

Influence of cleaning methods on zirconia ceramic bonding with long-elapsed time post-airborne-particle abrasion

Majed Al-Akhali ^{a,b,*}, Eglal Al-Dobaei ^{a,b}, Abdulaziz Samran ^{a,b,c}, Ahmed Wagdy Hashem ^{a,d}, Sebastian Wille ^a, Matthias Kern ^a

^a Department of Prosthodontics, Propaedeutics and Dental Materials, Dental School, Christian-Albrechts University, Kiel, Germany, ^b Department of Prosthodontics, College of Dentistry, Ibb University, Ibb, Yemen, ^c Department of Restorative and Prosthetic Dental Sciences, College of Dentistry, Dar Al Uloom University, Riyadh, Saudi Arabia, ^d Faculty of Dentistry, October University for Modern Sciences and Arts, Cairo, Egypt

Abstract

Purpose: To evaluate the influence of cleaning methods on bonding to zirconia ceramic over a long period after airborne-particle abrasion (APA).

Methods: A total of 140 disc-shaped zirconia specimens were divided into five groups based on the cleaning methods after a 72-hour delay between APA and bonding: A-(alcohol), V-(Ivoclean), K-(Katana Cleaner), P2-(Clearfil Ceramic Primer Plus after 72 hours), and P1-(Clearfil Ceramic Primer Plus directly after APA). The specimens were APA-treated with 50- μ m alumina particles (Al_2O_3) and stored at room temperature for 72 hours ($n=28$ /group). Surface free energy (SFE) was measured for 12 specimens per group using a goniometer. For each group, 16 Plexiglas tubes filled with composite resin were bonded to zirconia specimens using a self-adhesive luting resin. Tensile bond strength (TBS) was tested for subgroups of eight specimens after 3 days of water storage and 150 days with 37500 thermal cycles. Failure modes were categorized as adhesive or cohesive using light microscopy.

Results: SFE increased significantly in groups V (68.4 mN/m) and A (61.8 mN/m) ($P \leq 0.05$), but decreased in group K (33.1 mN/m) ($P \leq 0.05$). TBS after 3 days of water storage ranged from 60.7 MPa in group A to 51.6 MPa in group P2 without significant difference between the tested groups ($P > 0.05$), and after 150 days with thermocycling, it ranged from (45.4 MPa) in group K to 33.9 MPa in group P2 with a significant difference between groups K and P2 ($P \leq 0.05$).

Conclusions: Cleaning significantly affected the strength and durability of bonding in non-retentive zirconia restorations when there was a considerable time gap between APA and bonding.

Keywords: Zirconia, Ceramic, Airborne-particle abrasion, Elapsed time, Bonding

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1. Introduction

Zirconia (ZrO_2) dental restorations are widely used because of their biomechanical properties and aesthetic appeal[1–4]. Adhesive luting cements enhance the durability and fracture resistance of zirconia restorations when bonding to tooth structure, primarily through micromechanical roughening and cleaning via airborne-particle abrasion (APA) with alumina particles (Al_2O_3), followed by the application of an adhesive luting system containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP)[5–11]. Recent studies have explored various surface treatments to enhance the bond strength, such as tribochemical silica coating, which has been shown to outperform alumina blasting, particularly after aging[12], and glass-ceramic spray deposition (GCSD), which, when paired with 5% HF etching, significantly enhances the bond strength by improving

micromechanical interlocking and hydrophilicity[13].

The measurement of the contact angles formed by two dissimilar liquids with varying surface tensions serves to quantify the surface free energy (SFE) of solid surfaces[10]. Understanding the SFE of a solid surface can offer insights into the interaction dynamics between the surface and any liquid[14]. Previous investigations[15–18] have suggested that a heightened SFE of the zirconia bonding sur-

WHAT IS ALREADY KNOWN ABOUT THE TOPIC?

» Airborne-particle abrasion (APA) with alumina (Al_2O_3) followed by adhesive luting is a well-established method for achieving durable bonding to zirconia. However, studies have indicated that delaying the bonding procedure after APA can weaken the bond strength and compromise long-term adhesion.

WHAT THIS STUDY ADDS?

» This study explores different cleaning methods to improve the bond strength of non-retentive zirconia restorations, even when bonding is delayed after APA. These findings offer valuable insights into optimizing clinical protocols for long-term success.

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*Corresponding author: Majed Al-Akhali, Department of Prosthodontics, Propaedeutics and Dental Materials School of Dentistry Christian-Albrechts University, Arnold-Heller-Str. 3, House B, 24105 Kiel, Germany.

