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The Influence of Tooth Primer and Zirconia Cleaners on the Shear Bond Strength of Saliva-Contaminated Zirconia Bonded with Self-Adhesive Resin Cement

Vorrawatn Pornatitanakul [1], Awiruth Klaisiri [2,*], Tool Sriamporn [3,*], Somporn Swasdison [3] and Niyom Thamrongananskul [1]

1 Department of Prosthodontics, Faculty of Dentistry, Chulalongkorn University, Bangkok 10330, Thailand; 6470026332@student.chula.ac.th (V.P.); niyom.t@chula.ac.th (N.T.)
2 Division of Restorative Dentistry, Faculty of Dentistry, Thammasat University, Pathum Thani 12120, Thailand
3 College of Dental Medicine, Rangsit University, Pathum Thani 12000, Thailand; somporn.s@rsu.ac.th
* Correspondence: dentton@tu.ac.th (A.K.); tool.s@rsu.ac.th (T.S.); Tel.: +66-2986-9206 (A.K.); +66-2997-2200 (T.S.)

Abstract: This study investigated the effectiveness of Tooth Primer and cleaning agents in removing saliva contamination from zirconia as assessed using shear bond strength (SBS). A total of 175 rectangular specimens, 10 mm in diameter and 6 mm in thickness, were randomly divided into seven groups (n = 25 each): group 1, no saliva contamination (control); group 2, saliva-contaminated and not rinsed; group 3, saliva-contaminated and rinsed; group 4, saliva-contaminated, Ivoclean-treated, and rinsed; group 5, saliva-contaminated, Katana Cleaner-treated, and rinsed; group 6, saliva-contaminated, Tooth Primer-treated, and rinsed; and group 7, saliva-contaminated, Tooth Primer-treated, and not rinsed. All zirconia specimens from groups 1–7 were bonded to composite rods with Panavia SA Luting Multi cement. The bonded specimens were subjected to the SBS test using a universal testing machine. To assess the type of failure, the debonded surface was evaluated using a stereomicroscope. The SBS data were analyzed using one-way ANOVA with Tukey's post-test. The SBS values of groups 2 (0.90 $\pm$ 0.20 MPa) and 3 (1.35 $\pm$ 0.43 MPa) were significantly lower compared with the other groups (p = 0.00). The SBS value of zirconia decontamination using Ivoclean in group 4 (18.51 $\pm$ 3.01 MPa) was significantly lower than that of the control group (22.24 $\pm$ 2.37 MPa) (p = 0.00). However, the SBS values of groups 5 (20.92 $\pm$ 2.63 MPa), 6 (21.43 $\pm$ 2.81 MPa), and 7 (20.87 $\pm$ 2.35 MPa) did not significantly differ compared with the control group (22.24 $\pm$ 2.37 MPa) (p = 0.369, p = 0.861, p = 0.327, respectively). Moreover, SBS values did not significantly differ among groups 5 to 7 (p = 0.984, p = 1.00, p = 0.976, respectively). Regarding failure mode, groups 2 and 3 exclusively experienced adhesive failures. Groups 1, 4, 5, 6, and 7 exhibited adhesive and mixed failures. In conclusion, Ivoclean, Katana Cleaner, and Tooth Primer effectively mitigated the adverse effects of saliva contamination on the resin cement–zirconia interface. Furthermore, Tooth Primer can be used as a cleaner for saliva-contaminated zirconia surfaces, with or without rinsing.

Keywords: bond strength; resin cement; saliva contamination; zirconia cleaner

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1. Introduction

Zirconia, or zirconium dioxide (ZrO_2), has gained popularity in restorative dentistry due to its combination of esthetics and strength. Zirconia is a glass-free type of ceramic, and zirconia atoms are packed into a regular crystalline arrangement that has high mechanical strength [1]. Dental zirconia can be divided into four main generations, based on its chemical structure. The first generation is also known as conventional 3Y-TZP (3 mol% yttria-stabilized tetragonal zirconia polycrystal). Given its high degree of opacity, it is used as a framework material and veneered with traditional ceramics to create an esthetically pleasing effect. The second generation of zirconia features high translucency, achieved by reducing the amount of alumina additive to 0.05% by weight, while maintaining the yttrium oxide stabilizer at 3% by mol. The third generation of highly transparent zirconia is produced by increasing the amount of cubic phase structures through raising the yttrium oxide content to more than 4% by mol. In this generation, the translucency of zirconia is increased, but its strength is decreased. To balance these properties, manufacturers lowered the yttrium oxide content to 4%, yielding the fourth generation of zirconia [2].

At present, dental zirconia achieves both mechanical strength and esthetics, but the adhesion between zirconia and resin cement is weak. Several techniques, such as chemical and mechanical adhesion approaches, are available to enhance the bonding strength of zirconia. A typical method for improving surface roughness and providing mechanical retention is air abrasion [1].

Under carefully controlled in vitro conditions, a good ceramic–resin cement adhesion may be achieved, but it may not hold up as well in clinical settings, leading to a significant reduction in bond strength. When attempting a restoration in the mouth, it is difficult to avoid contaminating the restoration's inner surface with materials such as silicone indicator, blood, and saliva [3]. Saliva contamination is a common cause of reduced resin cement bond strength [3–6]. Saliva consists of proteins, enzymes, blood cells, bacteria, and various phosphate forms, such as phospholipids, in a water solution [7]. It interferes with bonding of the restoration to the tooth. Saliva may remain as a thin layer on the ceramic surface, blocking micromechanical and durable chemical bonds [8]. Salivary phosphates irreversibly bind to zirconia and the tooth surface, which makes removing them difficult. Consequently, saliva contamination can lead to issues when restorations contaminated with saliva are cemented, leading to adhesive failure. Numerous investigations have shown that saliva contamination interferes with the formation of stable bonds [4,9–11].

