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Effect of Silane Application and Heat Treatment on the Bond Strength of Translucent Zirconia to Glass-Reinforced Core Composite Using Universal Resin Cement: An in vitro Study

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Abstract

Background: Zirconia crowns are gaining popularity due to their excellent optical properties, minimal preparation requirements, high strength, and biocompatibility. Yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) are now widely used as alternatives to PFM crowns, implant abutments, and implants. However, achieving reliable bonding remains a significant challenge.

Aim: To evaluate the effect of silane application, with and without subsequent heat treatment, on the shear bond strength of translucent zirconia to glass-reinforced core composite using universal resin cement.

Materials and Methods: Thirty translucent zirconia blocks (12 mm × 12 mm × 3 mm) were CAD/CAM milled and fully sintered from UPCERA ST zirconia blanks. The specimens were divided into three groups based on surface pretreatment: non-surface treated group (NSTG), silanized group (SG), and silanized with subsequent heat-treated group (SHTG). Thirty composite resin cylinders (2 mm in diameter and 3 mm in height) were fabricated using polyethylene tubes and light-cured according to the manufacturer's instructions. A universal resin cement was used to lute the composite cylinders to the zirconia blocks, followed by light curing as per the manufacturer's guidelines. Each specimen was then embedded in polymethyl methacrylate (PMMA) resin using polyethylene molds. Shear bond strength was measured using a universal testing machine. Data were subjected to the Kruskal-Wallis test applied for overall comparisons and the Mann-Whitney U test for pairwise group comparisons. A p-value of <0.05 was considered statistically significant.

Results: Shear bond strength was significantly influenced by silanization & heat treatment. Group 3 (SHTG) had the highest shear bond strength (4.13 ± 0.91MPa), followed by Group 2 (SG). Group 1 (NSTG) showed the least bond strength.

Conclusion: Silane application at a 1% concentration significantly enhanced the bond strength between composite resin and zirconia. Subsequent heat treatment of the silanized zirconia surfaces resulted in an additional increase in bond strength.

Keywords: CAD-CAM, Composite, Resin Cements, Shear Bond Strength, Silanes, Zirconia.

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1. Introduction

The restoration of anterior teeth demands the careful selection of materials that balance esthetics, strength, and biocompatibility. Traditionally, metal-ceramic restorations were considered the gold standard due to their durability and favourable biological response. However, they present notable limitations such as extensive tooth preparation, inherent opacity, technique sensitivity, and limited translucency [1,2]. These drawbacks have driven a shift towards all-ceramic systems, which offer superior optical properties and esthetics. Yet, their increased translucency often comes at the cost of mechanical strength and fracture resistance [3].

Zirconia (zirconium dioxide), recognized as a "smart ceramic," has emerged as a material of choice for dental restorations, offering high strength, minimal tooth reduction, excellent biocompatibility, and favourable optical properties. Isolated in 1824 and introduced for orthopaedic applications in 1969 [4], zirconia exists in monoclinic, tetragonal, and cubic phases depending on temperature. Dental zirconia is yttria-stabilized and is available in various formulations such as 3Y-TZP (high strength, for posterior use), 4Y-TZP (balanced strength and translucency), and 5Y-TZP (high translucency, suitable for anterior regions). Its resistance to moisture and chemical stability further enhances its clinical applicability [3].

When restoring anterior teeth with compromised crown structure, the use of post and core build-ups is frequently required. These structures compensate for the loss of natural tooth strength and provide foundational support to restorations [5]. Materials historically used for core build-ups include amalgam, conventional composites, glass ionomer (GI) cement, and silver-reinforced GI (SRGI) [6,7]. However, the limitations of SRGI and GI-based materials have led to the development of improved alternatives [8]. One such material is ParaCore (Coltene), a dual-cure, thixotropic, glass-reinforced composite that offers both mechanical strength and favorable esthetics, making it a suitable core build-up material. The long-term success of zirconia restorations relies heavily on both their mechanical properties and the effectiveness of the cementation process. Conventional luting agents such as zinc polycarboxylate and GI cements have largely been replaced by resin luting cements, which offer enhanced adhesion, esthetics, and resistance to solubility [9]. Bonding zirconia remains a challenge due to its lack of a glassy phase, which renders hydrofluoric acid etching ineffective. While techniques such as airborne-particle abrasion, silane application, and functional monomers have been explored, no universally accepted protocol has been established [10]. Adhesive monomers like 10-MDP, found in cements such as SpeedCEM Plus (Ivoclar Vivadent), have shown promise in bonding to zirconia [10].

