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RESEARCH ARTICLE

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# Shear Bond Strength of CAD/CAM Highly Translucent Zirconia Bonded to Occlusal Dentin after Different Extrinsic Modifications: An *In vitro* Study

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**Abstract:**

***Introduction:*** The bond between highly translucent zirconia (HTZ) onlay restorations and the resin cement is challenging due to its non-glassy structure. This study compares the effect of different extrinsic modifications on the shear bond strength of HTZ to occlusal dentin after thermocycling.

***Methods:*** Forty human molars were prepared and stored in 0.5% chloramine. Ceramic discs (n = 40) were distributed into four groups. HTZ discs underwent airborne-particle abrasion (Group ZA), 30 μm diamond abrasion (Group ZB), and no treatment (Group ZN). All HTZ discs were treated with 10-MDP primer. Lithium disilicate discs (Group LD-control) were treated with about 4.5% hydrofluoric acid and silane. All discs were cemented to dentin using resin cement. SBS was measured using a universal testing machine, and fracture types were assessed.

***Results:*** Group ZA showed the greatest SBS among HTZ groups (Tukey's test; *p*-value < 0.05), though none exceeded the LD-control group (*p*-value < 0.0001). Most fractures occurred at the zirconia-resin cement interface, relatively less in group ZA (40%).

***Discussion:*** The current study highlights that combined mechanical and chemical treatment has the potential to strengthen the bond of HTZ to occlusal dentin. Moreover, it includes both the resin-zirconia and resin-dentin interfaces in the experiment that closely simulates the intracoronal restoration. One of the limitations is that the results may vary in the oral environment during the clinical trials because of external factors, such as saliva, food, and masticatory forces. Use of thermo-mechanical ageing could have overcome this limitation.

***Conclusion:*** Combined mechanical and chemical treatment improved HTZ bonding to dentin. Further clinical studies are needed to establish optimal surface modification protocols.

**Keywords:** Bond strength, Dental ceramic, Dental onlays, 10-MDP, Resin cement, Zirconia.

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## 1. INTRODUCTION

Polycrystalline tetragonal zirconia stabilized with yttrium at 3 mol% is ideal for onlays in posterior teeth with favorable qualities like biocompatibility, aesthetic appeal, and strong mechanical properties. Its qualities have improved despite its lack of purity due to the addition of minute amounts of yttrium oxide or hafnium [1, 2]. In recent times, it has shown emergence as a versatile and promising material among other dental ceramics. The opacity offered by the tetragonal phase of zirconia has been recently replaced with the cubic phase of monolithic zirconia that contains relatively more yttria stabilizers (3 mol%; partially stabilized zirconia), which offers translucency at a thickness of 0.5 mm [3, 4].

The high translucency of zirconia is obtained at the expense of its strength due to the reduction in the toughening during the transformation from the tetragonal to the monoclinic phase. This behavior is attributed to the increase in cubic biphasic stable phase (30-50%) that does not undergo phase transformation to stop the crack propagation [4, 5]. It is thus defined as a mixed cubic/tetragonal fully stabilized zirconia. The cubic phases were increased by adding yttrium oxide to 5 mol% in the third-generation zirconia [6]. Basically, the volume of the cubic crystals is greater than that of the tetragonal ones. This makes the material more translucent because light scatters less intensely at the residual porosities and grain borders. In addition, the cubic crystal structures emit incident light more uniformly in all spatial directions than the tetragonal ones because they are more isotropic [6].

Zirconia's structure and extrinsic modification are key factors in achieving a long-lasting bond between zirconia and the adhesive cement [7]. Many adhesion techniques have been studied to date, including zirconia extrinsic modifications and the use of adhesives, primers, and resin cements [1, 8, 9]. It is well known that the bonding of zirconia to teeth or any other substrate needs a strong resin bond [1]. There are various micro-mechanical bonding techniques that involve sandblasting with aluminum oxide, diamond grinding, and etching with hydrofluoric acid. Many chemical bonding techniques, such as tribochemical silica coating, porcelain coating, and application of coupling agents like 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and silanes, were also tried [1]. However, the physical properties and composition of zirconia differ significantly from ceramics, which contain silica. The low reactivity of zirconia with acid imposes a constraint on the acid etching process [10, 11]. Kern *et al*. proposed that a long-lasting, robust bonding to zirconia might be achieved using air particle abrasion, primers, and/or luting resins incorporated with 10-MDP, which was supported by the data from the clinical trials compared to laboratory studies on bond strength [12].

Filling the minute void between an indirect restoration and the tooth, with mechanical locking of the restoration in place to prevent dislodgement during function, is the main function of the luting agent/material [13]. Modern luting agents, such as resin, glass-ionomer, and resin-modified glass-ionomer, show a high success rate clinically [13–15]. Resin luting agents are unique in filling and sealing the tooth-restoration gap by forming the polymer matrix. Glass-ionomer cements, possessing adequate translucency, are easy to mix with good flow properties and adhesion to the tooth structure and base metals. They are primarily used for luting metal and metal-ceramic restorations, but can also be used with all-ceramic crowns (alumina/zirconia) [13]. On the other hand, a study contradicted the positive impact of air abrasion on zirconia's flexural characteristics [16].

