

Effect of cleaning agent, primer application and their combination on the bond strength of a resin cement to two yttrium-tetragonal zirconia polycrystal zirconia ceramics

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ABSTRACT

Objective: The objective of this study is to evaluate the effect of a cleaning agent and priming on the bond strength (BS) of a resin cement (RC) to zirconia ceramics after 24 h and 1 year of water-storage. **Materials and Methods:** Fifty-six Katana and 56 ZirCAD zirconia ceramic plates were prepared and each zirconia brand was divided into four groups ($n = 14$): (1) untreated; (2) treated with Ivoclean cleaning agent; (3) treated with Monobond Plus coupling agent; (4) treated with Ivoclean + Monobond Plus. Cleaning and coupling agents were applied to zirconia following the manufacturers' instructions. The RC was manipulated, inserted into tubes (0.75 mm diameter/1 mm height) that were positioned on the zirconia surfaces and light activated. Specimens were tested after 24 h and 1 year of water storage. A shear load was applied to the base of the RC cylinders (until failure). Data were analyzed by three-way ANOVA and Tukey tests ($\alpha = 5\%$). **Results:** No significant difference in BS was noted between zirconia ceramics, except when the combination of cleaning and coupling agents was used. This combination increased the BS for Katana zirconia. One year of water storage leads to a decrease in BS for all experimental groups. **Conclusion:** The combination of cleaning agent and priming can yield higher BS for Katana at 24 h. BS to zirconia ceramics reduced approximately 50% after 1 year.

Key words: Bond strength, coupling agents, resin cement, zirconia

INTRODUCTION

Ceramic materials are widely used in dentistry, because of their high esthetic potential, easily mimicking color, texture, and shape, while restoring function.^[1] These materials are composed of two distinct phases: Glassy and crystalline. However, due to high failure rates, brittleness, low fracture toughness and flexural strength, the use of feldspathic glassy

porcelains in the posterior dentition is rather reduced, while materials with high crystalline content, which have better mechanical properties, have increased. Among the materials with high crystalline content, yttrium-tetragonal zirconia polycrystal (Y-TZP) is a good option to combine favorable mechanical properties and esthetics. Studies have shown that

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Y-TZP ceramics have high flexural strength and modulus, adequate esthetics, biocompatibility, high fatigue resistance under cyclic loading test, and as a result, its application has expanded considerably in dentistry.^[2-4]

Unfortunately, the low adhesive potential is yet a disadvantage of Y-TZP dental ceramics. Unlike feldspathic porcelains that may undergo acid etching, Y-TZP materials are composed of approximately 96%–99% of crystalline structure with no glassy phase.^[3-6] This means that hydrofluoric acid cannot create micro retentions on the surface of crystalline ceramics since its use is restricted to the dissolution of the glassy phase. In addition, because of the absence of silica in its composition, silanization is not indicated. Thus, conventional luting procedures for porcelains are not recommended to Y-TZP restorations.^[4-9]

Studies have described many different luting protocols for Y-TZP. Methods of surface treatment, such as sandblasting with aluminum oxide followed by application of primers and resin cements (RCs) containing phosphate monomers show promising results. Phosphate ester groups present in some primers and RCs have been indicated because they can bond to metal oxides such as zirconium oxide.^[7-10] Some authors suggest that 10-methacryloyloxydecyl dihydrogenphosphate (10-MDP) monomer promotes chemical bonding of RC to zirconia ceramic,^[7] which also showed the ability to resist water storage. Later studies have confirmed these results.^[8-12]

Nonetheless, to increase bond strength (BS), bonding procedures must be performed in contaminant-free surfaces; otherwise, it could lead to reduced durability. Many cleaning protocols to achieve durable bonding have been proposed, such as water rinsing, acetone cleaning, 37% phosphoric acid application, ethanol, and sandblasting. Among them, sandblasting seems to be more efficient than organic solvents,^[13] such as phosphoric acid and acetone.^[13] Beyond surface roughening, its use is extended to surface decontamination from phosphorous compounds or silicon originated from “try-in” procedures in the mouth.^[13,14] It also improves surface reactivity and wettability, prerequisites to a strong adhesive bonding.^[15]