The contamination must be removed from the restoration's inner surface prior to cementation. It is important to make sure that the inner surfaces of the restorations are properly cleaned and decontaminated after the intraoral try-in in order to obtain a sufficiently durable adhesion between the tooth structure and the restorations. The cleaning methodologies should involve chemical or mechanical methods or a mix of both [12,13]. The mechanical technique involves sandblasting with aluminum oxide particles. The chemical techniques include cleaning solvents and acid treatment of the surface with substances such as phosphoric acid, acetone, alcohol, and acidulated phosphate fluoride. Prior research has shown that although each cleaning technique has benefits, none achieve sufficient bonding ability [12,13].

Several experiments have used extraoral zirconia cleaning solutions (Ivoclean and Katana Cleaner) to clean the saliva-contaminated zirconia surface prior to cementation [12,13]. These cleaning products are liquids composed of monomer (10-MDP), other ingredients, and no initiator and thus are unable to polymerize. Studies have demonstrated that applying these solutions and subsequently washing with water can increase the bondability of the resin cement material and zirconia. Nevertheless, there is limited information on how to decontaminate saliva-contaminated zirconia using a primer containing 10-MDP—a component commonly recommended by manufacturers for intraoral use to modify tooth structure. It is therefore of particular interest to use the Panavia V5 Tooth primer to clean saliva-contaminated zirconia surfaces both with and without rinsing to enhance the adhesion between zirconia and resin cement.

This research aimed to assess the efficiency of various cleaning agents in removing saliva contamination from zirconia, as evaluated based on the shear bond strength (SBS) of zirconia and self-adhesive resin cement. The null hypotheses were as follows: (1) saliva contamination would not significantly influence the bond strength of zirconia and self-adhesive resin cement; (2) the different decontamination protocols would not significantly influence the bond strength between saliva-contaminated zirconia and self-adhesive resin cement.

2. Materials and Methods

The experimental part of this study received ethical approval from the Human Research Ethics Committee of the Faculty of Dentistry, Chulalongkorn University (HREC-DCU), under the investigation identification number 2023-007.

2.1. Specimen Preparation

Blocks of pre-sintered 2nd generation zirconia (3 mol %-Cercon HT; Dentsply Sirona, Bensheim, Germany) were milled (Ceramil® mikro 5; AMANN GIRRBACH, Maede, Austria) to prepare 175 rectangular specimens ($10 \times 10 \times 6$ mm^3). After sintering (Ceramil® therm3; AMANN GIRRBACH, Maede, Austria), each zirconia specimen was inspected with a stereomicroscope at 40× magnification (SZ61, Olympus Corporation, Tokyo, Japan) to determine surface errors. Six defective specimens were rejected and replaced with six new specimens. Each specimen was placed in a 20 mm diameter by 15 mm high poly(vinyl chloride) pipe filled with epoxy resin prepared in accordance with the manufacturer's instructions (epoxy resins and hardeners, Shandong Deyuan Epoxy Resin Co., Ltd., Tai'an, China). After the epoxy resin had fully set, the specimens were polished using a polishing machine (NANO 2000, Pace Technologies, Tucson, AZ, USA) with 400-, 800-, and 1200-grit silicon carbide sheets at 250 rpm under a pressure of 50 N pressure using running water for 60 s. Subsequently, the specimens were sandblasted with 50 µm aluminum oxide particles (Korox; Bego, Bremen, Germany) using a sandblasting machine (Renfert Basic Quattro, Hamburg, Germany) for 15 s at a distance of 1 cm and a pressure of 2.5 bars. The specimens were then rinsed in an ultrasonic cleaning device (Ultrasonic cleaner VI, Yoshida Dental Trade Distribution Co., Tokyo, Japan) with 99% isopropanol for 10 min to remove any debris. The specimens were thoroughly dried using oil-free air spray. All specimen were re-examined under a stereomicroscope at 40× magnification.

The sample size was analyzed using the G*power program (Version 3.1.9.7, Heinrich-Heine Dusseldorf University, Dusseldorf, Germany). Based on the pilot investigation (n = 6 per group), 161 samples were required (effect size f = 0.37, α err prob = 0.05, power ($1-\beta$ err prob) = 0.95, number of groups = 7). Therefore, each group should contain a minimum of twenty-three samples. In this study, 25 samples were included per group to account for a 10% error rate. The specimens were then randomly assigned into 7 groups. Each group underwent different zirconia surface cleaning based on the following protocols:

Group 1:

- No saliva contamination;
- Panavia SA Luting Multi cement.

Group 2:

- Saliva contamination;
- Panavia SA Luting Multi cement.

Group 3:

- Saliva contamination;
- Rubbed with distilled water;
- Rinsed with distilled water;
- Panavia SA Luting Multi cement.

Group 4:

- Saliva contamination;
- Rubbed with Ivoclean;
- Rinsed with distilled water;
- Panavia SA Luting Multi cement.

Group 5:

- Saliva contamination;
- Rubbed with Katana Cleaner;
- Rinsed with distilled water;
- Panavia SA Luting Multi cement.