However, the highly translucent zirconia (HTZ) does not yield to etching, which consequently prevents precise identification of the adhesive processes [7]. Overall, there is no standard protocol that discusses the relative extrinsic modification method, which involves any mechanical or chemical, or a combination of chemical and mechanical methods for the bonding of HTZ to resins.

The present research mainly focuses on the comparison of different mechanical and chemical treatments on HTZ and their impact on shear bond strength (SBS) against occlusal dentin that represents a substrate for inlays and onlays. The primary intention of this study was to evaluate the shear bond strength of highly translucent zirconia treated with different extrinsic modifications against occlusal dentin after thermocycling. The secondary intention was to compare the quality of extrinsic modifications based on the frequency of fracture types. The null hypothesis considered was that there is no difference in the shear bond strength of HTZ to occlusal dentin due to different extrinsic modifications.

Although several studies have evaluated different surface treatments on zirconia, few reports have demonstrated HTZ adhering to occlusal dentin, which is a therapeutically relevant substrate for onlays and overlays, especially after thermocycling. The novelty of this study is in its direct comparative analysis of various mechanical and chemical surface treatments on HTZ, alongside thermocycling, to evaluate the clinically significant shear bond strength (SBS) results. Our research used occlusal dentin to accurately simulate the intraoral environment compared to previous assessments using flat enamel or artificial substrates.

## 2. MATERIALS AND METHODS

This *in vitro* study adhered to the Declaration of Helsinki and ethical approval (REC-45/05/891& CODJU-2316I dated November 5 and December 10, 2023, respectively) was provided by the Scientific Research Committee of Jazan University. The composition and manufacturer details of materials used in this study are given in Table **1**.

### 2.1. Sample Size

The power of the study was calculated using means and standard deviations from comparable prior research [17], with the help of G*Power software, the sample size determined was less than ten while assuming the power as 80%. Nonetheless, it was determined that ten samples in each group were sufficient.

**Table 1. Materials used for experiments.**

| Name | Specification | Composition | Product | Company |
|---|---|---|---|---|
| Highly translucent zirconia, | CAD/CAM ceramic, 98x16 mm, Shade A1 | (ZrO2+ HfO2+ Y2O3) ≥ 99%, other oxides (≤ 0.5).[Y2O3 (4.5-6%), Al2O3(≤ 0.5%)] | UpceraDental Zirconia HT | Upcera Dental Technology Co. Ltd., Shenzhen, China. |
| Glass-ceramic | CAD/CAM ceramic | Lithium di silicate,ZrO2 (0.0 – 8.0%), ZnO (0.0 – 8.0%), Al2O3 (0.0 – 5.0%), MgO (0.0 – 5.0%), K2O (0.0 – 13.0%), P2O5 (0.0 – 11.0%), Coloring oxides (0.0 – 8.0%) | IPS e.max CAD HT | IvoclarVivadent AG, Schaan, Liechtenstein |
| Self-adhesive dual cure resin cement | Medium viscosity, universal shade | UEDMA, BMEP, HEMA, TMPTMA, H2O, catalysts Fillers: Barium,glass, ytterbium trifluoride, inert minerals | Embrace wetbond | Pulpdent Corp., Watertown, MA, USA |
| Air abrasion unit | Powder | Precious corundum (Al2O3) of particle size 50 µm | Cobra | Renfert GmbH, Hilzingen, Germany |
| Ceramic primer | Liquid | 3-trimethoxysilylpropyl methacrylate,10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Ethanol | Clearfil ceramic primer plus | Kuraray Noritake Dental Inc., Okayama, Japan |
| Ceramic etchant | Gel | >5% Hydrofluoric acid | IPS Ceramic Etching Gel | IvoclarVivadent AG, Schaan, Liechtenstein |
| Silane | Liquid | Silane methacrylate, phosphoricmethacrylate and sulfide methacrylate | Monobond N | IvoclarVivadent AG, Schaan, Liechtenstein |

## 2.2. Teeth Specimens

Forty freshly extracted human permanent molars due to periodontal reasons were collected from adults with informed consent between December 11, 2023, and February 5, 2024, from the institute where permission was granted, and stored in 0.5% chloramine T (TCI America, Lake Forest, IL, USA) liquid for 7 days and then transferred to physiological saline to keep them hydrated. Acrylic resin cylinders (Ø28 mm x 30 mm height) were used to mount the teeth with the crown exposed. A unique holder was used to place the tooth inside the mold to guarantee exact alignment and parallelism. The occlusal surface was ground to expose the dentin with the help of a grinding unit (Automata A; Jean Wirtz, Dusseldorf, Germany) under water coolant. The mounted cylinders containing teeth were divided into 4 groups of 10 each by using specific software (Random Allocation Software, Version 2.0; Microsoft Corporation, WA, USA) [9-11, 18].

