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# Effect of Immediate Dentin Sealing on the Bond Strength of Monolithic Zirconia to Human Dentin

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### Clinical Relevance

One frequent clinical complication related to zirconia restorations is decementation. Immediate dentin sealing could increase the bond strength of zirconia to dentin when self-adhesive cements are used.

## SUMMARY

**Objective: This study evaluated the shear bond strength (SBS) of pretreated monolithic zirconia surfaces bonded to human dentin following immediate dentin sealing (IDS) using two different self-adhesive resin luting agents.**

**Methods and Materials: Sixty intact human third molars were collected, stored, sectioned appropriately, and molded according to ISO 29022:2013, resulting in 120 dentin specimens. Ceramic cylindrical specimens were fabricated using CAD/CAM technology and sintered as recommended (final bonding area A=2.56 mm$^2$). Specimens were randomly assigned to eight groups (15≥n≥14) depending on dentin conditioning method (IDS or delayed dentin sealing [DDS]), zirconia surface pretreatment (airborne particle abrasion [APA] with 50 µm $Al_2O_3$ particles at 3 bar for 10 seconds or tribochemical silica coating [TBC] with 30 µm CoJet particles at 2.8 bar for 10 seconds), and adhesive luting agent type (Panavia F2.0 [PAN] or PermaCem Dual Smartmix [PER]). Bonded specimens were water-stored (37°C, 24 hours) and subjected to SBS testing (50-kgF load cell, 1 mm/min). Fracture type was evaluated with stereomicroscopy. Data (MPa) were statistically analyzed using three-way analysis of variance (α=0.05).**

**Results: All factors significantly affected SBS values (*p*<0.001). Dentin conditioning method presented the greatest effect. Mean SBS values ranged from 12.603 MPa (PER-APA-DDS) to 40.704 MPa (PER-TBC-IDS). Based on the fracture type, adhesive failures at the luting**

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**agent–zirconia interface were the least common.**

**Conclusion: Bonding strategies for monolithic zirconia restorations could potentially benefit from IDS, regardless of the adhesive luting agent system used.**

## INTRODUCTION

The increasingly prominent trend toward tooth-colored ceramic restorations in dentistry has led to the development of dental materials that combine excellent optical and mechanical properties.[1,2] Toward this direction, monolithic zirconia restorations have been well integrated into everyday clinical practice. Unlike traditional silica-based ceramics that can provide predictable bond strengths, thanks to chemical adhesion combined with micromechanical retention, high crystalline ceramics, including monolithic zirconia, lack silica, and, subsequently, until recently they were believed to be incapable of providing similar levels of bonding.[3] In order to solve this problem, the cementation surface had to be roughened so that surface area and surface energy would increase. As a result, an increased surface wettability could be provided and subsequently increased micromechanical interlocking compared to the initial state.[4] However, there is increasing evidence that chemical bonding can be achieved with zirconia using methacryloyloxydecyl dihydrogen phosphate (MDP)–containing products, such as MDP containing cements and primers.[5-7] Thus, zirconia is gaining ground against other ceramic materials as a result of the latest improvements in the bonding techniques available.

As a result of the wide variety of possibilities and the amount of research that has been conducted on the surface pretreatments of zirconia ceramics prior to cementation and their effect on the bond strength, it is difficult to outline specific principles on the subject or to provide specific clinical protocols. However, a combination of mechanical and chemical treatments improve the bonding reliability as long as an adhesive luting agent is applied.[8] With regard to mechanical surface treatments to enhance zirconia bonding, airborne particle abrasion (APA) has shown high bond strength values,[9] although the bond presents low durability.[10,11] Various conflicting results[12-15] exist concerning the bond strength between APA-treated zirconia surfaces and adhesive cements, primarily due to factors such as alumina grain size, applied pressure, and roughness of the treated zirconia surface, as well as phase transformations and flexural strength reduction of zirconia surfaces related to alumina sandblasting. According to a recent meta-analysis,[16] there are some encouraging data about APA, as small particle size, low pressure, and appropriate duration all may enhance the flexural strength of zirconia even after artificial aging. In addition, after 12 hours of aging, the monoclinic phase appears to be decreased, which signals higher mechanical properties.[16]

A stronger chemical bond is formed when silica-coated alumina particles are air-blasted onto the zirconia surface (eg, tribochemical silica coating [TBC]). A silane coupling agent applied to the silica-coated surface creates a chemical bond with an adhesive resin and increased bond strengths. To date, the recommended strategy for successful zirconia bonding involves the use of APA or TBC along with a phosphate-based monomer as a luting-cement adhesion promoter. The 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP)–containing adhesive cements are most commonly recommended, as this monomer causes acidic decalcification, increasing the diffusion of resin in dentin, and binds to calcium ions or amino groups of the tooth structure, enhancing adhesion.[17] According to Nagaoka and others,[18] ionic bonding as well as hydrogen bonding have been observed between 10-MDP and zirconia. Moreover, one P-OH group of 10-MDP bonds with $Zr^{4+}$, whereas the other $OH^{-}$ group bonds with P=O of another neighboring 10-MDP molecule.[18] However, to reduce the complexity of bonding steps and to induce fast and simple clinical procedures, self-adhesive resin cements (SARCs) that do not require pretreatments (owing to their acidic functional monomer) have been introduced.[19,20] MDP containing SARCs develop a strong bond even after thermocycling.[21] On the other hand, both MDP-containing and non–MDP-containing SARCs show lower values compared to resin cements without self-adhesive capacity.[22] Simplified adhesive cements may be chosen for speed and ease of use, which may not always lead to quality care; however, there are cases, such as those involving intracrevicular margins, mandibular preparations for which moisture control is difficult, and other uncontrollable patient factors, that can render self-adhesive or conventional non-adhesive cements an alternative, despite their lower bond strength.