E-mail address: malakhali@proth.uni-kiel.de

face could potentially enhance the strength and longevity of resin bonding.

Contaminants, such as saliva, blood, and silicone disclosing media, can significantly impair the bond strength between zirconia and resin cement. Various cleaning methods, including alcohol[19–22], acetone[22], 37% phosphoric acid[17,19–25], sodium hypochlorite[26], hydrogen peroxide[26], sodium dodecyl sulfate[26], or additional APA[11,20–22,26–28], have been tested for their effectiveness in removing contaminants and enhancing bond strength. However, challenges in clinical dental practice have arisen owing to limited access to abrasion devices, especially after clinical try-ins. Consequently, chemical cleaning solutions have been explored as practical alternatives for ensuring effective cleaning and durable bonding[11]. In recent years, various universal cleaning solutions have been introduced in the dental market to enhance zirconia surface cleaning after contamination. Ivoclean, an alkaline cleaning paste, effectively removes contaminants from zirconia surfaces through a chemical gradient mechanism, enabling easy rinsing with water[11,19,26,27,29,30]. Similarly, the Katana Cleaner, an acidic ceramic cleaner containing MDP, has been designed for both intraoral and extraoral use. Its hydrophobic properties promote bonding to contaminants, whereas the exposed hydrophilic phosphate groups facilitate effective rinsing[31,32]. Angkasith *et al.*[27] demonstrated that if salivary contamination occurs after the application of an MDP primer, rinsing zirconia surfaces with water can effectively preserve bond strength.

The effect of the elapsed time between APA and bonding on microtensile bond strength was studied using computer-aided design/computer-aided manufacturing (CAD/CAM) polymer composite blocks and bovine dentin[33,34]. APA with 50 μm Al_2O_3 particles at pressures of 0.1 and 0.2 MPa, followed by adhesive bonding with an MDP-containing luting system, demonstrated that delaying bonding by seven days negatively impacted microtensile bond strength[33]. Additionally, APA of dental alloys significantly reduces the contact angle of water, enhancing the SFE, which persists for over 20 hours post-APA[35].

Based on clinical trials, minimally invasive protocols for oxide ceramic restorations recommend, as the gold standard[7], an intraoral try-in followed by cleaning and roughening of the zirconia bonding surface using APA with 50- μm Al_2O_3 particles at a pressure of 0.1 MPa before luting with an adhesive system[36–39]. However, in scenarios where an APA device is not readily accessible chairside, zirconia restorations undergo APA in dental laboratories. The duration between the APA procedure in the dental laboratory and the actual luting procedure in the dental office can vary significantly depending on factors such as the location and schedule of the dental laboratory. This interim period poses the risk of contaminating the bonding surface with air pollutants.

Our previous study[18] demonstrated the adverse effects of time delay between APA and resin bonding on zirconia ceramics, highlighting a critical limitation in clinical practice. Building on this foundation, this study aims to explore innovative solutions by investigating whether chemical cleaning methods can mitigate this disadvantage. By focusing on the efficacy of these cleaning protocols, particularly under delayed bonding conditions, this study provides a deeper understanding of strategies for optimizing resin bonding to zirconia and therefore, addresses a significant gap in the current literature.

The null hypothesis posited in this study was that zirconia SFE and tensile bond strength (TBS) would not be affected by different cleaning methods after a 72-hour interval between APA and the bonding procedure when utilizing an MDP-containing luting resin.

2. Materials and Methods

2.1. Specimen preparation

A total of 140 disc-shaped 3Y-TZP zirconia specimens (Cercon, DeguDent) with a diameter of 6.5 mm and a thickness of 3.4 mm were used in this study. Zirconia specimens were randomly assigned to five groups (n=28 per group) using Excel software (Excel 2019; Microsoft Corp). The sample size of this study was justified to follow previous studies carried out by our group with the same study design and sources of variation, and the sample sizes of this study gave acceptable standard deviations and allowed the statistical differentiation of the evaluated factors[11,18,22,23,30,40]. The specimens were divided into five groups according to the cleaning method after the elapsed time between the APA and the bonding procedure. These groups were designated as follows: alcohol (A), Ivoclean (V), Katana Cleaner (K), Clearfil Ceramic Primer Plus (P2), and Clearfil Ceramic Primer Plus applied directly after APA (P1). This resulted in 60 specimens (n=12 per group) being used to measure the SFE and 80 specimens (n=16 per group) to measure the TBS. The operator was calibrated by practicing specimen preparation for group A until consistent values were achieved with a maximum standard deviation of 20%, consistent with the results reported in the literature[11,18,41]. The study design is summarized in **Table 1**.

