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Evaluation of Chemical Treatment on Zirconia Surface with Two Primer Agents and an Alkaline Solution on Bond Strength

FC Lorenzoni • VP Leme • LA Santos
PCG de Oliveira • LM Martins • G Bonfante

Clinical Relevance

Achieving a reliable bond to zirconia is still a challenge. The chemical conditioning methods, which do not require the micromechanical topographical changes, enhanced the shear bond strength to zirconia.

*Fabio Cesar Lorenzoni, PhD candidate student, Department of Prosthodontics, Bauru School of Dentistry, São Paulo University, Bauru, Brazil

Viviane Paes Leme, DDS, Bauru, Brazil

Lívia Aguiar Santos, DDS, MS, Department of Prosthodontics, Bauru School of Dentistry, University of São Paulo, Bauru, Brazil

Pedro Cesar Garcia de Oliveira, DDS, MS, PhD, Department of Prosthodontics, Bauru School of Dentistry, University of São Paulo, Bauru, Brazil

Leandro de Moura Martins, DDS, MS, PhD , Federal University of Amazonas, School of Dentistry, Amazonas, Brazil

Gerson Bonfante, DDS, MS, PhD, Department of Prosthodontics, Bauru School of Dentistry, University of São Paulo, Bauru, Brazil

*Corresponding author: al. Octávio Pinheiro Brisola, 9-75, Bauru, São Paulo 17012-180, Brazil; e-mail: fcesarlorenzoni@yahoo.com.br

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SUMMARY

Objectives: **This study evaluated the effect of an alkaline solution and two 10-methacryloyloxydecyl dihydrogen phosphate (MDP)-based primer agents on bond strength to zirconia (yttria-stabilized tetragonal zirconium polycrystal [Y-TZP]) through the shear bond strength (SBS) test.**

Materials and Methods: **Sixty square-shaped Y-TZP samples were embedded in an acrylic resin mold, polished, and randomly assigned to one of six groups (n=10) according to treatment surface: group CR, no treatment (control); group NaOH, 0.5 M NaOH; group AP, Alloy Primer; group ZP, Z-Primer Plus; group NaOH-AP, 0.5 M NaOH + Alloy Primer; and group NaOH-ZP, 0.5 M NaOH + Z-Primer Plus. The resin cement (Rely X U100) was applied inside a matrix directly onto the Y-TZP surface, and it was light-cured for 40**

seconds. The samples were stored in distilled water at 37°C for 24 hours prior to the test, which was performed in a universal machine at a crosshead-speed of 0.5 mm/min. The data were analyzed by one-way analysis of variance and Tukey tests ($p<0.05$). Light stereomicroscopy and scanning electron microscopy were used to assess the surface topography and failure mode.

Results: The SBS was significantly affected by the chemical treatment ($p<0.0001$). The AP group displayed the best results, and the use of NaOH did not improve SBS results relative to either AP or ZP. The samples treated with Alloy Primer displayed mainly mixed failures, whereas those conditioned with Z-Primer Plus or with 0.5 M NaOH presented a balanced distribution of adhesive and mixed failure modes.

Conclusions: The use of a NaOH solution may have modified the reactivity of the Y-TZP surface, whereas the employment of a MDP/6-4-vinylbenzyl-*n*-propyl amino-1,3,5-triazine-2,4-dithione–based primer enhanced the Y-TZP bond strength.

INTRODUCTION

Fully ceramic systems are attractive materials because of their excellent properties, including high biocompatibility and esthetic potential.[1] The first use of zirconia as a biomaterial involved its use in artificial limbs,[2] whereas its introduction to clinical dentistry has been more recent. Three percent yttria-stabilized tetragonal zirconium polycrystal (Y-TZP) presents a flexural strength and fracture toughness of 1100 MPa[3] and 6.27 MPa $m^{1/2}$,[4] respectively, which are higher than those of alumina (514 MPa[5] and 4.78 MPa $m^{1/2}$[4]) or lithium disilicate (407 MPa[6] and 3.0 MPa $m^{1/2}$[7]) ceramics. Nowadays, Y-TPZ is a safe treatment option for single or multiple tooth replacements, including those located in the posterior region.[8–10]

