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Laboratory Research

Effect of Adhesive Cementation Strategies on the Bonding of Y-TZP to Human Dentin

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Clinical Relevance

A 10-methacryloxydecyl dihydrogen phosphate monomer–based universal adhesive primer is a viable alternative to air-abrasion surface conditioning when bonding zirconia to dentin.

SUMMARY

This study evaluated the effects of different adhesive strategies on the adhesion of zirconia to dentin using conventional and self-adhesive cements and their corresponding adhesive resins. The occlusal parts of human molars (N=80) were sectioned, exposing the dentin. The teeth and zirconia cylinders (N=80) (diameter=3.4 mm; height=4 mm) were randomly divided into eight groups according to the factors "surface conditioning" and "cement type" (n=10 per group). One conventional cement (CC: RelyX ARC, 3M ESPE) and one self-adhesive cement (SA: RelyX U200, 3M ESPE) and their corresponding adhesive resin (for CC, Adper Single Bond Plus; for SA, Scotchbond Universal Adhesive-SU) were applied on dentin. Zirconia specimens were conditioned either using chairside (CJ: CoJet, 30 μm, 2.5 bar, four seconds), laboratory silica coating (RC: Rocatec, 110 μm, 2.5 bar, four seconds), or universal primer (Single Bond Universal-UP). Nonconditioned groups for both cements acted as the control (C). Specimens were stored in water (37°C, 30 days) and subjected to shear bond strength (SBS) testing (1 mm/min). Data (MPa) were analyzed using two-way analysis of variance and a Tukey test (α=0.05). While surface conditioning significantly affected the SBS values (*p*=0.0001)

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(C<RC=CJ=UP), cement type did not (*p*=0.148) (CC=SA). The interaction terms were significant (*p*=0.014). Failure types were predominantly adhesive. Air-abrasion and the use of the universal primer improved the bond strength of zirconia to dentin compared to the control group, regardless of the type of resin cement used.

INTRODUCTION

Zirconia-based ceramics have superior mechanical properties compared to the other ceramic materials, such as glass-based ceramics.[1,2] This material has been used as the framework for crowns and fixed dental prostheses (FDPs) during the last two decades and, more recently, has been indicated for monolithic crowns or FPDs.[3] Since zirconia is totally crystalline and is not etchable like glass-based ceramics, the adhesion between resin cement and zirconia remains weak.[4] Retention of single crowns is primarily dependent on the preparation form,[5] but some restorations, such as inlay-retained or surface-retained resin-bonded FDPs, short or tapered crown preparations, or more conservative monolithic zirconia restorations, require more retention, which is usually obtained by adhesive cementation.

Several surface conditioning methods have been proposed to improve the adhesion between resin cement and zirconia.[6-14] Airborne particle abrasion with alumina particles has been used to increase the surface area and, consequently, the micromechanical retention between the zirconia surface and the resin cement.[9] Furthermore, air-abrasion with alumina particles coated by silica promotes micromechanical retention on the zirconia surface, similar to abrasion with alumina particles, with the advantage that silica is deposited on the zirconia surface.[6,15] This silica incorporated onto the zirconia surface will then be bonded to the resin cement by a silane coupling agent that acts as a link between the silica and the resin cement matrix.[11]

Since some authors have reported on the possible deleterious consequences caused by air-abrasion,[16,17] other surface conditioning methods have been proposed, such as application of an etchable glass layer onto the zirconia surface,[13] air-abrasion before zirconia sintering,[18] and etching zirconia with highly concentrated hydrofluoric acid.[19] In addition to these methods, chemical adhesion between zirconia and resin cement could be enhanced using resin cements or specific primers based on 10-methacryloxydecyl dihydrogen phosphate monomer (MDP).[20-25] In fact, bonding zirconia FDPs involves two aspects, namely zirconia-resin cement and resin cement–tooth interfaces. Consequently, it is important to employ a cementation strategy that achieves durable adhesion of resin cement to both the tooth and zirconia in order to guarantee the success of the restoration.[26] When conventional resin cements are used, conditioning the dental substrates with the corresponding adhesive resin is mandatory. In contrast, self-adhesive resin cements do not require any preconditioning of dental tissues, thereby saving clinical time. Hence, it can be anticipated that the combination of surface conditioning methods for both the tooth and zirconia and the type of resin cement affect the adhesion of zirconia.