The zirconia specimens were polished with rotating silicon carbide papers down to 600 grit under water cooling, followed by ultrasonic cleaning in 99% isopropanol for 3 min and drying with oil-free air. The zirconia specimens were airborne-particle abraded with 50- μm alumina particles (Al_2O_3) at a pressure of 0.1 MPa for 15 s at a distance of 10 mm, and the abrasion procedure was applied to the bonding surface of the specimens in horizontal and vertical directions to ensure that the complete surface was abraded[42]. All the specimens were ultrasonically cleaned in 99% isopropanol for 3 min. Cleaning with 99% isopropanol during specimen preparation was a standardized step applied uniformly across all groups before subjecting the specimens to the experimental cleaning methods. Finally, all airborne-particle-abraded zirconia specimens were stored in closed plastic boxes in a dark, dry place at room temperature (20 °C) for 72 hours. Finally, the airborne-particle-abraded zirconia specimens for the five groups were stored in closed plastic boxes in a dark, dry environment at room temperature (20 °C) for 72 hours.

2.2. Cleaning methods

The cleaning methods were implemented in five groups according to the elapsed storage time and before bonding, as outlined below. The compositions of the cleaning agents are listed in **Table 2**.

Group A: The specimens were ultrasonically cleaned in 99% isopropanol for 3 min and dried with an oil-free air stream directly before the bonding procedure (control group).

Group V: The specimens were cleaned with Ivoclean according to the manufacturer's instructions. The cleaning paste was applied to the zirconia-bonded surface using a brush for 10 s, followed by a reaction time of 20 s. Subsequently, the cleaning paste was rinsed

Table 1. Study design depicting the order of the trial steps

140 zirconia discs				
Polished with rotating carbide paper down to 600 grit				
Airborne-particle abrasion with 50- μm Al_2O_3 at pressure of 1 bar				
All discs were cleaned with 99% isopropanol in an ultrasonic bath for 3 minutes				
All zirconia discs were divided into five groups (n=28/group)				
The elapsed time between airborne-particle abrasion and bonding to zirconia was 72 hours				-
Cleaning with isopropanol Group A: n=28	Cleaning with Ivoclean Group V: n=28	Cleaning with Katana Cleaner Group K: n=28	Clearfil Ceramic Primer Plus Group P2: n=28	Clearfil Ceramic Primer Plus Group P1: n=28
-				Elapsed time 72 hours
SFE was measured of 12 specimens from each group				
Plexiglas tubes were filled with self-curing restorative composite resin (Clearfil Fil New Bond)				
All samples were bonded with PANAVIA 21 cement				
After bonding, each subgroup n=16 was divided into				
8 specimens from each subgroup stored in a distilled water bath at 37 °C for 3 days without thermal cycling to record the initial bond strength			8 specimens from each subgroup stored in a distilled water bath at 37 °C for 3 days and interrupted by thermal cycling between 5 and 55 °C in distilled water with a dwell time of 30 sec for 37500 cycles to test the hydrolytic stability of the obtained bond strength after the thermocycling	
Specimens were debonded in the universal testing machine (Zwick Z010; ZwickRoell Group) at a cross-head speed of 2 mm/min				
Light microscope was used to calculate the debonded area, which was assigned to adhesive or cohesive failure modes				
Data were tabulated and statistically analyzed				

Table 2. List of cleaning materials used in this study

Cleaning methods	Composition in wt%
Alcohol	Isopropanol ($\text{C}_3\text{H}_8\text{O}$) 99%
Ivoclean	Zirconium oxide (ZrO_2) 10–15%, water (H_2O) 65–80%, polyethylene glycol ($\text{C}_{2n}\text{H}_{4n+2}\text{O}_{n+1}$) 810%, sodium hydroxide (NaOH) \leq 1%, pigments, additives 4–5%
Katana cleaner	Water (H_2O), 10-methacryloyloxydecyl dihydrogen phosphate (MDP, $\text{C}_{14}\text{H}_{27}\text{O}_6\text{P}$), triethanolamine ($\text{N}(\text{CH}_2\text{CH}_2\text{OH})_3$), polyethylene glycol ($\text{C}_{2n}\text{H}_{4n+2}\text{O}_{n+1}$), stabilizer, dyes
Clearfil Ceramic Primer Plus	Ethanol ($\text{C}_2\text{H}_6\text{O}$) >80%, 3-trimethoxysilylpropyl methacrylate ($\text{C}_{10}\text{H}_{20}\text{O}_5\text{Si}$) <5% and 10-methacryloyloxydecyl dihydrogen phosphate (MDP, $\text{C}_{14}\text{H}_{27}\text{O}_6\text{P}$)

with water spray for 15 s, and the bonding surface was dried using an oil-free air stream immediately prior to the bonding procedure.