Whereas the main failure mode of all-ceramic systems involves bulk fracture (except for Y-TZP–based restorations),[11] the porcelain veneer fracture represents the most prevalent mechanical complication observed in Y-TZP–based restorations, showing rates of 15%[12] and 20%.[13] Several *in vitro*[14–18] and *in vivo*[12,13,19,20] studies have addressed the reasons behind the chipping of porcelain veneers. On the other hand, a recent three-year follow up[21] determined that 7% of all Y-TZP crowns placed in the molar region lost retention. Although the loss of retention has never been a topic of interest with Y-TZP restorations (because it has never been reported), efforts have been undertaken to establish ways to achieve a reliable bond between Y-TZP crowns and luting agents.[22–34] Comparatively, data from a literature review[11] showed a 2.8% loss of retention rate in all-ceramic systems (except for zirconia) after five years, while for a metal-ceramic system the loss of retention rate decreases to 0.7% after 10 years.[35] Thus, the effective bond strength between the substrate and ceramic plays an important role in enhancing the longevity of restorative treatments[25,29,36–40] and in preventing microleakage.[32]

Although alternatives such as sandblasting with Al_2O_3 particles,[37] tribochemical silica coating,[25] selective infiltration etching,[23,41] and experimental hot etching solution[41] have enhanced the zirconia bond strength, the concept employed for all of these methods relies on the micromechanical interlocking between the zirconia and the cement. Whereas sandblasting has been associated with microcracks, which may decrease the mechanical properties of Y-TZP,[42,43] neither selective infiltration etching or experimental hot etching solution has not been examined with regard to this defect to date. Furthermore, these procedures are invasive methods and often require special equipment. Moreover, there is no consensus about the most appropriate or reliable method with which to bond Y-TZP restorations.

Taking into account the above-mentioned concerns associated with sandblasting,[43] the chemical interaction between zirconia and the luting agent must be evaluated. The chemical adhesion potential of zirconia is low as a result of its inertness (ie, it presents a nonpolar surface),[44] which hampers its union with cements.[27] However, it has been discussed[45] that an increased availability of hydroxyl groups was found at the implant surface of a zirconia/alumina nanocomposite after 15 M sodium hydroxide solution (NaOH) treatment. In addition, durable bond strength may be achieved by employing acid monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP)-based primers.[32]

Considering that strong chemical bonds with zirconia have been proven to be difficult as a result of zirconia's nonreactive surface,[34] the present study sought to assess *in vitro* the effect of hydroxylation of the zirconia surface on shear bond strength (SBS) with either the combination or exclusion of two commercially available primers. The following null hypothesis was tested: no differences in SBS will be observed among the groups treated with the different primers with or without the use of NaOH.

MATERIALS AND METHODS

Four pre-sintered blocks of Y-TZP (IPS ZirCAD, InLab Blocks C15, Ivoclar Vivadent, Schaan/Liechtenstein, Germany) were sintered in a furnace (Sintramat, Ivoclar Vivadent) at 1500°C for eight hours. Subsequently, each block was cut on a machine saw (Isomet™ 1000, Buehler, Lake Bluff, IL, USA) at a speed of 500 rpm under constant irrigation with water, producing 60 square-shaped zirconia specimens with dimensions of $1 \times 10 \times 10$ mm. Next, each square-shaped specimen was embedded in acrylic resin mold (ie, so that one side of the Y-TZP square was not clouded by the resin acrylic) to be attached into the shear test jig (Figure 1). All of the materials used in this study are described in Table 1.