## 2.3. Preparation of Ceramic Samples

Forty cylindrical (Ø4 mm x 6 mm height) ceramic specimens were prepared (Fig. **1**). Thirty specimens were milled from pre-sintered HTZ blanks (Dental Zirconia HT; Upcera Dental Technology Co. Ltd., Shenzhen, China) using computer-aided design (Worknc CAM software; Hexagon AB, Stockholm, Sweden) and computer-aided manufacturing (CAD/CAM) on a wet milling machine (A52W, Upcera Dental Technology Co. using diamond-coated burs (OnePro Dental, Shandong, China), followed by final sintering at 1450°C (GT1 furnace; Upcera Dental Technology Co. Ltd.) as per the manufacturer’s instructions. Using random allocation software, the HTZ samples were also split into three experimental groups of ten. Another set of 10 discs was digitally milled from lithium disilicate glass (IPS e.max CAD; Ivoclar Vivadent AG, Schaan, Liechtenstein) blocks as per the manufacturer’s instructions. The final e.max restoration was created by digitally milling the glass ingots using diamond milling burs (Diamond RFID, Amann Girrbach AG, Maeder, Austria) in a milling machine (Ceramill Matik; Amann Girrbach AG) using the Dental CAD TruSmile Module (exocad GmbH, Darmstadt, Germany). This created ceramic blocks with the appropriate shape, which were subsequently heated at 850°C in a furnace (Program at CS6; Ivoclar Vivadent AG). Every specimen was polished using abrasive paper with grits of 600, 800, and 1000 while being water-cooled. All the pairs of non-bonded side ceramic discs and the sides of the respective mounted resin cylinders were color-coded to segregate group-wise prior to extrinsic modification.

## 2.4. Groups and Extrinsic Modification

The prepared ceramic specimens were mounted group-wise temporarily on condensation silicone impression material (Zeta plus; Zhermack SpA, Polesine, Italy) for convenience in handling during the extrinsic modification.

Group ZA-HTZ samples were exposed to air particle abrasion using alumina particles of 50 µm in size (Cobra, Renfert GmbH, Hilzingen, Germany) at a pressure of 2.5 bar positioned 10 mm away from the nozzle and directed perpendicular to the longitudinal axis of the HTZ disc surface for 15 seconds/mm$^2$ using a tabletop device (Vario Basic, Renfert GmbH, Hilzingen, Germany) and chemically treated with a liquid possessing 10-MDP (Clearfil primer plus; Kuraray Noritake Dental Inc., Okayama, Japan). A micro brush was used to apply a single layer of ceramic primer, which was then allowed to sit for 10 seconds and allowed to dry for 5 seconds.

Group ZB-Zirconia samples were abraded under water coolant with 30 µm grit round diamond bur (Bredent, Senden, Germany) under low speed (20000 rpm), and the chemical treatment was done with ceramic primer containing 10-MDP. The diamond bur was moved across the surface of the HTZ disc in a single direction and was replaced after using it on five samples.

Group ZN-No mechanical treatment was done in this group. Chemical treatment was performed with a ceramic primer containing 10-MDP.

Group LD (control)-4.5% hydrogen fluoride was used to treat the lithium disilicate glass ceramic samples for 20 seconds, followed by silanization with 3-methacryloxypropyl-trimethoxysilane (Monobond N; Ivoclar Vivadent AG) for 1 minute.

