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Laboratory Research

Effect of Resin Luting Systems and Alumina Particle Air Abrasion on Bond Strength to Zirconia

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Clinical Relevance

Alumina particle air abrasion (topographical alterations) of the inner surface of zirconia-based ceramic (Y-TZP) restorations and the application of universal primers (adhesives) containing multiple bond promoters (methacrylate monomers, including phosphate monomers such as 10-methacryloyloxi-decyl-dihydrogen-phosphate and silane) optimize the adhesion of Y-TZP to resin cements.

SUMMARY

This study aimed to evaluate the effect of different primer/resin luting agent combinations and alumina air abrasion on the adhesion to zirconia. Eighty blocks (4×4×3 mm) of Lava Frame Zirconia (3M ESPE) were produced and randomly assigned into eight groups (n=10) according to two zirconia surface treatments (untreated or air abrasion with 50-μm alumina particles) and four luting systems (SU: Scotchbond Universal/RelyX Unicem 2; ZP: Z-Prime Plus/Duo-link Universal; MB: Monobond Plus/Variolink II; and AP: Alloy Primer/ED Primer II/Panavia F 2.0). After the conditioning and primer applications, resin luting agents were manipulated and applied on the zirconia, using a matrix, to form a cylinder (2 mm in diameter×2 mm high), followed by photoactivation for 40 seconds. After that, the specimens were stored in distilled water (37 °C) for 120 days and then submitted to shear bond strength testing, followed by failure mode evaluation under an optical microscope (30×). A two-way analysis of variance and Tukey test (α=0.05) were used for data analysis. Alumina air abrasion (Al) promoted higher bond values for the three luting systems, except for SU, which showed the best results without air abrasion, while with air abrasion, Al-SU, Al-ZP, and Al-MB presented higher values compared to Al-AP. We concluded that the alumina air abrasion of zirconia

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surfaces seemed to be dispensable for the SU group, while air abrasion (topographical alterations) enhanced the adhesion of the ZP, MB, and AP groups.

INTRODUCTION

The use of zirconia-based ceramics (Y-TZP) has increased over the past decade, especially due to their superior mechanical and esthetic properties used as frameworks and monolithic restorations in posterior areas.[1-5] Despite the large clinical application of zirconia ceramics, the major drawback is related to its unpredictable bond with resin cements.[1-3,6] Zirconia is a densely sintered ceramic that offers chemical and dimensional stability and desirable physical properties, such as high flexural strength, high modulus of elasticity, and high fracture toughness compared to other ceramic materials.[4] On the other hand, polycrystalline ceramics are nonetchable by hydrofluoric acid since it does not contain amorphous silica in its composition.[4-6] Therefore, traditional surface treatment methods indicated for silica-based ceramic, such as hydrofluoric acid followed by silane coupling agent application, are impracticable on zirconia-based ceramics due to their high crystalline content and silica-free surfaces.[2,4-6]

The use of conventional cements, such as zinc phosphate or resin-modified glass ionomer cements, had been initially recommended by the manufacturers for luting zirconia restorations.[4] However, adhesive cementation has been shown to increase fracture resistance[4] and the fatigue resistance[7] and improve the longevity of ceramic restorations[8] besides sealing internal surface defects created by airborne particle abrasion.[9] Achieving a reliable adhesion to zirconia-based ceramics would further expand the application of this material.[4] However, it requires surface treatments based on physical and/or chemical treatments, such as air abrasion and zirconium oxide dedicated primers, that will promote the interaction between zirconia and luting substrate.

Air abrasion with aluminum oxide particles aims to roughen the internal surface of the ceramic restorations to optimize the adhesion area and promote better mechanical interlocking with the resin cement.[4,5] Although studies have shown that particle air abrasion promotes the improvement of resin bond to zirconia materials,[10,11] previous studies reported that this treatment creates surface microcracks and defects that can damage the mechanical properties of the material.[12-15] Hence, the use of zirconia primers has been studied as a substitute to air abrasion in order to promote the chemical bond to zirconia through phosphate monomers without a mechanical bond.