The number of articles published on this subject during the first half of the 2010s outnumbered those published during the entire previous decade.[23] The testing parameters of these laboratory studies present extreme variation, thereby inhibiting the establishment of a standard protocol for bonding zirconia restorations.[6] Some monolithic zirconia ceramics contain a percentage of $Y_2O_3$ that is higher than 5 mol%, as opposed to conventional zirconia, which contains 3 mol% of $Y_2O_3$.[24,25] Moreover, fully stabilized zirconia, which provides the highest level of translucency, can be obtained with $Y_2O_3$ content higher than 8 mol%.[26] There are no studies to investigate possible differences in bond strength between monolithic and conventional zirconia. However, the different chemical structure could potentially influence the bond strength of the material to dentin. Moreover, monolithic zirconia is a material of increased interest, and clinically it is becoming more widely used by clinicians because of the abovementioned advantages. Despite this fact, little research has been published regarding its bond strength to composite resin cements, while no studies incorporating human dentin exist. However, the strength and durability of the complex configuration of zirconia restorations, in situations in which resin cement, the zirconia surface, and the dentin surface are bonded together, are of paramount significance to their long-term survival.

The immediate dentin sealing (IDS) concept was initially presented nearly two decades ago by Magne, who described the technique's steps and benefits.[27] The procedure includes application and polymerization of a dentin bonding adhesive before the final impression for the indirect restoration is taken. The fabrication of a provisional restoration is followed by the use of a separating medium.[28] Traditionally, fourth- and fifth-generation adhesive agents (etch-and-rinse) have been proposed for the IDS technique.[29] Nonetheless, it has been reported[28] that sixth- and seventh-generation adhesives (self-etch systems) can also enhance bond strengths.[28] Apart from being beneficial to pulp protection, it was reported that IDS protects freshly cut dentin from contamination. In addition, collagen bundles of the hybrid layer are guarded from collapsing, and subsequently, the bonding procedures of indirect restorations result in higher bond strength values.[27,29] This statement has been reasserted recently.[30] The aforementioned dentin treatment immediately after dentin preparation could potentially facilitate the bonding of monolithic zirconia restorations. To the best of the authors' knowledge, no studies have been conducted to enlighten the issue.

The objective of this study was to evaluate the effects of 1) self-adhesive resin luting agent type, 2) monolithic zirconia ceramic surface pretreatment, and 3) dentin conditioning method on the bond strength of a monolithic zirconia ceramic to human dentin. Therefore, the null hypotheses formulated were that luting agent type, zirconia surface pretreatment, and dentin conditioning method do not influence the bond strength between monolithic zirconia and human dentin.

## METHODS AND MATERIALS

### Preparation of Dentin Specimens

Sixty intact human third molars that had fully erupted were collected, following the approval of the local bioethics committee, cleaned from organic remnants of the periodontal ligament, and stored in 1% aqueous chloramine-T solution at (4°C±4°C). The solution was replaced at least every two months until preparation of the specimens, and the teeth were stored for a maximum of six months in total. The roots of each tooth were resected and the crown was sectioned in the mesio-distal dimension in order for each buccal and lingual half to result in a separate specimen. The buccal or lingual surface of every buccal and lingual half, respectively, was used as the bonding surface. Each bonding surface was cut flat, placed facing the bottom of a cylinder mold, and then embedded in self-curing acrylic resin. The molds were placed in cool water in order to prevent a temperature rise of the embedded teeth during the acrylic resin's exothermic polymerization phase. Afterwards, the cylinder specimens were stored in distilled water until the cementation procedures. The specimens were randomly divided into groups of 15 using a research random assignment tool (www.randomizer.org). The bonding surface of every cylinder underwent water-polishing with 600-grit silicon carbide paper for 60 seconds to achieve proper surface standardization. The bonding procedures followed in less than four hours, according to ISO 29022:2013. During the four-hour span, the dentin conditioning procedures were also performed in order to ensure that all bonding procedures would take place early enough following the exposure of the dentin substrate. The substrate was carefully inspected to ensure that the zirconia specimens would be bonded exclusively to dentin.

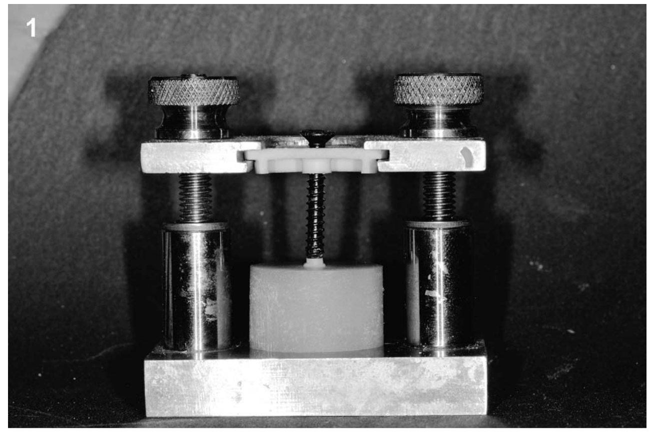
The teeth belonging to the IDS groups were then etched with 37% $H_3PO_4$ for 15 seconds and rinsed for 30 seconds with copious water. The primer and adhesive resin (Optibond FL, Lot No. 6080704, Kerr, Orange, CA, USA) were applied, air-thinned, and photopolymerized for 15 seconds using an LED polymerization device (Mini LED Black, Acteon Group, Mérignac, France; light intensity 1250 mW/cm$^2$) from a distance of 1 mm, which was standardized with the use of a ruler. The surface was then covered with glycerine gel and photopolymerized for 15 more seconds. The dentin specimens of the IDS and delayed dentin sealing (DDS) groups were then stored in distilled water until the bonding procedures.