Group 6:

- Saliva contamination;
- Rubbed with Tooth Primer;
- Rinsed with distilled water;
- Panavia SA Luting Multi cement.

Group 7:

- Saliva contamination;
- Rubbed with Tooth Primer;
- Panavia SA Luting Multi cement.

Table 1 lists the chemical compositions of the materials employed in this investigation. Additionally, Figure 1 displays the experiment design flowchart, and Figure 2 depicts the stage of specimen preparation prior to testing.

Table 1. Several types of materials used in the present investigation.

Material Name	Compositions
Zirconia Cercon HT (Dentsply Sirona, Bensheim, Germany) Lot No.: 18032881	ZrO_2 + H_fO_2 + Y_2O_3: ≥99%wt, Y_2O_3: 9.15–9.55%wt, H_fO_2: ≤5%wt, Al_2O_3: ≤0.5%wt, other oxides: ≤1%wt
Filtek Z350 XT (3M ESPE, St. Paul, MN, USA) Lot No: NF40079	Matrix: UDMA, TEGDMA, Bis-GMA, Bis-EMA Filler: SiO_2 nanofiller, ZrO_2 nanofiller, ZrO_2/SiO_2 nanocluster
Katana Cleaner (Kuraray America Inc., New York, NY, USA) Lot No.: C90023	Water, 10-MDP, poly(ethylene glycol), triethanolamine, stabilizer, dyes
Tooth Primer (Panavia V5) (Kuraray Noritake Dental Inc, Kurashiki, Okayama, Japan) Lot No.: BB0123	2-HEMA, 10-MDP, hydrophilic aliphatic dimethacrylate, accelerators, water
Panavia SA Luting Multi cement (Kuraray Noritake Dental Inc, Kurashiki, Okayama, Japan) Lot No.: BR0196	Paste A: Bis-GMA, 10-MDP, TEGDMA, 2-HEMA, hydrophobic aromatic dimethacrylate, silanated colloidal silica, silanated barium glass filler, dl-camphorquinone, catalysts, peroxide, pigments Paste B: Hydrophobic aromaticdimethacrylate, hydrophobic aliphatic dimethacrylate, surface-treated sodium fluoride, silanated barium glass filler, accelerators, pigments
Ivoclean (Ivoclar vivadent AG, Schaan, Liechtenstein) Lot No.: Z0304K	10–15% ZrO_2, 65–80% water, 2.5–10% poly(ethylene glycol), <2.5% NaOH

Abbreviations: UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; BisGMA, bisphenol A-glycidyl methacrylate; BisEMA, ethoxylated bisphenol A-diglycidyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 2-HEMA, 2-hydroxyethyl methacrylate.