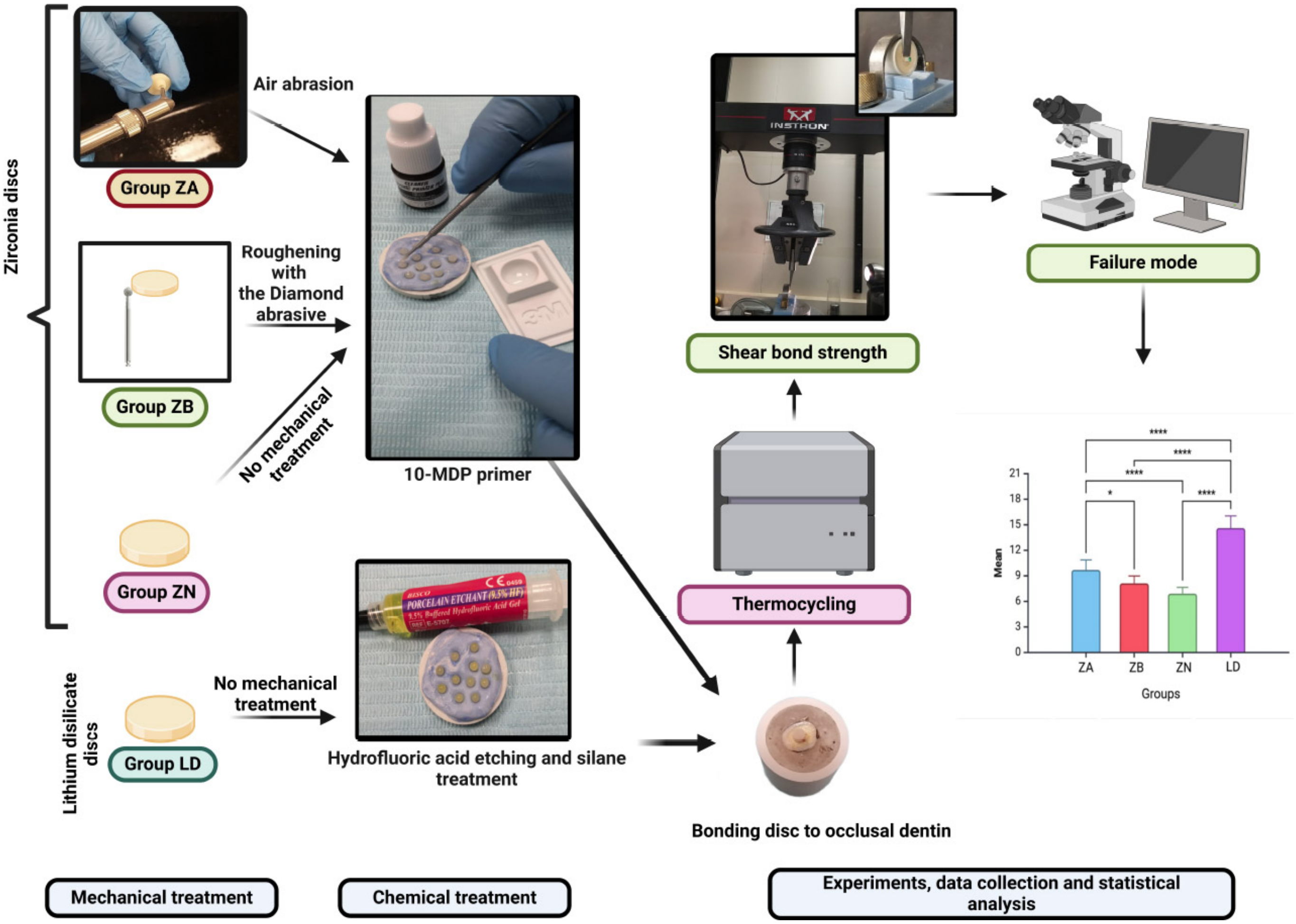

**Fig. (1).** Schematic representation of methodology followed in this study.

After cleaning for five minutes in acetone and five minutes in water using an ultrasonic cleaner (SW1500, CBM, Tokyo, Japan) [19, 20], each specimen was dried using an oil-free air spray. The air-abrasion procedures were carried out by a single experienced technician. All the ceramic specimens were subsequently bonded [18] to the corresponding teeth (Fig. **2A-C**) using self-adhesive resin cement (Embrace WetBond; Pulpdent Corp., Watertown, MA, USA) under static pressure of 30 N (Fig. **2A**) mimicking the thumb pressure on a surveyor (Dentalfarm SRL, Turin, Italy) for 2 minutes to ensure proper seating and a thin film of resin cement [21]. During this time, the extruded flash of luting resin was cleaned using a microbrush, and the interphase was protected from atmospheric oxygen using a gel (Liquid Strip, Ivoclar Vivadent AG). The samples were light-cured for 30 seconds with a light-curing unit (Bluephase Style, Ivoclar Vivadent AG) with a power intensity of 1100 mW/cm$^2$. After 3 minutes of setting time and an incubation period of 37°C, the samples were thermocycled (Thermocycler THE1100e/230; SD Mechatronic GmbH, Feldkirchen-Westerham, Germany) for 5000 heat cycles that alternate between 5°C and 55°C every 5 seconds and a holding time of 5 seconds in water [22].

## 2.5. Evaluation of Shear Bond Strength

The SBS was evaluated with the help of a universal testing machine under shear force (Instron 5965 Dual Column Tabletop Testing Machine, Canton, MA, USA). The bonded specimens were positioned firmly on a support that kept the plane of the bonded interface in line with the long axis of the crosshead. The sample was stressed in shear (Fig. **2D**) by moving the head at the rate of 1 mm/min until fracture occurred. The highest force at fracture was recorded, and the SBS was determined by applying the formula SBS (MPa) = load (N)/area (mm$^2$), where 12.57 mm$^2$ is the area of the disc face [23].