The Y-TZP surface was polished with 320-, 400-, and 600-grit carbide silicon papers[28] under abundant water irrigation in a polishing machine (Struers Polisher Metallographic DP-10, São Paulo, SP, Brazil). Subsequently, the Y-TZP surfaces were cleaned with 96° GL alcohol in an ultrasonic bath (Unique USC700, Indaiatuba, SP, Brazil) for three minutes.[46,47] The specimens were randomly divided into six experimental groups (n=10) according to the surface treatment employed, as follows: group CR (control): No treatment was provided to the zirconia surface; group NaOH: A 0.5 M NaOH was applied in excess to the zirconia surface for 60 seconds and then it was washed out with distilled water for 20 seconds and dried with a gentle oil-free air stream for 10 seconds; group AP: The Alloy Primer was applied on the zirconia surfaces in excess with a microbrush and it was allowed to react with the surface for 60 seconds. Afterward, a gentle oil-free air stream was directed for 10 seconds to dry the surface; group ZP: Z-Primer Plus was applied on the zirconia surfaces, also in excess, with a microbrush and it was allowed to react for 60 seconds. Subsequently, a gentle oil-free air stream was applied for 10 seconds to dry the surface; group NaOH-AP: As described for the NaOH group, the 0.5 M NaOH solution was applied on the zirconia surface; this was followed by the application of the Alloy Primer (as described for the AP group); and group NaOH-ZP: As described for the NaOH group, the 0.5 M NaOH solution was applied on the zirconia surface; this was followed by the application of the Z-Primer Plus (as described for the ZP group).

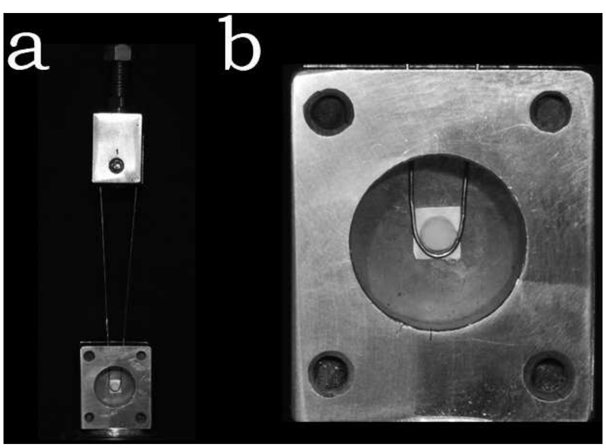

Figure 1. *Shear bond strength testing configuration.*

Afterwards, a polytetrafluoroethylene matrix of 3 mm internal diameter and 3 mm internal height was placed on the center of the Y-TZP square[28] and filled with the luting agent (Rely X U100, 3M ESPE, St Paul, MN, USA). Two minutes after the start of mixing, the cement was light-activated with a halogen light-curing unit (750 mW cm^{-2}; Optilight Plus, Gnatus, Ribeirão Preto, SP, Brazil) for 40 seconds. The preparation of the luting agent and the curing time followed the manufacturer's guidelines. Prior to the tests, all specimens were stored in deionized water in a chamber (Estufa de Secagem 410/1ND, Nova Ética, São Paulo, SP, Brazil) at 37°C for 24 hours. The SBS tests were performed in a universal testing machine (Emic-2000, São José dos Pinhais, PR, Brazil) with a 50-KgF load cell at a constant crosshead speed of 0.5 mm/min. The force was concentrated on the ceramic/cement interface. The shear bond strength (σ) values (MPa) were determined from the following equation:

$$\sigma = \frac{P}{A},$$

in which P is the maximum load (Newtons) required to produce the fracture, and A is the adhesive cross-sectional area (where $A = \pi r^2$). The r denotes the diameter of the bonded area divided by 2, which was measured with a digital caliper (Mitutoyo, Tokyo, Japan).

Additionally, the fractured surfaces were inspected with a light stereomicroscope (Zeiss Stereomicroscope Leica MZE, Mannheim, Germany) at 20× magnification, aided by an external light source (Leica CL5 150D). Using the grading method[26] employed to assess failure modes in zirconia/cement interfaces, the failures were classified into the following categories: 1) cohesive failures in cement; 2) adhesive failures between the ceramic and

Table 1: *Materials, Chemical Compositions, and Manufacturers of the Investigated Materials*