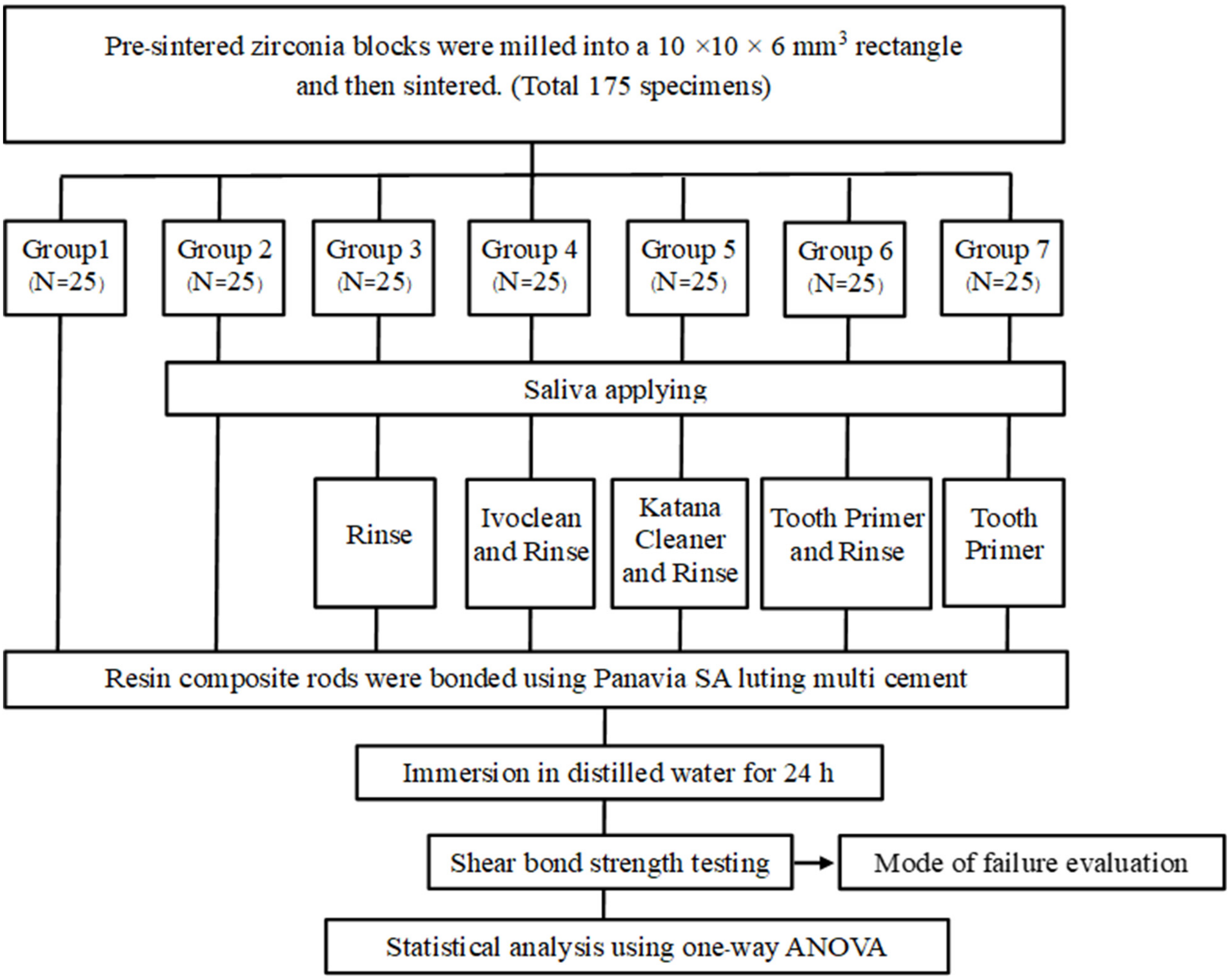

Figure 1. A flowchart of the experimental procedure used to analyze the shear bond strength and identify the type of failure.

2.2. Resin Composite Rod Preparation

Resin composite rods were fabricated using a poly(vinyl siloxane) mold with a hole size of 3 mm in diameter and 4 mm in thickness. The resin composite (Filtek Z350 XT, 3M ESPE, St. Paul, MN, USA) was inserted in the hole and light-activated for 40 s using an LED light-curing instrument (Elipar S10, 3M ESPE, St. Paul, MN, USA). After removing the resin composite rod from the mold, it was light-cured for an additional 40 s. Abrasives in the form of 50 µm aluminum oxide particles were used to sand the end of the resin composite rod under 2.5 bars of pressure and 1 cm away. Afterward, the specimens were washed in 99% isopropanol in an ultrasonic cleaning device for 3 min to remove debris. The specimens were dried using oil-free air spray and subsequently stored in a closed, clean plastic container.

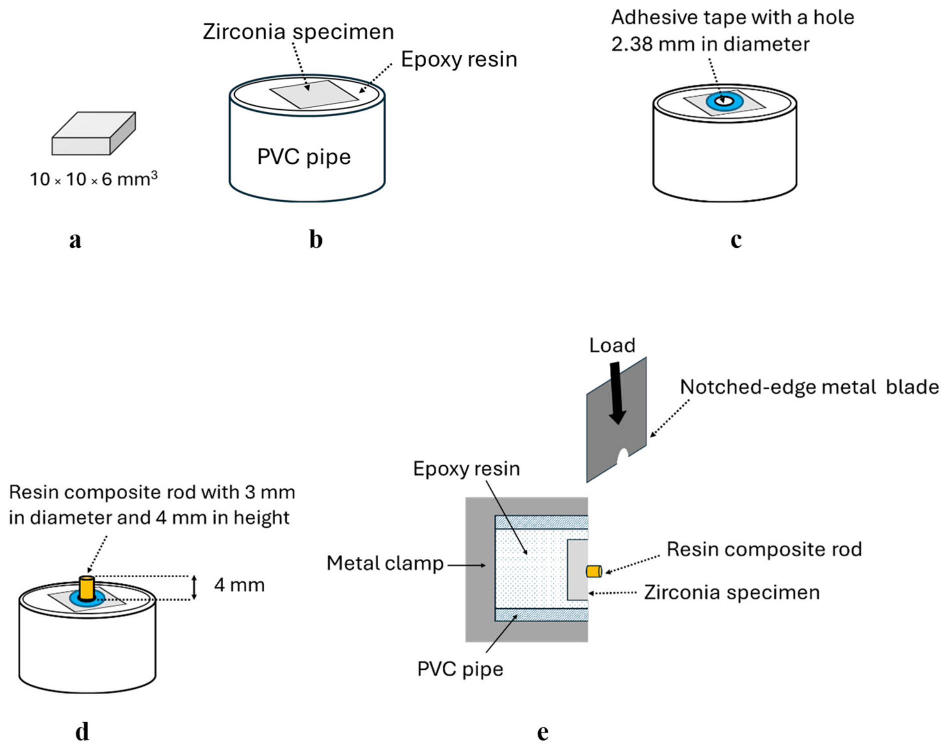

Figure 2. SBS test setup: (**a**), the dimensions of the sintered zirconia specimen; (**b**), the specimen was embedded in PVC pipe; (**c**), adhesive tape was firmly affixed to the specimen surface; (**d**), resin composite rod was bonded to the zirconia surface using resin cement; (**e**), the testing method was performed.

2.3. Saliva Collection

Unstimulated saliva from a single healthy person was collected over the same duration. To maintain standardization, the participant refrained from food, beverages, or other oral intakes for at least one hour before saliva collection [11]. The saliva was collected by pooling it in the mouth and then gently expelling it into a collection plastic tube. The sample was used within 15 min of collection.

2.4. Bonding Area Preparation

Single-sided adhesive tape (3M-ESPE St. Paul, MN, USA), 50 µm thick (ISO 4049:2019) [14], with a circular hole of 2.38 mm in diameter (ISO 29022:2013) [15], was firmly attached to the center of the specimens to control the cement film thickness and bonding area.