Recently, a new cleaning detergent to decontaminate the zirconia extra-orally has been developed.^[16] Its technology relies on an alkaline suspension of

zirconium oxide particles, which is likely to bond to phosphate contaminations, leaving behind a clean surface. However, in the authors’ opinion, clinicians have a tendency to overdo cleaning procedures to ensure a safe bonding to the restorative prosthesis. Therefore, in the present study, the consensus was to evaluate the BS of noncontaminated ceramics *in vitro*, associating sandblasting followed by the use of the previously mentioned cleaning detergent.

Thus, the purpose of this study was to examine the influence of a commercially available cleaning agent, before priming, and their effects on the BS of RC to two contaminant-free zirconia ceramics. The hypotheses to be tested were that (1) cleaning agent and priming would improve the BS to clean zirconia and (2) the long-term storage would not reduce resin BS to them.

MATERIALS AND METHODS

Specimen preparation and experimental groups

For the present study 56 Katana zirconia (lot # BNAHZ, Kuraray Noritake Dental, Tokyo, Japan) plates and fifty-six ZirCAD zirconia (lot # M60517, Ivoclar Vivadent AG, Schaan, Liechtenstein) plates with dimensions of 9 mm length × 5 mm width × 1 mm thickness were used. These plates were obtained from computer-aided design (CAD)/computer-aided manufacturing blocks, which were cut with a precision saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) under water-cooling and later sintered for 1 h at 1550°C.

Second, the zirconia plates were sandblasted (Microetcher, Danville) with aluminum oxide (50 µm, Danville Engineering Inc., San Ramon, CA, USA) for 15 s, at a distance of 10 mm and 2.5 bar pressure. Finally, the plates were ultrasonically cleaned with distilled water for 5 min and each zirconia brand was randomly divided into four groups ($n = 14$). The method of randomization for designation of plates into groups and specimen preparation was the sortition. The groups comprised:

- Untreated and Multilink Speed RC (lot #P62316, Ivoclar Vivadent AG) applied directly onto the surface (control)
- Surface treated with Ivoclean cleaning agent (lot #P66483, Ivoclar Vivadent AG) followed by RC application
- Surface treated with Monobond Plus coupling agent (lot #P50852, Ivoclar Vivadent AG) followed by RC application
- Surface treated with Ivoclean followed by Monobond Plus and RC.

First, Ivoclean was applied actively onto the surface for 60 s, rinsed with water stream for 20 s and air-dried. Secondly, a layer of Monobond Plus was applied for 60 s.

Four cylindrical transparent matrices (Tygon tubing, TYG-03, Saint-Gobain Performance Plastic, Maim Lakes, FL, USA) of 1 mm height × 0.7 mm diameter were positioned on the zirconia surfaces and an automix RC (Multilink Speed, Ivoclar Vivadent AG) was placed into the tubes with the assistance of a #5 explorer (Hu-Friedy, Chicago, IL, USA). Each cylinder filled with RC was light-activated for 20 s using a LED light-curing unit (approximately 1000 mW/cm², Valo, Ultradent Products Inc., South Jordan, UT, USA). Plastic matrices were removed to expose four RC cylinders per zirconia plate, which were stored in distilled water at 37°C for 24 h. Two resin cylinders were tested at 24 h, while the two remaining underwent testing after 1 year [Figure 1]. For this long-term storage, water had to be changed every 2 weeks.