## Preparation of the Zirconia Specimens

Blocks of monolithic zirconia (BruxZir, Lot No. BZ0004486, Solid Zirconia, Newport Beach, CA, USA) were used to fabricate cylinders (2×2.5 $mm^2$). Zirconia cylinders were then sintered as recommended by the manufacturer, resulting in final dimensions of 1.8 mm in diameter and 2 mm in height. The surface of the cylinder to be bonded to the dentin substrate underwent water-polishing in a polishing device (Ecomet III, Buehler, Lake Bluff, IL, USA) with 800-, 1000-, and 1200-grit silicon carbide papers for 60 seconds in order to ensure the presence of homogeneous surfaces. The zirconia specimens were, finally, ultrasonically cleaned (Branson 200, Branson Ultrasonics Corp, Danbury, CT, USA) for eight minutes in isopropyl alcohol.

## Surface Pretreatments of the Zirconia Specimens

Two different surface pretreatments were performed, namely APA and TBC. Half of the zirconia cylinders (N=60) were air-abraded with 50 μm $Al_2O_3$ particles for 10 seconds from a distance of 10 mm with a pressure of 3 bar. Afterwards, the specimens were cleaned ultrasonically for eight minutes in isopropyl alcohol and dried with oil-free air. The rest of the cylinders underwent air-abrasion with 30 μm silica-coated $Al_2O_3$ particles (CoJet Sand, Lot No. 633121, ESPE Dental AG, Seefeld, Germany) for 10 seconds from a distance of 10 mm with a pressure of 2.8 bar. It was ensured that the sandblasting was performed perpendicularly to the bonding surface in both cases with a custom-made device. The residues were then removed with oil-free air. After each surface pretreatment, one specimen was used for surface compositional analysis and morphological evaluation with scanning electron microscopy (SEM; Jeol J.S.M. 840A, Jeol, Tokyo, Japan) and energy-dispersive spectroscopy (SEM-EDS, INCA 250, Oxford Instruments, Oxford, UK). Finally, all zirconia specimens were silanized with a silane coupling agent (Monobond S, Lot No. V30663, Ivoclar Vivadent, Schaan, Liechtenstein) for 60 seconds and then air-dried. Silanization of all zirconia specimens was performed to avoid bias against the groups that were submitted to APA.



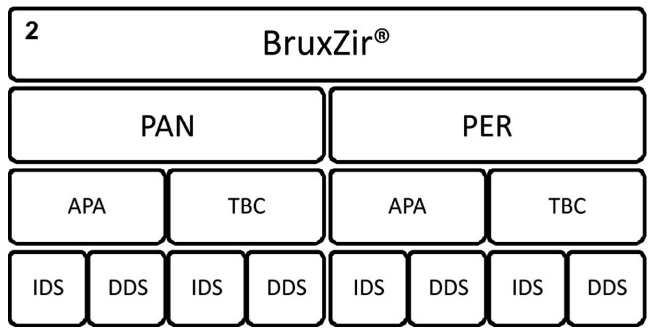



Figure 1. *Custom-made bonding clamp for the bonding procedures. The specimen is bonded and excess cement is removed immediately after its initial set.*

Figure 2. *Study groups according to luting agent type, zirconia surface pretreatment, and dentin conditioning. PAN: Panavia F2.0; PER: PermaCem Dual Smartmix; APA: airborne particle abrasion with 50 μm $Al_2O_3$ particles (3 bar, 10 seconds); TBC: tribochemical silica coating with 30 μm CoJet particles (2.8 bar, 10 seconds); IDS: immediate dentin sealing; DDS: delayed dentin sealing.*

## Bonding Procedures

Two adhesive luting agents were used to bond the zirconia specimens to the dentin substrate. PAN (Panavia F2.0, Lot No. 000052, Kuraray America Inc, Houston, TX, USA), a dual-cure adhesive MDP-containing cement, was used for half of the specimens, whereas PER (PermaCem Dual Smartmix, Lot No. 758470, DMG America, Chicago, IL, USA), a compomer cement consisting of an ionomer glass in a Bis-GMA–based matrix, was used for the rest. The manufacturers' instructions were followed, and the specimens were placed in a bonding clamp (custom-made device), as shown in Figure 1, until the removal of the cement excess. With regard to the IDS groups, the sealed dentin was air-dried and silanized for 60 seconds prior to the bondin of the zirconia specimen. Following the completion of the bonding procedure, the specimens were water-stored at 37°C for 24 hours before the shear bond strength (SBS) test was conducted. A total of eight groups were evaluated, as shown in Figure 2. All procedures were performed by the same operator in order to minimize variability in the bonding procedure of each specimen.