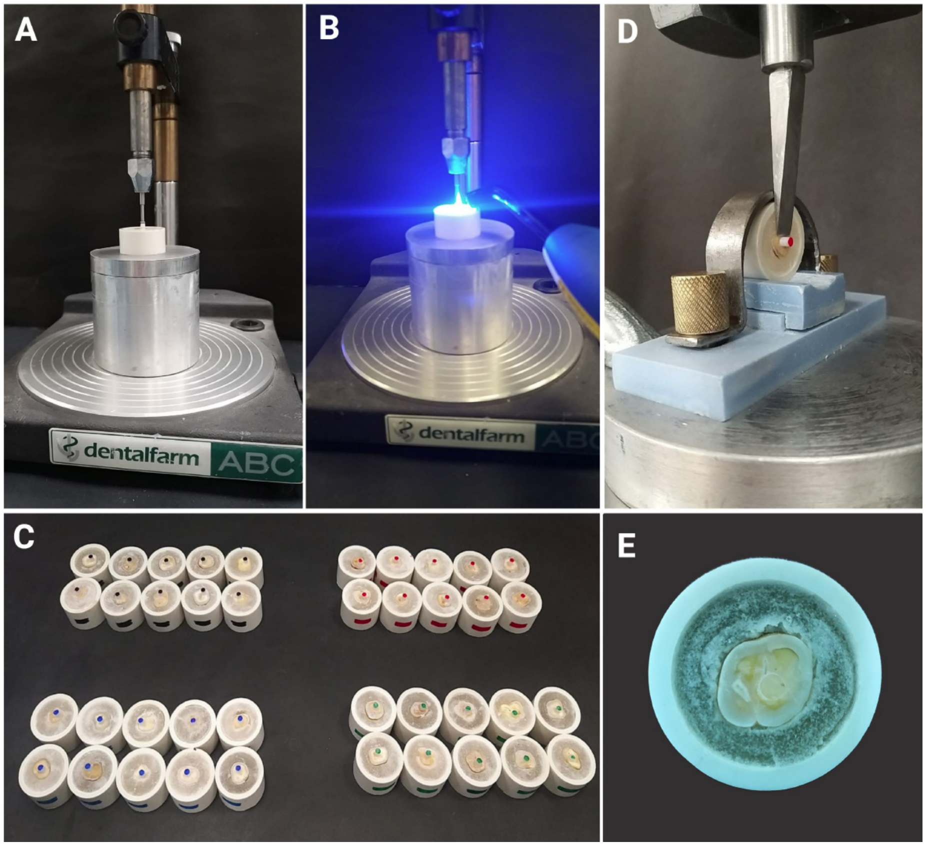

**Fig. (2).** Shear bond strength test. (**A**) Simulated pressure applied while bonding ceramic disc to occlusal dentin, (**B**) Light curing the resin cement, (**C**) Bonded samples of all four groups with color code, (**D**) Illustration of shear force on the universal testing machine, (**E**) Occlusal dentin after shear fracture for fracture type analysis.

## 2.6. Type of Fracture

The fracture type was analyzed using a computer-controlled microscope (KH-7700; Hirox Co., Ltd., Tokyo, Japan). The fractured dentin samples (Fig. **2E**) were observed on a digital microscope at 50x magnification. All the images were categorized by a single investigator under standardized room lighting conditions as follows:

- Adhesion fracture at the resin-dentin interface (less than 50% or no luting resin).
- Adhesion fracture at the resin-ceramic interface (100% resin cement without craters).
- Cohesion fracture in the resin cement (100% resin cement with irregular craters).
- Combination of both adhesion and cohesion fracture (100% resin cement with irregular craters).

## 2.7. Statistical Analysis

The data were analyzed using IBM SPSS Statistics for Windows (Version 29.0; IBM Corp., Armonk, NY). Mean and standard deviation (SD) were employed as descriptive statistics to characterize the data. One-way ANOVA in conjunction with post hoc analysis using Tukey's test was utilized to determine the significant difference in the multivariate analysis. The probability value of 0.05 is regarded as the significance level in all the statistical techniques mentioned above.

## 3. RESULTS

The mean SBS among the four groups (Table **2**) showed a highly significant (one-way ANOVA; $p$-value <0.0001) variation; group ZA showed a higher mean value than the other two experimental groups. The control (group LD) had the highest mean SBS of all the groups. The SBS in all the experimental groups (Table **3**, Fig. **3**) was significantly lower than that of the control (Tukey's test; $p$-value < 0.0001). The mean SBS of group ZA (9.7 ± 1.18) was significantly higher ($p$-value < 0.05) than that of groups ZB (8.11 ± 0.87) and ZN (6.9 ± 0.75). The mean difference in SBS between the groups ZB and ZN was not significantly different ($p$-value = 0.7942). The dentin face of fractured samples in the digital micrographs revealed that the zirconia samples predominantly exhibited adhesion fracture at the resin-ceramic interface (Table **4**, Fig. **4A-D**), whereas control samples exhibited adhesion fracture at the resin-dentin interface. Among the experimental groups, group ZA had the least (40%) fractures at the resin-ceramic interface.

**Table 2. Multivariate study of the shear bond strength data and their descriptive statistics.**

| Group | N | Mean | SD | 95% Confidence Interval | *p*-value |
|---|---|---|---|---|---|
| ZA | 10 | 9.70 | 1.18 | 8.85 to 10.54 | <0.0001 * |
| ZB | 10 | 8.11 | 0.87 | 7.49 to 8.74 | |
| ZN | 10 | 6.90 | 0.75 | 6.36 to 7.44 | |
| LD | 10 | 14.61 | 1.44 | 13.59 to 15.64 | |

**Note:** * One-way ANOVA; Significant at *p*-value <0.05; SD: Standard Deviation; ZA: Air-abraded HTZ; ZB: Diamond abrasion HTZ; ZN: Untreated HTZ; LD: Lithium disilicate discs.

**Table 3. Comparison of mean shear bond strength between groups of ceramic samples.**

| Groups | | Mean Difference | Standard Error | *p*-value | 95% CI of Mean Difference |
|---|---|---|---|---|---|
| ZA | ZB | 1.58 | 0.489 | 0.01326 * | 0.264 to 2.897 |
| | ZN | 2.8 | 0.489 | <0.0001 * | 1.479 to 4.112 |
| | LD | -4.92 | 0.489 | <0.0001 * | -6.236 to -3.603 |
| ZB | ZN | 1.21 | 0.489 | 0.07941 | -0.102 to 2.532 |
| | LD | -6.5 | 0.489 | <0.0001 * | -7.816 to -5.183 |
| ZN | LD | -7.71 | 0.489 | <0.0001 * | -9.031 to -6.398 |

**Note:** * Tukeys post hoc test; Significant at *p*-value < 0.05, CI: Confidence interval; Standard Deviation; ZA: Air-abraded HTZ; ZB: Diamond abrasion HTZ; ZN: Untreated HTZ; LD: Lithium disilicate discs.