Material	Composition and Batch No.	Manufacturer
e.max ZirCAD	Zirconium dioxide partially stabilized by yttrium in the tetragonal phase (Batch L 15418)	Ivoclar Vivadent, Schaan, Liechtenstein, Germany
Alloy Primer	Acetone, 10-ethacryloyloxydecyl dihydrogen phosphate (MDP), 6-(4-vinylbenzyl-*n*-propyl) amino-1,3,5-triazine-2,4-dithione (VBATDT) (C6415)	Kuraray Medical, Tokyo, Japan
Z-Primer Plus	Biphenyl dimethacrylate, hydroxyethyl methacrylate, ethanol (0900011498)	Bisco, Schaumburg, IL, USA
Rely X U100	Base: fiberglass, acrylic acid ester, dimethacrylate trietilenoglico, silica treated with silane and sodium persulfate	3M ESPE, St Paul, MN, USA
	Catalyst: fiberglass, substitute dimethacrylate, silica treated with silane, *p*-toluenesulfonate sodium and calcium hydroxide (349178)	
Sodium Hydroxide (NaOH)	NaOH 0.5 M, pH 13	Fórmulas Bauru, SP, Brazil

cement; and 3) mixed failures (a combination of adhesive and cohesive failures). Representative specimens from each group were examined under scanning electron microscope (SEM) (Model 3500S, Hitachi Ltd, Osaka, Japan). The data obtained from the SBS test were evaluated using one-way analysis of variance to analyze the chemical surface treatments of Y-TZP. A *post hoc* Tukey test was applied to identify pairwise differences among the tested groups. Both tests employed a pre-set significance level of 5%.

RESULTS

The SBS was significantly affected by the chemical treatment ($p<0.0001$). The highest bond strength values were observed in the NaOH-AP (12.71 MPa) and AP (11.94 MPa) groups and were statistically higher than in other groups. The bond strength value for the NaOH group (6.74 MPa) was comparable to values for the ZP (6.89 MPa) and NaOH-ZP (5.85 MPa) groups, although it was lower than those of the AP and NaOH-AP groups. The use of 0.5 M NaOH did not improve the SBS outcomes relative to either AP or ZP Primers. The CR group (3.69 MPa) showed the lowest bond strength. The average values of SBS, along with standard deviations and minimum and maximum values, are shown in Table 2.

The failure analysis showed that the AP and NaOH-AP groups mainly exhibit mixed failure modes, whereas the NaOH, NaOH-ZP, and ZP groups displayed a balanced distribution between adhesive and mixed failure modes. The results from inspections of the Y-TZP surfaces are presented in Table 3. SEM analysis of one representative failure mode from each group is illustrated in Figure 2. Neither group showed a cohesive failure type. No samples showed spontaneous debonding prior to testing.

Table 2: *Bond Strength, Standard Deviation (SD), and Minimum and Maximum Values Are Described in MPa*

Group	Bond Strength (±SD)[a]	Minimum Value	Maximum Value
CR	3.69 (1.45) A	1.22	6.01
NaOH	6.74 (1.91) B	3.72	9.21
AP	11.94 (3.30) C	8.46	20.07
ZP	6.89 (1.99) B	4.58	10.42
NaOH-AP	12.71 (1.86) C	9.50	15.02
NaOH-ZP	5.85 (2.12) AB	3.05	9.07

Abbreviations: AP, Alloy Primer; CR = control; NaOH, sodium hydroxide; ZP, Z-Primer Plus.
[a] *Identical letters show that the values were not statistically different (Tukey test, $p>0.05$).*

Table 3: *Failure Mode Classifications After Shear Bond Strength (SBS)*

Failure Mode	CR, %	NaOH, %	AP, %	ZP, %	NaOH-AP, %	NaOH-ZP, %
Adhesive	100	40	0	50	20	60
Cohesive	0	0	0	0	0	0
Mixed	0	60	100	50	80	40

Abbreviations: AP, Alloy Primer; CR = control; NaOH, sodium hydroxide; ZP, Z-Primer Plus.

DISCUSSION

Achieving a reliable and adequate bond strength to Y-TZP has proven to be difficult,[48] in part because of Y-TZP's microstructure and chemical composition, which prevent the action of the hydrofluoric acid etch (ie, it does not make any changes with regard to the surface morphology of zirconia).[49,50] Unlike glasses and porcelains,[48] it also fails to respond to silanization procedures.[51] This study evaluated the effect of application of 0.5 M NaOH solution either with or without the association of two primer agents (Alloy Primer and Z-Primer Plus) on bond strength to zirconia. The results revealed that all of the provided chemical treatments increased the SBS when compared with the CR group, which supports the rejection of the null hypothesis.