### Testing Procedure and Fractographic Analysis

Following water storage, the bonded specimens were placed in the appropriate fixture of the Universal Testing Machine (Testometric M350-10CT, The Testometric Company Ltd, Rochdale, UK) for the SBS test. Load until fracture followed with a 50-kgF load cell at a crosshead speed of 1 mm/min. The force at break was recorded through the Testometric software (WinTest Analysis CX, Version 3.5). SBS values were calculated with the formula $P/A$, where $P$ was the force at break (N) and $A$ was the bonded area (in $mm^2$). The fractographic analysis included an initial evaluation under a stereomicroscope (Stereo Discovery V40; Carl Zeiss, Oberkochen, Germany) at 40× magnification. This ensured the detection of cement residues on the zirconia and dentin surfaces, which would have been difficult to distinguish with the naked eye. The modes of failure were categorized as follows:

- Cohesive failure, in which more than two-thirds of the debonded surface presented luting agent residues on both the zirconia surface and the dentin substrate;
- Adhesive failure at the ceramic-cement interface (adhesive-zirconia [Zr]), in which less than one-third of the debonded zirconia surface presented luting agent residues;
- Adhesive failure at the dentin-cement interface (adhesive-D), in which less than one-third of the debonded dentin surface presented luting agent residues; and
- Mixed failure, in which adhesive and cohesive failures were present simultaneously.

Several typically debonded specimens from each of the observed failure types were sputter-coated with carbon, and SEM microphotographs were taken at various magnifications at 20 kW. EDS was performed on the back-scattered microphotographs received by SEM from the bonded surfaces after fracture (both on zirconia and dentin). Spot elemental analysis was performed at various areas of interest on each surface.

### Statistical Analysis

In order to ensure the validity of the statistical analysis and the normality of the results, a sequence of statistical control tests was performed (Shapiro-Wilk test, Levene test of homogeneity of variance, Cochran test, Dixon $Q$-test, Grubbs test for outliers, and the $Z$-score method) prior to the analysis. Three variables were studied (luting agent type, zirconia surface pretreatment, and dentin conditioning method), so three-way analysis of variance was selected as the statistical model. Bonferroni multiple comparison tests were performed to reveal statistically significant differences between groups. Descriptive statistics were calculated. All analyses were performed with the IBM Statistics SPSS 20.0 software ($\alpha=0.05$).

## RESULTS

Descriptive statistics are presented in Table 1. Based on the statistical analysis, a specimen from the group PER-TBC-IDS was excluded from the final results as well as the fractographic analysis. Following the Dixon and Grubbs tests, this specimen appeared to be an outlier, and based on the Tukey hinges theory, it had to be discarded from its respective group. Luting agent type and zirconia surface pretreatment as well as dentin conditioning method significantly affected SBS ($p<0.001$) (Table 2). As shown in Table 1, the dentin conditioning method appears to be the most detrimental factor, as it presents the largest effect size. The combination of luting agent type and zirconia surface pretreatment also presented a statistically significant effect on the SBS. From Bonferroni multiple comparison tests, the groups with APA exhibited better results with PAN compared to PER ($p<0.001$), whereas no difference was detected when TBC was used as the zirconia surface pretreatment ($p=0.648$). Furthermore, for groups bonded with PER, TBC showed better results compared to APA ($p<0.001$). On the other hand, groups bonded with PAN showed no statistical difference regardless of whether APA or TBC was applied ($p=0.519$).

As can be seen in Table 2, there are statistically significant differences among the groups [$F(7,111)=6.973$, $p<0.001$, $R^2=30.5\%$] with regard to the three variables evaluated. Greater effect size was recorded for the interaction between the luting agent system and zirconia surface pretreatment.

Representative SEM microphotographs following each zirconia surface pretreatment are presented in Figure 3. The as-received specimen presented the smoothest surface, with well-dispersed micropores. The APA specimen presented a lot of imperfections and irregularities and a quite-rough surface. The surface of the TBC specimen had no significant

Table 1: *Descriptive Statistics for Each Combination of Study Factors*[*]

| Groups | N | Min | Max | Median | Mean$^2$ | SD$^2$ | CV(%)SQRT |
|---|---|---|---|---|---|---|---|
| PER-TBC-IDS[a] | 14 | 12.358 | 104.351 | 40.537 | 40.704 | 3.803 | 9.34 |
| PER-TBC-DDS[b] | 15 | 9.53 | 51.146 | 21.989 | 24.305 | 1.300 | 5.35 |
| PER-APA-IDS[b] | 15 | 8.745 | 55.796 | 27.603 | 26.214 | 1.742 | 6.65 |
| PER-APA-DDS[c] | 15 | 0.247 | 32.878 | 15.306 | 12.603 | 2.310 | 18.33 |
| PAN-TBC-IDS[a] | 15 | 21.291 | 69.276 | 36.093 | 38.688 | 1.166 | 17.36 |
| PAN-TBC-DDS[b] | 15 | 7.247 | 61.557 | 30.333 | 29.376 | 2.161 | 7.36 |
| PAN-APA-IDS[a] | 15 | 22.46 | 77.415 | 40.767 | 39.942 | 1.346 | 3.37 |
| PAN-APA-DDS[b] | 15 | 15.635 | 73.745 | 30.564 | 33.408 | 1.769 | 5.29 |

*Abbreviations: APA, airborne particle abrasion; CV(%)SQRT, coefficient of variation square-root; DDS, delayed dentin sealing; IDS, immediate dentin sealing; Max, maximum; Min, minimum; PAN, Panavia; PER, PermaCem Dual Smartmix; SD, standard deviation; TBC, tribochemical silica coating.*
[*] *Different superscript letters present statistically significant differences revealed from Bonferroni multiple comparison tests ($\alpha$=0.05).*

alterations compared to the as-received, although at higher magnification it seemed slightly rougher, yet smoother compared to the APA one.

