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Effect of Cleansing Methods on Saliva-Contaminated Zirconia—An Evaluation of Resin Bond Durability

SA Feitosa • D Patel • ALS Borges
EZ Alshehri •MA Bottino • M Özcan
LF Valandro • MC Bottino

Clinical Relevance

Considering the bond strength data and absence of adhesive failures, it can be stated that an appropriate cleaning method (ie, a zirconium-oxide–based paste) can be helpful in restoring the resin bond strength to saliva-contaminated zirconia.

SUMMARY

The aims of this study were to investigate 1) the influence of cleansing methods after saliva contamination and 2) aging conditions (thermocycling and water storage) on zirconia shear bond strength (SBS) with a resin cement. One hundred and eighty zirconia specimens were sandblasted with 50 μm aluminum oxide particles, immersed in saliva for one minute (with the exception of the control group, [C]), and divided into groups according to the cleansing method, as follows: water rinse (W); 37% phosphoric acid gel (PA); cleaning paste (ie, Ivoclean®) containing mainly zirconium

Sabrina Alves Feitosa, DDS, MSc, PhD student, Department of Dental Materials and Prosthodontics, São José dos Campos Dental School, São Paulo State University, São José dos Campos, SP, Brazil / Department of Restorative Dentistry, Dental Biomaterials Division, Indiana University School of Dentistry, Indianapolis, IN, USA.

Dhara Patel, BDS, MSD student, Department of Restorative Dentistry, Dental Biomaterials Division, Indiana University School of Dentistry, Indianapolis, IN, USA.

Alexandre L. S. Borges, DDS, MSc, PhD, associate professor, Department of Dental Materials and Prosthodontics, São José dos Campos Dental School, São Paulo State University, São José dos Campos, SP, Brazil.

Eman Z. Alsheeri, BDS, MSD student, Department of Restorative Dentistry, Dental Biomaterials Division, Indiana University School of Dentistry, Indianapolis, IN, USA.

Marco A. Bottino, DDS, PhD, full professor, Department of Dental Materials and Prosthodontics, São José dos Campos Dental School, São Paulo State University, São José dos Campos, SP, Brazil.

Mutlu Özcan, DDS, PhD, full professor, Dental Materials Unit, Center for Dental and Oral Medicine, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, University of Zürich, Zurich, Switzerland.

Luiz F. Valandro, DDS, MSc, PhD, associate professor, Department of Restorative Dentistry, Division of Prosthodontics, Federal University of Santa Maria School of Dentistry, Santa Maria, RS, Brazil / Department of Dental Materials and Prosthodontics, São José dos Campos Dental School, São Paulo State University, São José dos Campos, SP, Brazil.

*Marco C. Bottino, DDS, MSc, PhD, assistant professor, Department of Restorative Dentistry, Dental Biomaterials Division, Indiana University School of Dentistry, Indianapolis, IN, USA.

*Corresponding author: 1121 West Michigan Street, Indianapolis, IN 46202, USA; e-mail: mbottino@iu.edu

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oxide (IC); and 70% isopropanol (AL). Scanning electron microscopy was done to qualitatively evaluate the zirconia surface after each cleansing method. For the SBS test, resin cement buttons were bonded to the specimens using a dedicated jig. SBS was evaluated according to standard protocols after 24 hours, 5000 thermal cycles (TC), or 150 days of water storage. Statistical analysis was performed using two-way analysis of variance and Tukey test ($p<0.05$). Data showed a significant effect for the 150 days of water storage, TC, and 24 hours of water storage (150 days < TC < 24 hours). Group comparisons showed that PA < AL and W < IC and C. SBS ranged from 10.4 to 21.9 MPa (24 hours), from 6.4 to 14.8 MPa (TC), and from 2.9 to 7.0 MPa (150 days). Failure analysis revealed a greater percentage of mixed failures for the majority of the specimens and a smaller percentage of adhesive failures at the ceramic-resin cement interface. Our findings suggest that Ivoclean® was able to maintain adequate SBS values after TC and 150 days of storage, comparable to the uncontaminated zirconia.