Bond strength test and failure pattern analysis

Microshear BS (μSBS) test was performed using a universal testing machine (EZ Test, Shimadzu Corp, Kyoto, Japan) at a crosshead speed of 0.5 mm/min until failure. The shear load was applied at the base of the RC cylinders with a loop wire (0.2 mm diameter). BS data were calculated using the peak of loading failure divided by specimen surface area and means were obtained in MPa. The factors under study considered for statistical analysis were: (1) “Type of zirconia” (ZirCAD or Katana), (2) “treatment” (none, cleaning agent, priming, and combination) and (3) “evaluating time” (24 h or 1 year). Data were analyzed by three-way ANOVA and Tukey’s *post hoc* test (α = 0.05), using SAS 9.3 Software (SAS Institute Inc., Cary, NC, USA).

Specimens were mounted on metal platforms, gold-coated and observed using scanning electron

microscope (JSM5600, Jeol Ltd., Tokyo, Japan). Failure pattern was classified as (1) Cohesive failure within RC; (2) adhesive failure between zirconia and RC; (3) mixed failure.

RESULTS

Table 1 displays means and standard deviations of μSBS of RC to the ceramics. The three-way ANOVA demonstrated that “type of zirconia” (P = 0.0320), “treatment” (P = 0.0102) and “evaluation time” (P < 0.0001) significantly influenced the μSBS results. For ZirCAD, different treatments did not influence μSBS (P > 0.05), while for Katana the combination of Ivoclean and Monobond Plus resulted in increased μSBS of the RC (P < 0.05). There was not a significant difference in BS following the same treatment among zirconia, except when the combination of Ivoclean and Monobond Plus was applied. This combination produced higher BS for Katana than ZirCAD (P < 0.05). At 1 year, water storage reduced the μSBS for both zirconia ceramics (P < 0.05).

Failure mode is presented in Table 2. Cohesive failure within RC was observed for all groups. In general, long-term storage changed the failure mode, except when the Katana zirconia treated with only the cleaning agent. ZirCAD showed low incidence of adhesive failure at 1 year. Mixed failures were not predominant for either zirconia ceramics, except for control group of Katana at 24 h. Figures 2-6 show representative failure modes of the groups tested.

DISCUSSION

Researchers and clinicians constantly face difficulties to promote durable BS to zirconia substrates. Methods

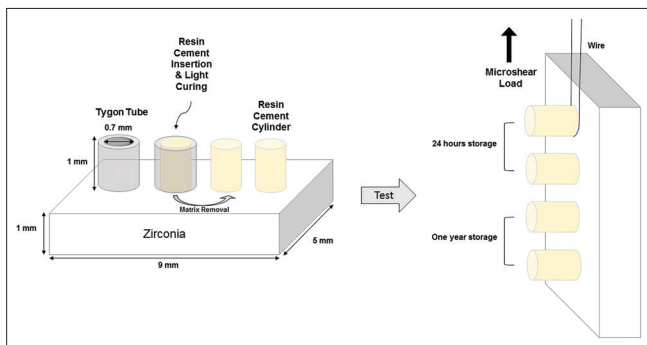


Figure 1: Schematic representation of specimen preparation

Table 1: Means (standard deviation) of bond strength of the resin cement to zirconia ceramics (in MPa)

Time (h)	Treatment	Zirconia	
		ZirCAD	Katana
24 h	Control	41.8 (3.2) ^{*A,a}	*39.8 (1.5) ^{*A,b}
	Ivoclean	38.8 (2.4) ^{*A,a}	*39.7 (1.4) ^{*A,b}
	Monobond Plus	40.2 (1.9) ^{*A,a}	*38.8 (3.5) ^{*A,b}
1 year	Ivoclean + Monobond Plus	39.3 (4.0) ^{*A,a}	44.6 (2.4) ^{*B,a}
	Control	21.7 (2.7) ^{A,a}	19.8 (3.5) ^{A,a}
	Ivoclean	20.2 (2.8) ^{A,a}	22.2 (1.4) ^{A,a}
	Monobond Plus	20.0 (2.0) ^{A,a}	22.6 (1.1) ^{A,a}
	Ivoclean + Monobond Plus	20.1 (1.7) ^{A,a}	23.0 (1.3) ^{A,a}