The objective of this study, therefore, was to evaluate the effect of different adhesive cementation strategies employing different surface conditioning methods based on air-abrasion or the use of universal primer only in conjunction with conventional or self-adhesive resin cements on the adhesion of zirconia to dentin. The null hypotheses tested were that surface conditioning and resin cement type would not affect the bond strength results.

METHODS AND MATERIALS

The types, brands, manufacturers, and batch numbers of the materials used in this study are listed in Table 1.

Tooth Selection

The Committee on Ethics in Research (CEP, Process 435.230) approved this study in which molars or premolars (N=80) were used. Soft tissue and debris were cleaned from tooth surfaces using periodontal instruments and were stored in distilled water (5°C) until the experiments. Each tooth was embedded in acrylic resin (JET, Artigos Odontológicos Clássico, Ltd., São Paulo, SP, Brazil) with the aid of a metallic mold (height: 15 mm; diameter: 25 mm) and a surveyor, leaving the coronal portion exposed and the long axis of the tooth parallel to the y-axis. Enamel was removed from the occlusal surface in a cutting machine (Labcut 1010, EXTEC, Enfield, CT, USA) to expose the dentin. This surface was polished for 60 seconds with silicone carbide paper (#600) to achieve a standard smear layer. The teeth were randomly allocated into eight groups (n=10 per group) according to surface conditioning and resin cement type using a random allocation program (www.randomizer.org) (Table 2). The teeth were stored in distilled water for one week prior to cementation.

Table 1: *The Types, Brands, Manufacturers, and Batch Numbers of the Materials Used in this Study*

Material Type	Brand	Manufacturer	Batch No.
Zirconia	VITA In-Ceram YZ	Vita Zahnfabrik, Bad Säckingen, Germany	28070
Conventional resin cement (CC)	RelyX ARC	3M ESPE, St Paul, MN, USA	305960
Self-adhesive resin cement (SA)	RelyX U200	3M ESPE	473396
Alumina particles coated with silica (30 μm)	CoJet	3M ESPE	351794
Alumina particles coated with silica (110 μm)	Rocatec	3M ESPE	269078
Silane coupling agent	RelyX Ceramic Primer	3M ESPE	286040
Phosphoric acid (37%)	Condac 37	FGM, Joinvile, SC, Brazil	180612
Total-etch, single-component adhesive resin	Adper Single Bond Plus	3M ESPE	297179
Universal primer (UP)	Scotchbond Universal	3M ESPE	458640

Zirconia Specimens

Zirconia blocks (Vita InCeram YZ, Vita Zahnfabrik, Bad Säckingen, Germany) were positioned in a cutting machine and zirconia disks (diameter=4.5 mm; height=5 mm) were obtained by cutting the blocks with a cylindrical bur (diameter=4.5 mm). Zirconia disks were wet-finished using silicone carbide paper (#1200), ultrasonically cleaned (Vitasonic®, Vita Zanhfabrik, Bad Säckingen, Germany) in distilled water, and dried. The cylinders were then sintered, according to the manufacturer's instructions, in a specific oven (Vita ZYrcomat, Vita Zahnfabrik). The final dimensions of the zirconia disks after approximately 20% shrinkage were 3.4 mm in diameter and 4 mm in height. The cementation surfaces of the disks were polished with #800, #1000, and #1200 silicon carbide papers in sequence under water cooling for 60 seconds in a polishing machine (PSK-2V, Skill-tec Comércio e Manutenção Ltd, São Paulo, SP, Brazil). The disks were again ultrasonically cleaned in isopropyl alcohol for five minutes.