**Table 4. Distribution of fracture types after subjecting to shear load.**

| Groups | N | Adhesive at Resin-dentin (%) | Adhesive at Resin-ceramic (%) | Cohesive (%) | Combined Fracture (%) |
|---|---|---|---|---|---|
| ZA | 10 | 2 (20) | 4 (40) | 2 (20) | 2 (20) |
| ZB | 10 | 1 (10) | 5(50) | 1 (10) | 3 (30) |
| ZN | 10 | 0 (0) | 6(60) | 2 (20) | 2 (20) |
| LD | 10 | 9 (90) | 0 (0) | 0 (0) | 1 (10) |
| Total | 40 | 12(30) | 15 (37.5) | 5 (12.5) | 8 (20) |

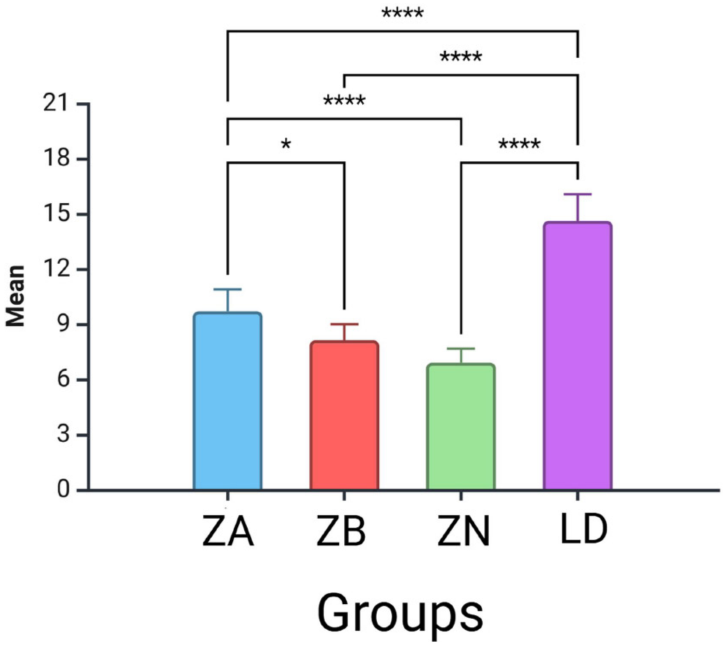

**Fig. (3).** Shear bond strength comparison in a bar graph among the groups.

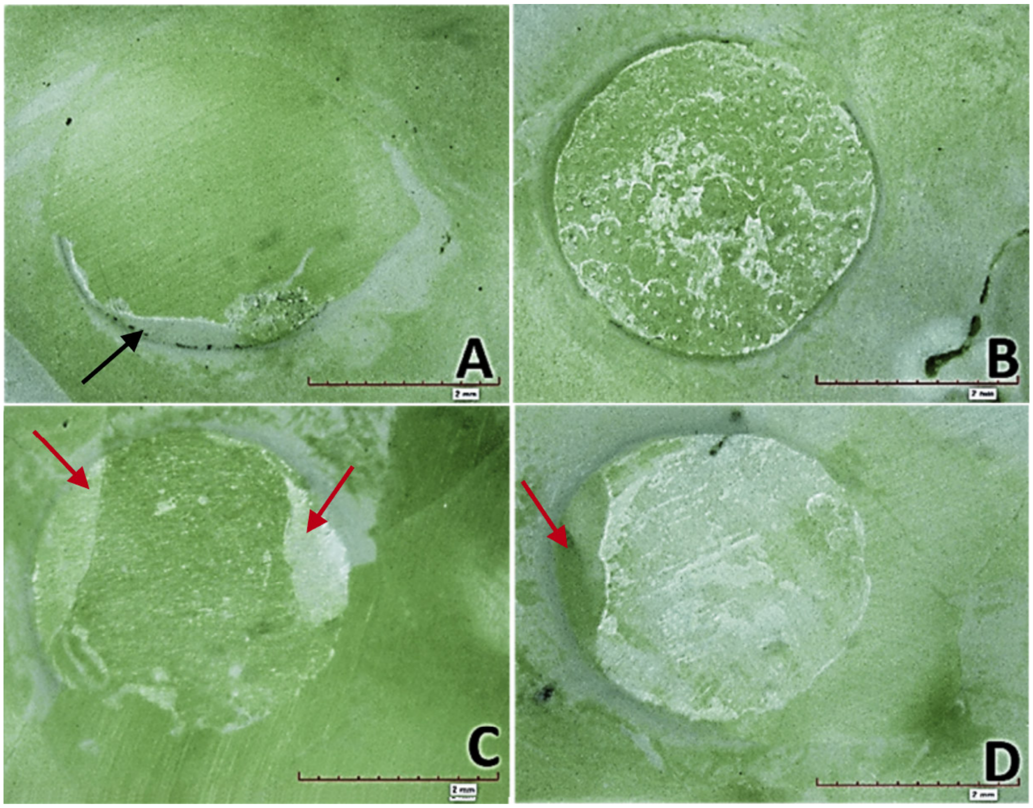

**Fig. (4).** Representative digital microscopic images of dentin after fracture. (**A**) Adhesion fracture at the junction of luting resin and dentin showing resin cement (black arrow), (**B**) Adhesion fracture at the junction of luting resin and ceramic, (**C**) Cohesion fracture showing small craters (red arrow), (**D**) Combination fracture showing irregular craters (red arrow).