Silanes, silicon-based organic compounds with bifunctional reactivity, have demonstrated efficacy in improving resin-zirconia adhesion. A 1% silane concentration is reported to be optimal for bonding to zirconia ceramics [11]. Moreover, research also found that heat treatment of silane-pretreated zirconia at 150°C for 60 minutes significantly enhanced bond strength compared to silane application alone [11]. Considering the inconsistencies in zirconia bonding protocols and the absence of a standardized approach, this study aims to evaluate the effect of silane application alone and in combination with subsequent heat treatment on the shear bond strength of translucent zirconia luted to a glass-reinforced core composite using universal resin cement. The null hypothesis states that there will be no significant difference in bond strength among the different surface treatments

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2. Materials and methods

2.1 Sample preparation

Thirty zirconia blocks (12 mm × 12 mm × 3 mm) were CAD/CAM milled using ARUM 5X-300D and fully sintered in a high-temperature furnace (TABEO-1/M/ZIRKON-100; MIHM-VOGT, Germany) from a zirconia blank (UPCERA ST, Shenzhen, China). All specimens were stored at 37 °C in 100% relative humidity for 24 hours, followed by wet grinding with 1200-grit silicon carbide paper to standardize the surface.

2.2 Preparation of composite cylinders

Thirty polyethylene tubes (2 X 3 mm) were used as molds to fabricate cylindrical composite resin specimens using a glass-reinforced core build-up composite (ParaCore; Coltene, Altstatten, Switzerland). The composite was placed into the molds and light-cured for 20 seconds using an LED curing unit (Woodpecker, Guilin, Guangxi, China), following the manufacturer’s instructions.

2.3 Bonding procedure

Resin luting cement (SpeedCEM Plus, Ivoclar, Schaan, Liechtenstein) was applied to the zirconia surface and bonded to the composite resin cylinders. To simulate finger pressure, a constant load of 50 N was applied using a standardized weight for 5 minutes. The specimens were then light-cured for 20 seconds according to the manufacturer's recommendations, and any excess luting cement was carefully removed.

The thirty zirconia specimens were randomly divided into three groups (n = 10 per group) based on the surface pre-treatment protocol employed:

Group 1 – Non-Surface Treated (NST): Specimens in this group received no surface treatment prior to bonding and served as the control.

Group 2 – Silane Treated (S): Zirconia surfaces were first etched with 9% hydrofluoric acid (Porcelain Etch, Ultradent, USA) for 90 seconds. The specimens were then rinsed thoroughly with distilled water and gently air-dried. A 1% silane coupling agent (Ultradent, USA) was applied using an applicator tip and allowed to sit for 60 seconds. Excess silane was removed by gentle air drying.

Group 3 – Silane with Heat Treatment (SH): These specimens underwent the same silane pretreatment as Group 2. Following silanization, the specimens were heat-treated at 150 °C for 60 minutes in a ceramic furnace (Programat P310; Ivoclar, USA) to enhance chemical bonding.

All specimens were embedded in polymethyl methacrylate (PMMA) resin cylinders (DPI RR Cold Cure, India) with the bonded interface exposed, ensuring accurate alignment for shear bond strength testing.

2.4 Evaluation of shear bond strength

The specimens were securely positioned in a custom-fabricated jig and mounted on a universal testing machine (Instron 3360, USA). A load was applied at a crosshead speed of 1 mm/min, directed at the interface between the zirconia surface and the bonded composite cylinder until bond failure occurred (Figure 1). The maximum load at failure was recorded in Newtons (N), and the shear bond strength (MPa) was calculated using the following formula:

Shear bond strength (Mpa) = F / π D H

Where 'F' is the maximum force (newtons) applied for bond failure in each sample, 'π is a constant with a value of 3.14, D is the diameter and 'H' is height of the composite cylinder (mm).