For each surface, four area EDS measurements (spectrum 1 to spectrum 4) were performed, and their mean values are presented in Figure 3. A significant increase of aluminum (Al) is recorded for the APA specimen, as is a significant increase of silicon (Si) for the TBC specimen.

The failure type evaluation under stereomicroscopy revealed that adhesive failures on the ceramic-cement interface were the least common (Table 3), indicating that bond strengths to the ceramic surface were superior compared to the bond strengths achieved by the luting agent on the dentin surface.

Representative SEM microphotographs of each failure type are presented in Figure 4, along with the respective EDS analysis. In Figure 4a and b, a mixed failure type is observed on a PER-APA-DDS specimen. This failure type was confirmed by EDS elemental analysis on the surface of both dentin and zirconia specimens. An adhesive-D failure is observed in Figure 4c and d. This was evidenced by the fact that only calcium and phosphorus (P) were detected on the exposed dentin surface in Figure 4c (spectrum 1), while on the surface of the specimen in Figure 4d, elements such as Si, ytterbium (Yb), and barium (Ba), along with the complete absence of Zr, suggest the presence of the luting agent only. The presence of Yb indicates that dentin bonding adhesive remained on the zirconia surface. In the case of the specimen in Figure 4e and f, most of the zirconia surface is exposed, suggesting an adhesive-Zr failure type. A small amount of the luting agent is present, as shown from the EDS analysis (spectrum 2, Figure 4f). Finally, for the specimen in Figure 4g and h, the vast majority of the specimen's surface is covered by

Table 2: *Results from the Three-way Analysis of Variance*

| Source | Type III Sum of Squares | df | Mean Square | *F* | *p*-value | Partial Eta Squared |
|---|---|---|---|---|---|---|
| Corrected model | 94.257 | 7 | 13.465 | 6.973 | <0.001 | 0.305 |
| Intercept | 3553.614 | 1 | 3553.614 | 1840.364 | <0.001 | 0.943 |
| Luting agent type | 26.461 | 1 | 26.461 | 13.704 | <0.001 | 0.11 |
| Zirconia surface pretreatment | 8.852 | 1 | 8.852 | 4.584 | 0.034 | 0.04 |
| Dentin conditioning method | 35.424 | 1 | 35.424 | 18.345 | <0.001 | 0.142 |
| Luting agent type × zirconia surface pretreatment | 17.977 | 1 | 17.977 | 9.31 | 0.003 | 0.077 |
| Luting agent type × dentin conditioning method | 5.243 | 1 | 5.243 | 2.715 | 0.102 | 0.024 |
| Zirconia surface pretreatment × dentin conditioning method | 0.036 | 1 | 0.036 | 0.019 | 0.892 | 0 |
| Luting agent type × zirconia surface pretreatment × dentin conditioning method | 0.268 | 1 | 0.268 | 0.139 | 0.71 | 0.001 |
| Error | 214.333 | 111 | 1.931 | | | |
| Total | 3854.054 | 119 | | | | |
| Corrected total | 308.59 | 118 | | | | |