Group K: The specimens were cleaned with a Katana Cleaner according to the manufacturer's instructions, and the cleaning paste was distributed on the surface using a brush for 10 s. Subsequently, the cleaning paste was removed with a water spray for 15 s, and the bonding surface was dried with an oil-free air stream directly before the bonding procedure.

Group P2: The specimens were activated by applying Clearfil Ceramic Primer Plus according to the manufacturer's instructions and were distributed on the bonding surface using a brush for 10 s, followed by a reaction time of 10 s. Subsequently, the entire adherent surface was dried using a mild, oil-free airflow immediately before the bonding procedure.

Group P1: The specimens were activated by applying Clearfil Ceramic Primer Plus before the elapsed storage time, according to the manufacturer's instructions. The specimens were then stored for 72 hours.

2.3. Surface free energy (SFE)

SFE measurements were conducted on representative specimens from the five groups before bonding at room temperature.

This was accomplished using a goniometer (drop shape analyzer DSA25E, Krüss) and the sessile drop technique. Two liquids with different surface tensions were employed to gauge the SFE. A 2- μL drop of deionized water or diiodomethane was deposited onto the abraded surface of a zirconia specimen, and the contact angles of both solutions were measured and computed using software (Advance 1.7.2.1). The Owen, Wendt, Rabel, and Kaelble (OWRK) method was used to derive the dispersive and polar energies of zirconia. The sum of the dispersive and polar energies represented the SFE of the investigated zirconia specimens[10].

A typical method for measuring SFE using two liquids with different surface tensions involves testing the same specimen. However, the surface area of a single specimen was insufficient to accommodate separate drops of water and diiodomethane. Therefore, square Plexiglas jigs measuring 40×40×10 mm were employed to hold the two specimens together side by side, treating them as a single specimen. One specimen was used to measure the contact angle of deionized water, and the other was used to measure the contact angle of diiodomethane.

2.4. TBS

The bonding procedure for the five groups were conducted following the designated cleaning methods after the specimens had been stored. Plexiglas tubes with a standard diameter of 3.2

Table 3. Means and standard deviations in mN/m of the surface free energy (SFE), dispersive, and polar of the different groups

Cleaning method	SFE	Dispersive	Polar
	Mean (SD)	Mean (SD)	Mean (SD)
Alcohol (A)	61.8 (2.5) ^B	38.2 (2.1)	23.6 (4.2)
Ivoclean (V)	68.4 (7.6) ^A	43.0 (1.3)	25.4 (8.1)
Katana (K)	33.1 (1.4) ^D	31.6 (1.9)	1.4 (0.5)
Primer 2 (P2)	51.0 (1.6) ^C	44.5 (1.4)	6.5 (0.9)
Primer 1 (P1)	56.5 (2.5) ^{BC}	42.4 (1.0)	14.2 (2.1)

Within the same column, means with the same superscript letter are not statistically different ($P > 0.05$). Within the same row, means with the same superscript are not statistically significantly different ($P > 0.05$).

mm[41,42] were filled with autopolymerizing restorative composite resin (Clearfil FII New Bond, Kuraray Medical, Osaka, Japan) and allowed to polymerize for 10 minutes before being bonded to the zirconia specimens ($n=16$). The filled Plexiglas tubes were bonded to the conditioned zirconia specimens using an autopolymerizing luting resin (Panavia 21, Kuraray Medical). To ensure perpendicular alignment of the tube axis to the zirconia bonded surface, an alignment apparatus under a load of 7.4 N was employed[30,42,43]. Excess cement was removed using a sponge pellet, and an air-blocking gel (Oxyguard II, Kuraray Medical) was applied around the bonding margins to prevent the formation of an oxygen-inhibited unpolymerized layer.

Eight specimens from each group were stored in distilled water at 37 °C for 3 days without undergoing thermal cycling, aiming to assess the initial bond strength[44]. Simultaneously, the remaining eight samples were stored in distilled water at a temperature ranging between 5 and 55 °C for 150 days, undergoing 37500 thermal cycles with a dwell time of 30 seconds each.