2.5. Saliva Contamination and Surface Modification Procedure

For groups 2–7, a micropipette was used to gather 50 microliters of saliva and apply it to the circular hole on the zirconia surface. The saliva was then spread with a disposable microbrush, gently dried for 20 s with oil-free air spray using a triple syringe from a mobile dental unit (under 2.5 bars of pressure at a distance of 1 cm), and left untouched for 120 s.

For group 3, after saliva contamination, fifty microliters of distilled water was collected using a micropipette and applied to the specimen. The saliva was spread on the specimen for 10 s with a microbrush, then washed with distilled water for 1 min. Subsequently, the specimen was dried completely for 20 s with oil-free air spray using a triple syringe.

For groups 4–7, after saliva contamination, fifty microliters of each primer or cleaning solution was collected by micropipette. The solution was applied to the specimen and spread for 10 s using a microbrush. Only groups 4–6 were washed with distilled water for 60 s and thoroughly dried for 20 s using oil-free air spray with a triple syringe.

2.6. Cementation

The Panavia SA Luting Multi cement (Kuraray Noritake Dental Inc., Aichi, Japan) was applied to a circular hole that serves as the zirconia's cementation surface. The resin composite rod was then placed over the hole and pressed using a durometer (ASTM D 2240 Type A, DPTC Instrument, Bonny Doon, CA, USA) with a constant weight of 1 kg. The constant weight was maintained during the cementation procedure. Residual cement was eliminated with a microbrush and light-cured for 20 s on all sides, with the light source placed as close as possible to the cement. The adhesive tape was carefully detached from the bonded specimens. All specimens were stored in distilled water within an environment chamber (Contherm 1200, Contherm, Lower Hutt, New Zealand) at 37 °C for 1 day, followed by immersion thermocycling for 5000 cycles at temperatures ranging from 5 to 55 °C with a 30 s dwell time and a 5 s transfer time.

2.7. Shear Bond Strength Test

The specimen was placed in the universal testing apparatus (EZ-S; Shimadzu Corporation, Kyoto, Japan) in a position that provided a shear force parallel to the zirconia and resin composite's interface. After careful placement of the notched-edge blade over the resin composite rod, the force was delivered at a steady crosshead speed of 1 mm/min until the sample broke. The maximum force (N) applied until failure was recorded. The SBS (measured in MPa) was computed using the bonded area (between resin cement and zirconia) and the maximum force. All procedures were performed under ambient room conditions. Following this, every specimen was examined to determine the failure modes.

2.8. Determining Failure Mode

A stereomicroscope (SZ61, Olympus Corporation, Tokyo, Japan) at 40× magnification was used to examine the broken parts. The failure type was assigned into three categories after fracture, as modified from Matinlinna et al. [16].

(a) Adhesive failure: Less than 40% of the self-adhesive resin cement is detectable on the zirconia surface.

(b) Cohesive failure: A minimum of 60% of the resin cement is visible within the zirconia surface, indicating failure of the self-adhesive resin cement.

(c) Mixed failure: Between 40% and 60% of the resin cement is observed on the zirconia surface, indicating a mixed failure including both cohesive and adhesive failures.

Using ImageJ software (http://imagej.net/ij/download.html (accessed on 14 June 2024)), the area of self-adhesive resin cement on the bonded zirconia surface was measured twice by two independent operators. The mean of the measurement area was then divided by the total bonded surface area, and the result was multiplied by 100. This calculation provided a percentage that represents the extent of the self-adhesive resin cement coverage on the zirconia surface.

2.9. Statistical Analysis

IBM SPSS. V29.0.1 (SPSS Inc., Chicago, IL, USA) was used to evaluate the quantitative data from the seven separate groups at a 95% confidence level. Normality was assessed using the Kolmogorov–Smirnov test at a significance level of 0.05. The equality of variation was examined using Levene's test. The data had equal variance and a normal distribution according to the findings. Consequently, the data were assessed using one-way ANOVA, and the differences between groups were then ascertained using a post hoc Tukey test ($p < 0.05$).

3. Results

The initial phase of the statistical analysis included testing a first-order model for the two research variables: contamination and cleaner. All pairwise comparisons were significant, suggesting that each variable independently influenced the bond strength outcome. Second-order implementation analysis further showed a significant interaction between contamination and cleaner, suggesting that the type of cleaner used affected the final SBS (Table 2).

Table 2. Shear bond strength (mean ± standard deviation (MPa)) and failure mode percentages (%).

Group	Mean SBS ± SD	Failure Mode Percentages (%)		
		Adhesive	Mixed	Cohesive
1. No saliva contamination, no additional cleaning (control)	22.24 ± 2.37 ^{A}	22 (88%)	3 (12%)	0 (0%)
2. Saliva without rinsing	0.90 ± 0.20 ^{B}	25 (100%)	0 (0%)	0 (0%)
3. Saliva, rubbed with distilled water, rinsed with distilled water	1.35 ± 0.43 ^{B}	25 (100%)	0 (0%)	0 (0%)
4. Saliva, rubbed with Ivoclean, rinsed with distilled water	18.51 ± 3.01 ^{C}	22 (88%)	3 (12%)	0 (0%)
5. Saliva, rubbed with Katana Cleaner, rinsed with distilled water	20.92 ± 2.63 ^{AC}	21 (84%)	4 (16%)	0 (0%)
6. Saliva, rubbed with Tooth Primer, rinsed with distilled water	21.43 ± 2.81 ^{AC}	22 (88%)	3 (12%)	0 (0%)
7. Saliva, rubbed with Tooth Primer without rinsing	20.87 ± 2.35 ^{AC}	23 (92%)	2 (8%)	0 (0%)

The different uppercase letters represent significant differences at $p < 0.05$.