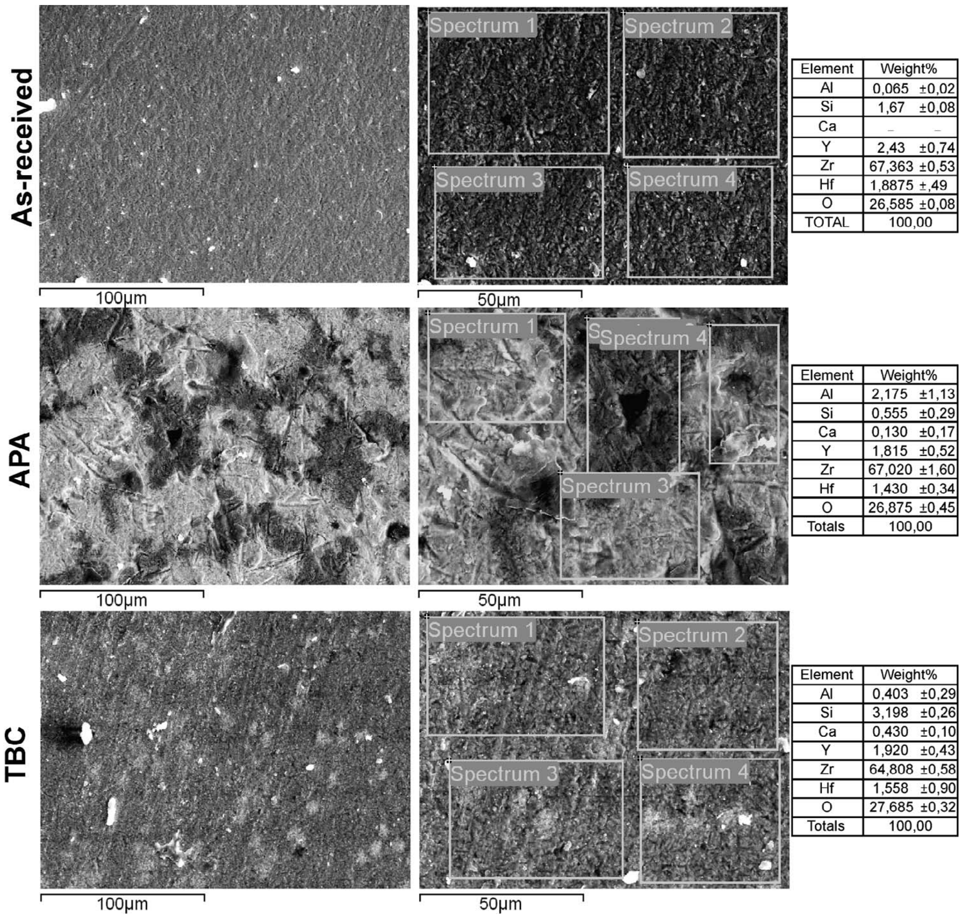



Figure 3. *Representative scanning electron microscopy (SEM) microphotographs following each zirconia surface treatment.*

a layer consisting of Al, Si, Ba, Yb, and P (Figure 4h, spectrums 1, 3, and 4), and only a minor area of zirconia surface is exposed (Figure 4h, spectrum 2). On the tooth surface, the presence of Si, Al, and Ba from the luting agent suggests a cohesive failure type.

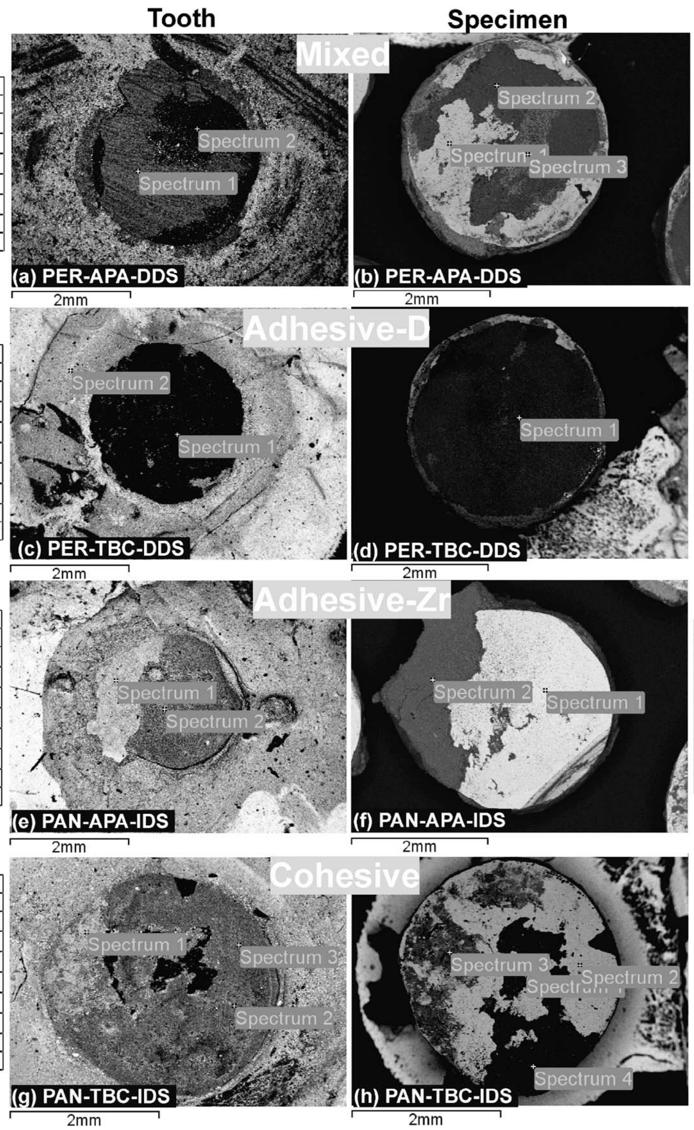
## DISCUSSION

This study evaluated the effects of adhesive luting agent type, monolithic zirconia ceramic surface pretreatment, and dentin conditioning method on the bond strength of a monolithic zirconia ceramic to human dentin.

Table 3: *Distribution of the Various Failure Types Within Each Group*

| | N | Mixed, % | Cohesive, % | Adhesive-D (Dentin), % | Adhesive-Zr (Zirconia), % |
|---|---|---|---|---|---|
| PER-TBC-IDS | 14 | 29 | 50 | 14 | 7 |
| PER-TBC-DDS | 15 | 7 | 86 | 7 | 0 |
| PER-APA-IDS | 15 | 60 | 20 | 20 | 0 |
| PER-APA-DDS | 15 | 26 | 13 | 61 | 0 |
| PAN-TBC-IDS | 15 | 73 | 13 | 7 | 7 |
| PAN-TBC-DDS | 15 | 79 | 7 | 7 | 7 |
| PAN-APA-IDS | 15 | 27 | 40 | 6 | 27 |
| PAN-APA-DDS | 15 | 46 | 13 | 26 | 15 |