The TBS was assessed using a universal testing machine (Zwick 2010, Zwick Roell Group) operating at a 2 mm/min crosshead speed. This evaluation involved employing a self-aligning chain-loop attachment to ensure precise testing conditions[21,30,42,45].

2.5. Failure mode

Following the TBS assessment, the debonded zirconia ceramic specimens were examined under a light microscope (Wild M 420, Wild) at 30× magnification. Failures were categorized as adhesive if the bond occurred at the zirconia surface or cohesive if the failure manifested within the luting or tube-filling composite resin. The areas of each failure mode were computed and expressed as percentages of the total bonding surface area for each experimental group. Representative debonded zirconia specimens were sputtered with a gold alloy conductive layer, approximately 15 nm thick and subsequently analyzed using a scanning electron microscope (SEM, XL 30 CP, Philips) operating at an acceleration voltage of 15 kV and a working distance of 10 mm. This analysis aimed to elucidate the failure modes observed under a light microscope and facilitate a comparative assessment[7,18,20].

2.6. Statistical analysis

The normal distribution of both the TBS and SFE data was examined using the Shapiro-Wilk test across all groups. The SFE data exhibited a normal distribution. Statistical analyses were performed

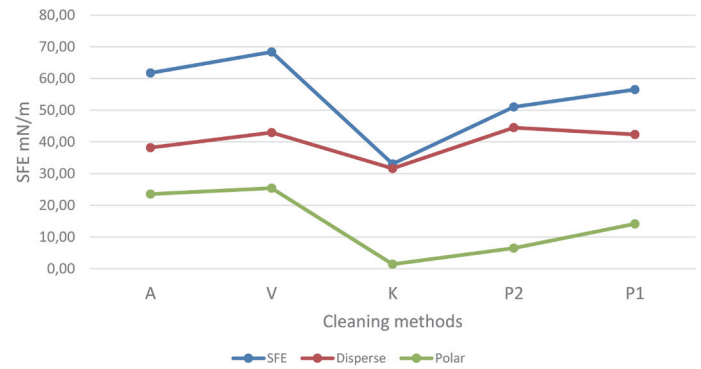


Fig. 1. Surface free energy with its dispersive and polar components of test groups showing the influence of cleaning methods after airborne-particle abrasion on the surface free energy (SFE). A: Alcohol, V: Ivoclean, K: Katana, P2: Clearfil Ceramic Primer Plus after 72 hours, P1: Clearfil Ceramic Primer Plus directly after APA.

using ANOVA and Tukey's HSD tests. Descriptive analysis was applied to the dispersive and polar energy data.

Given that the TBS data for certain groups did not follow a normal distribution, statistical analysis was conducted using the Kruskal-Wallis test and multiple pairwise comparisons of groups using the Mann-Whitney U test. To account for multiple testing, significance levels were adjusted using the Bonferroni-Holm correction method. Statistical analysis was performed using a statistical software program (IBM SPSS Statistics for Windows, Version 20.0, IBM Corp).

3. Results

3.1. Surface free energy (SFE)

The SFE test results revealed significant differences among the five groups, highlighting the impact of different cleaning methods applied after a long-elapsed time following APA, particularly between group K and the remaining four groups employing different cleaning methods ($P \leq .05$). The detailed SFE results for the five test groups are presented in **Table 3** and illustrated in **Figure 1**. Representative images of the measured contact angles of water on the airborne particle-abraded zirconia after cleaning are shown in **Figure 2**.

Group V showed a significantly higher SFE than the other four groups ($P \leq .05$), whereas group K showed a significantly lower SFE than the other four groups ($P \leq .05$). Moreover, the control group A showed significantly higher SFE ($P \leq .05$) than group P2. No statistically significant differences were found between groups P1, group P2, and control group A ($P > .05$).

3.2. Tensile bond strength

The TBSs of the five test groups are presented in **Table 4** and **Figure 3**. After 3 days of water storage, the highest TBS was 60.7 MPa for the control group A, while the lowest TBS was 51.6 MPa for the group P2, with no statistically significant differences between the five groups. After 150 days of water storage with thermocycling, the highest TBS was 45.4 for the group K, while the lowest TBS was 33.9 MPa for the group P2. There was a statistically significant difference between group K and group P2 ($P \leq .05$). However, there were no