Identical letters indicate no significant difference (P>0.05). Capital letters compare zirconia ceramics within the same treatment (row), and lower cases compare treatments within the same zirconia (column). *Same groups differ between 24 h and 1 year

Table 2: Failure pattern for tested specimens (in percentage)

Zirconia	Cleaning agent	Coupling agent	Evaluation time	Type 1	Type 2	Type 3
ZirCAD	No	No	24 h	40	45	15
ZirCAD	No	No	1 year	82	18	-
ZirCAD	Yes	No	24 h	25	60	15
ZirCAD	Yes	No	1 year	75	-	25
ZirCAD	No	Yes	24 h	18	67	15
ZirCAD	No	Yes	1 year	100	-	-
ZirCAD	Yes	Yes	24 h	25	60	15
ZirCAD	Yes	Yes	1 year	90	-	10
Katana	No	No	24 h	17	18	65
Katana	No	No	1 year	66	34	-
Katana	Yes	No	24 h	18	47	35
Katana	Yes	No	1 year	18	42	40
Katana	No	Yes	24 h	68	32	-
Katana	No	Yes	1 year	75	-	25
Katana	Yes	Yes	24 h	40	35	25
Katana	Yes	Yes	1 year	67	-	33

Type 1: Cohesive failure within resin cement, Type 2: Adhesive failure, Type 3: Mixed failure

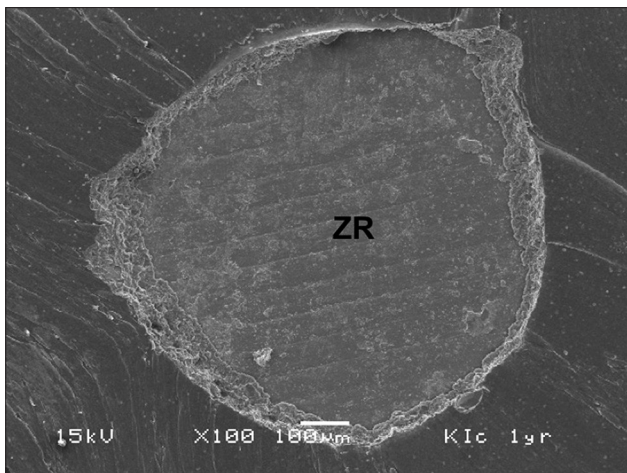


Figure 2: Scanning electron microscope photomicrograph illustrating an adhesive failure along the zirconia surface when zirconia (Katana) was cleaned with Ivoclean at 1 year ($\times 100$)

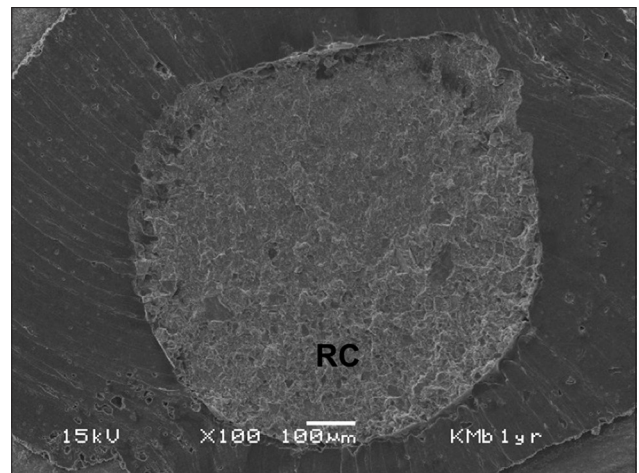


Figure 3: Scanning electron microscope photomicrograph illustrating a cohesive failure within resin cement when zirconia (Katana) was treated with silane coupling agent at 1 year ($\times 100$)