*Abbreviations: APA, airborne particle abrasion; DDS, delayed dentin sealing; IDS, immediate dentin sealing; PAN, Panavia; PER, PermaCem Dual Smartmix; TBC, tribochemical silica coating.*




| | spectrum 1 | spectrum 2 |
|---|---|---|
| Element | Weight% | Weight% |
| P | 19,74 | – |
| Ca | 39,15 | – |
| Al | – | 11,29 |
| Si | – | 21,87 |
| Ba | – | 28,57 |
| O | 41,11 | 38,28 |
| Totals | 100 | 100 |

| | spectrum 1 | spectrum 2 | spectrum 3 |
|---|---|---|---|
| Element | Weight% | Weight% | Weight% |
| F | – | 7,14 | 8,73 |
| Mg | – | – | 1,6 |
| Al | – | 10,73 | 10,74 |
| Si | 2,2 | 17,68 | 17,07 |
| P | – | 2,62 | 2,7 |
| Ba | – | 25,75 | 22,95 |
| Zr | 70,55 | – | – |
| O | 27,25 | 36,07 | 36,21 |
| Totals | 100 | 100 | 100 |

| | spectrum 1 | spectrum 2 |
|---|---|---|
| Element | Weight% | Weight% |
| P | 19,62 | – |
| Ca | 39,33 | – |
| Al | – | 3,67 |
| Si | – | 25,52 |
| Ba | – | 20,09 |
| Yb | – | 14,1 |
| O | 41,04 | 36,63 |
| Totals | 100 | 100 |

| | spectrum 1 |
|---|---|
| Element | Weight% |
| Si | 20,53 |
| Ba | 16,7 |
| Yb | 32,87 |
| O | 29,9 |
| Totals | 100 |

| | spectrum 1 | spectrum 2 |
|---|---|---|
| Element | Weight% | Weight% |
| Si | 1 | – |
| P | 19,52 | – |
| Ca | 37,96 | – |
| Al | – | 2,73 |
| Si | – | 33,98 |
| Ba | – | 19,84 |
| O | 41,51 | 43,45 |
| Totals | 100 | 100 |

| | spectrum 1 | spectrum 2 |
|---|---|---|
| Element | Weight% | Weight% |
| Al | – | 20 |
| Si | – | 7,28 |
| Ca | – | 18,84 |
| Sr | – | 17,15 |
| Zr | 74,03 | – |
| O | 25,97 | 36,73 |
| Totals | 100 | 100 |

| | spectrum 1 | spectrum 2 | spectrum 3 |
|---|---|---|---|
| Element | Weight% | Weight% | Weight% |
| Al | – | 1,57 | 1,16 |
| Si | – | 39,21 | 39,79 |
| P | 18,91 | – | – |
| Ca | 38,96 | – | – |
| Ba | – | 12,02 | 11,7 |
| Yb | – | – | – |
| O | 41,12 | 47,2 | 47,35 |
| Totals | 100 | 100 | 100 |

| | spectrum 1 | spectrum 2 | spectrum 3 | spectrum 4 |
|---|---|---|---|---|
| Element | Weight% | Weight% | Weight% | Weight% |
| Al | 3 | – | 1,84 | 1,35 |
| Si | 29,42 | 2,72 | 19,62 | 37,54 |
| Ba | 28,62 | – | 12,08 | 11,29 |
| Yb | – | – | 15,93 | – |
| P | – | – | – | 1,98 |
| Zr | – | 69,91 | 16,98 | – |
| O | 38,96 | 27,37 | 33,55 | 47,84 |
| Totals | 100 | 100 | 100 | 100 |

Figure 4. *Representative scanning electron microscopy (SEM) backscattered microphotographs of each failure type with the respective energy-dispersive x-ray spectroscopy (EDS) spot analysis.*

To the best of our knowledge, no studies have investigated the bond strength of monolithic zirconia ceramics to dentin, and, hence, the results of this study are being compared to those of studies in which conventional zirconia ceramics were used. The surface treatments that were selected were acknowledged to represent the most well-established mechanical surface treatments for zirconia ceramics, and silanization was performed as an additional step in all specimens of the experiment.[31,32] With regard to zirconia surface pretreatment, the results of this study showed that when TBC was applied, the highest values were recorded with PER, while when APA was applied, the highest values were recorded with PAN. Furthermore, PAN achieved significantly higher SBS values in all but one group (PAN-TBC-IDS). Panavia F2.0 is a self-etching, resin-based cement, which contains the functional monomer 10-MDP.[33] Apart from achieving a chemical bond with zirconia, this monomer enhances chemical adhesion to dentin.[34,35] PermaCem Dual Smartmix is a universal adhesive dual-curing compomer cement.[36] As anticipated for a resin-modified glass ionomer cement, there is a combination of properties from glass ionomer cements as well as resin-based cements. Thus, a polymerization reaction triggers the setting of the cement, and an acid-base reaction leads to the final maturation.[37] Studies have reported lower bond strengths for compomer cements compared to adhesive resin cements when used for the cementation of various restorative materials.[38-40] The MDP agent has been reported to chemically interact with the zirconia ceramic surface[41] and in particular to form strong bonds between the phosphate ester group of the monomer and the metal oxides, such as Al and Zr.[42,43] The intervention of the TBC particles could potentially prohibit the MDP mechanism from reaching its full effect, or a competitive mechanism between the silane coupling agent and the MDP monomer may exist. An additional contributing factor to these results may be the smoothest surface related with TBC, as higher surface roughness results in higher SBS when cementation is performed with PAN.[44-46] This is in agreement with the literature on MDP-based cements.[6,47] Furthermore, this is also in agreement with the findings of de Castro and others[48] and Nothdurft and others,[49] according to whom the initial bond strength of PAN is greater when APA is used as a surface treatment. The positive effect of TBC in the case of PER might be attributed to strong bond formation between the silicate-barium glass network of the cement with the silica layer following TBC. The compomer cement (PER) achieved inferior SBS values compared to the MDP-based cement (PAN), which is in accordance with the findings of previous studies.[9,50]