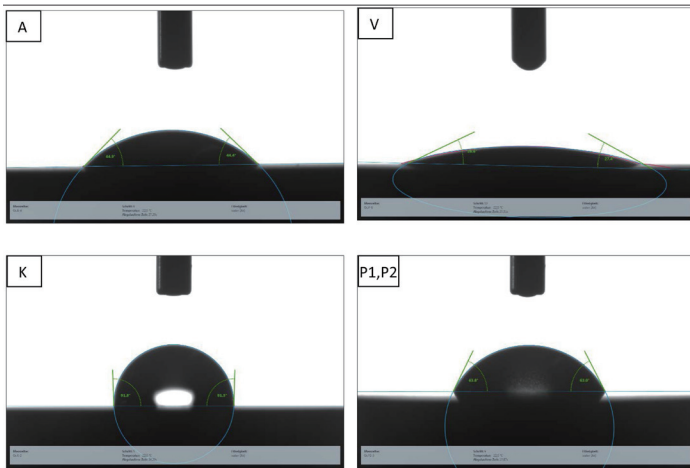


Table 4. Medians, means, and standard deviations of initial and final tensile bond strength of the test groups

Cleaning method	Storage time			
	3 days		150 days	
	Median	Mean (SD)	Median	Mean (SD)
Alcohol (A)	60.7 ^{Aα}	58.3 (10.8)	44.4 ^{ABβ}	41.9 (8.2)
Ivoclean (V)	60.5 ^{Aα}	60.0 (5.3)	37.0 ^{ABβ}	37.7 (3.4)
Katana (K)	56.0 ^{Aα}	56.5 (5.9)	45.4^{AB}	44.9 (6.9)
Primer 2 (P2)	51.6 ^{Aα}	51.5 (8.4)	33.9^{Bβ}	34.1 (3.7)
Primer 1 (P1)	53.1 ^{Aα}	53.7 (8.9)	37.0 ^{ABβ}	41.0 (10.5)

Within the same column, medians with the same superscript letter are not statistically significantly different ($P > 0.05$). Within the same row, medians with the same superscript Greek letter are not statistically significantly different ($P > 0.05$).

Fig. 2. Representative images of contact angles of water on the airborne-particle abraded zirconia after cleaning with A: Alcohol, V: Ivoclean, K: Katana, P1, and P2: Clearfil Ceramic Primer.

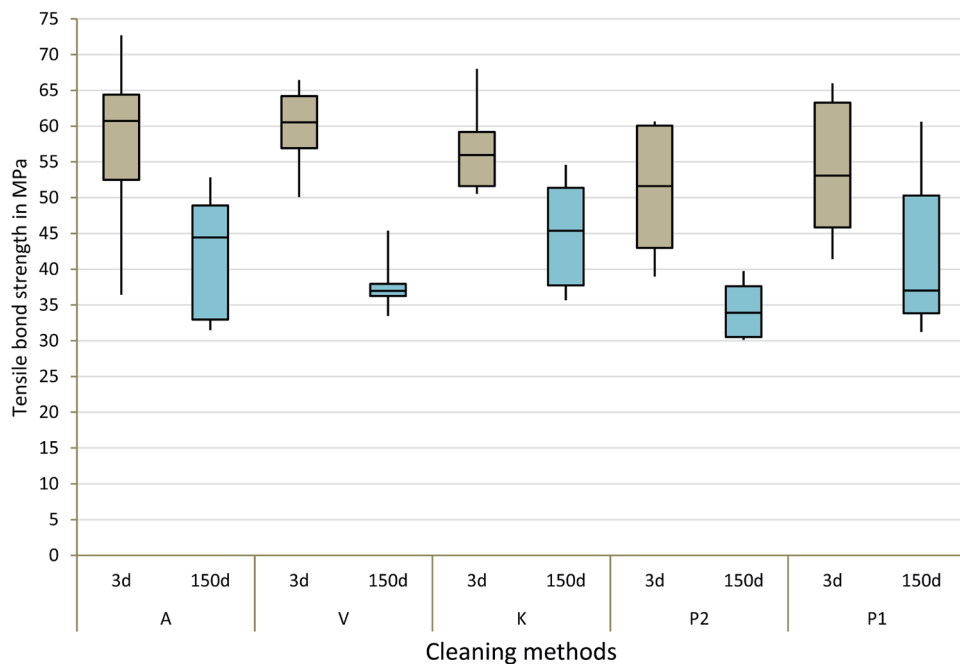


Fig. 3. Tensile bond strength of test groups in box plots showing medians, lower and upper quartiles, minima and maxima. A: Alcohol, V: Ivoclean, K: Katana, P2: Clearfil Ceramic Primer Plus after 72 hours, P1: Clearfil Ceramic Primer Plus directly after APA.

significant differences between groups K, A, P1, and V or between P2, V, P1, and A ($P > .05$).