3.1. SBS Test

The SBS value for group 2 did not significantly differ from that of group 3 ($p = 0.992$), and the SBS values of groups 2 and 3 were lower than those of the other groups ($p = 0.00$). The SBS value of zirconia decontamination using Ivoclean (group 4) was significantly less than that of uncontaminated group ($p = 0.00$). While the SBS values of zirconia decontamination using Katana Cleaner (group 5) or Tooth Primer (groups 6 and 7) were not significantly different compared with the uncontaminated group ($p = 0.369$, $p = 0.861$, $p = 0.327$, respectively). Nevertheless, no statistically significant differences in SBS values were noted among groups 5, 6, and 7 ($p = 0.984$, $p = 1.00$, $p = 0.976$, respectively).

3.2. Failure Mode Determination

Table 2 and Figure 3 illustrate the frequency of the identified failure categories within each group. Adhesive failure was the most prevalent failure mode, occurring in 100% of specimens in groups 2 and 3 and in over 80% of specimens in the remaining groups. In contrast, mixed failure was observed in all groups, except groups 2 and 3. None of the groups exhibited cohesive failure. Figure 4a–g shows the percentage of adhesive failures observed in groups 1 to 7, respectively, while Figure 4h–l shows the percentage of mixed failures in groups 1, 4, 5, 6, and 7, respectively.

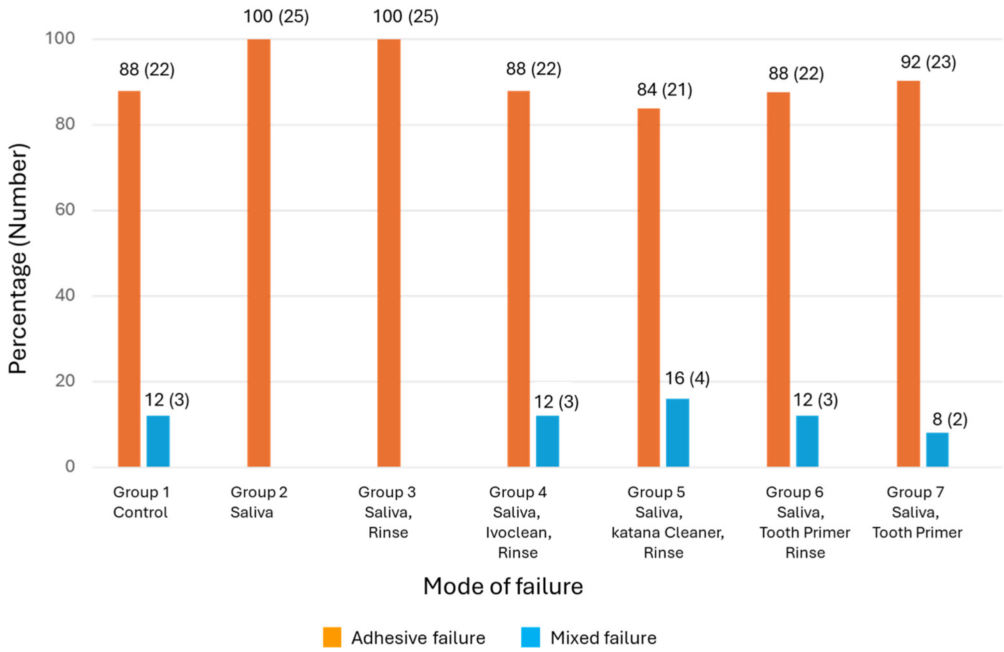

Figure 3. The failure mode percentages for each group after the shear bond strength test.

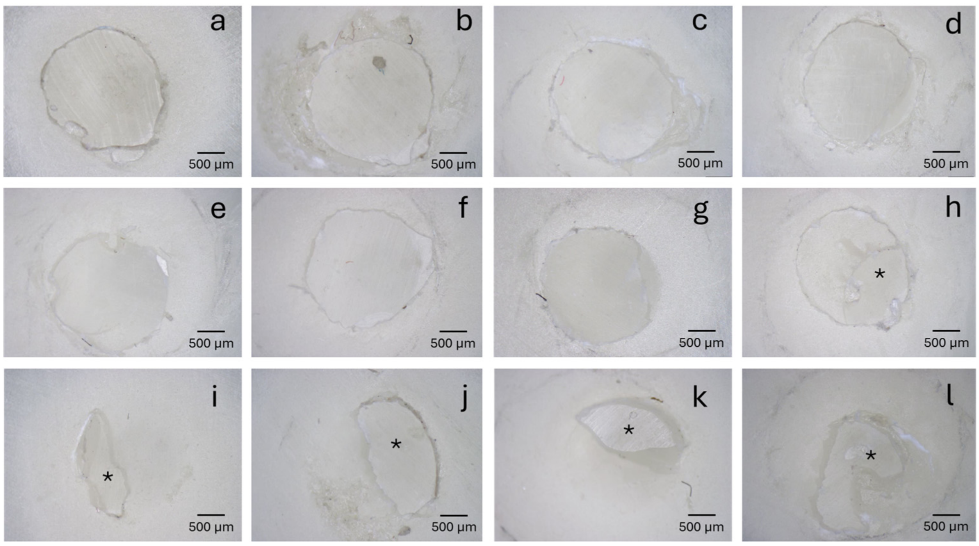

Figure 4. Stereomicroscope images at 40× magnification. Examples of adhesive failure from groups 1 to 7 are shown in (**a**–**g**), respectively, and examples of mixed failure from groups 1, 4, 5, 6, and 7 are shown in (**h**–**l**), respectively. The residual resin cement on the zirconia surface is denoted by the asterisk.