Phosphate monomers act as bifunctional molecules in which one end connects with the ceramic's metal oxides (such as aluminum and zirconium), while the other end copolymerizes with the resin cement matrix. Some examples of these bifunctional monomers are 10-methacryloyloxydecyl-dihydrogen-phosphate (10-MDP), 2-methacryloyloxyethyl dihydrogen, and 6-methacryloyloxyhexyl dihydrogen phosphate .[16] It has been found that MDP monomers promote a water-resistant chemical bond to densely sintered zirconia ceramics.[5,17] MDP monomers are also available in some resin cements, such as RelyX Unicem (3M ESPE, Maplewood, MN, USA) and Panavia (Kuraray Noritake Dental Inc, Chiyoda-ku, Tokyo, Japan). Today, several primers that claim chemical adhesion to zirconia are available on the market. However, more studies are necessary to verify their efficacy and long-term bond durability when combining different primers and resin cements.[18]

Thus, the aim of this study was to evaluate the effect of alumina particle air abrasion and different luting systems (primer/resin luting agent) on bond strength to zirconia after aging. The research hypotheses were 1) that regardless of the luting system adopted, alumina air abrasion would promote bond improvement and 2) that there would be no difference in bond strength among the different luting systems.

METHODS AND MATERIALS

The resin cements and primers used in this study are shown in Table 1. The evaluator was "blind" for some study conditions (cementation procedure, shear test, failure analysis).

Specimen Preparation

Eighty blocks (4×4×3 mm) Lava Frame (3M ESPE) zirconia were obtained, sintered as recommended by manufacturer, and embedded in autopolymerizing acrylic resin cylinders (Orthodontic Resin, Dentsply Caulk, Milford, DE, USA), keeping free a zirconia surface for bonding procedures. After the resin acrylic polymerization, the zirconia's exposed surface was ground finished with 800-grit silicon carbide abrasive (Auto Advanced, 3M ESPE) under running water in a polishing machine (Buehler Metaserv, Buehler, Düsseldorf, Germany) for 1 minute. After-

Table 1: *Information and Chemical Composition of Resin Cements and Zirconia Primers/Bonding Agents Used in the Present Study*

Commercial Brand	Lot Number	Specifications
RelyX Unicem2 Translucent, 3M ESPE	524950	Base paste: methacrylate monomers containing phosphoric acid groups, initiator components, silanated fillers, stabilizers, rheological additives. Catalyst paste: methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, rheological additives, pigments
Scotchbond Universal, 3M ESPE	525058	MDP phosphate monomer, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators, silane.
Duo-link Universal, Bisco	1400003516	Base paste: Bis-GMA, triethyleneglycol dimethacrylate, Glass Filler. Catalyst past: Bis-GMA, triethyleneglycol dimethacrylate, glass filler
Z-Prime Plus, Bisco	1400002857	Biphenyl dimethacrylate, MDP, ethanol
Variolink II Transparent, Ivoclar Vivadent	Base: T00900; catalyst: T00901	Base: 26.3%wt monomer (Bis-GMA, urethane dimethacrylate, triethylene glycol dimethacrylate), 73.4%wt filler. Catalyst: 22.0%wt monomer, 77.2%wt filler
Monobond Plus, Ivoclar Vivadent	S55075	Alcohol solution of silane methacrylate, phosphoric acid methacrylate, sulfide methacrylate
Panavia F2.0 Light, Kuraray	061229	Paste A: 10-methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic methacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica, dl-camphorquinone, catalysts, initiators, others Paste B: sodium fluoride, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic methacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, catalysts, accelerators, pigments, others
Alloy Primer ED Primer II, Kuraray	Alloy Primer: 0436AA ED Primer II Liquid A: 00322B Liquid B: 00196A	Alloy primer: acetone, VBATDT,[a] 10-MDP ED Primer II Liquid A: 2-hydroxyethyl methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate, N-methacryloyl-5-aminosalicylic acid, water, accelerators ED Primer II Liquid B: N-methacryloyl-5-aminosalicylic acid, water, catalysts, accelerators

[a] *Phosphate monomer 6-(4-vinylbenzyl-N-propyl)amino-1,3,5-triazine-2,4-dithione.*

ward, the specimens were ultrasonically cleaned for five minutes in deionized water and then wiped with 95%vol ethanol.