The long-term TBS of the five test groups decreased significantly after 150 days of water storage with thermocycling ($P \leq .05$), as shown in **Table 4** and **Figure 3**. The percentage changes in the TBS before and after thermocycling for each group is as follows: group A, 36.7%; group V, 63.5%; group K, 24.4%; group P2, 52.2%; and group P1, 43.5%.

3.3. Failure mode

All groups exhibited a predominantly cohesive failure mode, with a slight increase in the adhesive failure mode after 150 days in group P2. The observed failure modes of the test groups are illustrated in **Figure 4**.

4. Discussion

The strength and durability of resin bonding to zirconia ceramics are influenced by various factors, including contamination, clean-

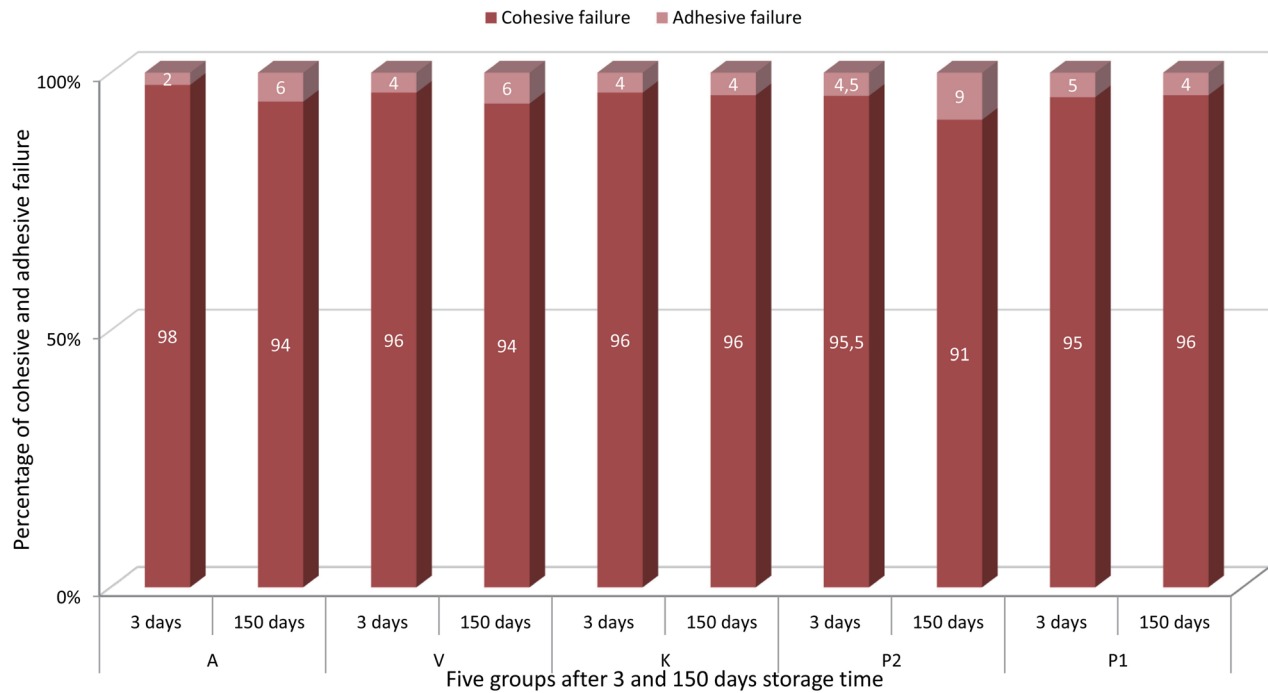


Fig. 4. Failure mode percentage of groups after 3 and 150 days of water storage with thermocycling. A: Alcohol, V: Ivoclean, K: Katana, P2: Clearfil Ceramic Primer Plus after 72 hours, P1: Clearfil Ceramic Primer Plus directly after APA.

ing procedures, surface roughness, hydrophilicity, and activation methods applied to the zirconia bonding surface. Additionally, the technique, type, and chemistry of the bonding system play pivotal roles in determining the effectiveness of the bond[11,17,24,30,46,47]. The rationale for including the groups P1 and P2 in this study was to evaluate whether applying the primer before or after the elapsed time before bonding could protect or reactivate the zirconia fitting surface and influence bond strength[27]. In laboratory investigations, artificial aging through long-term storage and thermal cycling is commonly employed to assess the durability and strength of bonds[11,30,42,43]. Conducting bonding tests in the laboratory under simulated long-term oral conditions is essential to establish a foundation for future clinical trials and provide preliminary evidence that clinicians can cautiously consider.