INTRODUCTION

Current advances in computer-aided design/computer-aided manufacturing technology have facilitated and expanded the use of high-toughness yttria-stabilized tetragonal zirconia ceramics (Y-TZP) as frameworks for fixed-partial dentures (FPDs) and more recently as full-contour restorations.[1-5] Regrettably, apart from its superior mechanical properties, when contrasted with glassy-matrix ceramics, and the finer esthetic and biocompatibility characteristics, as opposed to those of metallic FPD frameworks, the achievement of a durable adhesive bonding to structural Y-TZP ceramics remains a very difficult task.[6-20]

Meanwhile, another major issue pertaining to bonding of ceramic restorations relates to the potential of contamination before cementation. Zirconia shows a strong affinity toward the phosphate group found in saliva and other fluids.[21] After sandblasting and clinical try-in procedures, zirconia may become contaminated with saliva and/or blood, which reacts with the zirconia surface and makes bonding a challenge.[21] X-ray photoelectron spectroscopy (XPS) revealed that the organic coating formed after saliva contamination resisted complete removal with water rinsing, isopropanol, or phosphoric acid.[21] Nonetheless, while numerous studies[6-18] have shown an immediate (24-hour) increased bond strength between zirconia and resin cements after various surface conditioning methods, the potential contamination of the intaglio surface prior to cementation, as well as the maintenance of high bond strength values after long-term storage periods and/or thermocycling (TC) regimens, should be the primary goal. The null hypotheses tested were that 1) the cleansing methods would not negatively influence zirconia bonding; and 2) the aging conditions (ie, TC and 150 days of water storage) would not damage the bond strength between zirconia and resin cement.

METHODS AND MATERIALS

Specimen Preparation

One hundred and eighty zirconia (Diazir®, batch P02286, Ivoclar-Vivadent, Amherst, NY, USA) specimens (12×13×3 mm^3) were obtained from full-contour zirconia blocks with a diamond wafering blade mounted in a precision saw machine (Isomet 1000, Buehler, Lake Bluff, IL, USA). Specimens were sintered at 1500°C according to the manufacturer's instructions in a high-temperature furnace (Lindberg/Blue M, Asheville, NC, USA).[22,23] Specimens were embedded in acrylic resin (Bosworth Fastray™, Bosworth Company, Durham, UK), wet-finished with 600-1200-grit silicon carbide papers (LECO Corporation, Saint Joseph, MI, USA), and cleaned in an ultrasonic bath in distilled water for five minutes. All specimens were sandblasted with 50 μm aluminum oxide particles (Patterson Dental Supply Inc, batch 3150313, St Paul, MN, USA) for 30 seconds, under 2.8 bars and from a distance of approximately 10 mm.[24,25] Next, the specimens were rinsed with water, air-dried, and randomly distributed into five groups (N=36), as follows: control (C)—no saliva contamination; water rinse (W)—specimens were immersed in stimulated human saliva (IRB approval 1105005588) for one minute at 37°C, rinsed with water from a multifunction syringe (MFS) for 15 seconds, and then air-dried[21]; phosphoric acid (PA)—contamination with saliva followed by 37% phosphoric acid (Total Etch, batch R51858, Ivoclar-Vivadent) cleansing for 60 seconds, rinsed with water from MFS for 15 seconds, and air-dried[26,27]; Ivoclean® (IC)—contamination with saliva and cleansing with a commercially available cleaning paste (Ivoclean, batch R53033, Ivoclar-Vivadent), according to the manufacturer's instructions (briefly, it was applied on the bonding surface with a microbrush for 20 seconds and then rinsed with water from MFS); and isopropanol (AL)—contamination with saliva and immersion in 70% isopropa-