4. Discussion

In this study, natural saliva was used instead of artificial saliva. Although natural saliva is more variable than artificial saliva, it better mimics clinical conditions. To control variability, unstimulated saliva was obtained from a single healthy person at the same time of day and at least an hour after consuming food or liquids, which was used within 15 min of collection.

The durability of resin–ceramic bonds can influence the clinical performance of ceramic restorations [17–19]. Chewing and biting subject adhesively cemented zirconia restorations to tensile and shear forces [20]. This study aimed to examine the effects of cleaning protocols on the resin–zirconia interface. Although SBS testing may produce a less uniform stress distribution across the interface compared to tensile bond strength testing, it can still provide a good evaluation of the tested experimental groups when performed correctly and interpreted using fractography analysis. The SBS test is often employed to evaluate the bond ability of various dental ceramics [21,22]. Zirconia surfaces modified with alumina air abrasion could potentially facilitate saliva adherence during intraoral try-in because they are rougher and have more surface energy. Bond strength is influenced by modifications to the intaglio surface of zirconia restorations. Numerous studies have focused on optimizing protocols to enhance the bonding between dental zirconia restorations and resin cements [17–19,23–26].

The most widely used method for zirconia ceramic surface modification is airborne particle abrasion using 50 μm Al_2O_3 particles spaced 10 mm apart and under 2.5 bars of pressure. Thus, to replicate clinical scenarios, this approach was employed for specimen preparation in this study. Furthermore, a variety of resin cements, including auto-cured and dual-cured cements, can be used to cement the zirconia restoration. Specifically, one of the factors that contributes to clinical success is the strength of the bonding of the zirconia restoration to the resin cement interface given that shear pressures during masticatory function may influence the strength of the binding between restorations and tooth structure. According to this study's findings, saliva contamination considerably reduced the resin–zirconia bond strength. Consequently, the first null hypothesis was rejected. Organic substances, such as carbon (C) or nitrogen (N) elements, that remain on saliva-contaminated zirconia surfaces cannot be removed by rinsing with distilled water or submerging them in 99% isopropanol alcohol [13]. Therefore, the mean SBS values in groups 2 and 3 of the present study were significantly lower compared with the other groups. The SBS values in group 3 did not improve even after rubbing the contaminated specimen surface with a microbrush for 10 s and then washing it with distilled water for 60 s. This may explain why there was still residual saliva. Specifically, the chemically etched zirconia surface can adsorb phosphorous-containing compounds, including salivary phospholipids, as well as residual organic matter, such as salivary protein [9]. This contamination inhibits the chemical reaction between the hydroxyl group on the zirconia surface and the 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) functional monomer in the resin cement, resulting in a reduction in the resin–zirconia SBS. On the other hand, if 10-MDP makes direct contact with the etched zirconia surface, the bond strength is enhanced. The chemical adhesion between zirconia and 10-MDP relies on the phosphate group in the functional monomer. This reaction creates zirconium phosphate salts that are chemically stable. These salts improve the bonding between resin and zirconia because they can withstand heat and hydrolytic breakdown, thus making them stronger [27]. Moreover, the 10-MDP monomer has a methacrylate group, which can be used to copolymerize with other methacrylate-based monomers, such as methacrylate-based resin cement. This notion has been demonstrated by the chemical assessment of the zirconia-primed surface [28]. Furthermore, the 10-MDP molecule and other methacrylate phosphate functional monomers reduce the likelihood of phase transformation from tetragonal to monoclinic zirconia, thereby contributing to the preservation of zirconia's mechanical strength [29].

In the present study, we did not employ phosphoric acid or alcohol to decontaminate saliva since these substances can only partially remove saliva contamination from zirconia surfaces [10,30]. According to an earlier investigation, washing with phosphoric acid did not enhance the resin–zirconia bond because it has the ability to lower the surface energy of zirconia [30]. Additionally, a phosphorous residue may be deposited on the zirconia surface through the interaction between zirconia and phosphoric acid. This would hinder the chemical adhesion between the zirconia and 10-MDP molecules, subsequently weakening their bonding [12,31,32].

The shear bond strengths of zirconia decontaminated with Ivoclean, Katana Cleaner, and Tooth Primer decontamination are comparable. Therefore, the second null hypothesis, which states that there is no difference in the effects of the investigated decontamination techniques, was also not rejected. Additionally, unlike the decontamination or cleaning protocols utilized in this investigation, Katana Cleaner and Tooth Primer achieved a shear bond strength comparable to the control (uncontaminated) group, whereas Ivoclean did not. Ivoclean consists of zirconia particles in a hyper-saturated solution. Phosphorus is one of the contaminants that Ivoclean adsorbs when applied to contaminated substrates. Thus, contaminants can be eliminated from the zirconia surface. Recent X-ray photoelectron spectroscopy investigations indicated the effectiveness of Ivoclean for use as a ceramic cleaner since it removes deposits of organic substances, including saliva [13,33,34]. Moreover, Ivoclean increases the surface energy of the decontaminated ceramic [33]. This explains why the SBS of group 4 was significantly higher compared with groups 2 and 3. Statistical analysis revealed no significant difference among Ivoclean, Katana Cleaner, and Tooth Primer.