Many studies[10,11,15,16,21] have documented the ability of APA to enhance the SFE of zirconia bonding surfaces. Lümekmann *et al.*[15] reported that zirconia specimens treated with APA, followed by oxygen gas plasma, exhibited the highest SFE TBS. Previous studies have examined the impact of the time lapse between APA treatment and bonding on zirconia ceramics over 15 minutes, 1 hour, 4 hours, 24 hours, and 72 hours[18]; CAD/CAM polymer composite blocks over 1 and 7 days[33,34]; and on dental alloys over 20 hours[35]. They found that increased elapsed time after APA significantly affected the bond strength.

Al-Akhali *et al.*[18] demonstrated that an extended period between APA and the bonding process to zirconia resulted in a detrimental effect on both the SFE and TBS. However, the obtained bonds were durable and seemingly stable after 150 days of thermocycling. This was attributed to minimal contamination of the zirconia bonding surface caused by pollution with hydrocarbons, indicating that a composite luting resin containing an adhesive phosphate monomer

could still establish sufficient adhesive bonds with zirconia[25,48,49].

In this study, the disparate SFE among the five tested groups can be attributed to the hydrophobic (groups K, P2, and P1) and hydrophilic (groups A and V) nature of the cleaning materials used[27,31,32], as illustrated in **Figures 1 and 2**.

The TBS results of the five groups after three days of water immersion showed no statistically significant differences. However, after 150 days of thermal cycling, the specimens in group K exhibited significantly higher bond strength to zirconia ceramic compared to those in group P2 after 72 hours ($P \leq .05$), where group P2 showed relatively increased adhesive failure in comparison to the other test groups (**Fig. 4**).

In group P2, the primer was applied after 72 hours, which may have reduced adhesive bonding to zirconia due to contamination during the storage period. This contamination can negatively affect the bond strength. In contrast, in group P1, the primer was applied immediately after airborne particle abrasion and before the 72-hour storage period. This may have created a protective layer on the zirconia surface, helping to shield it from contamination and resulting in a more stable bond with less adhesive failure compared with P2[27].

The decrease in TBS observed in this study after 150 days of water storage with 37500 thermocycles may be attributed to degradation within the luting composite during long-term water storage with thermocycling. This rigorous aging process aims to evaluate the hydrolytic durability of the bond strength over time[30,42,43]. This degradation is often attributed to the adverse effects of water on the bonds between the resin matrix and silanated fillers, leading to the incomplete hydrolytic stability of the cement over prolonged periods of water exposure[11,50].

Therefore, the null hypothesis that zirconia SFE and TBS would not be affected by different cleaning methods after a 72-hour interval between APA and the bonding procedure when using an MDP-containing luting resin was partially rejected. The adverse effects of delayed bonding after APA on the bond strength to zirconia can be reduced by cleaning the zirconia bonding surface before bonding.

Our previous study[18] demonstrated the adverse effect of time delay on the resin bond strength of zirconia ceramics. The current study investigates whether chemical cleaning methods can mitigate this effect when bonding is delayed after airborne-particle abrasion. In the current study, bond strength values were initially much higher and remained elevated after 150 days compared to the 72-hour group in our previous study[18]. All groups in the current study exhibited sufficient bond strength after 150 days, nearly equivalent to the bond strength measured in the 15-minute elapsed time group in the previous study. However, we acknowledge that the inclusion of a control group without cleaning would have further strengthened the findings of this study. Therefore, group A was designated the baseline reference group[11,41]. The findings of this study are consistent with the results of several other studies, in which cleaning procedures using APA[11,17,20–22,31], Ivoclean[11,29,30,40], alcohol[11,30], and Katana Cleaner[29,32,40] were employed after contamination of the zirconia bonding surface to increase the adhesion strength to the zirconia ceramic.

5. Conclusions

Within the limitations of this study, the following conclusions can be drawn:

1. The role of SFE in influencing the bond strength is uncertain, as significant differences in the SFE do not correlate with significant differences in the bond strength.
2. Cleaning zirconia surfaces subjected to prolonged delays between airborne-particle abrasion (APA) and bonding using an MDP-containing cleaning agent demonstrated comparable bond strengths across groups, with no substantial reduction observed compared with the control group (A).
3. Future research should explore the impact of varying time intervals on delayed bonding, alternative cleaning protocols, and the long-term performance of different resin cement to further enhance our understanding of zirconia bonding mechanisms.

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Conflict of interest

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