Cementation Procedures

Zirconia discs were conditioned according to the experimental groups (Table 2), and the dentin substrates were conditioned following the resin cement manufacturer's recommendations. The cementation surfaces of the specimens were enclosed by adhesive tape (Scotch, 3M ESPE, St Paul, MN, USA) with a hole (diameter=3.4 mm) in the middle. For the groups cemented with the conventional resin cement (CC: RelyX ARC, 3M ESPE), dentin was etched with 37% phosphoric acid for 15 seconds, washed, and gently dried with absorbent paper. One coat of adhesive resin (Adper Single Bond Plus, 3M ESPE) was then applied for 10 seconds, gently air-dried for five seconds, and photo-polymerized for 20 seconds (Radii Cal, SDI, Victoria, Australia). For the groups cemented with self-adhesive cement (SA: RelyX U200), dentin was cleaned only with water and gently dried with absorbent paper.

The base and catalyst pastes of resin cements (CC and SA) were then dispensed in equal portions, mixed, and applied to the cementation surfaces of the zirconia discs. Next, each zirconia disc was placed on its corresponding dentin, in the region defined by the adhesive tape, and a load of 750*g* was applied. Excess cement was removed, and the cementation interface was photo-polymerized for 20 seconds from four directions (Radii Cal). After polymerization, the adhesive tape was removed by cutting with a blade. One operator performed all bonding procedures (MA).

Table 2: *Abbreviation of the Experimental Groups with Respect to Surface Conditioning Methods for Zirconia and the Conditioning Parameters*

Abbreviation of the Groups	Surface Cnditioning Type for Zirconia	Conditioning Parameters
C	No conditioning—control	—
CJ	CoJet + silane	Airborne particle abrasion (duration: 4 s; pressure: 2.5 bar; distance: 10 mm) + one coat silane, waiting for 5 min
RC	Rocatec + silane	Airborne particle abrasion (duration: 4 s; pressure: 2.5 bar; distance: 10 mm) + one coat silane, waiting for 5 min
UP	Universal primer	One coat primer, rubbing for 20 s, air-dry for 5 s

Table 3: *Mean Shear Bond Strength Values (MPa), Standard Deviations, and Distribution of Frequency of Failure Types in Numbers and Percentages per Experimental Group*

Groups	Mean (±SD)	Failure Type			
		Ad-CD, No. (%)	Ad-CC, No. (%)	Mixed, No. (%)	Coh-CM, No. (%)
CC-C	5.64 ± 2.8 [D]	0	10 (100)	0	0
CC-RC	17.52 ± 7.4 [ABC]	1 (10)	5 (50)	4 (40)	0
CC-CJ	20.17 ± 6.07 [AB]	0	5 (50)	4	1 (10)
CC-UP	24.93 ± 7.01 [A]	0	4 (40)	6 (60)	0
Total CC		2.5	60	35	2.5
SA-C	10.36 ± 3.87 [CD]	1 (10)	6 (60)	3 (30)	0
SA-RC	16.83 ± 6.81 [BC]	5 (50)	1 (10)	4 (40)	0
SA-CJ	15.54 ± 4.66 [BC]	10 (100)	0	0	0
SA-UP	17.96 ± 5.94 [ABC]	4 (40)	2 (20)	4 (40)	0
Total SA		50	22.5	27.5	—

Abbreviations: Ad-CD, adhesive between cement-dentin; Ad-CC, adhesive between cement-ceramic; Coh-CM, cohesive in cement; Mixed, Coh-CM + Ad-CD or Ad-CC. For group abbreviations, see Tables 1 and 2.
[a] Different letters indicate statistical difference between the experimental groups.

Prior to the bond strength test, specimens from all groups were immersed in distilled water at 37°C and stored for 30 days.