Zirconia specimens were randomly allocated into eight groups (n=10), as described in Table 2. Half of these groups did not receive zirconia surface treatment, while the other groups had the zirconia surface air abraded (aluminum oxide, 50 microns per 15 second, device sample distance of 10 mm, pressure of 87 psi, perpendicular to the surface, by linear motion), using a microetcher (Optiblast, Buffalo DentalManufacturing Inc, New York, NY, USA).

Cementation Procedure

After particle air abrasion, the primer agents were applied over the treated zirconia surface following the manufacturer's instructions (Table 2). A cylinder of resin cement was built on the ceramic surface using an Ultradent SBS device (Bisco, Schaumburg, IL, USA) with an inner diameter of 2 mm and height of 2 mm. The resin cements were manipulated following the manufacturer's instructions (Table 2). Resin cement cylinders were light cured (Bluephase style, Ivoclar Vivadent, Schaan, Liechtenstein) with an intensity of 1330 mW/cm^2 for 40 seconds. All specimens were prepared by the same operator to avoid interoperator variability.

Shear Bond Strength Test

After four months of storage in distilled water at 37°C for aging process, all specimens were submitted to the shear bond strength test in a universal test machine (Compact force gauge, Bisco) at a crosshead speed of 0.5 mm/min, using a flat rod as testing assembly (Figure 1). The bond strength R (MPa) was calculated using the following formula: $R = F/A$, where F is the load for specimen failure (N) and A is the cross-sectional interfacial area (mm^2).

Failure-Type Analysis

After the shear bond testing, the debonded surfaces were observed through an optical microscope (Microview Canada, Markham, ON, Canada) at 30× magnification to determine and classify the failure mode. The failure types were classified into the following categories: (A) adhesive at the zirconia-cement interface and (B) cohesive in the resin cement structure. Micrographs of representative

Table 2: *Testing Groups According to the Zirconia Surface Treatment and Cementation Strategy. Bonding Procedures, as Recommended by Manufacturers, Are Described as Footnotes*

Cementation Strategy	Zirconia Surface Treatment	
	Without	With
Scotchbond Universal/RelyX Unicem 2[a]	SU	Al-SU
Z-Prime Plus/Duo-link Universal[b]	ZP	Al-ZP
Monobond Plus/Variolink II[c]	MB	Al-MB
Alloy Primer/ED Primer II/Panavia F 2.0[d]	AP	Al-AP

[a] *Scotchbond Universal was applied on zirconia surface, with a microbrush, for 20 seconds and then air-dried for 5 seconds. The specimen was positioned on the Ultradent SBS device, and RelyX Unicem cement was manipulated by equal parts of both pastes.*
[b] *Two layers of Z-Prime Plus were applied over the zirconia surface and air-dried for 5 seconds. Then the specimen was positioned on the Ultradent SBS device, and the Duo-link Universal cement was manipulated by equal parts of both pastes.*
[c] *Initially, Monobond Plus was applied on the specimen-free surface and let stay for 60 seconds, then it was air-dried to remove possible primer excess. The specimen was positioned on the Ultradent SBS device, and the Variolink II cement was manipulated by equal parts of both pastes during 10 seconds.*
[d] *First, Alloy Primer was applied with a microbrush and let dry by itself. Then ED Primer II (A and B) was applied due its influence on resin cement polymerization, let stay for 30 seconds, and then gently air-dried. Finally, the specimen was positioned on the Ultradent SBS device, and Panavia cement was manipulated by equal parts of both pastes mixed for 20 seconds.*

samples were taken using a scanning electron microscope (S-2500 Hitachi Scanning Electron Microscope, Hitachi High Technologies America, Inc, Schaumburg, IL, USA).