## 4. DISCUSSION

This study's distinctive addition is its emphasis on bonding HTZ to occlusal dentin, a substrate that more accurately simulates the intraoral circumstances for onlay restorations. Although several research studies have evaluated the shear bond strength of zirconia with different surface treatments, the majority of them have failed to include both the clinical substrate (occlusal dentin) and thermal aging modeling (thermocycling) in their methodologies. Our present work filled the gap by assessing several extrinsic surface modifications (combination of mechanical + chemical and chemical alone) on HTZ and quantifying their SBS values to occlusal dentin after thermocycling. The results provide significant insight into the long-term performance expectations of HTZ-based indirect restorations.

While our study did not experimentally assess various dentins, previous reports indicate that bond strength differs by dentin region, mostly owing to structural variations such as tubule density and intertubular dentin composition. For instance, elevated bond strengths are observed in superficial dentin, which has markedly lower values owing to enhanced permeability [24]. Furthermore, experiments comparing superficial, middle, and deep dentin have shown that bond strength diminishes with increasing depth (from around 37 MPa in superficial dentin to around 21 MPa in deep dentin) [25].

Considering that occlusal dentin exhibits traits akin to middle dentin, including moderate tubule density and diminished permeability, our SBS findings for Group ZA (9.7 ± 1.18 MPa) align with clinically relevant values identified in these dentin regions.

The bond strength of materials to tooth structure has been evaluated and compared using a variety of testing methods [26, 27]. The SBS test is widely used due to its simplicity, minimal sample preparation, quick procedure, and results [28]. Due to the absence of proper conditioning of dental hard tissue, self-adhesive cementation is simpler to handle and more effective than standard cementation composites [29] and hence used in this study. Thermocycling with temperature variations was performed to simulate the intraoral conditions and to assess the long-term SBS, which is a more reliable outcome with regard to the clinical setting [30].

Lithium disilicate had a strong SBS in this investigation, and none of the tested groups exceeded those values. This is most likely the result of hydrofluoric acid etching, which breaks down the glassy matrix and produces tiny surface defects and pits by selectively reacting with silicon dioxide. Therefore, the micromechanical retention between the luting resin and the glass structure in the control group is improved, resulting in a stronger and longer-lasting bond [31].

In contrast to lithium disilicate ceramics, zirconia needs both mechanical and chemical preparation owing to its polycrystalline structure. In our investigation, the control group (LD) exhibited the greatest SBS, consistent with other findings [1, 32] that affirm the bondability of glass ceramics. The SBS of HTZ in Group ZA (9.7 ± 1.18 MPa) neared clinically accepted levels and was significantly greater than that of untreated zirconia, which, when well processed, may provide performance similar to ceramics while exhibiting enhanced mechanical qualities, making it a formidable option for posterior restorations [33].

As opposed to traditional 3Y-TZP zirconia, HTZ materials (4Y-PSZ and 5Y-PSZ) possess elevated yttria concentrations, which improve translucency but diminish transformation toughening and surface reactivity. Recent comparative research by Silveira *et al*. (2022) assessed the bonding efficacy of resin cement before and after thermocycling across zirconia generations 3Y-TZP, 4Y-PSZ, and 5Y-PSZ. The research indicated that only 4Y-PSZ exhibited stable binding strength with aging, whereas both 3Y and 5Y variants had considerable declines after thermocycling [34]. This corresponds with our results in Group ZA, where the integration of mechanical and chemical pretreatment of HTZ produced much superior SBS upon thermocycling in comparison to untreated HTZ. These data underscore the critical need for dental practitioners to understand the behaviour of zirconia while choosing it in the treatment plan.

The cleansing, activation, and roughening of the luting face of restoration are crucial preconditions for a strong and long-lasting chemical interaction. As far as zirconia is concerned, such chemical interaction can only be accomplished by light mechanical extrinsic modification with a blasting machine rather than chemical conditioning with hydrofluoric acid [29]. The purpose of the air particle abrasion process is to increase the surface area and the number of hydroxyl groups that are accessible for the 10-MDP to bond [35]. The bifunctional MDP monomer's phosphate ester group chemically attaches itself to the hydroxyl group of zirconia forming a Zr-oxyphosphate (Zr-O-P) chemical bond [29, 33].