Table 1: *Materials, Manufacturer, Batch Number, and Composition*

Materials		Manufacturer	Batch No.	Composition
Zirconia	Diazir Full-Contour	Ivoclar-Vivadent, Amherst, NY, USA	P02286	Y-TZP
Phosphoric acid	Total Etch 37%	Ivoclar-Vivadent, Amherst, NY, USA	R51858	Distilled water, phosphoric acid (85%), thickener, pigments
Clean paste	Ivoclean	Ivoclar-Vivadent, Schaan, Liechtenstein	R53033	Zirconium oxide, water, polyethylene glycol, sodium hydroxide, pigments, additives
Silane	Monobond Plus	Ivoclar-Vivadent, Amherst, NY, USA	R50513	Alcohol solution of silane methacrylate, phosphoric acid, methacrylate and sulfide methacrylate
Resin cement	Multilink Automix	Ivoclar-Vivadent, Amherst, NY, USA	S04093	Dimethacrylate, HEMA, barium glass, ytterbium trifluoride, spheroid mixed oxide

Abbreviations: HEMA, 2-hydroxyethyl methacrylate; Y-TZP, yttria-stabilized tetragonal zirconia ceramics.

nol for two minutes and rinsed with water from MFS for 15 seconds and air-dried. Two additional zirconia specimens were prepared to assess the surface morphology after the different cleaning methods (ie, groups C, W, PA, IC, and AL). Briefly, zirconia specimens were mounted on Al stubs, sputter-coated with Au-Pd alloy, and imaged at various magnifications using a scanning electron microscope (SEM, JSM-6390, JEOL, Tokyo, Japan).

All bonding procedures were carried out immediately after the contamination and cleansing steps. The same individual bonded all the study specimens. The materials, manufacturers, compositions, and batch numbers are listed in Table 1.

Bonding Procedure

After the specimens received the assigned cleansing regime, a silane agent (Monobond Plus, batch R50513, Ivoclar-Vivadent) was applied with a brush and left undisturbed for one minute, and then the solvent was air-dried. Resin cement buttons (ca 2.15 mm in height and 2.38 mm in diameter) were fabricated using a specially fabricated jig (Ultradent, South Jordan, UT, USA) with a cylindrical Teflon mold over each zirconia specimen. The resin cement (Multilink® Automix, batch S04093, Ivoclar-Vivadent) was applied into the mold and then photopolymerized (Demi L.E.D. Dental Curing Light, Kerr Corporation, Middleton, WI, USA), following the manufacturer's instructions. The curing light intensity was measured before bonding procedures (ca 1200 mW/cm^2) using a radiometer (Cure Rite, Curing light meter, Caulk, Dentsply International Inc, Milford, DE, USA).

Aging Conditions

The specimens (N=36) of each group were assigned into three subgroups (n=12), as follows: 1) no aging (ie, the specimens were kept in water for 24 hours at 37°C before testing); 2) TC: the specimens were thermocycled before testing (5000 cycles, 8°C to 48°C, dwell time of 30 seconds, transfer time of 10 seconds)[28]; and 3) water storage: the specimens were kept in water at 37°C for 150 days before testing. The water was changed every other week. No evidence of any bacterial and/or fungal growth was seen; however, the pH was not monitored.

Shear Bond Strength and Failure Analysis

Shear bond strength (SBS) was determined using a dedicated jig (Ultradent) attached to the Universal Testing Machine (ElectroPuls E3000 All-Electric Test Instrument, Instron Industrial Products, Grove City, PA, USA). The load was applied to the adhesive interface until failure at a crosshead speed of 1 mm/min. The maximum stress to produce fracture was recorded (N/mm^2=MPa). The fractured interfacial zones on the zirconia specimens were examined under optical microscopy, and the mode of failure was identified as follows: cohesive resin cement—cohesive failure in the resin cement; Cohesive-ceramic—cohesive failure in the ceramic; and mixed—adhesive failure combined with cohesive failure in the resin cement, adhesive—within any of the substrates or interfaces.[10] Representative specimens were examined under a scanning electron microscope (SEM) (JSM-6390, JEOL, Tokyo, Japan). Images were taken after sputter coating the specimens with gold at different magnifications.