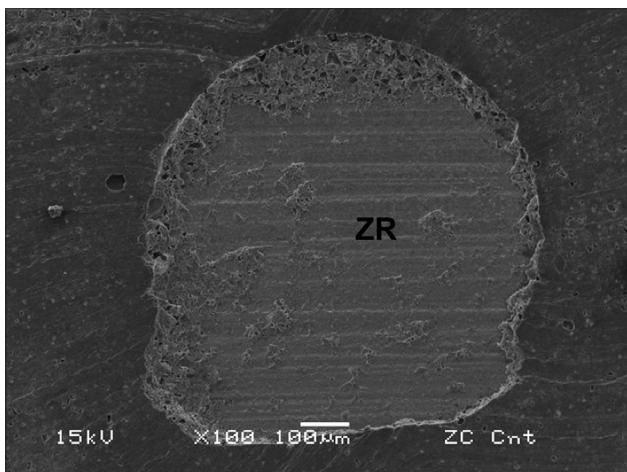


Figure 4: Scanning electron microscope photomicrograph illustrating an adhesive failure along the zirconia surface when zirconia (ZirCAD) was untreated (control) at 24 h ($\times 100$)

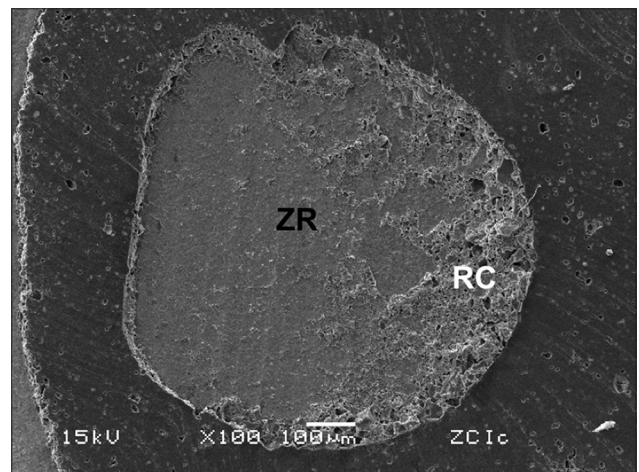


Figure 5: Scanning electron microscope photomicrograph illustrating a mixed failure when zirconia (ZirCAD) was cleaned with Ivoclean at 24 h (resin cement; zirconia; $\times 100$)

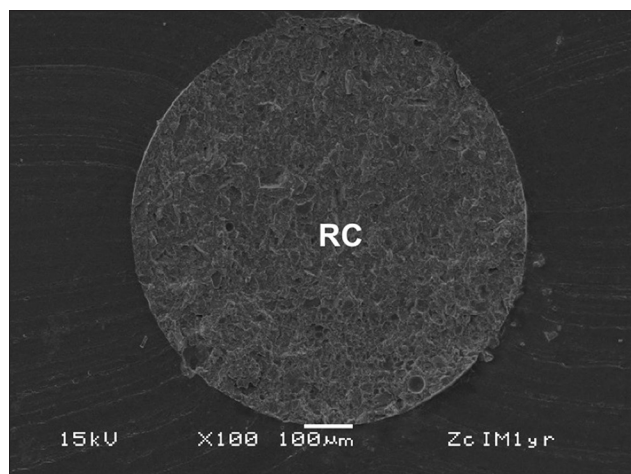


Figure 6: Scanning electron microscope photomicrograph illustrating a cohesive failure within resin cement when zirconia (ZirCAD) was cleaned with Ivoclean and primed with coupling agent at 1 year ($\times 100$)

to enhance bonding rely solely on the alteration of surface topography and/or chemistry by means of sandblasting or tribochemical silica-coating.^[6,8-12] Nonetheless, this may not guarantee durable bonding^[7] and therefore a effective luting protocol seems to be unknown. In the present study, the authors evaluated the effect of the application of a cleaning agent (Ivoclean) followed by priming (Monobond Plus), on contaminant-free, sandblasted surface by assessing resin-cement μ SBS. By the results obtained in this study, one could partly accept the first hypothesis, since the combination of Ivoclean and Monobond Plus increased μ SBS only to one commercially-available material (Katana). However, the second hypothesis must be rejected because 1 year of water storage reduced BS approximately 50% for all experimental groups when compared to 24 h of storage.