2.5 Statistical analysis

Data analysis was done using the Statistical Package for Social Sciences (SPSS version 26.0, IBM Corporation, USA). Overall comparisons of shear bond strength were analyzed using the Kruskal-Wallis test. Pair-wise comparisons were done using the Mann-Whitney U test. The level of significance was set at p less than 0.05.

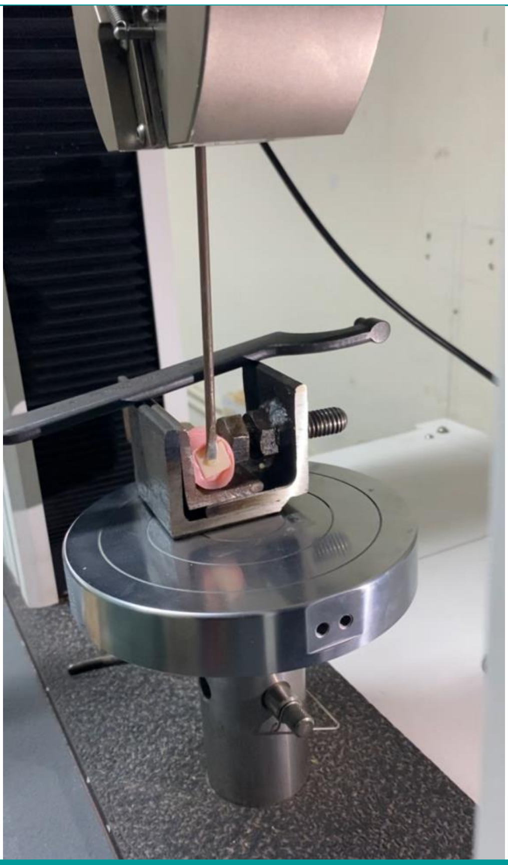

Figure 1. Specimen is subjecting to shear bond strength test on the universal testing machine.

3. Results

The mean shear bond strength (MPa) of specimens from different groups is presented in Table 1. In this study, the shear bond strength of zirconia luted to a core composite with a self-adhesive resin cement was significantly affected by silanization. The silane and the heat-treated group showed more SBS (4.13 ± 0.91 MPa) followed by the silane-treated group (2.52 ± 0.83 MPa). The no-surface-treated group (Group 1) showed the least shear bond strength (0.88 ± 0.27MPa) among the groups. The Kruskal-Wallis analysis displayed a significant difference ($p<0.001$) in SBS among the three groups (Table 1). On pair-wise comparison, significant differences were observed between all the groups (Table 2).

Table 1. Comparison of mean shear bond strength (MPa)

Groups	Mean ± SD#	Kruskal-Wallis	p-Value
Group 1 (Non-surface treated)	0.88 ± 0.27	23.110	<0.001*
Group 2 (Silane-treated)	2.52 ± 0.83		
Group 3 (Silane heat-treated)	4.13 ± 0.91		

#Standard Deviation, and *Statistically significant.

Table 2. Pair-wise comparison of shear bond strength between the groups.

Groups		Significance
Group 1	Group 2	<0.001*
	Group 3	<0.001*
Group 2	Group 3	0.001*

*Statistically significant.

4. Discussion

Zirconia has rapidly gained prominence due to its esthetics and better mechanical properties, such as high strength, fracture toughness, and durability against wear. Its non-magnetic nature, electrical insulating capabilities, low thermal conductivity, and resistance to corrosion in both acidic and alkaline environments make it highly versatile. Furthermore, its modulus of elasticity, which closely resembles that of steel, along with a thermal expansion coefficient similar to iron, underscores its suitability for use in restorative dentistry [12,13].

However, the bonding ability of zirconia is a questionable reason to replace it with other restorative materials. Zirconia is a chemically inert substance; traditional adhesive chemistry is ineffective on zirconia surface [14]. Also, acid etchants such as hydrofluoric acid do not selectively etch the surface for simple micromechanical attachment. Bonding of any two dissimilar materials requires the substrate and adherent to have a clean surface for maximum bond strength. Removal of impurities from the surface results in an increase in surface energy and wettability of the surfaces to be bonded. This process of removing impurities and increasing the surface area is known as surface conditioning [15].