Statistical Analysis

Two-way analysis of variance was used to examine the effects of both the cleansing method and the aging condition on SBS. Comparisons were adjusted for multiple testing using the Tukey method, with an overall significance level of 5%. The SBS data were found to have a log-normal distribution, so the

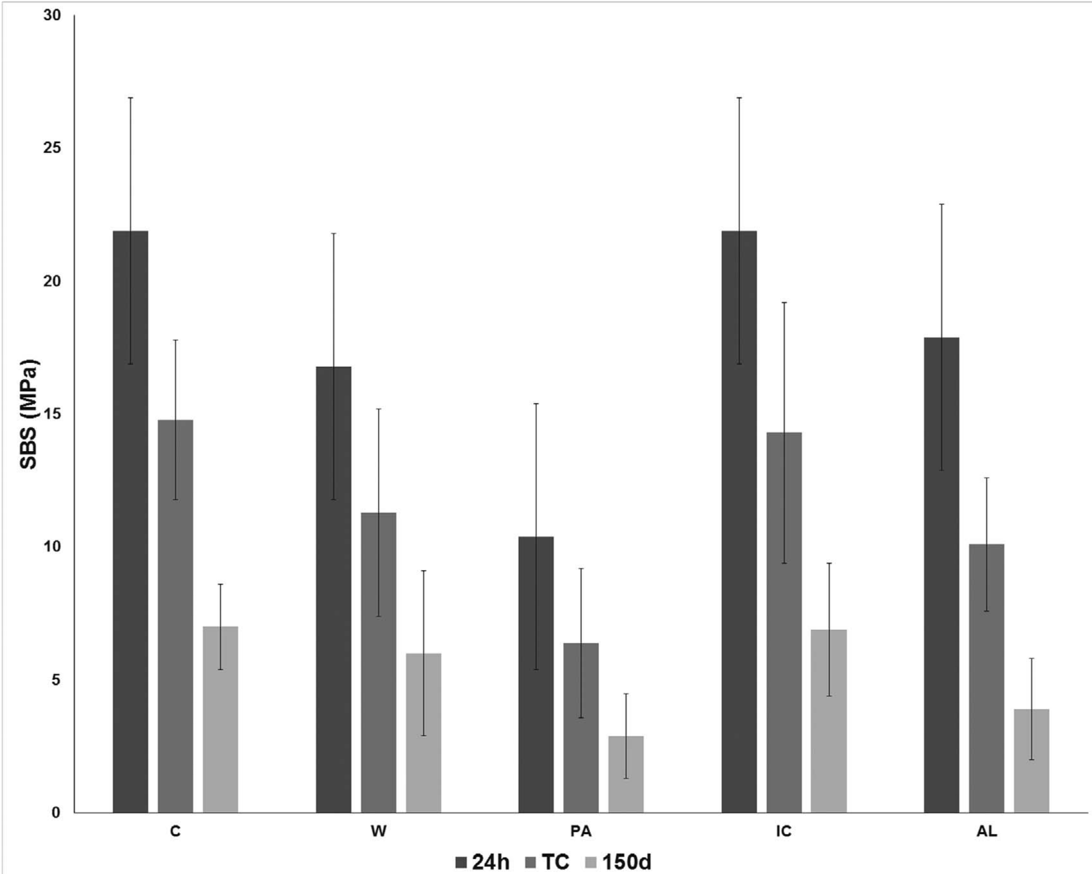

Figure 1. *SBS values after 24 hours, thermocycling, and 150 days.*

analyses were performed on the transformed data. The means along with the 95% confidence intervals were calculated using the transformed data and were then converted back to the original scale to allow the results to be more easily interpreted.