Data Analysis

Statistical analysis was performed using the software Statistix 8.0 for Windows (Analytical Software Inc, Tallahassee, FL, USA). Bond strength data (MPa) were subjected to two-way analysis of variance (ANOVA) and Tukey tests ($\alpha=0.05$); p-values

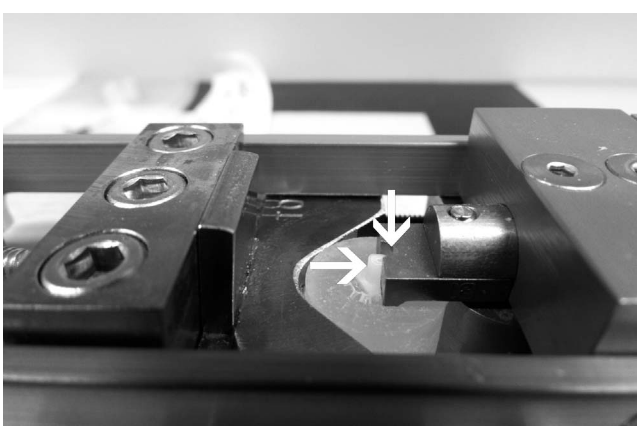

Figure 1. *Picture of the test setup adopted for the shear bond strength testing. It notes the proper fit between the resin cement cylinder (→), built over the zirconia block, and the flat rod testing assembly (↓).*

less than 0.05 were considered to be statistically significant in all tests. Multiple comparisons were made by repeated measures tests at a significance level of 0.05.

RESULTS

Two-way ANOVA revealed that the cementation approach ($p<0.0001$) and the zirconia surface treatment ($p<0.0001$; Al_2O_3>untreated) showed a significant effect on the bond strength between the zirconia and the resin cement. Interaction between the factors surface treatment and cementation strategy ($p=0.0062$) was also significant.

Surface treatment by particle air abrasion (Al_2O_3) statistically increased bond strength values for all luting systems, except for Scotchbond Universal/RelyX Unicem 2 (Table 3).

Comparing the different luting systems without air abrasion, Scotchbond Universal/RelyX Unicem 2 promoted the highest bond values, while Z-Prime Plus/Duo-link Universal and Monobond Plus/Variolink II were intermediaries, and Alloy Primer/ED Primer II/Panavia F 2.0 showed the lowest bond strength values (Table 3). When surface treatment with aluminum oxide particles was performed, it potentiated the bonding of Z-Prime Plus/Duo-link Universal and Monobond Plus/Variolink II cementation strategies, which achieved values statistically similar to Scotchbond Universal/RelyX Unicem 2 but higher than Alloy Primer/ED Primer II/Panavia F 2.0 (Table 3).

No pretest failures occurred in this study. The percentages of adhesive and cohesive failure after test are represented in Table 3. Representative micrographs of tested samples are shown in the Figure 2.

DISCUSSION

The first hypothesis (bond improvement with the use of alumina air abrasion regardless of the luting system) was partially accepted once the zirconia surface treatment increased bond strength values when compared to untreated surface, except for the luting system that used Scotchbond Universal/RelyX Unicem 2.

Air abrasion with aluminum oxide particles with a cross section of 50-125 mm is considered as the main zirconia surface treatment prior to the cementation procedure due to the results achieved and ease of chair-side procedures.[19-21] Alumina air abrasion aims to roughen the zirconia surface, producing microretentions and increasing the adhesion ar-

Table 3: *Data Analysis of Shear Bond Strength Test Results. Presented are the means and Standard Deviations (MPa) and Tukey Tests of Testing Groups. Percentages of Adhesive (A) and Cohesive (B) Failures in Each Group Are Also Described*[a]

Cementation Strategy	Bond Results		Failure Types	
	Alumina Air Abrasion			
	Without	With	Without	With
Scotchbond Universal/RelyX Unicem 2	19.6 ± 5.6 Aa	23.2 ± 5.2 Aa	A: 60%	A: 40%
			B: 40%	B: 60%
Z-Prime Plus/Duo-link Universal	11.1 ± 6.3 Bb	21.2 ± 5.2 Aa	A: 40%	A: 30%
			B: 60%	B: 70%
Monobond Plus/Variolink II	11.8 ± 3.3 Bb	25 ± 7.1 Aa	A: 70%	A: 40%
			B: 30%	B: 60%
Alloy Primer/ED Primer II/Panavia F 2.0	4.7 ± 1.3 Bc	9.1 ± 2 Ab	A: 100%	A: 100%
			B: 0%	B: 0%

