when the bonding agents were applied to the original ceramic surface after cleaning with 96% isopropanol, in accordance with previous studies with alumina ceramics.[8,31] Low bond strength values or those spontaneously debonded before testing have been found in other studies, when the zirconia surface was cleaned by alcohol.[26-27] For these groups, 100% of the specimens that bonded with Variolink II, RelyX U100 and Maxcem debonded spontaneously after long-term storage and thermocycling. Four specimens of the Panavia F 2.0 group failed prior to testing (pretesting failures). Water sorption may have caused hydrothermal degradation during aging.[1,9,15,19,25,32-35] The weak adhesion noted for these conditions may also be attributed to poor chemical and micromechanical bonding.[5,7,34]

With regard to the conditioning methods performed on the zirconia surfaces, several *in vitro* studies have shown that, not only cleaning, but also airborne-particle abrasion, is an essential step for achieving a durable bond to high-strength ceramics.[9,15,23] In the current study, the silica coating showed higher bond strengths when compared to cleaning with 96% isopropanol. These current results, which are similar to other *in vitro* tests, suggest that applying a physical-chemical conditioning method is recommended for ensuring the long-term success of bonding to zirconia, regardless of the type of resin cement used.[7,19,36] The resin cements tested showed statistically better bond strength values when luted to silica-coated zirconia surfaces than when cemented to the specimens cleaned with 96% isopropanol, independent of aging. This may be due to the ease of penetration of the self-adhesive cement through the roughened Y-TZP surfaces, facilitating micro-mechanics interlocking of the resin to the ceramic[37] and the silica-modified surface becoming chemically more reactive to the resin through silane coupling agents.[4-5,11] Some studies have confirmed that only the application of silane (without silica coating) cannot promote the adhesion of a resin composite cement onto zirconia, since the Al–O–Si bonds in the siloxane film at the zirconia-resin interface are not durable due to hydrolytic effects in oral conditions.[11,13,17,38] Additionally, this study did not indicate statistically significant differences among the four subgroups of silica-coated specimens at 24 hours of water exposure. In fact, according to the manufacturers, bonding with this self-adhesive cement can be achieved without any pretreatment steps.

Various studies have used thermocycling and water storage as clinically relevant parameters to identify the performance of bonding methods and materials.[8,30] Considering the influence of water storage in bond strength durability of resin cements luted to zirconia, it has been demonstrated that water sorption may cause cement hydrothermal degradation after storing.[35,39] In these studies, the shear bond strengths showed a significant decrease after thermocycling. Conventional bis-GMA based resin-luting cements did not demonstrate a durable long-term bond to high-strength ceramic materials when using silica coating or sandblasting.[9-10,29,40] In the current study, when silica coating was used with Variolink, and RelyX U100, a conventional bis-GMA resin-luting cement and a self-etching resin cement, respectively, increased bond strengths were observed over time.

The specimens treated with 96% isopropanol recorded a remarkable percentage of premature failures, which was in accordance with other published works with alumina ceramics.[8,31] Residual cement was mainly observed when the cements were luted to silica-coated zirconia surfaces, which may be due to micromechanical retention created by the air-abrading procedure. Pure cohesive failures within the resin cement were only minimally recorded when Panavia F 2.0 and RelyX U100 were luted to the silica-coated ceramic. The adhesive failure mode was found in all groups, as indicated by light microscopy and confirmed by SEM. Mixed and cohesive fracture patterns are clinically preferable to total adhesive types of failure, since adhesive failure is usually attributed to low bond strength values.[28]

Although there are studies indicating that air abrasion using Al_2O_3 particles affect the surface of zirconia ceramics, leading to a reduction in the flexural strength of these ceramics,[41] there are other studies that have shown that air abrasion using Al_2O_3 particles[25,33,41,43-45] and silica coating using the Rocatec system (110 μm)[46] might even strengthen zirconia ceramics, suggesting that air abrasion induced a tetragonal to monoclinic phase transformation on the Y-TZP surface. However, Qeblawi and others[43] reported that no changes in the flexural strength of Y-TZP occurred when silica-coated alumina particles (30 μm) were applied on the Y-TZP surface.

The findings of the current study require rejection of the first hypothesis, as the resin cement with a MDP functional monomer did not provide the highest adhesion to the ceramic when compared to the other cements studied. The second hypothesis was accepted, as the silica coating increased the cement-ceramic bond strength values. The third hypothesis was accepted, as 90 days of water storage and thermocycling affected the bond strengths for both conditionings. However, the results of this current experiment only provide an indication of the possible performance of resin-luting agents to zirconia ceramics. These conclusions must be refined, because the clinical environment is more complex than *in vitro* tests.

CONCLUSIONS

From this study, the following results can be concluded:

1. Tribochemical silica coating promoted higher, more stable bond strengths, independent of the resin cement used.
2. Cleaning with 96% isopropanol resulted in unstable and low bond strengths, regardless of the type of cement used.
3. The longevity of the resin-Y-TZP interface depends on the type of luting agent.

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