RESULTS

Figure 1 and Table 2 show means and standard deviations of SBS (in MPa). The interaction between groups and the effect of TC and water storage was not significant (p=0.47), indicating that the

Table 2: *Mean (95% Confidence Interval) Shear Bond Strength (SBS; MPa)*[a]

Groups	C		W		PA		IC		AL	
24 h	20.5	a	16.4	a	10.1	a	21.5	a	17.4	a
	(16.1, 26.1)		(14.1, 19.0)		(8.6, 11.9)		(18.9, 24.4)		(14.7, 20.6)	
	A		AB		B		A		A	
TC	14.5	a	10.7	a	5.9	a	13.5	a	9.8	b
	(12.7, 16.6)		(8.6, 13.3)		(4.4, 7.8)		(10.8, 16.8)		(8.4, 11.4)	
	A		A		B		A		AB	
150 d	6.8	b	5.1	b	2.5	b	6.5	b	3.5	c
	(5.8, 7.9)		(3.4, 7.7)		(1.7, 3.6)		(5.2, 8.3)		(2.5, 4.8)	
	A		AB		C		A		BC	

Abbreviations: AL, isopropanol; C, control; IC, Ivoclean cleaning paste; PA, phosphoric acid gel; TC, thermocycling; W, water rinse.
[a] *Lowercase letters in the same column imply statistical similarity among conditions within the groups ($p<0.05$). Uppercase letters in the same row imply statistical similarity among groups within condition ($p<0.05$).*