Although the surface treatment with 0.5 M NaOH did not increase statistically the SBS results when combined with either AP or ZP Primers, it improved the bond strength when compared with the CR group. Two main reasons might explain such a finding. First, it can be speculated that there was activation of the zirconia surface due to the increased availability of hydroxyl groups (OH) after the application of the 0.5 M NaOH. This factor may have favored the acid-base reaction between the metal oxides present on the zirconia surface with both cement and primer agents, which are admittedly acidic. Second, the surface energy may have been raised (mainly the polar component), which could increase the wettability of the zirconia surface, and, as a result, it may have favored the bond reacting between the metal oxides on the zirconia surface and the functional monomer present in the

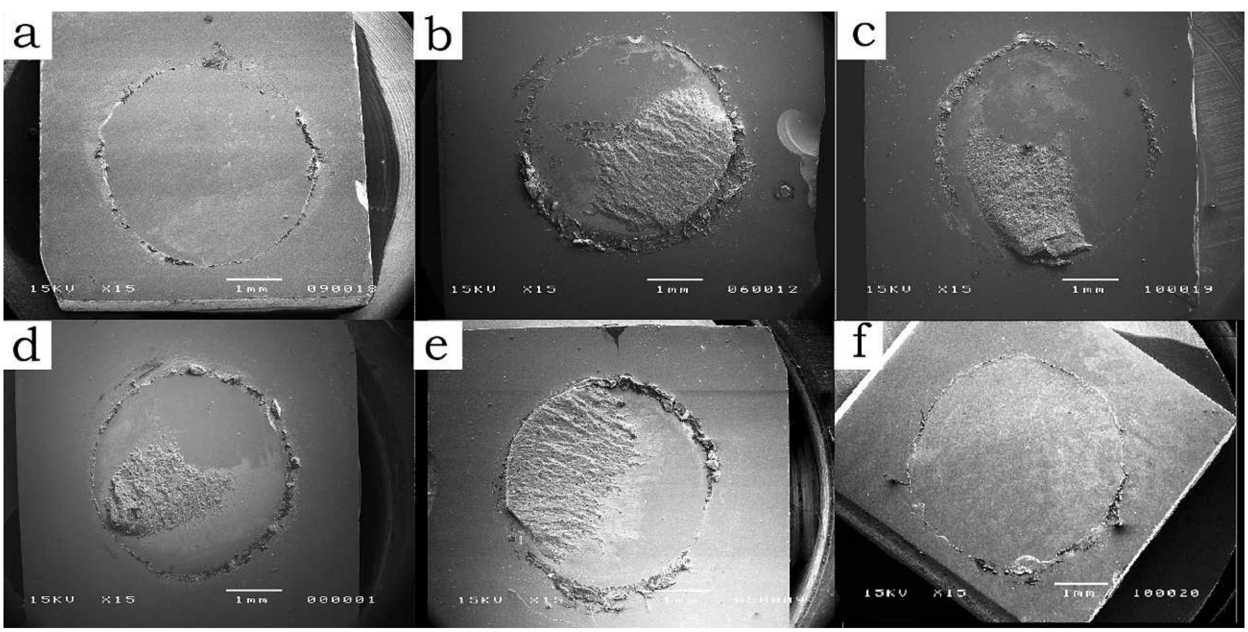

Figure 2. *SEM images showing representative failure modes of each group after SBS testing. (a, f) Images from CR and NaOH-AP groups, respectively, depicting the adhesive failure mode. Images (b), (c), (d), and (e) show the mixed failure mode for AP, ZP, NaOH, and NaOH-AP groups, respectively.*

composition of both primer agents and the resin cement evaluated in this study. When the 0.5 M NaOH solution was associated with Alloy Primer, the best SBS result was recorded. However, this outcome may be more significantly related to the composition of the primer than to the benefits provided by the NaOH solution, since the AP and NaOH-AP results were statistically similar. In addition, this strong base did not affect the performance of the primer itself because there were no statistical differences between the AP and NaOH-AP groups or between the ZP and NaOH-ZP groups. However, the result of the NaOH group was lower when compared with a value limit 10–13 MPa suggested as the minimum for acceptable clinical bonding {Thurmond, 1994 #23}.