[a] *Different uppercase letters mean statistical difference between surface treatment groups, keeping unaltered the cementation strategy; different lowercase letters mean statistical difference among cementation strategy groups, keeping unaltered the surface treatment.*

ea.[4,5,20] In this way, like hydrofluoric acid treatment on silica-based ceramics, alumina air abrasion allows a mechanical interlocking between these acid-resistant ceramics and the resin luting/ceramic primer.

Although air abrasion has been proven to be a successful treatment, enhancing the bond strength,[10,11,22-25] its positive influence on resin adhesion to zirconia is contradictory. Murthy and others[26] evaluated the effect of different surface treatments on shear bond strength between zirconia and resin luting agents and observed no significant differences on bond strength values between control (no surface treatment) and alumina air abrasion (with either 110 μm or 250 μm) groups. Foxton and others[27] obtained a durable (six-month) bond to Procera All Ceram and Procera All Zirkon using a ceramic primer containing MDP-phosphate monomer without any additional surface treatment (alumina air abrasion or erbium laser treated). These findings are in accordance with the findings observed in the present study when considering the Scotchbond Universal/RelyX Unicem 2 luting system, in which air abrasion did not improve the adhesion to zirconia. Air abrasion had a positive role by increasing bonding values for the luting systems with Z-Prime Plus/Duo-link Universal, Monobond Plus/Variolink II, and Alloy Primer/ED Primer II/Panavia F2.0, which is in agreement with several studies.[10,11,22,23,25,28,29] Furthermore, Amaral and others[24] and Inokoshi and others[30] reported that air abrasion is required when luting zirconia, even when using novel primers and universal adhesives. Therefore, the fact that Scotchbond Universal/RelyX Unicem 2 did not present increased bonding values after alumina air abrasion may be related to its chemical composition. This primer has been proposed as a substitute to air abrasion (morphological modifications) for zirconia surface treatment prior to the luting procedure due to the chemical adhesion to zirconia given by its differentiated chemical composition (Table 1), including multiple bond promoters, as reported below.

In this sense, the second null hypothesis (no difference in bond strength among the different luting procedures) was denied since different luting systems resulted in different bond strengths. Without surface treatment with air abrasion, Scotchbond Universal/RelyX Unicem 2 obtained the highest bond strength values. However, when alumina air abrasion was performed, it potentiated the adhesion of Z-Prime Plus/Duo-link Universal and Monobond Plus/Variolink II, resulting in similar bond strength of Scotchbond Universal/RelyX Unicem 2 (groups with the highest bond strength values). Even with an increase in bond strength after air abrasion, Alloy Primer/ED Primer II/Panavia F 2.0 presented the lowest bond values. Recent investigations have reported that the selection of the luting agent is one of the most important factors for luting zirconia restorations.[20,25,31-34]

As mentioned, another alternative surface treatment to zirconia is the use of ceramic primers for the chemical bond between the zirconia surface and the resin luting agent through phosphate monomers. They act as bifunctional molecules and improve the wettability of the ceramic surface and bond strength to resin cements by chemical interaction.[35] Thus, a luting approach that includes phosphate monomers could enhance, for example, bond strength and restoration longevity.[24,25,28,29,32,36-38] Inokoshi and