Testing Procedure and Failure Analysis

Specimens were mounted in the jig of the Universal Testing Machine (AGS-X10kN, Shimadzu, Kyoto, Japan), and a shear force was applied to the adhesive interface until failure occurred. The specimens were positioned in the testing machine so that the adhesive interface was perpendicular to the horizontal plane, and load was applied with a knife-edge device until failure. The specimens were loaded at a crosshead speed of 1 mm/min, and the stress-strain curve was analyzed with the software program. The bond strength was calculated according to the formula $SBS = F/A$, where SBS is the shear bond strength in MPa, F was the load required for fracture (N), and A was the bonded area ($\pi x r^2$, where π=3.14 and r=1.7 mm/A=9.07 mm^2) of the specimen (mm^2).

All debonded specimens were initially evaluated under a stereomicroscope (75×) (Discovery Z-20, Zeiss, Jena, Germany). The most prevalent failure types were further observed with a scanning electron microscope (Inspect S50, FEI Company, Eindhoven, The Netherlands) (MA and FC). The types of failures were classified as follows: Ad-CD (adhesive between cement-dentin), Ad-CC (adhesive between cement-ceramic), Coh-Cer (cohesive in ceramic), Coh-D (cohesive in dentin), Coh-CM (cohesive in cement), and Mixed (cohesive in cement+Ad-CD or Ad-CC).

Statistical Analysis

Statistical analysis was performed using Statistix 8.0 software for Windows (Analytical Software Inc, Tallahassee, FL, USA). Initially, data were subjected to normality and homogeneity tests. Bond strength data (MPa) were submitted to two-way analysis of variance. Multiple comparisons were made with the Tukey post hoc test (α=0.05), with the shear bond strength as the dependent factor and surface conditioning protocols (three levels) and the resin cement types (two levels) as the independent factors. p-values of less than 0.05 were considered to be statistically significant in all tests.

RESULTS

While surface conditioning significantly affected the SBS values (p=0.0001), cement type did not (p=0.148). The interaction terms were significant (p=0.014) (Table 3).

Nonconditioned control groups presented the lowest mean SBS with both CC and SA cements. CJ and RC air-abrasion methods did not show significant difference for both CC and SA cements. The use of UP only, without air-abrasion protocols, presented mean SBS values that were not significantly different from CJ and RC conditioning methods for both cement types.

No pretest failures were observed at the end of water storage. Most failures were adhesive (Table 3). For the CC cement, the failures were predominantly Ad-CC. Conversely, for the SA cement, the failures were mainly Ad-CD. The most representative failure types are shown in Figure 1A-C.