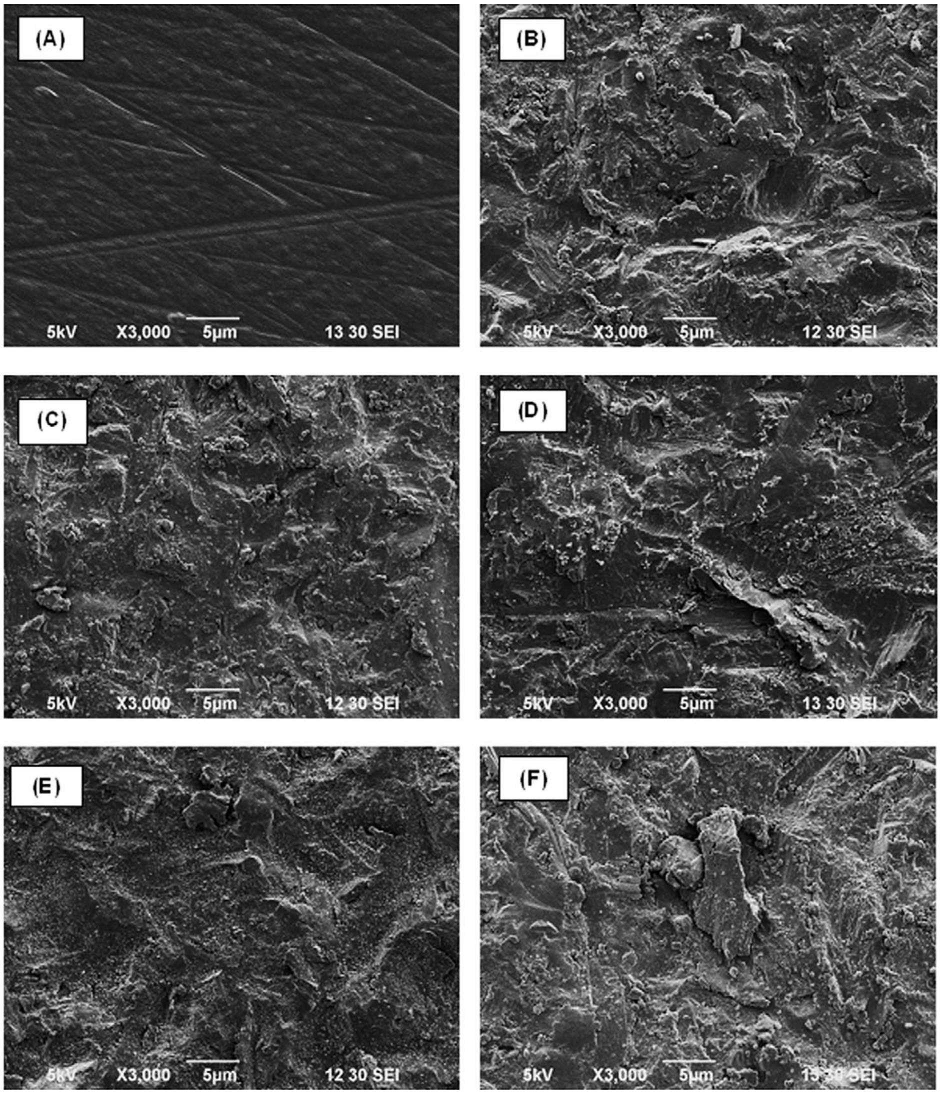

Figure 2. *(A-F) Representative SEM micrographs (3000× magnification) of (A) FCZ surface; (B) FCZ surface after sandblasting, sb; (C) FCZsb after saliva contamination, c; (D) FCZsbc and cleaned with H_3PO_4; (E) FCZsbc and cleaned with Ivoclean®; and (F) FCZsbc and cleaned with isopropanol.*

condition comparisons are valid for all groups and that the group comparisons are valid for all conditions. The effect of TC and water storage comparisons showed the following results: 150 days < TC < 24 hours. The overall group comparisons showed that phosphoric acid < isopropanol and water < cleaning paste and the control group. Figure 2 displays representative SEM micrographs for the zirconia surface morphology after the different cleaning regimens. No obvious morphological differences can be seen among the sandblasted groups (2B-F).

Table 3: *Percentage of Failure Modes Observed in Groups After Shear Bond Strength (SBS) Testing*

	Group														
Condition	**Control, %**			**Water,%**			**H_3PO_4, %[a]**			**Ivoclean, %**			**Isopropanol, %**		
Failures	**Adhesive**	**Cohesive**	**Mixed**	**Adhesive**	**Cohesive**	**Mixed**	**Adhesive**	**Cohesive**	**Mixed**	**Adhesive**	**Cohesive**	**Mixed**	**Adhesive**	**Cohesive**	**Mixed**
24 h	16	0	84	16	0	84	33	0	67	0	0	100	0	0	100
TC	7	0	93	25	0	75	7	0	93	0	0	100	0	0	100
150 d	0	0	100	0	0	100	7	0	93	0	0	100	0	0	100