Prior research demonstrated that a coating of zirconia primer is a relatively pragmatic approach to strengthening the link between zirconia and the luting resin when compared to air particle (50 µm) abrasion using aluminum oxide [36, 37]. Application of a zirconia primer preceded by air abrasion was advantageous in improving the SBS [10, 17]. However, the large size of the air particles produced micro-cracks, which deteriorated the mechanical characteristics of zirconia [10, 38, 39]. The size of particles was chosen as 50 µm in this study to increase the durability of the bond between zirconia and the resin cement. This study found that group ZA had the highest mean SBS, followed by groups ZB and ZN, and the mean difference was statistically significant (*p*-value < 0.05). Therefore, our study confirmed the fact that the combined mechanical (air abrasion) and chemical (10-MDP) extrinsic modifications strengthened the bond of zirconia compared to chemical or mechanical treatment alone. Hence, the null hypothesis was completely rejected.

Zirconia adheres to the tooth in two stages: first, directly to the resin cement, and second, indirectly to the tooth surface through the resin cement [40]. The first junction between the zirconia and the luting resin is the weakest in clinical settings. Fractures that are due to poor bonding at the second junction between the luting resin and dentin are not because of zirconia, as the bonding of the resin cement has not failed with the HTZ but with the dentin. Having such non-significant test results usually represents that the link between the luting resin and the dentin is compromised. The maximum frequency of fracture for the experimental groups was observed at the weakest zirconia-resin cement interface (37.5%), while that of the control was at the resin-dentin interface (90%). However, the frequency of fracture at the weak interface is relatively less in group ZA, meaning that air abrasion has significantly improved the bond strength of HTZ to resin cement. Cohesive and combined fractures of samples represent the possible error in the polymerization of resin cements.

This research used a self-adhesive dual-cure resin cement for the luting technique, which incorporates 10-MDP monomer, recognized for its chemical affinity to zirconia. The cement was immediately applied to the prepared zirconia surface and positioned into occlusal dentin with moderate finger pressure, followed by light curing. This approach was selected for its clinical efficacy and less susceptibility to technique variations.

While the present work is confined to *in vitro* assessment, several longitudinal clinical investigations have shown that zirconia restorations adhered using resin-based luting agents exhibit consistent clinical results. Therefore, while our research provides laboratory data, it substantiates the long-term viability of these materials in clinical use.

### 4.1. Strengths and Limitations

The current study highlights that combined mechanical and chemical treatment has the potential to strengthen the bond of HTZ to occlusal dentin. Moreover, it includes both the resin-zirconia and resin-dentin interfaces in the experiment that closely simulates the intracoronal restoration. One of the limitations is that the results may vary in the oral environment during the clinical trials because of the external factors such as saliva, food, and masticatory forces. Use of thermo-mechanical ageing could have overcome this limitation. Moreover, the thickness of HTZ used in this study is not compatible with that of the actual restorations in the range of 0.5 mm to 1.0 mm.

## CONCLUSION

Within the limitations of this *in vitro* study, extrinsic modifications of highly translucent zirconia (HTZ) significantly affected its bonding performance. In the presence of ceramic primer, air-abraded HTZ showed the highest SBS (9.7 ± 1.18 MPa), which is higher than diamond bur-abraded (8.11 ± 0.87 MPa) and non-mechanically treated HTZ (6.9 ± 0.75 MPa, *p*-value < 0.05). The control group involving lithium disilicate ceramic achieved the highest overall SBS. In the event of failure, the incidence of fracture was relatively less at the junction of air-abraded HTZ and luting resin (40%), indicating bond durability. These results highlight the effectiveness of combining mechanical and chemical pretreatments to optimize bonding with HTZ. Further, we encourage clinical trials to validate these results.

## AUTHORS' CONTRIBUTIONS

The authors confirm contribution to the paper as follows: N.H.A.: Writing – review & editing, Conceptualization, Methodology; T.S.V.: Writing – original draft, Conceptualization, Methodology, Project administration, Investigation; H.F.A.: Formal Analysis, Methodology, Data Curatison; A.A.Y.: Formal Analysis, Methodology; W.H.H.: Formal Analysis, Methodology, Data Curation; M.M.A.M.: Writing – review & editing, Conceptualization, Methodology, Investigation; S.N.B.: Writing – review & editing, Visualization, Supervision, Validation; M.I.K.: Writing – review & editing, Supervision, Visualization, Validation. All authors have read and agreed to the published version of the manuscript.

## LIST OF ABBREVIATIONS

SBS = Shear Bond Strength

HTZ = Highly Translucent Zirconia

MDP = 10-Methacryloyloxydecyl Dihyydrogen Phosphate

## ETHICS APPROVAL AND CONSENT TO PARTICIPATE

This ethical approval (REC-45/05/891& CODJU-2316I dated November 5 and December 10, 2023, respectively) was provided by the Scientific Research Committee of Jazan University, Saudi Arabia.

## HUMAN AND ANIMAL RIGHTS

All procedures performed in studies involving human participants were in accordance with the ethical standards of institutional and/or research committee and with the 1975 Declaration of Helsinki, as revised in 2013.

## CONSENT FOR PUBLICATION

Forty freshly extracted human permanent molars due to periodontal reasons were collected from adults with informed consent between December 11, 2023, and February 5, 2024

## AVAILABILITY OF DATA AND MATERIALS

The data and supportive information are available within the article.

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None.

## CONFLICT OF INTEREST

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