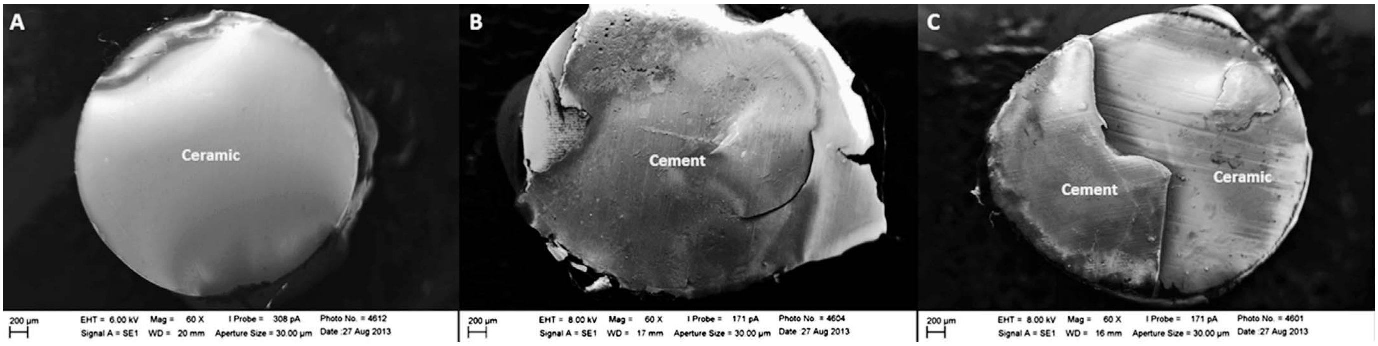

Figure 1. *(A-C) Scanning electron microscopy (SEM) micrographs (60×) of representative failure types after debonding (A) Ad-CC (adhesive between cement-ceramic), (B) Ad-CD (adhesive between cement-dentin), and (C) Mixed failure (Cohesive in cement+Ad-CD or Ad-CC).*

DISCUSSION

This study was undertaken in order to evaluate the effect of different adhesive cementation strategies employing two air-abrasion methods or the use of universal primer only in combination with conventional or self-adhesive resin cements for the adhesion of zirconia to dentin. Based on the results of this study, since surface conditioning significantly affected the adhesion results but the cement type did not, the first null hypotheses could be rejected.

It is necessary to use adhesive cementation strategies to enhance the adhesion of zirconia restorations, especially with the smaller areas available for adhering to teeth, such as inlay-retained FDPs.[25] In addition, the use of resin cements could improve the fracture resistance of all-ceramic restorations.[27] Furthermore, a ceramic restoration presents two interfaces (ceramic-resin cement and resin cement–dentin), and an adhesive-only approach can create a union between resin cement and teeth. Although it is important to measure the adhesion of ceramic restorations to the tooth substrate for better clinical significance,[9,28] most studies[13,15,22,23] to date have focused on the adhesion between zirconia and resin cement without considering the tooth aspect. In that respect, the present study could be considered more clinically relevant where the results showed that the interaction between the ceramic–resin cement–tooth interfaces is pertinent in this scenario. This was more evident not through the bond strength results but through the variations in modes of failure types.

This result corroborated with those of previous studies[29,30] in which the results showed that surface conditioning promoted higher bond strength values compared with the nonconditioned groups. For the air-abrasion surface conditioning, two particle types were used in this study (Rocatec: 110 μm, CoJet: 30 μm). Both chairside and laboratory air-abrasion versions showed similar results. This indicates that roughness did not dictate the adhesion in the resin cement–zirconia-dentin complex. With these methods, silica coating was achieved where air-abrasion with alumina coated by silica particles promoted adhesion between the silane coupling agent and the silica adhered on the zirconia surface due to the impact.[15] These results are in agreement with those of some previous studies[4,31,32] in which both particle types were compared in terms of the bond strength between resin materials and nonetchable ceramics. However, in these studies, tooth substrate was not involved. In another study[28] with similar methodology to that used in this study, both particle types presented comparable bond results. Accordingly, smaller particle size (30 μm) could be advised for air-abrasion of zirconia as this particle type would create less mechanical damage to zirconia, and chairside air-abrasion devices are certainly more cost-effective than laboratory ones.[33]

In the present study, the results expressed by the use of UP only are remarkable as a result of the fact that in these groups air-abrasion was not practiced. According to the study of Amaral and others,[23] air-particle abrasion and UP application on zirconia promoted bond strength values similar to those of the groups air-abraded with CoJet. Nevertheless, the highest bond strength values among the groups were achieved by this primer, without air-abrasion, in that study and among other primers in another study.[34]

From the clinical point of view, one single adhesive promoter for the cementation of etchable and nonetchable ceramics or other restorative materials, tooth substrate, and in intraoral repairs would eliminate multiple surface conditioning methods.[35] In its chemical composition UP, in this case Scotchbond Universal, contains MDP, dimethacrylate, 2-hydroxyethyl methacrylate, Vitrebond copolymer, filler, ethanol, water, initiators, and silane. The mixture of these constituents could also hinder the adhesion between resin-tooth and resin-zirconia pairs, as they react differently on these two substrates.[36] Since this solution is a simplified adhesive, it contains a greater quantity of solvents.[37] Hence, if the substances could be used separately for each indication they would be more effective on each individual substrate, producing more durable results. Yet, in this study, UP was effective on the dentin-resin cement–zirconia complex.