[a] *Group H_3PO_4: two pretest failures during thermocycling (TC).*

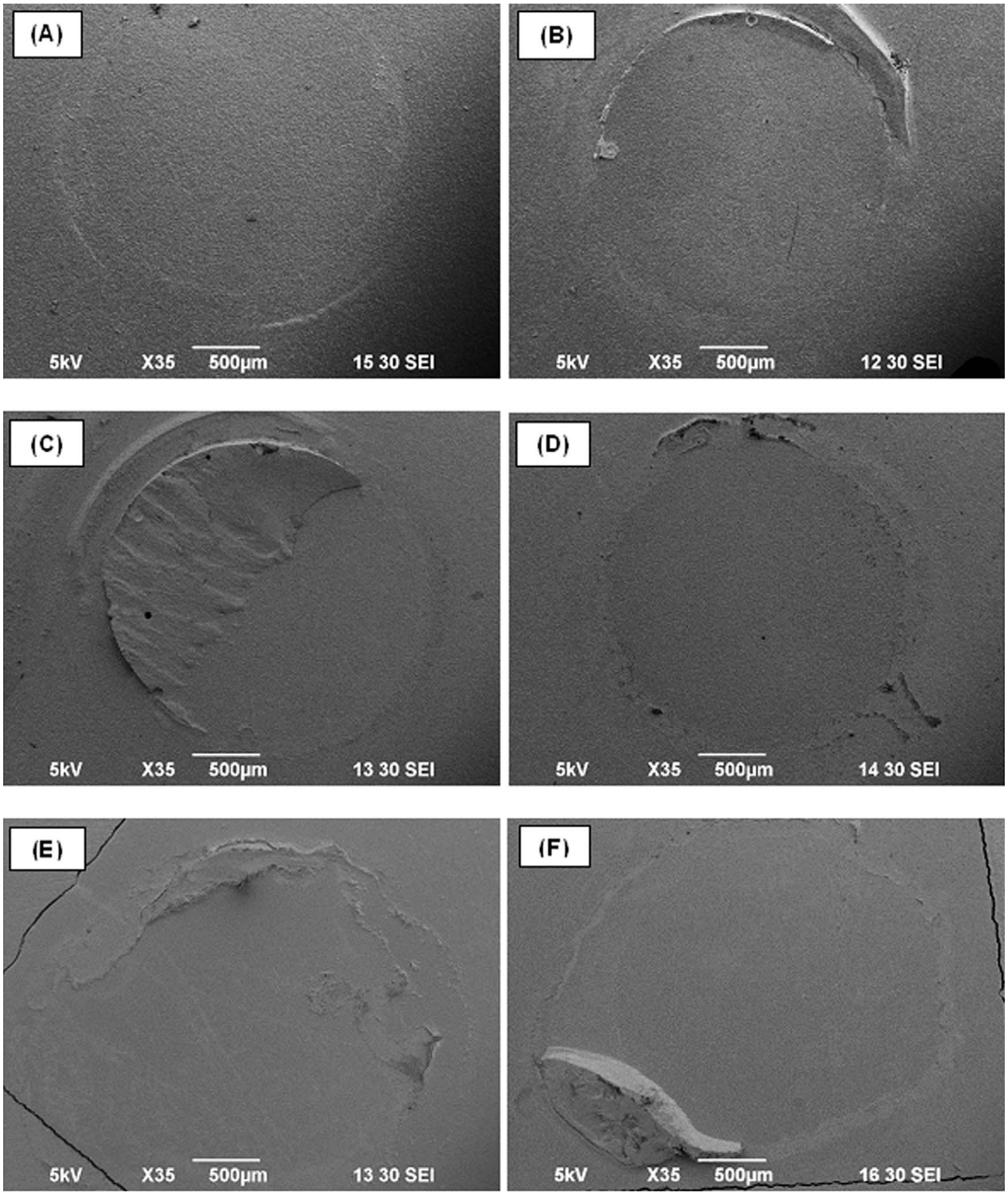

Figure 3. *Representative SEM micrographs of the debonded FCZ surface. Saliva, 24 hours (A): The failure mode was classified as adhesive; Ivoclean, 24 hours (B): The failure mode was classified as mixed with a small amount of composite resin cement on the FCZ surface. Saliva TC (C): The failure mode was classified as mixed with a significant amount of composite resin cement on the FCZ surface; Ivoclean TC (D): The failure mode was classified as adhesive. Saliva, 150 days (C): The failure mode was classified as mixed; and Ivoclean, 150 days (D): The failure mode was classified as mixed.*

Failure analysis revealed a larger percentage of mixed failure (M) for the majority of the specimens and a smaller percentage of adhesive failure at the ceramic-resin cement interface (Table 3). The water group presented a few mixed failures with small amounts of resin cement. Figure 3 shows representative SEM micrographs of the failure modes of the group that utilized the zirconium-based cleaning paste (Ivoclean®) vs control at the three conditions tested.