Regarding dentin conditioning method, it was shown that IDS significantly improves the bond strength of monolithic zirconia to dentin, regardless of the type of cement or zirconia surface treatment, the highest values were recorded for the IDS groups. This finding has already been published with regard to other types of indirect bonded restorations.[29] Higher microtensile bond strength was recorded, with a distinct and thicker hybrid zone, when ceramic inlays[51] or crowns[52] were adhesively bonded to immediately sealed dentin surfaces. In the present study, the positive effect on the bond strength was more profound for the PER groups, as almost double the values were recorded for the IDS groups bonded with this self-adhesive cement.

The surface treatment of the zirconia disks was followed by silane application. Silanes are hybrid molecules, the structure of which consists of an inorganic and an organic part. As a result, silanes are bifunctional molecules. They have been used to promote adhesion between resin composite materials and ceramics consisting of a glassy matrix.[53,54] Silanes increase the wettability and micromechanical interlocking of ceramic surfaces[3,55] as well as the flowability of the luting agent.[56] These molecules have been considered ineffective when used to improve the bond strength of Y-TZP ceramics to resin cements because of the lack of silica in this type of ceramic.[42,57] Hence, TBC has been required in order for silane application to result in bond strength increase, although the bond's durability has been questioned.[23,42] Nonetheless, a significant bond strength improvement as a result of silane application alone has also been found.[58] It was subsequently decided to follow both APA and TBC with silanization.

The fractographic analysis in conjunction with the SBS values of the present study led to the observation that there is a potential correlation between the cohesive type of failure and the application of PER, which is in agreement with the findings of Turker and others.[50] Simultaneously, PER showed the smallest percentage of adhesive failure in the cement-ceramic interface. This means that the cohesive failures of PER are attributed basically to the mechanical failure of the cement within its mass, due to the stronger bond with both the zirconia and the dentin surface and the limited capacity of the cement to withstand such high forces. On the other hand, PAN exhibited mostly mixed failures, likely indicating improved mechanical strength. As luting cement film thickness seems to have an effect on the microtensile bond strength of resin cements to ceramics,[59] further research is indicated to clarify if the higher values of PAN might be associated with higher film thickness.

Despite the limitations of the bond strength test methods,[60] the SBS test was selected for the present experiment. SBS tests, in general, result in value inhomogeneity due to the incidence of cohesive failures in the mass of either substrate (ceramic or dentin).[61-63] However, the fabrication of specimens for a SBS test is rather simple[64] compared to that of specimens for a TBS test.[65] Specimens with a bonding area of approximately $A = 2.56\ mm^2$ were used, which is significantly smaller compared to an area of $7\ mm^2$ that would result in a macro-SBS test.[66] It has been proven that the incidence of a critical flaw in the bonded interface is minimized as the area of the bonded zirconia specimens gets smaller.[64,67]

The evaluation of the bond strength between the ceramic material and adhesive cements is more clinically relevant when a dentin substrate is used.[48,68-70] There are a few studies evaluating the bond strength of monolithic zirconia ceramics to adhesive luting agents.[71,72] However, these studies did not simulate the complex clinical scenario by including a dentin substrate in their methodology, ultimately testing the bond strength of various cements on the monolithic zirconia surface.

Previously published studies evaluating the effect of IDS on dentin bond strength have included in their methodology either a provisionalization period[29,73]or an artificial aging period of the treated teeth before the luting procedures.[30] This stage has been omitted in the methodology of the present study, as luting procedures followed within four hours after the exposure of the freshly cut dentin substrate. Thus, based on the hypothesis that provisional restorations and temporary cements ideally provide full protection of the prepared tooth structure from microleakage, as well as on the fact that the incorporation of such a stage would compromise the experiment's standardization, it was decided that the zirconia specimens would be bonded to the IDS-treated tooth specimens within the four-hour timespan. This should be considered a limitation of the present experiment. Moreover, no thermomechanical cycling was used for the aging of the specimens. Instead, a period of 24 hours of water storage was used, according to the guidelines of ISO 29022:2013. However, this should also be considered a limitation of the study, as there was no simulation of the oral environment between the bonding of the specimens and the testing procedure. Thus, the durability of the bond strength after artificial aging was not revealed. Finally, the failure-type classification in the present study was based on evaluation under the stereomicroscope. Nonetheless, morphological and compositional analysis of the debonded interfaces with SEM-EDS is more reliable and should be implemented for this purpose in future studies.

## CONCLUSION

Based on the results, all three null hypotheses were rejected, as luting agent type, zirconia surface pretreatment, and dentin conditioning method significantly affected the bond strength of monolithic zirconia to dentin. Bonding strategies for monolithic zirconia restorations could potentially benefit from IDS, regardless of the adhesive luting agent system used. Moreover, MDP-containing luting agents are more effective when combined with airborne particle abrasion.

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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 Bio-ethics Committee of Aristotle University of Thessaloniki. The approval code for this study is 36/25-05-2017.

### Conflict of Interest

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

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