The results of this study showed no significant effect of resin cement type. However, it has to be noted that the bond strength values obtained were related to a scenario with two interfaces. If adhesion had been measured only between resin cements and dentin, the results could have been very different as a result of the differences between the adhesion mechanisms of the two cements. The conventional resin cements require preconditioning of the tooth substrate before cementation followed by the application of the adhesive system chosen, either an etch-and-rinse (three- or two-step) or self-etch (two- or one-step) system.[37] In this study, a two-step etch-and-rinse adhesive system was used, which removes the smear layer and exposes the collagen fibers through application of phosphoric acid, followed by the application of a one-bottle adhesive.[37] After phosphoric acid etching, the surface wettability of dentin increases and better adhesion is achieved.[38] On the contrary, the self-adhesive resin cements do not require conditioning of the tooth substrate. Self-adhesive cements contain acidic monomers, which etch dentin,[39] but without phosphoric acid etching they cannot interlock micromechanically with dentin, which results in lower bond strength values.[40]

The failure types clearly indicate the differences between the effects of conventional resin and self-adhesive cements, in that, with the self-adhesive cement, adhesive failures were more commonly observed between the cement and dentin (Ad-CD). On the other hand, with conventional resin cement, adhesive failures between resin cement and zirconia (Ad-CC) were more frequent. One could conclude that the mechanism of adhesion between cement and dentin may have guided the bond strength values. However, it is important to emphasize that although the failure patterns were different, the bond values for the whole system were similar and in accordance with those reported by other studies[9,22,41] in which the self-adhesive resin cements also obtained good results.

The methodology used in this study attempted to approximate the complex clinical scenario.[28] Nevertheless, in the shear bond strength test, the manner in which the load is applied to the specimen interface typically generates an inhomogeneous stress distribution.[5] Despite this irregular distribution, when there is a "double interface" (CC and CD), in theory, the failure would initiate from the weaker interface.[11] The adhesive failure types (Ad-CD) observed in this and other studies[11] are a consequence of the weakest interface between resin cement and dentin, due to the use of self-adhesive resin cement or a conventional resin cement followed by the use of a self-etch adhesive system. Self-adhesive cements do not create favorable resin tags, but because of MDP in their composition, adhesion to zirconia substrate was enhanced. Conversely, using a microtensile bond test to study the adhesion between zirconia and resin cement with the same cements used in this study, de Castro and others[9] found no adhesive failure between cement and dentin but more cohesive failures in the cement. Hence, the adhesion between zirconia and cement seems to be stable even after some aging.[9,11] In this context, the type and duration of aging need to be critically evaluated when interpreting results.

The use of human teeth in this study could be regarded as a limitation because of differences between the ages of the collected teeth. Nonetheless, a random distribution was used to control for this factor.[42] Other aging protocols, with longer duration of water storage or thermal and mechanical cycling, should be considered in future studies in order to challenge the interfacial hydrolysis and cement degradation.[43,44] Future investigations should also verify the results of this study using other test configurations.

CONCLUSIONS

Based on this study, the following could be concluded:

1. Both chairside and laboratory air-abrasion protocols and the use of universal primer without air-abrasion improved the bond strength of zirconia to dentin with conventional and self-adhesive cements compared to nonconditioned control groups.

2. Conventional resin and self-adhesive cements showed similar mean bond strength of zirconia to dentin after 30 days of water storage.
3. While conventional resin cement presented more frequent failures between cement and zirconia, self-adhesive cement showed mainly adhesive failures between cement and dentin.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Federal University of Rio Grande do Norte (UFRN). The approval code for this study is Process n° 435.230.

Conflict of Interest

The authors 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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