Other studies have tested the same cleaning agent, but using different protocols.^[13,14,17] In such cases, tested specimens were exposed to contaminants (e.g., saliva) prior to the application of the product in order to evaluate its potential to remove remnant contamination that could be originated during try-in procedures. However, the present study focused on the application of cleaning agent after sandblasting since it is believed that the latter should be done only after try-in procedures, therefore creating a clean and reactive surface without excessively damaging the material. Moreover, to assess Ivoclean ability to improve resin BS to zirconia, the present study evaluated not contaminated surfaces to verify if Ivoclean interferes negatively or positively on BS.

A previous study^[13] showed no statistical difference between cleaned, sandblasted zirconia specimens and the combination of sandblasting followed by cleaning with Ivoclean, on saliva contaminated specimens. These results may suggest that air abrasion is an efficient method on removing contaminations and the use of Ivoclean cannot increase BS any further. For the Katana group, the combination of Ivoclean and Monobond Plus yield higher BS to the RC (44.6 MPa). This was the 1st time in the literature that the combination of these materials (Ivoclean + Monobond Plus) showed higher BS than the control group without contamination. The primer used in the present study (Monobond Plus) is comprised metacrylated phosphoric acid ester and according to the manufacturer, its application range from oxide ceramic, glass ceramics, and metal. For zirconia, this claim can be supported by the fact that many studies have shown the ability of phosphoric primers to bond to metal oxides, and because crystalline ceramics such as zirconia are composed mainly by ZrO_2 , such interaction is plausible.^[6,8-11]

Another important information brought in the study was a significant difference of BS after Ivoclean + Monobond Plus application between zirconias. Although the composition of the ceramic materials is considered similar (as specified by both material safety data sheet), they are delivered in different states. Katana zirconia provided for the study comes in a green state CAD blocks, but the ZirCAD comes in pre-sintered CAD blocks. The authors assume that the inherent porosity present in the green state material could contribute to a rougher surface and increased free surface area, even after sintering consequently yielding higher BS. However, these statements are based on personal opinions only and must be scientifically assessed to confirm such presumption.

A recent study evaluates the chemical bonding between 10-MDP and tetragonal zirconia, and the effect of pH reaction conditions on BS. They reported that the application of MDP in alkaline conditions showed higher BS than that obtained in acid conditions because in alkaline conditions allowed better formation of MDP-zirconia bonds.^[18] The cleaning agent used in this study also has high pH, which might be responsible for improving the BS when used in combination with primer.

Analysis of fractured interfacial zone shows a predominance of adhesive failures for all ZirCAD

groups evaluated after 24 h, which might be related to low interaction between RC and zirconia. After 1 year of water storage, cohesive failure within RC was prevalent, might be because the degradation of RC cylinder. For Katana control group, mixed failure occurred more frequently, while adhesive failure tended to happen when the cleaning agent alone was applied before the RC. Groups that used Monobond Plus showed greater amounts of cohesive failures within RC at 24 h, which might be related to an efficient BS (short-term tests) after the application of Ivoclean + Monobond Plus, which is consistent to the results found in the study for this group.

CONCLUSION

The present study findings conclude that:

- The treatment proposed (cleaning agent, zirconia primer, and the combination of both) produced low effect in increasing BS in most of the groups
- At 24 h, only the combination of cleaning and priming agents produced higher BS for Katana zirconia
- Storing the specimens for 1 year reduced BS in approximately 50% for all groups tested.

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Conflicts of interest

There are no conflicts of interest.

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