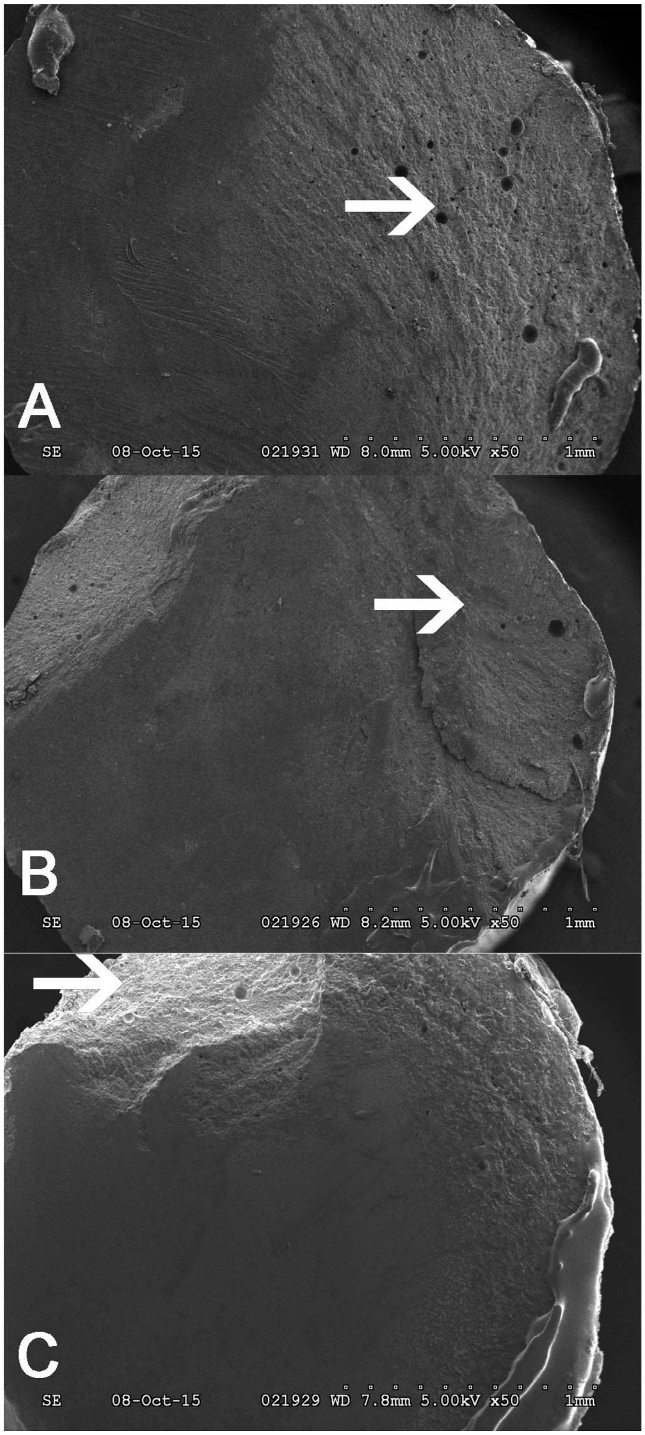

Figure 2. *Representative scanning electron micrographs of the resin cement debonded zirconia surfaces. It notes that the part of resin cement fractured (→) due to typical biomechanical behavior of the shear testing. Similar failure profiles were even observed between groups with the highest (A is representative of the SU group, and B is representative of the SU-Al group) and lowest (C is representative of the AP group) bond strength values.*

others[30] and De Souza and others[35] observed that the application of an MDP-based adhesive might improve the bond strength to zirconia. On the other hand, Cristoforides and others[39] observed that an MDP-containing liner is not effective for zirconia Y-TZP composite repairs. 10-MDP is one of the well-known phosphate monomers. Being in use for more than 20 years, it has achieved promising results in adhesion. Its phosphoric acid group bonds chemically to zirconia atoms, while the double bonds on the other end of the molecule copolymerize with the resin monomers; in addition, they are able to create ionic bonds with calcium from hydroxyapatite.[17,39,40] In the present study, it was observed that primers/adhesives that associate multiple adhesion promoters—10-MDP and others—appear to promote efficient adhesion to zirconia.