In the present study, the non-treated group exhibited lower shear bond strength (SBS) compared to the silane-treated and the silane-plus-heat-treated groups. Among these groups, the specimens that underwent silane treatment exhibited a higher SBS than the untreated specimens. Notably, specimens that underwent silane treatment followed by heat treatment demonstrated the highest SBS among all groups.

Previous studies have shown that silane-based surface conditioning is one of the effective methods for enhancing adhesion to zirconia, which aligns with the findings of the current study [16,17]. Chemically, silanes are organic compounds containing silicon (Si) atoms, structurally similar to orthoesters, and are known for their bifunctional reactivity [17,18]. One end of the silane molecule contains an organic functional group (typically a vinyl group $-CH=CH_2$) capable of polymerizing with the organic matrix, such as methacrylate. Organosilanes typically possess one organic substituent and three hydrolyzable substituents. In most surface treatments, the alkoxy groups of trialkoxysilanes undergo hydrolysis to form silanol groups. The silanization process involves four stages: hydrolysis of the labile groups, condensation into oligomers, hydrogen bonding of the oligomers with hydroxyl (-OH) groups on the substrate, and finally, through drying or curing, the formation of a covalent bond with the substrate accompanied by water elimination [4,19].

Previous research has also reported that silane treatment alone results in weaker adhesion compared to silane application followed by heat treatment. Research reported that the heating zirconia pre-treated with silane at 150°C for 60 minutes significantly increased the mean bond strength—by approximately 45%—compared to silane treatment alone [20]. These findings are consistent with the results of the present study, where the silane and heat-treated group exhibited a higher mean bond strength (4.13 ± 0.91 MPa) than the silane-only group (2.52 ± 0.83 MPa). This represents an approximate 64% increase in bond strength with the addition of heat treatment following silane application.

Heat treatment of the silane solution appears to enhance bond strength, potentially due to the condensation of free silanol groups into siloxane oligomers [21]. This process also likely facilitates the removal of hydrophilic components from the silane film, promoting the completion of the condensation reaction between silanol groups at the zirconia/resin interface. As a result, this may support better adhesion of the “hydrophobic” resin to the zirconia surface by forming a uniform and stable interfacial silane nanolayer [22].

Consequently, the application of silane coupling agents followed by heat treatment may fulfil clinical requirements for bonding zirconia restorations, thereby contributing to the long-term success and durability of dental restorations [23]. The findings of this study suggest that the combination of silane application with heat treatment can significantly improve zirconia-resin bonding.

The type of resin cement plays a critical role in determining the quality and durability of the bond to ceramic substrates. Self-adhesive resin cements offer ease of application and reduced technique sensitivity but may exhibit lower bond strength compared to adhesive resin cements that require separate etching and priming steps [24]. The presence of functional monomers such as 10-MDP in resin cements has been shown to enhance chemical bonding, particularly to zirconia, by forming stable bonds with metal oxides on the ceramic surface. Studies have shown that the interaction between the functional components of the resin cement and the surface treatment of the ceramic significantly affects the bond performance and longevity of restorations in clinical settings [25-27]. However, in the present study, only one resin cement such as universal self-adhesive resin cement, was used.

The present study demonstrated that the application of silane followed by heat treatment significantly increased the shear bond strength of zirconia ceramics. However, as this was an in vitro investigation, it does not fully replicate the complexities of the oral environment, which is dynamic and subjected to various masticatory forces and thermal fluctuations. Additionally, the study was limited to the use of a single resin luting agent. Therefore, future research should consider in vivo conditions and explore the use of different types of luting agents to better validate and generalize the findings.

5. Conclusion

In this study, 1% silane demonstrated improved bonding between zirconia and composite resin. Furthermore, the subsequent heat treatment of zirconia pre-treated with silane significantly enhanced the bond strength—showing an approximate 65% increase compared to the silane-only group (Group 2) and nearly a 90% increase compared to the non-silanized group (Group 1). However, further research is necessary to address the challenges associated with zirconia bonding and to improve the long-term durability of zirconia restorations under clinical conditions.

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