DISCUSSION

The challenge in promoting a strong, reliable bond between the intaglio (ie, the internal surface of zirconia restorations to resin luting agents) lies in achieving a surface free of the contaminants that often result from intraoral try-in procedures. Previous studies have reported on different cleansing protocols, such as water,[21] alcohol (70%-96% isopropanol),[21,29] phosphoric acid (35%-37%),[21,27,29,30] and additional airborne particle abrasion (Al_2O_3).[21,31] Here, we evaluated the effect of water, H_3PO_4, isopropanol, and a fairly new cleaning paste (Ivoclean®) on the resin/zirconia SBS bond durability. The results of the present study led us to accept the null hypothesis that the cleansing method would not negatively influence zirconia bonding (Table 2) and to reject our second hypothesis, since a significant effect of the aging, especially after 150 days of water storage, promoted a significant reduction in bond strength. The group comparisons after 24 hours showed that all groups presented lower results after 150 days, except group AL, which presented statistically differences after TC and after 150 days.

It is worth mentioning that prior studies[21,30] reported that water rinsing may not be effective to remove some saliva contaminants from the zirconia surface.[21] Studies using XPS showed that H_3PO_4 seems to be an effective cleansing method with which to remove organic contaminants from saliva and blood,[21,27,29] although it, leaves phosphorous residues that could negatively impair bonding ability.[27] As a result, the adhesion between zirconia and resin cement was shown to decrease, consequently changing the surface energy,[21] being unable to reestablish the original bond strength value of the uncontaminated zirconia surface,[30] a finding that is in agreement with the results of the present study. Accordingly, this film associated with water storage and TC changes the bonding interface, which can explain some adhesive failures (Figure 3) presented in groups cleaned with water and H_3PO_4.[30]

Some authors[21] have suggested that an additional particle abrasion may provide good bonding results after contamination, comparable to that seen in groups without contamination. However, the use of a second particle abrasion could be controversial as a result of the potentially deleterious effect on zirconia phase transformation that could possibly weaken the zirconia ceramic.[32]

Several testing methodologies, namely macroshear, microshear, macrotensile, and microtensile tests, have been suggested for evaluation of the bond strength of resin-based materials to dental ceramics where load is applied in order to generate stress at the adhesive joints until failure occurs. Hence, for the test to measure the bond strength values between an adherent and a substrate accurately, it is crucial that the bonding interface should be the most stressed region, regardless of the test methodology being employed. Shear tests have been criticized for the development of nonhomogeneous stress distributions in the bonded interface. On the other hand, conventional tensile tests also present some limitations, such as the difficulty of specimen alignment. Even though the microtensile test allows better specimen alignment and a more homogeneous stress distribution, during cutting procedures the adhesive joint may suffer from early debonding, yielding to high numbers of pretest failures, especially with a zirconia substrate.[33] There is still no consensus in the dental literature with regard to the best surface conditioning method for adequate adhesion of the resin cement to highly crystalline, oxide-based ceramics, but SBS can be useful in ranking materials or systems rapidly. The best outcome could then be tested with more sophisticated methods.

A fairly new cleaning agent called Ivoclean®, which is an alkaline suspension of zirconium oxide particles (ZrO_2), has recently entered the market. In the present study, the Ivoclean group showed bond strength results comparable to those of the control group after TC and water storage. Even though TC and water storage (150 days) reduced the SBS values, the results showed that the Ivoclean and control groups maintained similar SBS values. On the basis of the present study, additional studies, for example, one that makes use of chemical composition analyses through XPS, are suggested to understand the mechanism of Ivoclean® on the saliva-contaminated zirconia surface.

CONCLUSIONS

In conclusion, our findings suggested that a cleansing protocol for zirconia ceramics must be considered after exposure to saliva. The zirconium-based cleaning paste applied on the contaminated zirconia surface is the most effective method, being comparable with the effectiveness of the uncontaminated zirconia control group.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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