Scotchbond, as a universal adhesive, can be used in both tooth and indirect restoration surfaces, including metals, composites, glass-containing ceramics, and nonglass ceramics. This property is given by its composition (Table 1), which contains multiple adhesion promoters, such as methacrylate monomers, 10-MDP, polyalkenoic acid copolymer, and silane. The resin cement RelyX Unicem 2 also presents (Table 1) methacrylate monomers containing phosphoric acid groups. In the current study, this luting approach yielded the highest bonding values, as observed by Amaral and others,[24] De Souza and others,[35] and Seabra and others.[41] This finding was assigned to the adhesive chemical composition, whose MDP molecules may have interacted chemically to zirconium and aluminum (Al_2O_3) oxide particles due to their affinity to metallic oxides. Amaral and others[24] have also observed that Scotchbond Universal was effective in promoting durable bond to zirconia even without previous air abrasion with silica or alumina.

Hence, the present luting system may be a safe alternative for a stable bond to zirconia without the need of additional surface treatments, as the use of air abrasion may generate damage to the zirconia surface, including the presence of microcracks.[12-15] Moreover, as a universal adhesive, it simplifies the clinical procedures, reducing the number of steps involved in an adhesive luting system.[24] The superiority of the luting system using Scotchbond Universal, despite the fact that Panavia F 2.0 (Table 1), Alloy Primer, and ED Primer II (Table 1) also present 10-MDP and other phosphate monomers, methacrylates, and dimethacrylates in their compositions as adhesion promoters, was also observed previously.[25,42] On the other hand, several authors have observed that the Panavia luting system was superior to other luting agent/primer associations, such as AZ Primer/ResiCem (Shofu, Kyoto, Japan),[43] Metal/Zirconia Primer/Multilink (Ivoclar Vivadent),[43] Monobond S/Multilink (Ivoclar Vivadent),[34] and Porcelain Liner M/SuperBond (SunMedical Co, Moriyama, Japan).[34] Furthermore, Piwowarczyk and others[44] reported superior adhesion of RelyX Unicem and Panavia F2.0 to zirconia after aging when compared to zinc phosphate and modified glass-ionomer cements.

The inferior results achieved by Panavia F in the present study might have been affected by bonding procedures. During the bonding protocol, the Alloy Primer was applied first on the zirconia surface, followed by ED Primer. The manufacturer recommends ED Primer to be applied over the remaining tooth structure to keep contact with both tooth structure and the Panavia luting agent and recommends Alloy Primer to be applied over the zirconia to react with its surface and with the Panavia luting agent. Although no teeth have been used in the present study, as mentioned, ED Primer was applied after Alloy Primer and may have interfered with the reaction between Alloy Primer and Panavia. A study by Özcan and others[34] had also observed inferior or no adhesion to zirconia after artificial aging when following the manufacturer's instructions regarding Panavia F2.0. During initial tests for the present study, it was attempted to remove ED Primer II from the Alloy Primer/Panavia F2.0 luting protocol, but no adhesion to zirconia was achieved.

The occurrence of cohesive failures by shear testing can be considered as a limitation of this investigation since it may lead to misinterpretation of the bond performance of tested materials.[45-47] Instead of an indication of strong bonding, cohesive failures are explained by the mechanics of the test and the brittleness of the materials involved.[45] It may affect the accurate assessment of the interfacial bond strength, precluding a correct evaluation of each studied variable effect. Another limitation was the absence of thermocycling since only water storage at 37°C was performed, even though 150 and 300 days of distillated water storage might be a useful method for aging cement-zirconia adhesion interfaces, producing similar results to water storage associated with 12,000 thermocycling cicles.[48]

Although the use of ceramic primers seems to be part of a promising luting protocol by enabling to reduce time-consuming and critical clinical steps, more studies are still necessary to confirm the long-term efficiency of primers as bond promoters to zirconia.

CONCLUSION

The topographical alterations of the zirconia surface via alumina particle air abrasion provided enhanced resin bonding to the zirconia surface. The use of universal primers (adhesives) containing multiple bond promoters (methacrylate monomers, phosphate monomers such as 10-MDP, and silane), such as Scotchbond Universal, is a promising alternative to improve the adhesion of resin luting agents to zirconia.

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Conflict of Interest

The authors of this article 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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