Katana Cleaner is a commercial product specifically formulated for cleaning zirconia ceramic surfaces. The cleaning solution consists of 10-MDP, triethanolamine, poly(ethylene glycol), stabilizer, water, and dyes. Upon applying and rubbing Kanata Cleaner onto the saliva-contaminated restoration's intaglio surface, the hydrophobic portion of the 10-MDP molecule adheres to the contaminant and lowers surface tension by encircling it. The contaminant is subsequently removed when rinsed with water [35,36]. In addition, the 10-MDP molecule can interact with the hydroxyl groups of the hydrated oxide layer on the zirconia surface, which cannot be rinsed off with water, thereby enhancing the bond strength between the resin cement and zirconia interphase. Previous investigations have determined the bonding ability and formation of chemical interactions at the interface. Consequently, numerous investigations have demonstrated its efficacy on zirconia surfaces contaminated with blood and saliva [35,37–40]. Tooth Prime is a self-etching solution that the manufacturer recommends for use intraorally to condition prepared teeth. It is composed of 10-MDP, 2-hydroxyethyl methacrylate (2-HEMA), hydrophilic aliphatic dimethacrylate, accelerators, and water. When applied to the tooth structure, this self-etching primer modifies the smear layer on the dentin surface and accelerates the polymerization of the Panavia V5 resin cement upon contact. Moreover, this product is used intraorally to modify base metal alloys, as well as zirconia, such as in base metal cores or zirconia implant abutments. However, in the present study, this solution was used to clean or modify saliva-contaminated zirconia, and the results were comparable with the groups treated with Ivoclean and Katana Cleaner. In group 6, Tooth Primer was washed off with distilled water after being applied to the contaminated surface and created a high SBS comparable to the control group; this may be due to the similar action of the 10-MDP molecule present in Katana Cleaner. Although Tooth Primer was not rinsed off in group 7, the SBS was still as high as that of the control group. It is hypothesized that the Tooth Primer may denature saliva and that the rubbing motion activates the 10-MDP molecule. This molecule can then compete with the salivary phosphate molecule to interact with the hydroxyl groups on the zirconia surface. Consequently, using Tooth Primer to decontaminate saliva by rubbing it in and not washing it off can enhance the bonding ability at the zirconia–resin cement interface.

In contrast to earlier research [41–45], Tooth Primer from Panavia V5, a manufacturer-recommended primer for modifying dentin, was also used in this study to prime the zirconia surfaces and modify saliva-contaminated zirconia surfaces before luting with resin cement. These findings demonstrated that Tooth Primer can strengthen the bonding and alter the saliva-contaminated zirconia surface regardless of whether it has been washed off. As a result, Tooth Primer can serve as a surface-modifying agent for dentin and zirconia in routine clinical practice. This discovery could reduce the amount of reagent used in dental clinics, benefitting dental practices. Furthermore, this finding might result in the development of a novel multifunctional solution that can be used for surface modification of both intraoral (tooth) and extraoral (restorations) materials.

Regarding the mode of failure, this investigation employed the notched-edge shear bond strength testing technique. Here, the crosshead with a notched edge ensures more accurate force distribution, resulting in a more precise measurement. This setup is more representative of adhesive shear strength compared to the peel strength, which is typically produced using a crosshead with a straight edge. According to the failure mode results, 100% of adhesive failures were observed in groups 2 and 3, while more than 80% of adhesive failures were observed in the remaining groups. Therefore, the SBS values were closer to the adhesive shear strength. Groups 2 and 3 showed 100% adhesive failure for every low SBS value (0.90 ± 0.20 and 1.35 ± 0.43 MPa, respectively); this may imply that saliva and residual saliva left on the zirconia surface act as a barrier, preventing the interaction of the PO_3H_2 group from the 10-MDP molecule with the hydroxyl group (-OH) of the hydrated oxide layer of the zirconia. Regarding the mixed failure mode, it could be due to the interfacial bond strength between the resin cement and the zirconia being greater in certain areas than the strength of the resin cement itself. Alternatively, it could be due to a defect within the resin material that caused the failure. As a result, some resin cement was visible on the zirconia surface. Additionally, no cohesive fractures were observed in any of the zirconia ceramics. This is because sintered zirconia has better mechanical properties [46].

The study's limitations are that the specimens' surfaces were flat, but the dental restorations' surfaces are curved. In addition, only Panavia SA Luting Multi cement was used. As a result, the findings do not fully represent all situations encountered in dental clinics. In further studies, a method will be employed to show how the MPD molecule and the saliva-contaminated zirconia surface form a chemical bond after Tooth Primer treatment without rinsing.

5. Conclusions

The effect of eliminating human saliva on the resin–zirconia SBS was investigated in this work. Cleaners containing zirconium oxide powder (Ivoclean), 10-MDP functional monomer (Katana Cleaner), and a primer containing 10-MDP functional monomer (Tooth Primer) were found to overcome the adverse effects of saliva on resin–zirconia SBS. The resin–zirconia bond strength values obtained did not differ significantly after decontamination with these solutions. Moreover, the primer containing the 10-MDP functional monomer (Tooth Primer) can be used as a zirconia surface cleaner with or without rinsing, as it generates a high bond strength. For clinical use, Tooth Primer can be applied as a primer to the tooth structure as well as to zirconia restorations.

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