Y-TZP surface conditioning with primers has been proposed to increase the bond strengths between cements and zirconia[52]; however, the composition of these primers can result in different bond strength values[6] because they contain different monomers (eg, MDP, 4-methacryloxy- ethyl trimellitate anhydride, thiophosphate methacryloyloxyalkyl derivates, and zirconate coupler).[33] The Alloy Primer was specifically developed to enhance the bond strength in metal surfaces (fabricant information). Therefore, as a result of its chemical composition, the Alloy Primer can result in an improved performance when used in metal alloys. This primer presents two active monomers, MDP and 6-4-vinylbenzyl-*n*-propyl amino-1,3,5-triazine-2,4-dithione (VBATDT), that set up bonds to precious and nonprecious metal oxides, respectively. MDP has been considered an important monomer in bonding to zirconia[31]; thus, it was chosen for evaluation. In this study, the best SBS results were found in the AP and NaOH-AP groups. Considering the composition of the Alloy Primer, it can be speculated that the interaction between MDP and VBATDT (ie, because MDP aids in VBATDT's reaction with the precious metal oxides, which improves the bond strength) produced more reliable bond strength to the zirconia oxide layer. Therefore, this interaction was not investigated in the present study. The NaOH-AP and AP groups were associated with mixed failure modes, which may indicate that adhesions between the cement and ceramics were effective. This finding may be related to the double carbon bonds located at the end of both MDP and VBATDT molecules, which may have bonded to monomers present in the cement composition. High bond strength between Alloy Primer and Rely X U100 have been reported, even after aging.[32]

On the other hand, the results from the NaOH-ZP and ZP groups were greater than those of the CR group, although they were lower when compared with the AP and NaOH-AP groups. This may be related to Z-Primer Plus composition; it contains organophosphate and carboxylic acid monomers.[33] Although both primers present the MDP molecule, the differences found between the two may be a sign that monomers other than MDP can affect the bond strength.[32] Moreover, the Z-Primer Plus produced better SBS values when associated with methacrylate-based monomer (Bis-GMA)–based composites because the presence of acid monomers may weaken the links between Z-Primer Plus and the methacrylate group of the self-etching resin luting cements.[33] The adhesive failure was the main mode associated with the ZP and NaOH-ZP groups (pointing to weaknesses in the zirconia-resin cement bond interface); however, such outcomes do not agree with the findings of a previous report[33] that observed mixed failures as the main failure mode. However, those authors[33] sandblasted all zirconia surfaces with 50 μm aluminum oxide (Al_2O_3) particles. A cohesive failure in zirconia has never been reported with this type of test,[28] and, as expected, it was not found in the present study.

Several *in vitro* studies have attempted to increase zirconia bond strength. Although sandblasting seems to be a mandatory condition to achieve a reliable bond strength to zirconia,[52,53] it may induce defects in the zirconia surface, which may serve as crack initiation sites,[54] decreasing the long-term survival of all-ceramic crowns.[42,43] Many other attempts, such as those involving selective infiltration etching,[22] application of fused glass micro pearls,[48] and the tribochemical silica coating process,[24,55] also have displayed positive results on Y-TZP bond strength, but all of them depend on mechanical damages in the Y-TZP surface. Nevertheless, efforts should be directed to achieving a chemical protocol that does not require any special equipment or produce mechanical damage in the Y-TZP surface. As such, the use of MDP-based primers and alkaline solution seem to contribute to this approach.

This study presented outcomes limited to initial testing after 24 hours in a wet condition. However, the effect of hydroxylation on surface zirconia and its interaction with primer agents should further be evaluated through the use of a thermocycling method, which is often employed to simulated clinical conditions.[52,56,57] However, Y-TZP surface pretreatments proposed in the literature still warrant further clinical prospective controlled studies to evaluate their real effect on Y-TZP restoration in the long term.

CONCLUSIONS

Within the limitations of this study it was possible to conclude that the use of a 0.5 M NaOH solution modified the reactivity of the zirconia surface, whereas the application of a MDP/VBATDT-based primer enhanced the bond strengths between flat zirconia surfaces and resin luting.

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Conflict of Interest Declaration

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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